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NBS Special Publication 642, Suppl. 2

Construction Materials for Coal Conversion

Performance and Properties Data
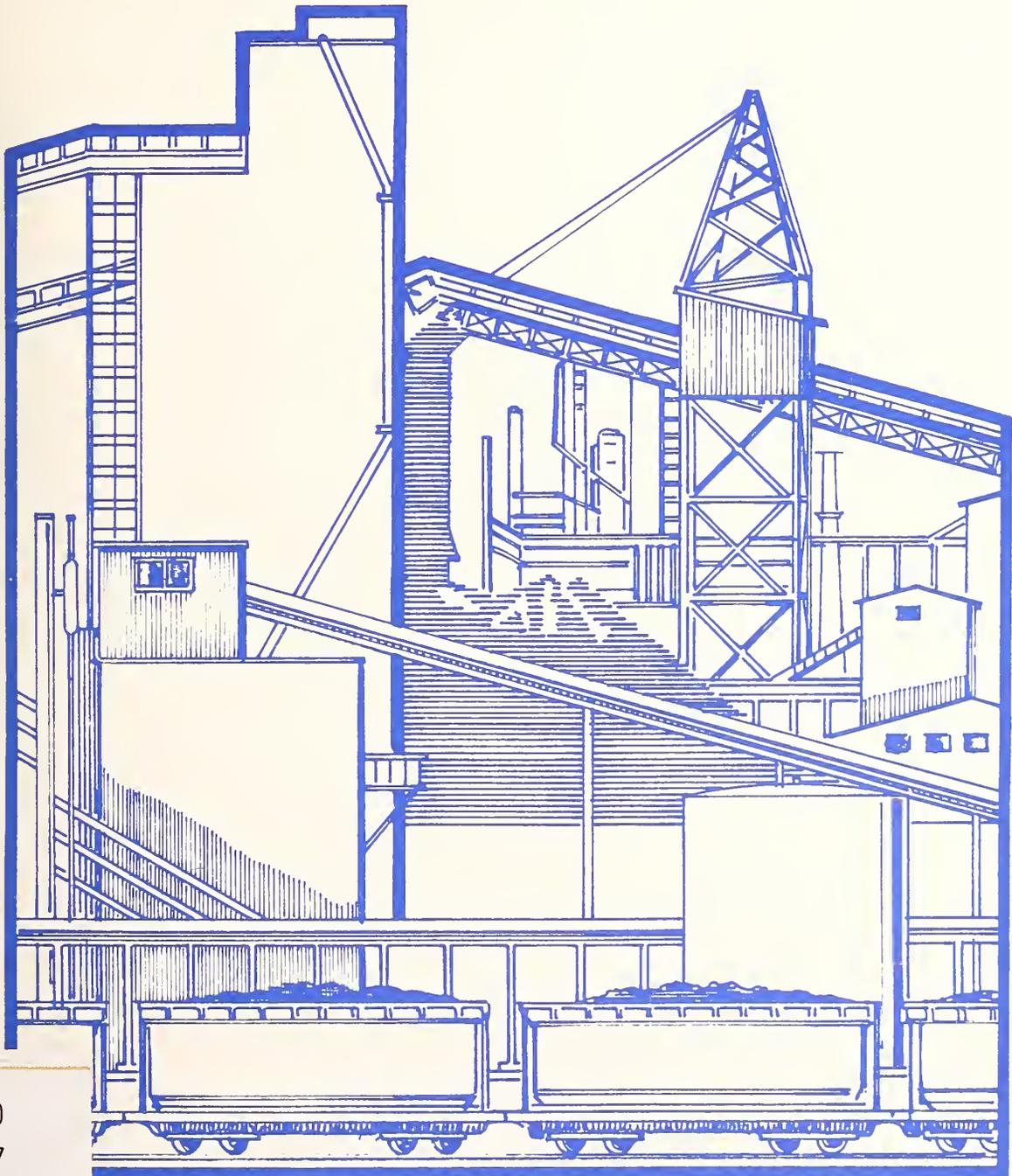
Supplement 2

*T*he National Bureau of Standards[1] was established by an act of Congress on March 3, 1901. The Bureau's overall goal is to strengthen and advance the nation's science and technology and facilitate their effective application for public benefit. To this end, the Bureau conducts research and provides: (1) a basis for the nation's physical measurement system, (2) scientific and technological services for industry and government, (3) a technical basis for equity in trade, and (4) technical services to promote public safety. The Bureau's technical work is performed by the National Measurement Laboratory, the National Engineering Laboratory, the Institute for Computer Sciences and Technology, and the Institute for Materials Science and Engineering.

The National Measurement Laboratory

Provides the national system of physical and chemical measurement; coordinates the system with measurement systems of other nations and furnishes essential services leading to accurate and uniform physical and chemical measurement throughout the Nation's scientific community, industry, and commerce; provides advisory and research services to other Government agencies; conducts physical and chemical research; develops, produces, and distributes Standard Reference Materials; and provides calibration services. The Laboratory consists of the following centers:

- Basic Standards[2]
- Radiation Research
- Chemical Physics
- Analytical Chemistry

The National Engineering Laboratory

Provides technology and technical services to the public and private sectors to address national needs and to solve national problems; conducts research in engineering and applied science in support of these efforts; builds and maintains competence in the necessary disciplines required to carry out this research and technical service; develops engineering data and measurement capabilities; provides engineering measurement traceability services; develops test methods and proposes engineering standards and code changes; develops and proposes new engineering practices; and develops and improves mechanisms to transfer results of its research to the ultimate user. The Laboratory consists of the following centers:

- Applied Mathematics
- Electronics and Electrical Engineering[2]
- Manufacturing Engineering
- Building Technology
- Fire Research
- Chemical Engineering[2]

The Institute for Computer Sciences and Technology

Conducts research and provides scientific and technical services to aid Federal agencies in the selection, acquisition, application, and use of computer technology to improve effectiveness and economy in Government operations in accordance with Public Law 89-306 (40 U.S.C. 759), relevant Executive Orders, and other directives; carries out this mission by managing the Federal Information Processing Standards Program, developing Federal ADP standards guidelines, and managing Federal participation in ADP voluntary standardization activities; provides scientific and technological advisory services and assistance to Federal agencies; and provides the technical foundation for computer-related policies of the Federal Government. The Institute consists of the following centers:

- Programming Science and Technology
- Computer Systems Engineering

The Institute for Materials Science and Engineering

Conducts research and provides measurements, data, standards, reference materials, quantitative understanding and other technical information fundamental to the processing, structure, properties and performance of materials; addresses the scientific basis for new advanced materials technologies; plans research around cross-country scientific themes such as nondestructive evaluation and phase diagram development; oversees Bureau-wide technical programs in nuclear reactor radiation research and nondestructive evaluation; and broadly disseminates generic technical information resulting from its programs. The Institute consists of the following Divisions:

- Ceramics
- Fracture and Deformation [3]
- Polymers
- Metallurgy
- Reactor Radiation

[1] Headquarters and Laboratories at Gaithersburg, MD, unless otherwise noted; mailing address Gaithersburg, MD 20899.
[2] Some divisions within the center are located at Boulder, CO 80303.
[3] Located at Boulder, CO, with some elements at Gaithersburg, MD.

Construction Materials for Coal Conversion
Performance and Properties Data
Supplement 2

NBS Special publication

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Special Publication 642, published in 1982, and SP 642, Supplement 1, published in 1983, were the first two books issued in this set of three publications. This 1985 publication, Supplement 2, like Supplement 1, contains revisions to SP 642 as well as many new pages of data, but is not a complete volume by itself. The pages of Supplement 2 are intended to be merged with the pages of SP 642 and of Supplement 1 according to the instructions given below. The first book, SP 642, is available from the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402, stock number 003-003-02442-2, price $16.00 (add 25% for other than U.S. mailing). Supplement 1 is available under stock number 003-003-02550-0 for $14.00 (plus 25% for other than U.S. mailing).

For the 1982 publication, the cutoff dates for the data varied with each project reviewed since the reports on hand at the time the data were abstracted varied with each project. The 1983 Supplement brought the coverage of data for metals and alloys up-to-date through December 31, 1982. This 1985 Supplement brings the refractory data up-to-date through December 31, 1983. Since publication of SP 642, plans for the projected supplements changed with respect to the coverage and contents of the volumes. These changes are reflected in the revised Introduction and Tables of Contents.

The combined publications, SP 642, Supplement 1, and Supplement 2 include data generated in over 80 projects carried out by approximately 40 organizations. All three books are bound so as to permit easy removal and insertion of pages and are three-hole punched to fit any standard ring binder (see page x to obtain complimentary binders). All pages are dated so the most current version of updated pages can be identified easily. Pages in Supplement 2 carrying a 1981, 1982, or 1983 date are included in order to provide a complete recto and verso leaf to merge with the pages of SP 642 and Supplement 1 and have not been revised or changed in any way. A 1984 or 1985 date on a page implies new or revised data have been printed.

In order to have a single volume containing the updated book sections simply substitute and add the Supplement 2 pages according to the following instructions. These instructions assume that SP 642 and Supplement 1 have been merged already according to the directions given in the Preface to Supplement 1.

Replace old SP 642 and Supplement 1 pages	with new Supplement 2 pages
pages iii through xiv	pages iii through xiv
A.2.0 pages 1 of 2 and 2 of 2	A.2.0 pages 1 of 2 and 2 of 2
A.2.2.1 page 1 of 1 through A.2.2.2.1 page 3 of 3	A.2.2.1 page 1 of 1 through A.2.2.2.1 page 3 of 3
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A.9.0 page 1 of 1	A.9.0 page 1 of 1

INTRODUCTION

Background

The nationwide program initiated in the 1970's by the U.S. Department of Energy and its predecessor agencies to foster development of a viable coal conversion industry featured a strong effort in materials testing and development. The data base for construction materials existing in the 1970's was inadequate for satisfactory materials selection for some component areas of plants for advanced coal conversion and use, e.g., 1) valves and piping exposed to high velocity, high-temperature streams of coal char and ash in turbulent flow, 2) metal components inside gasifier pressure vessels containing corrosive sulfide gases and operating at high temperatures and pressures, 3) vessels exposed to corrosive coal liquids and erosive slurries at high temperatures and pressures, and gas-quench waters at more moderate temperatures, and 4) pressure vessels larger than any in existence.

To expand the necessary data base, candidate materials were tested in controlled laboratory experiments, in coal gasification and liquefaction pilot plants and, in a few cases, in commercial coal-fired utility plants. About forty organizations including industrial, university, and government laboratories, and non-profit research institutions were involved in generating the materials properties and data reported in this compilation. Some materials evaluations were obtained from diagnostic failure analyses of actual plant components in about twenty pilot plants. About 500 alloys, including commercially available and developmental compositions and weld metals, were tested. The list of refractory materials tested contains over 200 commercially available and developmental refractories. To collect and disseminate the data generated by the DoE-sponsored materials testing and research projects, the NBS/DoE Fossil Energy Materials and Components Performance and Properties Data System was established.

Construction Materials for Coal Conversion--Performance and Properties Data, NBS Special Publication 642, a reference book for the fossil fuel industry, is a result of the combined effort of the two data centers which constituted the NBS/DoE Fossil Energy Materials and Components Performance and Properties Data System. This System was established at the National Bureau of Standards under the sponsorship of the Department of Energy and its predecessor, the Energy Research and Development Administration.

The first of the data centers was the Materials and Components Plant Performance Data Center, established in 1976 as the Failure Information Center. NBS was given the responsibility for collecting, abstracting, and disseminating information about operating events and materials and components failure analyses submitted by coal conversion pilot plant personnel and failure analyses laboratories under a voluntary information and data exchange program. The second of the data centers at NBS was the Materials Properties Data Center, established to collect, abstract, and disseminate construction materials properties and testing performance data. Emphasis was placed on the results of work by the materials research contractors to the Department of Energy.

Data compiled by the two data centers have been tabulated and summarized for inclusion in this publication. The first book focused on coal gasification, that form of coal conversion for which there was the largest amount of available information in the library of the data centers. With Supplements 1 and 2, mate-

rial in areas of interest such as coal liquefaction, and direct combustion have been included.

The looseleaf format was designed to permit data to be added and revised easily. The cutoff date for the projects reviewed in Supplement 1, dealing largely with alloys, is the contractors' reporting period ending December 31, 1982. The cutoff date for the projects reviewed in Supplement 2, dealing largely with refractories, is the contractors' reporting period ending December 31, 1983. Portions of the book remain incomplete either because no data were readily available in the source documents which were applicable to the component area or because the data were limited, and it was felt that definitive statements and recommendations would be premature. Although portions of this book are incomplete, it was felt that the data contained would be of sufficient interest and use to the fossil energy community to warrant publication.

A number of the same materials research projects are continuing and new projects have begun sponsored by the Department of Energy since the cutoff dates for this compilation. The reports of both continuing and new projects are being published in the Oak Ridge National Laboratory Advanced Research and Technology Development Fossil Energy Materials Program Quarterly Progress Reports available from the National Technical Information Service.

Organization of the Book

The book is divided into four major parts with the following headings--

A.   Materials Considerations and Performance Data
B.   Materials Testing and Research Results
C.   References
D.   Indexes

Part B contains summaries of performance and properties data generated by the DoE-sponsored research projects. Data are presented in graphical and/or tabular format with details of test conditions and procedures given in footnotes. Part A consists of discussions of the Part B data in the context of application to specific component areas of coal conversion plants. Part A also contains data and discussion on the performance of materials and components in actual pilot plant use.

Part A deals with materials needs and materials data for coal conversion plants by component area. It is anticipated that the user, in looking for information, will be interested in data for materials in a specific application. The major headings and subheadings for component area sections in Part A are:

1.   Coal Handling and Preparation Equipment, including;
         Conveying Equipment
         Grinding and Crushing Equipment
         Drying Equipment
         Fines Control Equipment
         Coal Pretreatment Equipment

2. Vessels (includes gasifiers, "dissolvers", devolatilizers, lockhoppers, etc.)
        Pressure-Containing Shell
        Refractory Linings and Components--Dry-Bottom Vessels
        Refractory Linings and Components--Slagging Vessels
        Metal Internal Components

3. Product Clean-Up Equipment
        Solids Separation Equipment
            Scrubbers
            Cyclones
        Cooling-Down Systems
            Quench Systems
            Heat Exchangers
        Gas Removal Systems (carbon dioxide, sulfur compounds, etc.)

4. Water-Gas Shift Equipment

5. Methanation Equipment

6. Compressors

7. Piping
        Gas Lines
        Solids Transfer Lines
        Slurry Lines
        Liquids Lines

8. Pumps
        Slurry Pumps
        Liquids Pumps

9. Valves
        Gas Valves
        Liquids Valves
        Slurry and Solids Valves

10. Direct Combustion Systems

Each component area title above is a heading for the following subsections--

1. Operating Requirements

These paragraphs are brief discussions outlining the major problems of that component area which must be considered in choosing suitable materials of construction.

2. Performance Data

There are three possible divisions of this subsection which may appear for each component area.

2.1 Plant Experience

Under this heading, there are tables of data taken from the files of the Materials and Components Plant Performance Data Center, and from some contractors' reports, with discussion of the data. The quality and completeness of the original information from which these tables were prepared is highly variable, and this fact must be taken into account in making use of the data.

2.2 Component Test and Development

For some component areas there is a subsection with this heading containing data obtained either by testing prototypes or off-the-shelf components on test stands, or in constructing and testing portions of, or models of, components. Such data are not available for all component areas and this subsection does not appear at all for most component areas. The data are taken from the Materials Properties Data Center files.

2.3 Materials Evaluation

These subsections contain summaries and discussions of data tables and graphs appearing in Part B of this book which are pertinent to the component area under consideration. The data in Part B are the results of testing and research programs utilizing small sample specimens rather than actual component pieces. Section B.0, the introduction to Part B, contains a full explanation of its contents. Since much of the data in Part B are applicable to more than one component area, it was decided to place them together in one section. The data are, therefore, simply summarized and discussed in these "Materials Evaluation" subsections.

The units used in the text, tables, and graphs in these subsections correspond to those used in the individual reports under discussion. The compilers did not convert all data to a common system of units. It is recognized that a common system of units is highly desirable, and that mixed units result in a wide variation in reporting of data causing possible confusion and requiring the user to exercise great care. Conversion of all the data in the book to a common system of units, however, would have been a very costly effort.

The numerical data should be viewed with caution. In many tests the number of samples per material per test is few, often only one, so no statistical significance is attached to the values. In most cases, complete characterization of the materials with preparative and thermomechanical history is lacking. The user, therefore, must bear in mind that the data should be used for guidance only and are not suitable for inclusion in design calculations. Such use of the data is at the sole risk of the user, and no responsibility for such use can be taken either by the compilers of the data or the sponsors of this compilation project.

It should be pointed out that most of the testing programs utilized specimens of commercially available materials. These samples should be considered as representatives of various classes of materials; the inclusion of brand names merely serves to identify and help characterize the materials. The designation of brand names should not be construed as an endorsement of any product or manufacturer. The materials are usually given the designation the authors of the original reports assigned although this practice causes some inconsistency in the book. This inconsistency is especially noted for alloys for which the designations given may or may not follow any one of the standard systems such as AISI, ASTM, or ANSI.

Much of the laboratory testing for which the data are discussed in this subsection was performed utilizing a "typical" or "simulated" coal gasification atmosphere. The composition was given as 18 percent CO, 12 percent CO_2 24 percent H_2, 5 percent CH_4, 1 percent NH_3, with varying amounts of H_2S (0.1 to 1.5 percent), and the balance H_2O. In many reports it is clearly indicated

that the above was an input composition, and equilibrium compositions at operating temperature and pressure were often given too. Some reports indicated that the above composition was the equilibrium one and others did not make any clear indication at all. The compilers have included the composition in the footnotes to tables as given in the reports.

Part B, "Materials Testing and Research Results", contains data from the Materials Properties Data Center files. The testing and research results have been arranged in four major categories: Corrosion effects, chemical reactions, and phase changes; Erosion, erosion/corrosion, and abrasion effects; Mechanical properties testing; and Physical properties testing. See Section B.0 for more information about the data in Part B.

Part C consists of complete references for the material contained in other sections of the book. References for data have been identified by numbers in square brackets which follow the titles of tables or graphs in Part B or appear elsewhere in the text or tables of Part A. Most of the references given are to reports of contractors to the Department of Energy and its predecessor agencies. See B.0, the Introduction to Part B, and also the Introduction given in Part C, for fuller information about the handling of the references.

Part D contains materials indexes which permit the user to locate sections dealing with specific alloys or refractories and also a reference concordance which permits the user to locate all of the sections which contain data from a given reference.

NOTICE ABOUT USE OF THE BOOK IN LOOSELEAF FORM

    These pages are three-hole punched to fit any standard ring binder. The
binding permits easy removal of the pages for insertion and use in such a binder.

    Binders for this publication are available from Dr. H. M. Ondik, Materials
Building, Room A229, National Bureau of Standards, Gaithersburg, MD 20899; tele-
phone (301) 921-2900.

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* See individual sections for detailed tables of contents.

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* Sections included in SP-642, Supplement 1, and Supplement 2 combined.

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A.2.2.1 OPERATING REQUIREMENTS

Refractory linings are used for thermal insulation and improved erosion
resistance. These linings in gasifier vessels must cope with the most severe
conditions of the gasification process. In dry-bottom (i.e. non-slagging)
processes, the maximum temperatures are limited by the ash-slagging tempera-
tures of coals. Slagging temperatures are composition dependent and range
from 2000 °F to ~2900 °F. In actual non-slagging processes being proposed
temperatures range from 800 °F (~700 K) to 2400 °F (~1590 K) and pressures
range from 100 to 1000 psi. In all gasifiers, there will be entrained solids
of coal, char, ash and, in some processes, dolomite. Gaseous components may
include H_2, CO, CO_2, H_2O, H_2S, N_2, NH_3, CH_4, organic sulfur compounds, chlo-
rides, hydrocarbons, organic acids, and at lower temperatures, tar and oils.
These constituents create both an erosive and corrosive environment.

Possible problems could arise from phase changes or reactions within the
refractory material at elevated temperatures or pressures as well as reactions
between the refractory and components of the gasification atmosphere, primarily
with steam, hydrogen, and carbon monoxide. Thermal and mechanical stresses
may be substantial, especially during start-up and shut-down procedures and
there are also questions of the mechanical strength and creep under high
stress for monolithic linings. Coping with these problems will require a
careful blend of material selection, design considerations, and close control
of process conditions. Most likely, a multiple-layered refractory will be
necessary, i.e, a dense concrete or brick layer exposed to the process condi-
tions backed up by an insulating porous layer to protect the metal pressure
shell from excessive temperatures. Unfortunately, operating conditions in
gasifiers are sufficiently different from other severe industrial processes,
such as blast furnaces and petrochemical facilities, that substantial tech-
nology transfer is uncertain.

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A.2.2.2.1 PLANT EXPERIENCE

Information concerning the performance of refractories in actual use in coal gasification plants is limited to data gathered from only three pilot plants. The usual operation of a pilot plant subjects the refractories to the shock of many more temperature and pressure cycles than would normal commercial scale operation so that good performance on a pilot scale should indicate better commercial use performance.

Minimal information is available for insulating refractories. Phase changes which occurred during operation in one pilot plant for one alumino-silicate insulating castable are given in Section A.2.2.2.1.1. Comparison of the compounds present initially in the refractory with those found in used material taken from around a sample port adjacent to the steel vessel shell where the temperature was about 95 °C (200 °F) indicated marked changes. The initial material contains, in order of apparent abundance, first the calcium aluminate bonding phases, anorthite ($CaAl_2Si_2O_8$) and gehlenite ($Ca_2Al_2SiO_7$) and simple calcium aluminate ($CaAl_2O_4$), followed by the aggregate materials, quartz (SiO_2) and mullite ($Al_6Si_2O_{13}$). The exposed refractory contains none of the bonding phases of the original material, all of the calcium present being in the form of calcium carbonate, indicating exposure to and reaction with carbon dioxide under conditions which permitted decomposition of the bonding phases. The silica present in the original bonding phases exists largely as cristobalite in the exposed material, and the mullite and quartz aggregate of the original material seem relatively unaffected. Some gibbsite (hydrated alumina) has also formed. The sample was described as very friable, which is consistent with the breakdown of the strong bonding phases and the lack of formation of new strong bonding phases. Such changes may not be serious for the insulating refractory in a vessel provided the hard-faced inner portion of the refractory lining remains intact. If, however, the hard-faced liner should fail, permitting reactive process gases to penetrate to the insulation, the degraded insulation would no longer provide protection from chemical and thermal attack to the metal shell.

More information is available for performance of hard-faced refractories from one coal gasification pilot plant. The pilot plant operation involved circulation of char or lignite with limestone to take up excess carbon dioxide. At the time plant operation was ended, samples of the refractory lining of the gasifier were taken. Two different calcium-aluminate-bonded medium-alumina refractories had been in use, an abrasion-resistant castable in the lower "boot" section (and as vapor stops at several heights) and a dense castable in the rest of the vessel. A full description of the sampling and analysis of the refractory is contained in Dobbyn et al. [18]. Visual examination of the refractory revealed cracking, but all cracks were tight and the lining offered good protection to the vessel shell. Some minor chipping and spalling had occurred along crack edges in the lower fluidized-bed portion of the vessel, but none along cracks in the upper vessel region (freeboard). General erosion was so slight that original casting marks were still visible on the refractory surface. Core samples were drilled from the lower part of the vessel, but the refractory in the upper portion proved so hard that it was only possible to obtain chips.

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Physical properties of the samples were measured: apparent porosity, water absorption, apparent specific gravity, and bulk density (see Section A.2.2.2.1.5). Eight samples of each material were chosen randomly from the core specimens. The data, summarized below, show that the uniformity of the installed refractory was good and that no one portion of the refractory was affected by exposure more than another.

Refractory	Property	Average Value for 8 Samples	Maximum Percent Deviation From Average
Abrasion-Resistant Castable	Apparent Porosity	30.17%	4.6
	Water Absorption	15.30%	6.6
	Apparent Specific Gravity	2.820	0.8
	Bulk Density	1974 kg/m^3	1.6
Dense Castable	Apparent Porosity	30.35%	3.8
	Water Absorption	15.42%	5.4
	Apparent Specific Gravity	2.826	0.4
	Bulk Density	1968 kg/m^3	1.6

X-ray diffraction studies of laboratory-prepared, fired refractory showed that the major constituents were the mullite and cristobalite aggregate compounds with calcium aluminate bonding phases. Examination of the refractory from the vessel lining (see A.2.2.2.1.2) indicated a number of reactions had taken place during the operation of the gasifier. The amount of bonding phases in proportion to aggregate materials had increased markedly. The original calcium aluminates, $CaAl_2O_4$ and $CaAl_4O_7$, had reacted with the silica in the presence of the steam in the gasifier atmosphere to form anorthite ($CaAl_2Si_2O_8$), a strong bonding phase. In some samples, the anorthite apparently was the most abundant compound. Gehlenite ($Ca_2Al_2SiO_7$), a good bonding phase, also formed and some of the calcium in the circulating lime may have been involved in this reaction. Appreciable amounts of nepheline, $(Na,K)_2Al_2Si_2O_8$, were also formed, the alkali probably being supplied by the lignite which was circulated during the second half of the operation of the plant. Nepheline is also a good bonding phase. In general, these bonding phases were better crystallized and more abundant in samples taken from the hot face of the refractory than in samples deeper in the refractory cores.

Changes in the compressive strength and erosion resistance of the refractories are consistent with the enhancement of the bonding phases. Comparison of the compressive strength of the liner refractories with that of laboratory-prepared, fired samples indicates large increases due to exposure in the gasifier atmosphere (see A.2.2.2.1.3). The exposed samples showed wide-ranging percent increases in strength for both refractories.

Refractory	Increase in Strength of Exposed Samples
Abrasion-Resistant Castable	24, 40, 42, 70, 100, 131, 175, 186 %
Dense Castable	82, 95, 99, 127, 182 %

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Erosion losses of samples subjected to SiC abrasive (see A.2.2.2.1.4) indicate increased erosion resistance for exposed material compared with control specimens (laboratory-prepared and fired). Material loss of the exposed samples was generally considerably less than the control samples.

Refractory	Increase in Erosion Resistance of Exposed Samples
Abrasion-Resistant Castable	25, 37, 44, 46, 56, 60, 68 %
Dense Castable	46, 57, 59, 62, 64, 72 %

In this pilot plant environment, two medium-alumina refractories performed well and the properties were apparently enhanced due to chemical reactions which occurred in the hydrothermal gasifier atmosphere.

Many refractories, especially those including iron-containing compounds, can experience deterioration in the presence of CO-containing atmospheres over long time periods. A little information concerning carbon penetration of the refractory lining of one pilot plant gasifier vessel is available. After about 1000 hours in a reducing atmosphere at 1000-1550 °F a core specimen was drilled from the refractory lining in the high-temperature reaction zone of the vessel. The carbon content distributed through the core sample is shown in Section A.2.2.2.1.6. The lining consisted of a dense 50% alumina castable reinforced with 310 SS fibers at the hot face backed with a low-iron 50% alumina insulating castable. The analysis shows that carbon penetrated almost nine inches from the hot face, well into the insulating layer. Only these data are available from plant experience but more extensive laboratory testing has been done on CO degradation (see Section A.2.2.2.3.1).

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POROSITY AND DENSITY[a] FOR SAMPLES OF THE GASIFIER REFRACTORY LINING[b] OF

A GASIFICATION PILOT PLANT[c][18]

MATERIAL

Sample No.[d]	Apparent Porosity[a] %	Water Absorption[a] %	Apparent Specific Gravity[a] x 1000	Bulk Density[e] kg/m^3
ABRASION-RESISTANT CASTABLE[b]				
1	31.578	16.295	2832	1938
2	30.481	15.485	2832	1968
3	30.057	15.224	2823	1974
4	30.465	15.511	2825	1964
5	30.069	15.369	2798	1956
6	29.718	14.987	2821	1983
7	29.446	14.811	2818	1988
8	29.238	14.688	2813	1991
DENSE CALCINED FLINT CLAY CASTABLE[b]				
1	31.001	15.928	2821	1946
2	30.543	15.495	2838	1971
3	30.584	15.613	2822	1959
4	30.150	15.279	2825	1973
5	29.192	14.596	2825	2000
6	30.124	15.269	2823	1973
7	30.678	15.684	2822	1956
8	30.498	15.514	2828	1966

[a] Apparent porosity, water absorption, and apparent specific gravity were all measured according to ASTM C20-70.

[b] Lining hot face consisted of a 6-in. layer of an abrasion resistant castable (~37% SiO_2, 57% Al_2O_3, 6% CaO; Lo-Abrade, A.P. Green) in the lower "boot" section of the vessel and the same material was used as vapor stops at several heights; the rest of the vessel had a hot face lining of a dense calcined flint clay castable (~40% SiO_2, 50% Al_2O_3, 10% CaO; KS-4V, A.P. Green).

[c] Conoco Lignite Gasification Pilot Plant, CO_2 Acceptor Process, was in operation about five and one-half years; the time the system operated fully at temperature (1500-1600 °F) and pressure (150 psig) was about 3/4 year; the number of pressure cycles to which the refractory was subjected was at least 175, the number of temperature cycles at least 123, the number of runs 72; gaseous environment varied widely from run to run but steam was one of the major reactants to which the refractory was subjected; in approximately one half of the runs char was used, in the latter half lignite was used.

[d] Core samples were drilled with a 2-in. water-cooled diamond core drill which yielded a core 1-3/4 in. in diameter; samples were from various locations within the portion of the vessel occupied by the char(lignite)-dolomite fluidized bed; core samples could not be taken from higher levels because of the extreme hardness of the material in the upper regions; samples were prepared from both the hot face end of the cores and from the cold end adjacent to underlying insulation or the vessel shell; samples are in random order since there was little difference in the values from location to location.

[e] Bulk density was determined from the measured dimensions and weight of samples.

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CARBON MONOXIDE PENETRATION[a] OF THE REFRACTORY LINING[b] OF A HIGH-BTU
GASIFIER REACTOR[c][91]

ESTIMATED TEMPERATURE PROFILE

DISTANCE FROM HOT FACE (in.)

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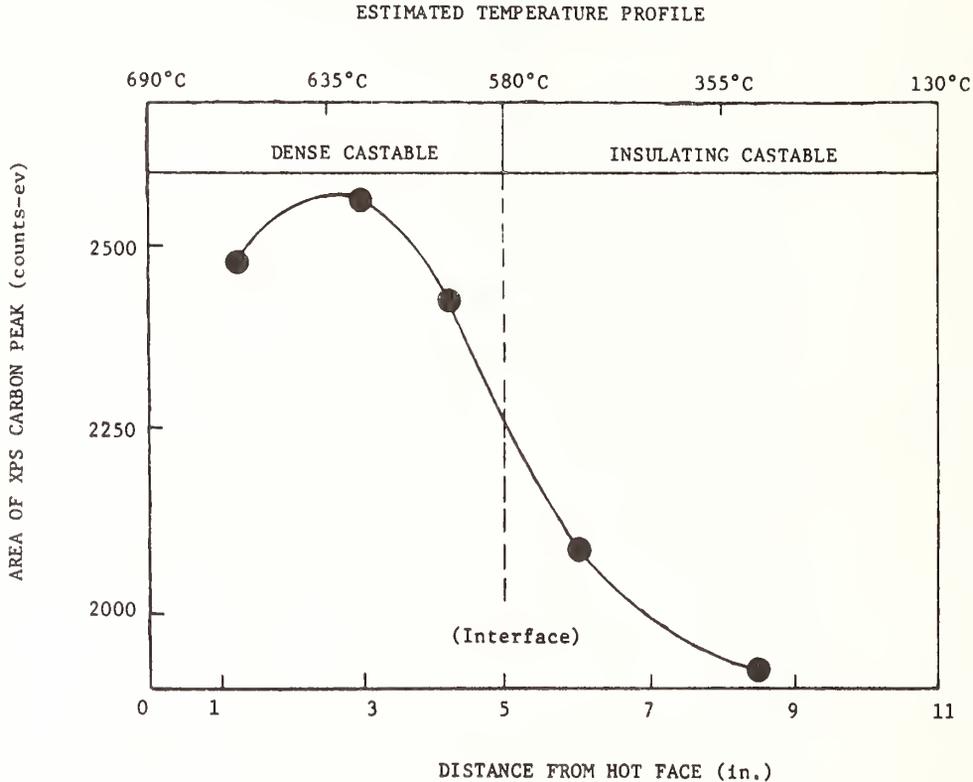
[a]As measured by x-ray photoelectron spectroscopy (XPS). Immediately before
obtaining the XPS scans, specimens were ion-milled with argon to remove
surface hydrocarbon contamination.

[b]A 1-in. diameter core, about 11 in. long was drilled from the refractory
lining of the barrel section of the high-temperature reaction zone of the
vessel. The lining consisted of a 5-in. dense layer of a high-strength
prototype castable (Mix 32C, Babcock & Wilcox) reinforced with 310 SS
fibers; the remainder of the lining was a commercial low-iron 50% alumina
insulating castable (HW-2800, Harbison-Walker).

[c]The high-Btu reactor operated by the Institute of Gas Technology (IGT).
The lining had been in service nearly 1000 hours in a reducing atmosphere
of H_2, CO, CH_4, CO_2, and H_2O at 500 psig in temperatures ranging 1000 °F-
1550 °F.

A.2.2.2.2 COMPONENT TEST AND DEVELOPMENT

Since it is vital to maintain the integrity of a refractory lining and therewith the capability of the refractory to protect the covered metal from chemical attack and thermal effects, it is important to use proper installation techniques to minimize the effect of thermal shock causing cracking and spalling. Of prime importance is the initial drying-out and heating up of the lining after installation. Experimental work has been done with small cast panels and test pressure vessels to determine the effect of anchor types and spacing, installation techniques, firing rates, and temperature on crack patterns and sizes and on shrinkage.

In one set of tests, small (15 in. x 18 in.) panels were prepared with 12-inch thick refractory (Section A.2.2.2.2.1). Various anchor types were used, with various firing schedules, and the crack patterns formed were observed. The 12-inch refractory layer consisted of about seven inches of 50 percent alumina lightweight commercial insulating castable and about five inches of 90 percent alumina dense generic castable for most of the tests; one test panel consisted of 12 inches of 50 percent alumina dense generic castable. Most of the firing schedules utilized heating rates of 100 °F/hour, but, in two tests using faster rates (260 °F/ hour and 480 °F/hour), the panels spalled explosively. In general, the longer the holding times at low temperature and the slower the heating and cooling rates, the fewer and smaller were the cracks which formed.

A second set of tests (see A.2.2.2.2.2) was performed using a test pressure vessel of seven-foot overall height, four-foot working height, and an inner diameter of five feet, which was heated internally and pressurized with steam. The vessel was lined a number of times and the refractories subjected to various patterns of heating and cooling cycles. The linings were examined visually after each cycle and cores were also taken and examined. Crack widths were measured and crack patterns observed. The same refractories were used as had been used in the small panel tests, a 7.5-inch thick 50 percent alumina insulating layer, and a 4.5-inch layer of the 90 percent and 50 percent alumina dense castables. One test was performed with a 42 percent alumina insulating castable and an acid-resistant mortar next to the vessel shell. Just as had occurred in the panel tests, a high heating rate after a relatively short holding period at lower temperatures caused explosive cracking and spalling. Because the crack pattern was found to be related to the pattern of the anchors and because many cracks propagated through the hard-face layer to the insulation, sometimes to the shell, the rest of the tests were done with linings installed in such a way that space between anchors was increased and bonding barriers between refractory and anchors and between refractory layers existed. These barriers, materials which are destroyed during firing, were a thin layer of ceramic paper fiber or plastic film between the layers of dense and insulating refractory; ceramic paper, plastic film, or a coating of silicone grease between the vessel shell and the insulating refractory; and a thin wrap of masking tape or asphalt-based tape around the anchors. From the tabulation in A.2.2.2.2.2, the following general conclusions may be drawn. Increasing the space between anchors provides fewer sites for crack pattern formation. Placement of a bonding barrier between the dense and insulating layers of the lining is an effective method of preventing propagation of cracks through the interface. Although cracking patterns

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frequently follow anchor spacings, coating the anchors does reduce this rela-
tionship and much finer and fewer cracks result. Coating the metal shell also
provides for fewer cracks in the insulation layer. For both the high- and
medium-alumina refractories, installation with bonding barriers improved per-
formance with respect to crack formation and propagation. As the linings were
heat cycled, there was no appreciable increase in crack formation during second
or even third heating cycles, even when the vessel was steam pressurized as
part of the cycle. Cracking and spalling were greatly reduced by the slow
heating rates.

     Core samples of these test refractory linings were not only examined
visually, but some physical and mechanical property measurements were also
made. Density measurements were made by immersion, volume, and mercury poros-
ity techniques. The values reported in Section A.2.2.2.2.4 are averages of ten
(in some cases, five) samples from each location, each from separate drill
cores. Measurements include values for both hot face and cold ends of the
cores. Examination of the data shows that there is no real difference in
refractory densities attributable to the heat treatment or the location of the
samples or the test method, compared with the densities for laboratory-prepared
specimens (controls). The following table demonstrates this fact for the dense
castables; the 50 percent alumina insulating castable data has greater scatter,
but the average is in good agreement. The data for the 42 percent alumina are
limited but the values indicate about an eight percent density decrease between
as-cast and post-test core samples.

Refractory	Density of Controls	Firing Temp. of Controls	Average All Core Samples, Tests & Methods	Maximum Difference Average & Core	Difference Core Average & Controls
90% castable	166 kg/m^3	2000 °F	177 kg/m^3	2.3%	6.6%
50% castable	136	1850	140	3.9	2.9
50% insulation	85	1500	85	19.	0.

The porosity data show much greater scatter but no obvious trends to indicate
changes due to exposure to the thermal or pressure cycling. Unfortunately, as-
cast or fired porosity data are not available for comparison.

     Cold crushing strength tests (see A.2.2.2.2.5) and diametral compression
tests yielding splitting tensile strength data (see A.2.2.2.2.3) were performed
on samples taken from the cores of the liner to determine the effect of thermal
cycling on the refractory strength. The data are not extensive, but examina-
tion of these results given in Table A.2.2.2.2a indicates possible trends. As
is to be expected, higher temperatures tend to increase strength. Although the
data are sparse, there is an indication that the presence of steam somewhat
weakens the 90 percent alumina dense castable but strengthens the 50 percent
alumina dense castable to a marked extent. The 50 percent alumina insulating
refractory seems not to be affected in a consistent way, probably because

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penetration of steam completely through the dense hard-face layer of the lining is minimal, and even the effect of temperature is not consistent. The data for the 42 percent alumina insulating refractory (A.2.2.2.2.3) is too sparse for any conclusion although it must be noted that the test specimens lost strength compared with as-cast specimens. These data, indicating an effect of steam on high- and medium-alumina castables, are consistent with the information included in Sections A.2.2.2.1 and A.2.2.2.3.

Refractory	Maximum Exposure Temperature	Splitting Tensile Strength	Cold Crushing Strength
90% dense castable (hot face values)	1200 °F	985 psi	8004 psi
	2000	1870	13160
	1950	1880	15672
	1850 + steam	1563	9939
50% dense castable (hot face values)	1200	260	2964
	1700	700	
	1850	845	
	1850	435*	
	1200 + steam	638	
	1850 + steam	715	
	1850 + steam	879	5732
50% insulating castable (interface values with 90% hard face)	800	200	1750
	1500	220	1293
	1500	340	2081
	1400 + steam	230	1341
50% insulating castable (interface values with 50% hard face)	800	270	2248
	1700	200	
	1850	320	
	1200 + steam	401	
	1400 + steam	558	2228

*Lining with 42 percent alumina insulating castable.

Table A.2.2.2.2a

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CRACKING AND SHRINKAGE OF REFRACTORIES SUBJECTED TO VARYING HEATING SCHEDULES[a] AND ANCHOR CONFIGURATIONS
IN A TEST PRESSURE VESSEL[b][26]

Lining Material[c]	Anchor Configuration	Heating Schedule[a]	Crack Width[d]		% Shrinkage[e]		Gap Width[f]	Observations
			ver.	hor.	ver.	hor.		
90% Al_2O_3 generic dense castable[g]/ 50% Al_2O_3 insulating[h] castable	12 in. vertical spacing, 18 in. circumferential spacing, V anchors	200 °F for 16 h, 400 °F for 16 h, 100 °F/h to 1000 °F, 1000 °F for 3 h, 100 °F/h to 1200 °F, cool at 50 °F/h to ambient.					NA	Crack pattern seen to be related to anchor spacing; no cracks observed to have propagated from dense to insulating layer.
		400 °F for 16 h, 100 °F/h to 1000 °F, 1000 °F for 3 h, 100 °F/h to 2000 °F, 2000 °F for 5 h, cool at 50 °F/h to ambient.	up to 1/8 in.				NA	Cracks enlarged and new cracks formed; cracks continuous from dense layer through interface and insulation, many almost to shell; cracks intersect at anchor and/or refractory interface.
90% Al_2O_3 generic dense castable[g]/ 50% Al_2O_3 insulating[h] castable	12 in. vertical spacing, 18 in. circumferential spacing, V anchors, coated with 0.020-0.030 in. masking tape	200 °F for 16 h, 400 °F for 16 h, 100 °F/h to 1000 °F, 1000 °F for 3 h, 100 °F/h to 1200 °F, cool at 50 °F/h to ambient.	0.028	0.102	0.2	0.2	NA	Little or no anchor/refractory bond, but anchors still affect crack formation; crack patterns similar to above test; one 5-in. diameter spalled area.
90% Al_2O_3 generic dense castable[g]/ 50% Al_2O_3 insulating[h] castable; ceramic fiber paper 0.030 in. thick placed between vessel shell and insulation, and between dense layer and insulation.	No anchors	100 °F/h to 200 °F, 200 °F for 16 h, 100 °F/h to 400 °F, 400 °F for 16 h, cool at 50 °F/h or slower to ambient.	0.005	0.005	0.05	0.05	NM	Numerous fine cracks on the hotface but no horizontal and vertical pattern as before.
		100 °F/h to 400 °F, 400 °F for 8 h, 100 °F/h to 1000 °F, 1000 °F for 3 h, 100 °F/h to 1200 °F, cool at 50 °F/h or slower to ambient.	0.017	0.010	0.09	0.11	NA	Increased cracking but still in random pattern (more random than in above linings); explosive cracking/spalling at 800-1000 °F (16.7 h) due to high heating rate; caused 1/2-1 in. wide crack around entire circumference of hotface; no cracks propagated through interface.
		100 °F/h to 400 °F, 400 °F for 5 h, 100 °F/h to 1000 °F, 1000 °F for 3 h, 100 °F/h to 2000 °F, 2000 °F for 5 h, cool at 50 °F/h or slower to ambient.	0.020	0.018	0.14	0.17	0.125	Little change in crack pattern from last heat; still no great degree of crack propagation through interface; insulation heavily randomly cracked, well-bonded to shell (ceramic paper penetrated by cement phase at installation).
90% Al_2O_3 generic dense castable[g]/ 50% Al_2O_3 insulating[h] castable; silicone coated ceramic fiber paper 0.046 in. thick between vessel shell and insulation; plastic film 0.004 in. thick between dense layer and insulation.	12 in. vertical spacing, 30 in. circumferential spacing for standard Y anchors; 36 in. circumferential spacing for Longhorn Y anchors to anchor insulation to shell independently; most but not all anchors coated with 0.100 in. thick asphalt-based tape[i]	50 °F/h to 1000 °F, 100 °F/h to 1200 °F, cool at 50 °F/h or slower to ambient.	0.016	0.009	0.07	0.10	NA	Fairly fine cracks, no propagation through interface; still related to anchor locations.
		50 °F/h to 1000 °F, 100 °F/h to 1850 °F, 1850 °F for 10 h at 120 psig steam, depressurize and cool at 50 °F/h or slower to ambient.	0.031 0.032[1]	0.017	0.16 0.36[1]	0.21	0.090	Only slight increase in cracking, pattern more random than in other tests with anchors.
50% Al_2O_3 commercial castable[j]/ 50% Al_2O_3 insulating[h] castable; 0.004 in. thick plastic film at vessel shell and between dense and insulating layers.	Same specifications as above but with all anchors coated with tape	50 °F/h to 1000 °F, 100 °F/h to 1200 °F, cool at 50 °F/h or slower to ambient.	0.007	0.005	0.04	0.04	NM	Fewer and finer cracks than in previous tests at 1200 °F, no gap at dense component-insulation interface.

(Table Continued)

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CRACKING AND SHRINKAGE OF REFRACTORIES SUBJECTED TO VARYING HEATING SCHEDULES[a] AND ANCHOR CONFIGURATIONS

IN A TEST PRESSURE VESSEL[b][26], Continued

Lining Material[c]	Anchor Configuration	Heating Schedule[a]	Crack Width[d] ver.	Crack Width[d] hor.	% Shrinkage[e] ver.	% Shrinkage[e] hor.	Gap Width[f]	Observations
(See previous page)		50 °F/h to 1000 °F, 100 °F/h to 1850 °F, hold 3 h while pressurizing to 140 psig, 1850 °F and 140 psig steam for 10 h, depressurize and cool at 50 °F/h or slower to ambient.	0.034	0.030 0.024[l]	0.21	0.26 0.26[l]	∿0.05	Less cracking than in tests with 90% Al_2O_3 dense refractory; no cracks propagated through refractory interface.
50% Al_2O_3 commercial castable[j]/ 50% Al_2O_3 insulating castable[h]; vessel shell was coated with silicone grease, 0.004 in. thick plastic film between dense layer and insulation.	Average spacing between anchors 30 in., standard Y anchors; no independent anchors; all anchors coated with 0.100 in. thick asphalt-based tape[i]	50 °F/h to 1000 °F, 100 °F/h to 1200 °F, steam pressurize from 350 °F at 15 psig/h, hold at 1200 °F and 150 psig 48 h, depressurize and cool at 50 °F/h to ambient.	0.007	0.005	0.04	0.04	NM	Less cracking than in previous linings; only very fine cracks in dense layer except one large one (30-40 mils); cracks follow anchor spacing; very few cracks in insulation, cracks which propagated through interface extended only a few inches into insulation from dense layer.
		50 °F/h to 1000 °F, 100 °F/h to 1850 °F, steam pressurize from 350 °F at 15 psig/h, hold at 1850 °F and 150 psig 35 h, depressurize and cool at 50 °F/h to ambient.	0.004	0.012 0.020[l]	0.03	0.07 0.48[l]	0.050	Less cracking than in other linings tested at 1850 or 2000 °F; most cracks did not propagate entirely through each component.
50% Al_2O_3 commercial castable[j] with 4 wt% 310 SS fiber added[k]/ 50% Al_2O_3 insulating castable[h]; vessel shell was coated with silicone grease, 0.004 in. thick plastic film between dense layer and insulation.	Average spacing between anchors 30 in., standard Y anchors; all anchors coated with 0.100 in. thick asphalt-based tape[i]	50 °F/h to 1000 °F, 100 °F/h to 1700 °F, 1700 °F for 10 h, cool at 50 °F/h or slower to ambient.	0.012[m]		0.10[m]		0.050	Only very fine cracks on hotface, pattern of cracks similar to earlier tests, running horizontally and vertically; cracks did not propagate through the component; least shrinkage of all linings tested so far.
		50 °F/h to 1000 °F, 100 °F/h to 1700 °F, pressurize with air to 100 psig, hold at 1700 °F and 100 psig 10 h, depressurize and cool at 50 °F/h or slower to ambient.	0.009[m]		0.095[m]		0.050	No additional cracking, cracks still very fine.
		100 °F/h to 1850 °F, 1850 °F for 10 h, cool at 50 °F/h or slower to ambient.	0.012[m]		0.13[m]		>0.050	No additional cracking, some cracks widened, but few visible to the naked eye; no propagation to the interface from the hotface; some fine cracks in insulation but none propagated to the shell.
		>250 °F/h to 1700°F, cool at >150 °F/h to ambient, same cycle repeated.	0.014[m]		0.13[m]		0.055	Only minor additional cracking of dense refractory; further cracking of insulation, not considered severe.

(Table Continued)

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CRACKING AND SHRINKAGE OF REFRACTORIES SUBJECTED TO VARYING HEATING SCHEDULES[a] AND ANCHOR CONFIGURATIONS

IN A TEST PRESSURE VESSEL[b][26], Continued

Lining Material[c]	Anchor Configuration	Heating Schedule[a]	Crack Width[d] ver.	hor.	% Shrinkage[e] ver.	hor.	Gap Width[f]	Observations
50% Al$_2$O$_3$ commercial castable[j] with 4 wt% 310 SS fiber added[k]/ 42% Al$_2$O$_3$ lightweight insulating castable[n]/ acid-resistant mortar[o] next to vessel shell; shell sandblasted, no bonding between shell and mortar; 0.004 in. thick plastic film between mortar and insulation and between insulation and dense layer.	30-36 in. spacing between anchors, standard Y anchors with V legs all oriented vertically; all anchors coated with 0.080 in. thick asphalt-based tape[i]	25 °F/h continuous heat to 1850 °F and slow cool.	0.005[m]			0.019[m]	0.056	Dense component had some very fine, hairline cracks; no propagation very far into layer; insulation cracked badly, many cracks propagated to the shell; lining had little shrinkage.

[a] Linings were subjected to the conditions given in the table, but were frequently cored and sampled, then patched before being subjected to the next test cycle.

[b] Test pressure vessel has an inner diameter of 5 feet, a 7-foot overall height with a 4-foot working height; vessel was heated internally.

[c] Eight individual different linings are described in the Lining Material column. All but the last lining had an insulation layer 7.5 in. thick with a 4.5 in. thick dense layer. The last lining had a 0.5 in. thick layer of mortar against the vessel shell, a 7 in. thick insulation layer, with a 4.5 in. thick dense layer. For several tests bonding barriers were placed between the insulation and the vessel shell and at the interface between the refractory layers.

[d] Cracks were measured at 90° intervals horizontally around the lining (hor.) and at three 15 in. intervals vertically (ver.). The crack widths given, in inches, are average values.

[e] Linear shrinkage calculations assume summation of crack widths accounts for shrinkage in vertical and horizontal directions from the as-cast condition.

[f] Gap width, reported in inches, was measured between the dense and insulating components; NA = not determined, NM = not measurable.

[g] DOE 90 generic preparation: 70% tabular alumina (T-61, Alcoa), 5% calcined alumina (-325 mesh), 25% calcium aluminate cement (Casting Grade CA-25, Alcoa).

[h] Commercial lightweight insulating castable, calcium aluminate bonded (Litecast 75-28, General Refractories).

[i] Presstite, Virginia Chemicals.

[j] Coarse grain size, high-strength 50% alumina prototype castable (B&W Kaocrete XD50, Mix 36C, Babcock & Wilcox).

[k] 1-in. long fibers of 310 SS (Ribtec 310).

[l] Average of cracks in the vertical and horizontal directions for the insulating component of the lining.

[m] Average of cracks in vertical and horizontal directions.

[n] Commercial lightweight castable, 42% alumina (B&W Kaolite 2300 LI, Babcock & Wilcox).

[o] Acid-resistant mortar (HES, Pennwalt Co.) was applied directly on the sandblasted vessel shell and cured by heating to 200 °F over a 20 h period and soaking at 200 °F for 35 h before application of the insulating refractory.

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TENSILE STRENGTH DETERMINED BY DIAMETRAL COMPRESSION TESTING[a] OF REFRACTORY SAMPLES
FROM LININGS[b] OF A TEST PRESSURE VESSEL[c][26]

Heating Schedule[d]	Lining Material[b]	Sample Location[e]	Tensile Strength[a]
First cycle: 200 °F for 16 h, 400 °F for 16 h, 100 °F/h to 1000 °F, 1000 °F for 3 h, 100 °F/h to 1200 °F, cool at 50 °F/h to ambient. Second cycle: 400 °F for 16 h, 100 °F/h to 1000 °F, 1000 °F for 3 h, 100 °F/h to 2000 °F, 2000 °F for 5 h, cool at 50 °F/h to ambient.	90% Al_2O_3 dense generic castable[f] 50% Al_2O_3 lightweight insulating castable[g]	Hot face Interface	1870 psi 220
One cycle: 200 °F for 16 h, 400 °F for 16 h, 100 °F/h to 1000 °F, 1000 °F for 3 h, 100 °F/h to 1200 °F, cool at 50 °F/h to ambient.	90% Al_2O_3 dense generic castable[f] 50% Al_2O_3 lightweight insulating castable[g]	Hot face Interface	985 200
First cycle: 100 °F/h to 200 °F, 200 °F for 16 h, 100 °F/h to 400 °F, 400 °F for 16 h, cool at 50 °F/h or slower to ambient. Second cycle: 100 °F/h to 400 °F, 400 °F for 8 h, 100 °F/h to 1000 °F, 1000 °F for 3h, 100 °F/h to 1200 °F, cool at 50 °F/h or slower to ambient. Third cycle: 100 °F/h to 400 °F, 400 °F for 5 h, 100 °F/h to 1000 °F, 1000 °F for 3 h, 100 °F/h to 2000 °F, 2000 °F for 5 h, cool at 50 °F/h or slower to ambient.	90% Al_2O_3 dense generic castable[f] 50% Al_2O_3 lightweight insulating castable[g]	Hot face Interface Interface	1880 1110 340
First cycle: 50 °F/h to 1000 °F, 100 °F/h to 1200 °F, cool at 50 °F/h or slower to ambient. Second cycle: 50 °F/h to 1000 °F, 100 °F/h to 1850 °F, 1850 °F for 10 h at 120 psig steam, depressurize and cool at 50 °F/h or slower to ambient.	90% Al_2O_3 dense generic castable[f] 50% Al_2O_3 lightweight insulating castable[g]	Hot face Interface Interface Cold face	1570 1185 230 255
First cycle: 50 °F/h to 1000 °F, 100 °F/h to 1200 °F, cool at 50 °F/h or slower to ambient. Second cycle: 50 °F/h to 1000 °F, 100 °F/h to 1850 °F, hold 3 h while pressurizing to 140 psig, 1850 °F and 140 psig steam for 10 h, depressurize and cool at 50 °F/h or slower to ambient.	50% Al_2O_3 commercial castable[h] 50% Al_2O_3 lightweight insulating castable[g] 50% Al_2O_3 commercial castable[h]	Hot face Interface Interface Cold face Hot face	250 305 270 250 715
First cycle: 50 °F/h to 1000 °F, 100 °F/h to 1200 °F, steam pressurize from 350 °F at 15 psig/h, hold at 1200 °F and 150 psig 48 h, depressurize and cool at 50 °F/h to ambient. Second cycle: 50 °F/h to 1000 °F, 100 °F/h to 1850 °F, steam pressurize from 350 °F at 15 psig/h, hold at 1850 °F and 150 psig 35 h, depressurize and cool at 50 °F/h to ambient.	50% Al_2O_3 commercial castable[h] 50% Al_2O_3 lightweight insulating castable[g] 50% Al_2O_3 commercial castable[h] 50% Al_2O_3 lightweight insulating castable[g]	Hot face (As-cast)[i] Interface(As-cast) Interface(As-cast) Cold face(As-cast) Hot face Interface Interface Cold face	638(521)[i] 575(552) 401(362) 370(387) 879 781 558 333
First cycle: 50 °F/h to 1000 °F, 100 °F/h to 1700 °F, 1700 °F for 10 h, cool at 50 °F/h or slower to ambient.	50% Al_2O_3 castable[h] with 4 wt% 310 SS fiber[j] 50% Al_2O_3 lightweight insulating castable[g]	Hot face Interface Interface Cold face	725 430 140 180

(Table Continued)

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TENSILE STRENGTH DETERMINED BY DIAMETRAL COMPRESSION TESTING[a] OF REFRACTORY SAMPLES

FROM LININGS[b] OF A TEST PRESSURE VESSEL[c][26], Continued

Heating Schedule[d]	Lining Material[b]	Sample Location[e]	Tensile Strength[a]
Second cycle: 50 °F/h to 1000 °F, 100 °F/h to 1700 °F, pressurize with air to 100 psig, hold at 1700 °F and 100 psig 10 h, depressurize and cool at 50 °F/h or slower to ambient.	50% Al_2O_3 castable[h] with 4 wt% 310 SS fiber[j]	Hot face / Interface	700 / 710
	50% Al_2O_3 lightweight insulating castable[g]	Interface / Cold face	200 / 220
Third cycle: 100 °F/h to 1850 °F, 1850 °F for 10 h, cool at 50 °F/h or slower to ambient.	50% Al_2O_3 castable[h] with 4 wt% 310 SS fiber[j]	Hot face / Interface	845 / 765
	50% Al_2O_3 lightweight insulating castable[g]	Interface / Cold face	320 / 210
Fourth cycle: >250 °F/h to 1700 °F, cool at >150 °F/h to ambient, repeat same cycle.	50% Al_2O_3 castable[h] with 4 wt% 310 SS fiber[j]	Hot face / Interface	510 / 740
	50% Al_2O_3 lightweight insulating castable[g]	Interface / Cold face	315 / 210
One cycle: 25 °F/h continuous heat to 1850 °F and slow cool.	50% Al_2O_3 castable[h] with 4 wt% 310 SS fiber[j]	Hot face (As-cast)[i] / Interface(As-cast)	435(475)[i] / 420(450)
	42% Al_2O_3 lightweight insulating castable[k]	Interface(As-cast) / Cold face(As-cast)	40(90) / 70(90)

[a] Splitting tensile test designed around ASTM test C496-71 was used; specimens were 1-in. thick cylinders, sectioned from 2-in. diameter drill cores; specimens were compressed to failure between 1/32-in. aluminum sheets along a diameter direction. Units are psi.

[b] The linings correspond, in order, to the linings in Section A.2.2.2.2.2. In the above table the long dashed lines separate the tests on a given lining. The dotted lines separate the tests done on the same linings after the specific heating cycle.

[c] Test pressure vessel has an inner diameter of 5 feet, a 7-foot overall height with a 4-foot working height; vessel was heated internally.

[d] Heating schedules are the same as those given in Section A.2.2.2.2.2.

[e] Samples were taken at different stages with respect to the heating cycles to which they were subjected. The first linings were core drilled only after all the heating cycles were completed; later linings were core drilled after each cycle and then patched for the next cycle. Also, samples examined for the early linings were not taken from the complete core depth as they were for the later tests.

[f] DOE 90 generic preparation: 70% tabular alumina (T-61, Alcoa), 5% calcined alumina (-325 mesh), 25% calcium aluminate cement (Casting Grade CA-25, Alcoa).

[g] Commercial lightweight 50% alumina insulating castable, calcium aluminate bonded (Litecast 75-28, General Refractories).

[h] Coarse grain size, high-strength 50% alumina prototype castable (B&W Kaocrete XD50, Mix 36C, Babcock & Wilcox).

[i] Values in parentheses are the as-cast values.

[j] 1-in. long fibers of 310 SS (Ribtec 310).

[k] Commercial lightweight castable, 42% alumina (B&W Kaolite 2300 LI, Babcock & Wilcox).

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EFFECT OF HEAT CYCLING[a] ON THE DENSITY AND POROSITY OF REFRACTORY FROM LININGS OF A TEST PRESSURE VESSEL[b][26]

Lining Material	Sample Location[c]	Heat Cycle Maximum Temperature[d]	Maximum Exposure Temperature[e]	Density, [f] lb/ft^3			Porosity, [f] %	
				Immersion	Mercury	Volume	Immersion	Mercury
90% Al$_2$O$_3$ dense generic castable[g]	Hot face	2000 °F	2000 °F	173		177	17.1	17.4
	Interface		1800	176		176	16.4	15.2
50% Al$_2$O$_3$ insulating castable[h]	Interface		1500	80		81	36.6	21.8
	Cold face		400	88		74	37.7	18.7
90% Al$_2$O$_3$ dense generic castable[g]	Hot face	1200	1200	181		175	15.2	9.5
	Interface		950	180.6		174	14.8	9.3
50% Al$_2$O$_3$ insulating castable[h]	Interface		800	78.5		74.7	35.1	19.7
	Cold face		210	78		74.8	32.4	24.0
90% Al$_2$O$_3$ dense generic castable[g]	Hot face	2000	1950	177	178.6	176	15.7	15.8
	Interface		1650	176	178.0	174	16.8	16.2
50% Al$_2$O$_3$ insulating castable[h]	Interface		1500	85.6	89.7	82.9	36.2	43.8
	Cold face		450	87.2	96.8	85.3	26.5	44.3
90% Al$_2$O$_3$ dense generic castable[g]	Hot face	1850 steam[i]	1800	179	180	176	13.6	11.3
	Interface		1600	179	180	175	14.2	14.1
50% Al$_2$O$_3$ insulating castable[h]	Interface		1400	76	74	76	39.6	28.2
	Cold face		450	77.5	87.7	78	41.4	34.7
50% Al$_2$O$_3$ commercial castable[j]	Hot face	1200	1200	141	143	139		12.8
	Interface		950	141	145	139		12.4
50% Al$_2$O$_3$ insulating castable[h]	Interface		800	87	87	85.8		29.1
	Cold face		210	86	88.5	85.8		34.2
50% Al$_2$O$_3$ commercial castable[j]	Hot face	1850 steam[i]	1800		139	137		19.9
	Interface		1600		139	136		18.9
50% Al$_2$O$_3$ insulating castable[h]	Interface		1400		86	83		36.5
	Cold face		450		89	85		45.2
50% Al$_2$O$_3$ commercial castable[j]	Hot face	1200 steam[i]	1200	144[k]	138.8[k]			14.5
	Interface		950	145.5[k]	139[k]			13
50% Al$_2$O$_3$ insulating castable[h]	Interface		850	101.5[k]	90.5[k]			29
	Cold face		350	98[k]	87.8[k]			30
50% Al$_2$O$_3$ commercial castable[j]	Hot face	1850 steam[i]	1850	142[k]	139[k](140.7)[l]			18
	Interface		1550	141.5[k]	139[k](141.3)[l]			18
50% Al$_2$O$_3$ insulating castable[h]	Interface		1400	98[k]	84.7[k](86.1)[l]			31
	Cold face		500	95.5[k]	88.6[k](85.2)[l]			34
50% Al$_2$O$_3$ castable[j] plus 310 SS fibers[m]	Hot face	1700				144		
	Interface					142		
50% Al$_2$O$_3$ insulating castable[h]	Interface					83		
	Cold face					82.5		
50% Al$_2$O$_3$ castable[j] plus 310 SS fibers[m]	Hot face	1700				143		
	Interface					142		
50% Al$_2$O$_3$ insulating castable[h]	Interface					84		
	Cold face					84		
50% Al$_2$O$_3$ castable[j] plus 310 SS fibers[m]	Hot face	1850				144		
	Interface					140		
50% Al$_2$O$_3$ insulating castable[h]	Interface					88		
	Cold face					88		
50% Al$_2$O$_3$ castable[j] plus 310 SS fibers[m]	Hot face	1700				142		
	Interface					144		
50% Al$_2$O$_3$ insulating castable[h]	Interface					82.3		
	Cold face					84.2		

(Table Continued)

EFFECT OF HEAT CYCLING[a] ON THE DENSITY AND POROSITY OF REFRACTORY FROM LININGS OF A TEST PRESSURE VESSEL[b][26], Continued

Lining Material	Sample Location[c]	Heat Cycle Maximum Temperature[d]	Maximum Exposure Temperature[e]	Density,[f] lb/ft^3			Porosity,[f] %	
				Immersion	Mercury	Volume	Immersion	Mercury
50% Al_2O_3 castable[j] plus 310 SS fibers[m]	Hot face Interface	1850 °F				142.2(144.5)[l] 140(143.4)[l]		
42% Al_2O_3 insulating castable[n]	Interface Cold face					59(64)[l] 62(68)[l]		

[a] The refractory linings were installed, subjected to various heat cycling schedules, and core drilled for testing at various stages. For some of the heating cycles the vessel was sealed and pressurized. The linings and heating schedules are those given in Section A.2.2.2.2.2. The long dashed lines in the above table separate the individual linings; the dotted lines separate different heating cycle data for the same lining.

[b] Test pressure vessel has an inner diameter of 5 feet, a 7-foot overall height with a 4-foot working height; vessel was heated internally. Linings consisted of 7.5-in. thick insulation layer and 4.5-in. thick dense component.

[c] Samples were taken from drill cores; the designations indicate the distance of the sample from the hot face surface: Hot face = 1/2 in., Interface (dense layer) = 4 in., Interface (insulation) = 5 in., Cold face = 11 in.

[d] The temperatures, in °F, correspond to the maximum temperatures designated in each heating cycle, see Section A.2.2.2.2.2.

[e] The maximum temperature, in °F, actually experienced by the designated portion of the lining. These values are not given for all samples in the original reports.

[f] Densities and porosities were determined by immersion, volume, and mercury porosity techniques; density values are each the average of 10 samples from each location, each taken from separate drill cores, except for values marked with k which are the average of 5 samples; porosity values are the average of 2 samples. Sample densities may be compared with densities (volume, lb/ft^3) of laboratory samples: 90% Al_2O_3 material from cast bars, as-cured 179, dried at 250 °F 175, fired to 2000 °F 166, fired to 1800 °F 165; 50% Al_2O_3 insulation, from bars corresponding to linings with 90% Al_2O_3 dense component, as-cured 79.4, dried at 250 °F 76.0, fired to 1500 °F 85, fired to 400 °F 82; 50% Al_2O_3 insulation for linings with 50% Al_2O_3 dense component, as-cast values reported varied 86 to 100.2, dried at 250 °F from 84.6 to 89.2, fired to 1400 °F 85, fired to 500 °F 82; 50% Al_2O_3 dense component, as-cast values reported varied from 141 to 147.4, dried at 250 °F from 137.7 to 141.6, fired to 1850 °F 136, fired to 1550 °F 136.

[g] DOE 90 generic preparation: 70% tabular alumina (T-61, Alcoa), 5% calcined alumina (-325 mesh), 25% calcium aluminate cement (Casting Grade CA-25, Alcoa).

[h] Commercial lightweight 50% alumina insulating castable, calcium aluminate bonded (Litecast 75-28, General Refractories).

[i] The vessel was steam-pressurized at 120-140 psig for these cycles.

[j] Coarse grain size, high-strength 50% alumina prototype castable (B&W Kaocrete XD50, Mix 36C, Babcock & Wilcox).

[k] These values are the average of 5 samples, see footnote f.

[l] Values in parentheses are as-cast values.

[m] 1-in. long fibers of 310 SS (Ribtec 310).

[n] Commercial lightweight castable, 42% alumina (B&W Kaolite 2300 LI, Babcock & Wilcox).

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COLD CRUSHING STRENGTHS[a] OF REFRACTORY FROM LININGS OF A TEST PRESSURE VESSEL[b][26]

Lining Material[b]	Sample Location[c]	Maximum Exposure[d] Temperature, °F	Cold Crushing Strength[a] psi
90% Al$_2$O$_3$ dense generic castable[e]	Hot Face	2,000	13,160
	Interface	1,800	11,787
50% Al$_2$O$_3$ castable insulation[f]	Interface	1,500	1,293
	Cold Face	400	1,778
90% Al$_2$O$_3$ dense generic castable	Hot Face	1,200	8,004
	Interface	950	9,439
50% Al$_2$O$_3$ castable insulation	Interface	800	1,750
	Cold Face	210	1,324
90% Al$_2$O$_3$ dense generic castable	Hot Face	1,950	15,672
	Interface	1,650	8,611
50% Al$_2$O$_3$ castable insulation	Interface	1,500	2,081
	Cold Face	450	2,413
90% Al$_2$O$_3$ dense generic castable	Hot Face	1,800[h]	9,939
	Interface	1,600	7,392
50% Al$_2$O$_3$ castable insulation	Interface	1,400	1,341
	Cold Face	450	1,563
50% Al$_2$O$_3$ commercial castable[g]	Hot Face	1,200	2,964
	Interface	950	2,575
50% Al$_2$O$_3$ castable insulation	Interface	800	2,248
	Cold Face	210	2,098
50% Al$_2$O$_3$ commercial castable	Hot Face	1,800[h]	5,732
	Interface	1,600	4,910
50% Al$_2$O$_3$ castable insulation	Interface	1,400	2,228
	Cold Face	450	2,332

[a] Each value is the average of the results of testing 5 samples from each location, each taken from separate drill cores. Values may be compared with hot crushing strength values for laboratory samples of the same materials: 90% Al$_2$O$_3$ castable, at 2000 °F, 8455 ± 420 psi, at 1500 °F, 9130 ± 1480 psi; 50% Al$_2$O$_3$ castable insulation, at 1500 °F, 5330 ± 430 psi, at 500 °F, 3490 ± 180 psi; 50% commercial castable, at temperatures between 950 °F and 1800 °F, values ranged around 3300 psi.

[b] Test pressure vessel has an inner diameter of 5 ft., a 7-ft. overall height with a 4-ft. working height; vessel was heated internally. Linings consist of 7.5 in. thick insulation layer and 4.5 in. thick dense component.

[c] Samples were taken from drill cores; the designations indicate the distance of the sample from the hot face surface: Hot Face = 1/2 in., Interface (dense layer) = 4 in., Interface (insulation) = 5 in., Cold Face = 11 in.

[d] Linings were subjected to a variety of heating cycles with varying patterns of holding at various temperatures and some cycles with the vessel sealed and pressurized; temperatures listed are the maximum values reached by the samples during the heating cycles to which they were subjected.

[e] DOE 90 generic preparation: 70% tabular alumina (T-61, Alcoa), 5% calcined alumina (-325 mesh), 25% calcium aluminate cement (Casting Grade CA-25, Alcoa).

[f] Commercial lightweight 50% Al$_2$O$_3$ insulating castable, calcium aluminate bonded (Litecast 75-28, General Refractories).

[g] Coarse grain size, high strength 50% Al$_2$O$_3$ prototype castable (B&W Kaocrete XD50, Mix 36C, Babcock & Wilcox).

[h] The vessel was steam pressurized at 120-140 psig for these cycles.

A.2.2.2.3.1 CHEMICAL AND PHASE CHANGES

OVERVIEW

A knowledge of the chemical and physical changes in refractories in use in coal conversion vessels is necessary from two points of view: first, the formation of the initial refractory and second, the reaction of the refractory to its use environment. The formation of the initial refractory is normally dependent on a series of reactions which occur during its preparation and firing which may include hydration, dehydration, and formation of new chemical species through reaction of preexisting compounds. The reaction products, will ultimately determine the chemical, physical, and mechanical properties of the materials. In addition to the chemical composition, the physical attributes, i.e., aggregate size and size distribution, may play a significant role in defining the properties of a refractory. One further complication should be noted. The performance of any refractory material can be greatly influenced by the installation technique used.

During use, potential problem areas include chemical reaction of the refractory constituents with components of the gasification atmosphere to produce compounds with less desirable properties, i.e., lower melting point, less erosion resistance, lower mechanical strength, or having large volume changes. To a large extent the integrity, strength, and chemical and physical resistance is primarily dependent upon the formation and retention of strong bonding phases occurring in the cement portion as opposed to the aggregate of the refractory. The reactions and changes involving cement portions of refractory castables are found to be of particular importance. A knowledge, therefore, of cement bonding phases and their stability ranges is critical to the successful application of refractory linings.

LABORATORY TESTS

THE EFFECT OF CONSTITUENTS OF COAL GASIFICATION ATMOSPHERES on the mineral phases present in various refractories is tabulated in a number of subsections in B.1.2.

Eleven alumina refractories were examined for phases present after curing and drying, after firing to 1100 °C, and after exposure of fired samples to steam, to hydrogen, to carbon monoxide, and to a mixed gas environment like that given in Section B.0 for various times at 1100 °C and 1000 psi (see B.1.2.5). The phases were identified by means of x-ray diffraction. The refractories tested had alumina contents ranging from 95 percent to 45 percent. Some were castables, both calcium aluminate bonded and phosphate bonded, and some were bricks. One of the test samples was neat calcium aluminate cement. The data are not complete for all refractories under all test conditions.

Hydrogen had little effect generally on high-alumina materials except for two brick refractories. One of the two observed component phases, β-alumina (β-$NaAl_{11}O_{17}$), disappeared after hydrogen exposure of these high-alumina bricks. The only effect observed due to CO exposure was the decrease in certain calcium aluminates ($CaAl_2O_4$, $CaAl_4O_7$, $CaAl_{12}O_{19}$, and $Ca_2Al_2SiO_7$) in the high- and medium-alumina calcium aluminate-bonded castables. One might expect weakened materials because of this loss since these compounds are bonding phases. Steam or the mixed gas, which is intended as a simulated coal gasification atmosphere, had a

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greater effect, especially with respect to medium-alumina (45 to 60 percent)
calcium aluminate-bonded castable or brick refractories. High-alumina castables
or brick showed less change. The major change in castables appeared with regard
to bonding phases, the various calcium aluminate compounds. The calcium alumi-
nates tended to disappear and another bonding phase, anorthite ($CaAl_2Si_2O_8$),
to form. Neat calcium aluminate cement when tested only showed a slight increase
in β-$NaAl_{11}O_{17}$ in steam. This same compound disappeared under steam exposure in
the 99 percent alumina brick. The medium-alumina brick showed apparent conver-
sion in steam of the α-cristobalite to tridymite.

THE EFFECT OF STEAM is shown more directly in a series of experiments which
subjected alumina castable refractories to hydrothermal treatment (see B.1.2.6
and B.1.2.7). The hydrothermal treatment consisted of exposing samples to satu-
rated steam in a pressure vessel at varying temperatures or, at a single tempera-
ture followed by heating to various test temperatures. The refractories were
generic and commercial calcium aluminate-bonded castables, three of which were
medium-alumina (50, 55.6 and 55 percent) and one high-alumina (95 percent). For
medium-alumina castables the general trend as the temperature is raised is to
observe fewer different constituent phases in the samples. The hydrothermal
treatment reduces the number of phases also. For medium-alumina materials the
phases which are consistently present upon exposure to heat and steam are mullite
($Al_6Si_2O_{13}$), cristobalite (SiO_2), and anorthite ($CaAl_2Si_2O_8$). Other phases,
particularly hydrated phases, tend to disappear with temperature. The one high-
alumina castable tested formed gibbsite ($Al_2O_3 \cdot 3H_2O$) and boehmite ($Al_2O_3 \cdot H_2O$)
at the lower temperatures and these compounds disappeared at higher temperatures.
A combination of steam and carbon dioxide was used as a test media for a 94.4 per-
cent alumina generic castable bonded with calcium aluminate cement (see B.1.2.8).
The refractory was exposed to 70% H_2O-30% CO_2 in a pressure vessel at temperatures
up to 910 °C. X-ray diffraction analysis of the mineral phases showed the forma-
tion of hydrated phases $Al_2O_3 \cdot 3H_2O$, $Ca_3Al_2O_6 \cdot 6H_2O$, $Al_2O_3 \cdot H_2O$ as well as calcium
carbonate and β-alumina (β-$NaAl_{11}O_{17}$). α-Alumina, $CaAl_4O_7$ and $CaAl_2O_4$ are the
original constituents which remain present but in varying amounts.

Another series of experiments performed on a medium-alumina generic castable
(55.6 percent alumina) (see B.1.2.9) showed that the same 70% H_2O-30% CO_2 composi-
tion at temperatures up to 910 °C increased the amount of the anorthite component
of the refractory but no evidence of calcium carbonate was reported. Only trace
amounts were found when the atmosphere was 50% H_2O-50% CO_2 and none was detected
when the atmosphere was pure carbon dioxide.

THE EFFECT OF HEAT TREATMENT on the mineral phases of high- and medium-
alumina castables is given in Sections B.1.2.10 and B.1.2.34. The results of
heating the refractories to 1350 °C is consistent with the pattern seen in the
experiments with steam and various gases. The very high alumina (75 percent
tabular alumina/25 percent calcium aluminate cement) generic castable in B.1.2.10
and the commercial castable in B.1.2.34 are largely α-alumina with smaller amounts
of $CaAl_2O_4$, $CaAl_2O_4 \cdot 10H_2O$ and $Al_2O_3 \cdot 3H_2O$. These small amounts of the hydrated
phases appear at low temperatures (from ambient to 200 °C) and disappear at
higher temperatures. At the higher temperatures $CaAl_4O_7$ and $CaAl_{12}O_{19}$ appear.
The differences between the constituents for the generic and the commercial
castable are probably due to small differences in the constituents of the orig-
inal mixes. The medium-alumina refractories (75 percent bauxite or kaolin ag-
gregate with calcium aluminate cement) maintain a fairly consistent composition
with regard to major constituents (mullite, cristobalite, α-alumina, $CaAl_2O_4$,

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$CaAl_4O_7$, etc.) over the lower temperature ranges with anorthite appearing at 800 °C and becoming a major constituent at 1200 °C. The two refractories made with calcined kaolin aggregate did show formation of appreciable amounts of anorthite at 1200 and 1350 °C. A 50 percent alumina insulating castable showed traces of anorthite formation at 1200 °C. The heat stability of phases in these same refractories was studied by differential thermal analysis and the results appear in Section B.1.2.11. It is apparent that the data in B.1.2.11, with all refractories showing changes between 250-300 °C, are consistent with the dehydration of the hydrated calcium aluminates above 200 °C as indicated by the x-ray diffraction data in B.1.2.10.

THE MINERAL PHASES OF NEAT CALCIUM ALUMINATE CEMENT as affected by steam was studied in a pressure vessel so constructed that specimens could be examined in situ by energy dispersive x-ray diffraction (see B.1.2.12). Temperature was varied from 25 °C to 1000 °C and pressure from ambient to 1130 psig for time periods from 1 to 46 hours. The initial compounds, $CaAl_2O_4$, $CaAl_4O_7$ and α-alumina, are diminished in proportion as hydrated aluminas and calcium aluminates are formed. These hydrated compounds remain the major phases up to about 400 °C when boehmite ($Al_2O_3 \cdot H_2O$) begins to dissociate. At and above 500 °C there is no evidence for the existence of any hydrated compounds. The major constituent of the cement at 600 °C and above is $CaAl_4O_7$ with a very small amount of α-Al_2O_3. In other experiments samples of five neat calcium aluminate cements were exposed to simulated coal gasification atmospheres with and without H_2S, both steam saturated and unsaturated. For some tests specimens were exposed not only to the vapor phase but were also immersed in the liquid in the vessel. After the exposures the specimens were examined by x-ray diffraction and differential thermal and thermogravimetric analyses (see B.1.2.20). One cement was the same high-purity (79 percent alumina, 18 percent calcia) cement tested and reported in B.1.2.12. The initial materials, primarily alumina and $CaAl_2O_4$, formed boehmite and calcite in the simulated coal gasification atmosphere (see B.0) at 500 °C and 500 psi pressure. The exposure periods were 10, 20, and 30 days. Exposures to the coal gasification atmosphere for the same periods of time at 1000 °C and 1000 psi produced no boehmite as shown by the x-ray diffraction and the differential thermal and thermogravimetric analyses. Exposures to other steam-containing or steam-saturated atmospheres or immersion in the test vessel liquid for as little as 10 days showed the strong formation of boehmite as long as the temperature was below 500 °C. Differences in pressure (410 or 1000 psig) seemed to make little difference to the boehmite formation.

Two other cements, both 72 percent alumina and ∿25 percent calcia, tested in the same ways, showed similar compounds were formed with the addition of $CaAl_4O_7$ as an important phase which was not found in the higher alumina cement. Two medium-alumina cements were tested, one 58 percent alumina with 33 percent calcia and one 44 percent alumina with 36 percent calcia. (Some of the original reports indicated the presence of titania in the cements but the compositions given were not consistent.) For these cements alumina was not generally present, the major constituent phases being $CaAl_2O_4$, $CaAl_4O_7$, and $Ca_2Al_2SiO_7$. The data for these cements also show boehmite formation at the lower temperatures and a lack of boehmite at the high temperatures.

TESTING IN COAL GASIFICATION ATMOSPHERE was also performed on seven high-alumina (91 to 95 percent) castables and four medium-alumina (54 to 59 percent) castables under the same conditions as the testing of the neat calcium aluminate cements (see B.1.2.17, B.1.2.18 and B.1.2.21). In addition to the coal gasifica-

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tion atmosphere, some tests were done in steam-carbon monoxide atmospheres at
465 psi and 390, 500, and 1000 °F for 10 day periods. The same steam-saturated
vapor testing and immersion in test vessel liquid was also performed with the
castable refractories as were done with the neat cements. The effects of ex-
posure were studied using the same methods as with the neat cements. The results
for all the high-alumina dense castables were essentially the same. The initial
material consisted of alumina. Boehmite and calcite formed as major constituents
in addition to the alumina as the materials were exposed to the coal gasification
atmosphere at 500 °C and 500 psi, or at 447 °C and 1000 psi. Tests in steam-
carbon monoxide at 465 psi and 500 °C and below also showed alumina, boehmite and
calcite as major constituents. Just as with the neat cements the boehmite was
not found in the high-alumina castables after exposure to these atmospheres when
the temperature was 1000 °C. In the medium-alumina dense and lightweight insu-
lating castables, boehmite was not found under any conditions except in minor and
trace quantities by means of x-ray diffraction. The differential thermal and
thermogravimetric analyses showed some amounts of boehmite in the medium-alumina
materials comparable to those found in the high-alumina castables.

Cycling tests (see B.1.2.21) were run specifically to determine the effect
of steam-saturated vapor and liquid immersion exposure to temperature-pressure
cycling on the phases present in seven alumina castables, especially with regard
to boehmite formation and decomposition. The test atmospheres included steam
alone and steam-saturated coal gasification gases. The cycling tests included
exposure to the steam-saturated coal gas test atmosphere for five days followed
by firing at 1000 °C in air for 18 hours. Four dense castables were tested and
three insulating castables. The dense castables included 93 percent alumina,
87 percent alumina, 64 percent alumina-28 percent silica, and 58 percent alumina-
30 percent silica and the data for these show that the boehmite which forms at or
below 500 °C is decomposed by the firing at 1000 °C. Other calcium aluminates do
not persist with this treatment. The x-ray data show mostly alumina present with
calcite as a minor phase for the high-aluminas, and mullite as the major phase
and some alumina and calcite present for the medium-aluminas. The three insu-
lating low-aluminas tested also show an absence of boehmite, but the data are
sparse for two of these materials. Further tests of these same high- and low-
alumina castables in saturated and unsaturated carbon monoxide/steam/hydrogen
sulfide atmospheres are in agreement with the above results with respect to the
presence of boehmite only below 500 °C. The data are incomplete for the stated
tests, however (see Section B.1.2.32). Specimens of a high-alumina phosphate-
bonded ramming mix apparently showed no real changes during the testing. In
Section B.1.2.31 the effect of immersion in the test vessel liquid on the calcia
content of four of the castables is given. The higher the carbon monoxide
content of the test atmosphere, the more calcia was leached from the refractories.

THE EFFECT OF EXPOSURE TO ALKALI ON THE ALKALI CONTENT of some refractories
was tested. Nine refractories were exposed to alkali-gas, and alkaline solution-
alkali gas. These refractories included two high-alumina calcium aluminate-
bonded dense castables (one commercial product and one generic preparation),
three medium-alumina calcium aluminate-bonded dense castables (all commercial
products), one dense high-alumina phosphate-bonded castable (commercial), one
high-alumina phosphate-bonded ramming mix (commercial), one medium-alumina cal-
cium aluminate-bonded insulating castable, and one medium-alumina fired brick
(commercial). The results of a variety of exposures to alkali are given in
B.1.2.4 expressed as Na_2O-K_2O content of exposed samples in weight percent. Not
all samples were tested in all of the various exposures. Table A.2.2.2.3.1a

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| Refractory and Cement | Alkali Content in Weight Percent | | | | | |
| | Prefired Samples | | Minimum for All Exposures | | Maximum for All Exposures | |
	K_2O	Na_2O	K_2O	Na_2O	K_2O	Na_2O
Dense 95% tabular Al_2O_3 castable (Castolast G) high-purity calcium aluminate (CA-25)	0.008	0.018	0.24	0.33	2.40	1.4
Dense 90% tabular Al_2O_3 castable (Generic Preparation) high-purity calcium aluminate (CA-25)	0.01	0.20	0.24	0.22	1.5	1.5
Dense 55% Al_2O_3 fire clay calcine castable (Lo-Abrade) high-purity calcium aluminate (CA-25)	0.54	0.28	1.4	0.51	5.00	2.0
Dense 45% Al_2O_3 fire clay calcine castable (H.S. Brikcast BF) medium-purity calcium aluminate (Refcon)	0.12	0.06	0.82	0.30	3.3	2.0
Dense 45% Al_2O_3 fire clay calcine castable (B & W Kaocrete D) low-purity calcium aluminate (Secar 50)	0.26	0.04	1.8	0.52	3.5	2.0
Dense 90% tabular Al_2O_3 castable (Resco Cast AA-22) phosphate bond	0.008	0.07	0.70	0.33	2.4	1.1
Lightweight 50% Al_2O_3 insulating castable pyrophyllite aggregate (Litecast 75-28) high-purity calcium aluminate (CA-25)	---	---	3.00	1.20	6.00	2.70
90% tabular Al_2O_3 ramming mix (Brikram 90R) phosphate bond	0.02	0.15	1.1	0.53	2.20	1.10
45% Al_2O_3 fired super duty brick, clay aggregate (KX-99)	---	---	1.30	0.29	2.10	0.65

Table A.2.2.2.3.1a (see text for legend)

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summarizes the data. It is readily seen that appreciable amounts of alkali are
taken up by the refractories even if one considers only the minimum values. If
the ratios of exposed alkali content (minimum or maximum) to initial alkali
content are examined, it is notable that the increase in potassium content is,
for most of the refractories, many times greater than the increase in sodium
content. Fireclay-based refractories tended to show the least increase but they
had a higher initial alkali content than the high-alumina refractories. The
exposure methods included exposure to alkali vapor only as well as soaking in
alkali solutions.

A second series of tests was performed using a variety of methods to intro-
duce potassium to the testing system in order to simulate a catalytic coal gasi-
fication atmosphere. Only potassium compounds were included since potassium
rather than sodium is taken up by the refractories to the greater extent. Seven
tests were performed, five in a simulated catalytic coal gasification gas and two
in a steam-argon atmosphere. Alkali addition to the atmosphere was introduced
variously by placing crucibles of molten KOH in the test chamber, by injection of
an aqueous solution of KOH at such a rate that the atmosphere was saturated with
KOH, and by packing holes in the specimens with KOH-impregnated coal. Some
specimens were not only exposed to the vapors of KOH from the crucibles but were
also submerged in the molten alkali. The results are reported in Section B.1.2.33
for the eight refractories tested. Not all refractories were subjected to all
the environments. Those refractories subjected to direct contact with molten KOH
were damaged severely. For the shortest exposure times (50 hours), either in the
simulated gasification atmosphere or in steam-argon with molten KOH as an alkali
source, no absorption of KOH from vapor phase transport was detected. For longer
times (100 hours or more) considerable absorption by vapor phase transport oc-
curred. Shorter (100-500 hour) exposures to the KOH-impregnated coal had little
effect on the refractories tested but after 2000 hours the specimens were de-
graded and large amounts of potassium were found to have migrated throughout the
specimens. The effect of the increase of alkali content on the mechanical prop-
erties of the refractories is discussed in A.2.2.2.3.4.

Limited testing was performed on refractories in a gas mixture simulating
the atmosphere in the devolatilizer section of a fixed-bed slagging gasifier
(Section B.1.2.36). The refractory specimens were subjected to alkali attack by
means of packing NaOH crystals into holes drilled in the specimens. A number of
the refractories tested were of the high-chromia spinel-type found to show good
resistance to slag corrosion (see Section A.2.3.2.2.1). The visual observations
and some x-ray diffraction results are reported after 50 and 100 hours of expo-
sure at 1000 °C and 360 psig. A fused-cast chromium magnesium-iron spinel ex-
hibited no evidence of attack and several fireclay super duty bricks apparently
performed well, showing little or no change after 100 hours. Ramming mixes of
varied types in general performed poorly as did a variety of alumina-bearing
refractories. X-ray diffraction showed that either carnegieite, β-alumina, or
gibbsite had formed causing volume expansion in alumina-containing materials.

THE EFFECT OF A "HIGH-BTU" GAS MIXTURE (21.7% H_2, 17.0% CO, 11.6% CO_2, 4.7%
CH_4, and 45% H_2O) ON THE SILICA CONTENT of a 55 percent alumina castable at
980 °C and 1000 psi for 250 hours was studied (see B.1.2.2). The refractory, a
dense alumina-fireclay calcined aggregate with calcium aluminate cement, was
exposed in the form of bars, powder, and crushed material, and the silica content
measured for intervals of exposure of 50 hours. Exposure of the bar samples to
gas with a calculated superficial velocity of 0.04 ft/min for varying times re-

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sulted in no significant change in silica content (about 1 weight percent). Ex-
posure of powder resulted in a slight increase in silica content (maximum of
4 weight percent). Scanty test data for crushed samples exposed to gas with a
calculated superficial velocity of 1.4 ft/min indicated a possible decrease in
silica content (maximum of 6 percent). It should be noted that the data are
limited but do not indicate serious silica loss under the test conditions.

THE EFFECT OF DIFFERENT ENVIRONMENTS ON A HIGH-ALUMINA PHOSPHATE-BONDED
REFRACTORY was tested (Section B.1.2.3). The refractory, a 90 percent alumina
ramming mix, was analyzed for P_2O_5 content by wet chemical methods after the test
exposures. There was an increase in phosphorus oxide content (compared with
cured but unexposed samples) after air-firing at 1100 °C for 24 and 250 hours,
after exposure to steam at 1100 °C and 1000 psi for 250 hours, after exposure to
CO at 1100 °C and 1000 psi for 125 hours, and after exposure to carbon dioxide at
1100 °C and 800 psi for 250 hours. Samples exposed to a simulated coal gasifica-
tion environment (see B.0) at 1000 °C and 1000 psi for 160 hours showed no sig-
nificant change in phosphorus oxide content. The P_2O_5 content of a sample ex-
posed to H_2 at 1100 °C and 1000 psi for 250 hours was drastically reduced from
2.97 weight percent to 0.03 weight percent. There was apparently no loss of
strength (see footnote, B.1.2.3) due to this loss of binder. Three high-alumina
phosphate-bonded ramming mixes were subjected to a simulated coal gasification
atmosphere at 500 °C and 500 psi and at 1000 °C and 1000 psi. The exposure times
for both sets of conditions were periods of 10, 20, and 30 days (see B.1.2.19).
X-ray diffraction analyses of the exposed materials compared with analyses of
air-fired samples showed no major changes due to exposure. The basic components
given are alumina and forms of aluminum phosphate. The exposures to the coal
gasification atmosphere seem to enhance the crystallinity of the berlinite form
of aluminum phosphate for at least two of the materials since the berlinite is
listed as increasing from trace amounts to minor or major phases after exposure.
It is difficult to say how much of this effect is due just to the increased
temperature. The exposures seem to have little other compositional effect on
these refractories. One of these ramming mixes (90 percent alumina) is included
in Section B.1.2.5 in which the effect of various gaseous exposures on mineral
phases was investigated by x-ray diffraction. In B.1.2.5 the phases are listed
as alumina, with quartz and cristobalite, and no aluminum phosphate, which should
be present, is mentioned. It should be pointed out that aluminum phosphate
occurs in forms which are structural analogs of the various forms of silica, i.e.
quartz, cristobalite, and tridymite. The x-ray diffraction peaks of the various
forms of silica and aluminum phosphate are extremely close together and mask each
other. This masking effect probably is the reason that no aluminum phosphate is
mentioned in Section B.1.2.5 and no silica forms are mentioned in B.1.2.19 al-
though small amounts were probably present.

THE EFFECT OF CARBON MONOXIDE ON IRON-CONTAINING REFRACTORIES was investi-
gated since iron may be present as an impurity in many refractories and it has
been implicated in carbon monoxide disintegration of refractories. Generic
preparations of three types of refractory were tested: 90 percent and 50 percent
alumina castables, both calcium aluminate-bonded, and a 90 percent alumina phos-
phate-bonded ramming mix (see B.1.2.13). The refractories were doped with vary-
ing amounts of iron, 0.5, 1.0, and 2.0 weight percent either as metallic iron or
Fe_2O_3. Samples were exposed to carbon monoxide at 500 °C for 100 hours at 200 psi
and 600 psi, to carbon monoxide at 1000 psi for 50 hours at 400, 450, 500, and
550 °C, to 80% CO-20% H_2O at 500 °C for 100 hours at 200 psi and 500 psi, and to

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a simulated coal gasification atmosphere (14% CO, 5% CO_2, 45% H_2, 17% H_2O, 17% CH_4, 0.1% H_2S) at 500 °C at atmospheric pressure for 1000 hours and 600 psi for 400 hours. Exposures to CO and CO-H_2O caused complete or partial disintegration of Fe-doped samples. Undoped samples were not seriously affected except for the 50 percent alumina material which showed serious degradation in CO at 600 psi. Exposure to the mixed gases of the coal gasification atmosphere did not seem to affect doped or undoped samples at all at atmospheric pressure but minor disintegration did occur at 600 psi. Results of visual examination were also reported for a series of carbon monoxide exposures which were performed to find the effect of exposure on the refractory strengths (see Sections A.2.2.2.3.4 and B.3.2.54). The various tests involved the inclusion of varying amounts of either CO_2, NH_3, H_2S, H_2O and H_2 with the CO to see the effect of each of these gases upon the action of the CO. The results are given in Section B.1.2.27. For the two castables tested, no appreciable effect was noticed except for color changes when the iron dopant was in the form of Fe_2O_3. When the dopant was metallic iron cracking and spalling was common. The exception to this degradation was the testing with CO-H_2O combinations which showed no changes. The ramming mix also showed no spalling in the CO/H_2O exposures but varying degrees of spalling and cracking in all other gas combinations. Samples of the two castables were also alkali-impregnated by soaking in alkali carbonate solution (see B.1.2.28). The specimens with alkali but no iron dopant were undamaged but alkali-impregnated samples with 1.0 weight percent Fe disintegrated after 100 hours at 500 °C in CO. Carbon monoxide exposure of a stainless steel fiber-reinforced 50 percent alumina castable was destructive (see Section B.1.2.29). Prefiring of an iron-doped 90 percent alumina castable before carbon monoxide exposure did not prevent disintegration of the specimens (see Section B.1.2.30).

Two dense castables, identified only as having 90 percent and 50 percent alumina were exposed to several high-Btu mixed gases and then examined for apparent changes (see Section B.1.2.35). Specimens of each either were doped with 0.1 percent iron, or contained 310 stainless steel fibers. Three insulating castables were also tested, a 95 percent alumina, a 55 percent alumina, and a 40 percent alumina. Specimens of each were doped with "rust" with no other details given. The presence of the 310 SS fibers apparently had a deleterious effect on both dense castables in the simulated coal gasification atmosphere like that described in Section B.0 with 1% H_2S. The exposure was only for 58 hours. The plain castables and the specimens doped with 0.1 percent iron were unaffected. A similar gas composition except for the absence of ammonia and hydrogen sulfide had an unfavorable effect on the plain dense castables and on the insulating castables with surface popouts being prominent.

SILICON CARBIDE AND SIALON REFRACTORIES have been tested for the effect of the mixed gas environment on the compounds present in these materials (see B.1.2.1). The SiC, whether oxynitride-, silicate-, or direct-bonded, completely disintegrated after 1000-hour exposure to 24% H_2, 18% CO, 12% CO_2, 5% CH_4, 1% H_2S, 2% NH_3, and 38% H_2O at 980 °C and 1000 psi. The breakdown of the material is due to formation of SiO_2 (as cristobalite with traces of quartz). The SiAlON samples remained intact but an outer layer of mullite and cristobalite formed which indicated some chemical effect of the gases on the material. Whether this outer layer indicates the beginning of disintegration or a protective layer is not known. Further testing is necessary since the data are limited.

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PILOT PLANT TESTING

THE EFFECT OF EXPOSURE TO COAL GASIFICATION PILOT PLANT CONDITIONS is shown
in Section B.1.2.16. Specimens of various refractories were placed on test racks
in a total of nine locations within six pilot plants. Not all refractories were
tested in all locations, and for some plants there was only one exposure, for
others two or three exposures. Rotating rod abrasion testing of exposed speci-
mens was performed at ambient temperature and at 1400 °F and the results compared
with abrasion tests performed on unexposed samples. The data are expressed as
volume loss of material and depth of penetration of the test rod. The tests are
not meant as a measure of abrasion or erosion resistance but as a measure of
changes which may have occurred in the bonding of the refractories as a result of
exposure in the plant. The full conditions to which the samples were exposed are
not available. Although run conditions are given they cannot be directly related
to the times for exposure of the refractories. Exposure times, when given, are
approximate, as are the concentrations of the chemical constituents of the atmo-
spheres, the temperatures, and the pressures. During plant operation these
variables fluctuated widely and the effect on the refractories is unknown. Not
all specimens were subjected to abrasion testing at 1400 °F. Evaluation of the
numerical data is difficult since percentage differences for exposed versus
unexposed data do not seem equally reliable for all cases and multiplicate sam-
ples were not tested. The results for separate exposures in the same location
differ widely for many of the materials.

Table A.2.2.2.3.1b contains a qualitative summary of the data in B.1.2.16
and constitutes an attempt to indicate materials which should be tested further
and those which may not be worth testing further. In the light of all of the
uncertainties outlined above, the data must be viewed with caution. The plant
locations in Table A.2.2.2.3.1b are:

Synthane 1	gasifier off-gas
2	gasifier fluidized bed
CO$_2$ Acceptor 1	gasifier off-gas
2	dolomite regenerator off-gas
BI-GAS	gasifier off-gas
Battelle 1	gasifier off-gas
2	combustor off-gas
U-GAS	gasifier off-gas
HYGAS	gasifier off-gas

The symbols used in the table carry the following meaning;

nt   the material was not tested in that location

-    the percent change in data between unexposed and exposed
     samples is generally less than or about equal to 50 percent in
     either direction

W   worse--the percent change in data between unexposed and
     exposed specimens generally is greater than 50 percent and
     indicates a weakening of the refractory bond after
     exposure

A.2.2.2.3.1   A.2.2 Refractory Linings and Components--Dry-Bottom Vessels
page 10 of 11   A.2.2.2 Performance Data
9/85   A.2.2.2.3 Materials Evaluation

Results of Refractory Coupon Testing in Pilot Plant Locations

Refractories	Synthane 1	Synthane 2	CO$_2$ Acceptor 1	CO$_2$ Acceptor 2	BI-GAS	Battelle 1	Battelle 2	U-GAS	HYGAS
CASTABLES, CALCIUM ALUMINATE-BONDED									
90+% Al$_2$O$_3$ dense (Purolite 30)	W	nt	W	W	nt	nt	nt	nt	nt
90+% Al$_2$O$_3$ dense (Greencast-97)	nt	nt	W	W	nt	nt	nt	nt	W
90+% Al$_2$O$_3$ light (Greencast-97L)	W	I	I	W	nt	nt	nt	nt	nt
90% Al$_2$O$_3$ dense (Castolast G)	W	I	W	W	nt	nt	nt	W	W
60% Al$_2$O$_3$ dense (Mizzou Castable)	B	B	I	W	I	?	?	I	B
54% Al$_2$O$_3$ dense (Super Brikcast A)	B	B	B	I	B	nt	nt	I	B
54% Al$_2$O$_3$ lightweight insulating (Litecast 75-28)	W	W	W	W	W	nt	nt	nt	W
52% Al$_2$O$_3$ light (Litecast 80)	W	nt	W	W	nt	nt	nt	nt	nt
PHOSPHATE-BONDED REFRACTORIES									
90+% Al$_2$O$_3$ dense castable (Resco Cast AA-22)	I	I	W	W	W	W	W	W	nt
90% Al$_2$O$_3$ ramming mix (Brikram 90)	I	W	I	B	I	B	B	B	I
73% Al$_2$O$_3$ dense ramming mix (Blu Ram)	I	nt	nt	nt	nt	nt	nt	nt	nt
73% Al$_2$O$_3$ dense ramming mix (Blu Ram H.S.)	I	nt	nt	nt	nt	nt	nt	nt	nt
60% Al$_2$O$_3$ ramming mix (Wasp #60)	I	I	W	B	W	I	nt	I	nt
85% Al$_2$O$_3$ dense fired brick (Chemal 85 B)	W	I	W	W	nt	nt	nt	nt	I
85% Al$_2$O$_3$ fired brick (Altex 85-B)	W	W	I	I	nt	nt	nt	nt	W
80+% Al$_2$O$_3$ fired brick (Alumex P-8)	W	I	I	I	nt	I	I	nt	W
ALUMINA BRICKS									
99+% Al$_2$O$_3$ self-bonded dense fired (H-W Corundum)	I	I	W	W	nt	W	nt	nt	I
99+% Al$_2$O$_3$ self-bonded fused-cast dense (Monofrax A)	B	B	I	B	nt	B	nt	B	nt
98+% Al$_2$O$_3$ self-bonded light insulating fired (Alfrax B 101)	W	I	W	W	nt	nt	nt	nt	I
90+% Al$_2$O$_3$ self-bonded dense fired (Kricor)	I	I	I	I	I	I	nt	nt	nt
77% Al$_2$O$_3$ self-bonded light insulating fired (B&W Insalcor)	W	I	W	W	nt	nt	nt	nt	W
70% Al$_2$O$_3$ vitreous bond fired (Mul-8)	I	I	I	I	nt	I	nt	nt	W
70+% Al$_2$O$_3$ vitreous fired (Hi Lumite 70 D)	W	I	W	I	I	nt	nt	nt	I
70% Al$_2$O$_3$ vitreous dense fired (Alumex 70-HD)	nt	nt	I	I	nt	nt	nt	nt	nt
60% Al$_2$O$_3$ tar-impregnated vitreous fired (Ufala TI)	I	I	I	I	?	nt	I	I	I
45-50% Al$_2$O$_3$ clay vitreous bond, high-fired super-duty (KX-99)	I	I	I	I	I	I	I	nt	I
MISCELLANEOUS BRICKS									
SiC silicon nitride-bonded dense fired (Refrax 20)	W	W	W	B	nt	nt	nt	I	nt
SiC oxynitride bond (Crystolon 63)	W	W	W	B	nt	nt	nt	nt	nt
SiC clay-bonded (Harbide)	nt	nt	nt	nt	nt	?	?	nt	nt
Alumina-zirconia-silica fused-cast (AZS)	W	nt	nt	nt	?	nt	nt	nt	nt
Zirconium silicate vitreous bond (Zircon)	nt	I	W	nt	nt	nt	nt	nt	nt
90% Al$_2$O$_3$/10% Cr$_2$O$_3$ solid solution fired (CS 612)	nt	nt	I	I	I	nt	nt	nt	nt

Table A.2.2.2.3.1b (see text for legend)

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B   better--the percent change in data between unexposed and
        exposed specimens is generally greater than 50 percent and
        indicates a strengthening of the refractory bond after
        exposure

?   data are incomplete--data given only for the unexposed
        sample or only for the exposed sample

The criteria used in the assignment of these symbols are completely ar-
bitrary. Some trends may be seen in examining Table A.2.2.2.3.1b. Among the
dense alumina castables the 60 percent alumina showed more promising performance
than the 90 percent alumina. The medium-alumina light castables do not perform
well but if used as insulating layers covered with dense castable these materials
would not be expected to face the same conditions as the denser materials. The
phosphate-bonded materials, whether castable, ramming mix, or brick, showed er-
ratic performance and need further testing for specific applications. Alumina
bricks of varying alumina content were tested. No trends are easily observed
based on alumina content or brick type except that generally the insulating
bricks showed poorer performance. In general, the medium-alumina castables and
the alumina bricks showed the best overall performance of the categories of
materials tested. SiC bricks received insufficient testing and in general per-
formed very poorly where tested. The other bricks listed, two zirconium-contain-
ing and one alumina-chromia, underwent so little testing that no conclusions are
possible.

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A.2.2.2.3.2 EROSION

OVERVIEW

Erosion occurs in various parts of coal conversion systems as abrasive
particles of coal, ash, char, or dolomite are driven by fluid pressures against
surface scales and metal or refractory surfaces. Some variables which affect
erosion include: type of erodent, erodent particle velocity, shape and hardness,
angle of impingement, temperature of material impacted, condition (including
hardness) of impacted material surface. Each of these variables is important in
determining the rate at which an erosion process takes place. It is important
that any testing methodology which is used to evaluate and rank material perfor-
mance takes into account all of these variables. Attention to each of these
testing variables has been considered in the testing methodology which is re-
ported in Sections B.2.1.1-.14, B.2.1.21-.22, and B.2.1.46-.51. Highlights of
this testing methodology include the following:

1. Erosion weight loss shows a mixed dependence on angle of impingement. In
   considering the effect of impingement angle on erosion, the character of the
   materials being subjected to the erosion must be considered, whether ductile
   (most metallic materials) or brittle (ceramic materials). For ductile
   (metallic) materials, weight loss shows a peak in the angular range 10-20°,
   usually followed by a systematic decrease as impingement angle approaches
   90° (see Section B.2.1.47). For brittle (ceramic) materials, the maximum
   erosion occurs at 90°. Examples of such performance are the data in Sections
   B.2.1.11 and B.2.1.14, which show erosion data typical of that for ductile
   materials, and Sections B.2.2.10 and B.2.2.13 (data at 20 °C), which show
   data typical of brittle materials. (Some variations of brittle materials
   behavior are to be seen at high temperatures because of phase changes in the
   refractories.)

2. Erosion weight loss is dependent upon the type of erodent. The rate of
   erosion tends to decrease as the hardness of the erodent decreases. Since
   char, ash, and coal particles are not as hard as silicon carbide or aluminum
   oxide, test results from the latter erodents will be conservative relative
   to a coal conversion environment. Most erosion test results reported in
   Sections B.2.1.1-.14, B.2.1.21-.22, and B.2.1.46-.51 were obtained with
   Al_2O_3 as the erodent. Sometimes SiC, SiO_2 or pumice was used as an erodent.
   Erosion due to char, ash, and coal particles is yet to be thoroughly evaluated.

3. Erosion weight loss increases with increasing erodent velocity in the range
   10 to 100 m/s. Data in B.2.1.5, B.2.1.13, B.2.2.7, B.2.2.11, B.2.2.12, and
   B.2.1.49 show the trend for both ductile and brittle materials. Some of the
   erosion data included in this book have been normalized by dividing the
   weight of sample lost by the weight of erodent used. Generally, a greater
   amount of erodent creates a greater material loss, although when very large
   amounts of erodent are involved there is not a corresponding increased
   erosion effect, probably because erodent particles are acting against each
   other. Section B.2.2.8 contains data showing an initial decrease in erosion
   at larger particle flux and then the approach of a steady state.

4. Erosion weight loss increases with increasing particle size in the range
   five to 50 μm. Particle size and erosion are generally directly propor-
   tional. In Sections B.2.1.9 and B.2.1.13 there are data showing increased
   material loss with increased particle size.

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5. Erosion weight loss depends upon the temperature of the material impacted,
    and may increase or decrease with increasing temperature, depending upon the
    material. Data for alloys in Sections B.2.1.5, B.2.1.9, B.2.1.10, B.2.1.11,
    and B.2.1.12 do not indicate a definite trend. Conflicting results are also
    indicated for refractory materials in B.2.2.8, B.2.2.11, B.2.2.12, and
    B.2.2.13. Although data in B.2.2.8 and B.2.2.13 show increased erosion loss
    with increasing temperature, the results in B.2.2.11 and B.2.2.12 are con-
    flicting. For both alloys and refractories, the results seem more dependent
    on the response of individual materials to increased temperature in terms of
    possible changes in the properties rather than a direct effect of temperature
    as a parameter on the phenomenon of erosion.

6. Erosion weight loss depends upon the hardness of the material impacted and
    tends to decrease slightly as the material hardness increases.

LABORATORY TESTS

    THE EROSION OF CASTABLE REFRACTORIES was studied using 150 μm SiC particles
at several particle velocities and temperatures. The SiC particles are much
harder and, therefore, subject the test materials to more severe erosion than the
char and ash in a coal gasifier. Investigators [22] have found that erosion of
castables occurs primarily within the cement matrix until enough of the matrix
has been eroded away from the harder aggregate particles to permit dislodging of
the aggregate particles by the erodent.

    THE EFFECT OF IMPINGEMENT ANGLE AND TEMPERATURE is to be seen in B.2.2.10
and B.2.2.13. A high-alumina (95 percent) calcium aluminate-bonded commercial
castable was subjected to erosion at varying impingement angles at 25 °C and
1000 °C (see B.2.2.13). The curve of material loss vs. angle at 25 °C for 28 m/s
particle velocity fits the pattern expected for the erosion of a brittle material,
i.e., less material loss at low angles and a maximum loss at 90°. At 1000 °C and
39 m/s particle velocity the behavior is similar to that expected of ductile
materials with a maximum material loss at low angles, evidence that there is
plastic flow of the cement matrix occurring at high temperatures. The investi-
gators [22] found small pieces of the SiC erodent embedded in the surface of the
matrix and aggregate at the high temperature, also a behavior typical of ductile
materials. Three other castable refractories were similarly tested at 25 °C and
1000 °C, but the particle velocities were more than twice that used for the tests
just described, 72 m/s at 25 °C and 93 m/s at 1000 °C (see B.2.2.10). Two of the
castables tested were calcium aluminate-bonded generic preparations, one high-
alumina (∿95 percent), and one medium-alumina (∿56 percent). The third material
was a high-alumina (∿90 percent) phosphate-bonded commercial castable. The low
temperature performance for all three castables is similar to that discussed in
the previous tests, in general lower material loss occurring at low angles,
greater loss occurring at high angles. The behavior at 1000 °C for the three
castables shown in B.2.2.10 differs considerably from that of the castable shown
in B.2.2.13. The investigators ascribed the difference in performance to the
higher particle velocities used in the tests in B.2.2.10. Although the erosion
at the higher temperatures occurred by a ductile flow mechanism, the higher
particle velocities caused deep channels and steps to form. The presence of
these channels and steps effectively resulted in a 90° exposure angle for all
impingement angles used. The erosion loss curves, therefore, do not resemble
those normally found for ductile materials, the loss at the higher particle
velocities being less sensitive to the angle than the losses at lower velocities.

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The low-temperature tests do not show greatly increased material loss as the
impingement angle increases.

THE EFFECT OF PARTICLE VELOCITY AND TEMPERATURE can be seen in Section
B.2.2.12, where erosion data are shown for various particle velocities and sev-
eral temperatures for tests at 90° impingement angles. For the high-alumina
castables studied, the material loss increased with increasing particle velocity
for any given temperature. For a given velocity, the material loss is lower the
higher the temperature, apparently due to the higher ductility at high tempera-
tures discussed above with regard to B.2.2.10 and B.2.2.13. The 90° impingement
angle is that angle for which the erosion loss is a minimum for ductile materials.

THE EFFECT OF HYDROTHERMAL TREATMENT ON EROSION RESISTANCE OF CASTABLES is
shown in B.2.2.7, B.2.2.9, B.2.2.11, and B.2.2.17. Test samples of castable
refractories were treated by sealing the samples in pressure chambers with water
and heating to various temperatures and pressures for varying periods of time.
Erosion testing at ambient temperatures using the SiC erodent was then performed
on these samples. A commercial 95 percent alumina castable bonded with calcium
aluminate was subjected to erosion testing after hydrothermal treatment at two
different temperatures (B.2.2.7). Samples treated at 210 °C showed improved ero-
sion resistance compared to untreated specimens, but samples treated at 510 °C
showed decreased erosion resistance. Erosion data for the same castable as well
as a commercial 55 percent alumina calcium aluminate-bonded castable are given in
B.2.2.9 where the erosion loss is plotted against the temperature of the treat-
ment. The erosion behavior for the high- and medium-alumina castables is mark-
edly different. The high-alumina shows an initial improvement in erosion resis-
tance with treatment up to about 200 °C. Treatment above about 350 °C changes
the refractory so that the loss of material upon erosion testing has increased
about ten times. This drastic change in erosion resistance is apparently due to
the formation of hydrated aluminas and hydrated calcium aluminates and their
subsequent decomposition (see A.2.2.2.3.1). The formation of the hydrated com-
pounds apparently strengthens the cement matrix initially but the decomposition
with no other suitable bonding phases being formed weakens the matrix consider-
ably. The medium-alumina refractory, however, showed little change in erosion
resistance, that little change which does occur being an improvement in erosion
resistance. The improvement is in accord with the formation of strong bonding
phases upon exposure to hydrothermal conditions which is discussed in A.2.2.2.3.1
(see also A.2.2.2.1 and A.2.2.2.2). Similar hydrothermal treatment was given to
two calcium aluminate-bonded medium-alumina generic castables (one 50 percent
alumina, one 56 percent alumina) which were erosion tested with SiC at 25 °C and
1000 °C (see B.2.2.11). The data clearly show that for both of these medium-
alumina castables, also, the hydrothermal treatment has increased the erosion
resistance, whether the erosion tests are conducted at 25 °C or 1000 °C.

THE EFFECT OF EXPOSURE TO VARIOUS ATMOSPHERES AT HIGH TEMPERATURE AND
PRESSURE ON THE EROSION RESISTANCE of a variety of refractory materials has been
studied. The material loss for part of the testing was measured in accordance
with ASTM standard test method C704-72 and for part of the testing in accordance
with ASTM C704-76a. After curing and drying, the refractories were fired in air
for 250 hours at 1000 or 1100 °C and the erosion loss of the fired materials
measured. Air-fired samples were then exposed to the different atmospheres and
the erosion resistance tested. The atmospheres were steam, hydrogen, carbon
monoxide, carbon-monoxide/water in a 3.5 composition ratio and a 7.0 ratio,
carbon dioxide, and a mixed gas simulating a coal gasification environment:

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24 percent hydrogen, 18 percent carbon monoxide, 12 percent carbon dioxide, five percent methane, two percent ammonia, one percent hydrogen sulfide, and 38 percent steam. The full results are given in B.2.2.14, B.2.2.15, and B.2.2.26 and are summarized qualitatively in Table A.2.2.2.3.2a. Not every refractory was tested under all conditions.

The hydrogen and the simulated coal gasification atmospheres had no serious effect on the erosion resistance of the refractories tested. Only an insulating castable seemed unfavorably affected by the carbon monoxide, the 45 percent alumina dense castable unfavorably affected by the carbon dioxide, and a fireclay castable by the $CO/H_2O = 7.0$ atmosphere. The steam exposure had the greatest effect, and that was an enhancement of the erosion resistance of three medium-aluminas (the two calcium aluminate-bonded castables, the dense 55 percent alumina and the lightweight 50 percent alumina castables, and the 45 percent alumina brick). One intermediate alumina was unfavorably affected by the steam which does not fit the pattern for steam-exposed medium-alumina refractories [due to error in recording of air-fired or steam-exposed values?].

Two dense castables, identified only as having 90 percent and 50 percent alumina, were exposed to several high-Btu mixed gases and then tested for changes in erosion resistance with respect to the resistance of air-fired specimens (see Section B.2.2.25). Specimens of each either were doped with 0.1 percent iron, or contained 310 stainless steel fibers, or were treated by soaking in an alkali carbonate solution before air firing or gas exposure. In general, the 90 percent alumina castable showed deteriorated erosion resistance after 1000 hour exposures to the three mixed gases, two of which were CO-containing. The short (58 hour) exposures did not indicate any serious deterioration. The 50 percent alumina itself, however, did not seem very seriously affected by the long exposures. This performance is consistent with the results of other medium-alumina castables exposed to gases with high steam content (30-40 percent). The short term exposure results indicate some possible degradation especially for the specimens containing stainless steel fibers.

THE EFFECT OF CARBON MONOXIDE EXPOSURE ON EROSION of iron-doped castables is reported in Section B.2.2.20. Two generic castables, one 90 percent alumina and one 50 percent alumina, doped with 0.1 weight percent of iron or Fe_2O_3, were exposed to CO-containing gases of several compositions at 500 °C and 1000 psi for times up to 50 hours. Exposed specimens were then tested using a modified version of test procedure ASTM C-704, with SiC erodent. For the high-alumina castable a number of the specimens were not prefired before exposure and the presence of the iron dopant in these samples was disastrous in a CO-0.8% H_2S atmosphere. The samples disintegrated completely. The undoped specimens did not respond well to the CO atmosphere but the presence of H_2S seemed to restrict the disintegration. The effect of iron doping on the 50 percent alumina material is apparently also the complete disintegration of unfired samples. There seems a trend for the lower-alumina castable to be more affected by the atmospheres with regard to erosion resistance than the high-alumina refractory.

Specimens of these same castables were prepared with stainless steel fibers added for reinforcement and exposed to CO-containing atmospheres at 500 °C and 1000 psi for 50 or 100 hours (see Section B.2.2.21). These specimens were not prefired at 1100 °C but held at 500 °C for 12 hours before test exposure. Prefiring seems to have a bad effect on the performance of the 50 percent alumina material but an erratic effect on the performance of the 90 percent alumina

Meaningful Change in Erosion Resistance After Exposure as Compared to Air Fired Values (+ greater/- poorer resistance)

Refractory	Steam	H_2	CO	$CO/H_2O=3.5$	$CO/H_2O=7.0$	CO_2	Mixed Gas
Dense 95% Al_2O_3 castable, CA bond* (Castolast G)	none	none	none			none	none
Dense high-Al_2O_3 castable (Plicast L-22-84)	none			none	none		
Dense high-Al_2O_3 castable (Greenkon-33)	none				none		
Dense high-Al_2O_3 castable (H-W 9/79)	none			none	none		
Dense 55% Al_2O_3 castable, CA bond (Lo-Abrade)	+	none	none			none	
Dense 45% Al_2O_3 castable, CA bond (H.S. Brikcast BF)						-	none
Dense intermediate-Al_2O_3 castable (Resco Cast RS-17E)	-			none	none		
Dense fireclay castable (Plicast HyMOR 3000)	none			none	none		
Fireclay castable (H-W 8/80)	none			none	-		
Lightweight 50% Al_2O_3 insulating castable, CA bond (Litecast 75-28)	+	none	-			none	none
90% Al_2O_3 ramming mix, phosphate bond (Brikram 90R)	none	none	none			none	none
Dense 90% Al_2O_3 castable, phosphate bond (Resco Cast AA-22)	none			none		none	none
High-Al_2O_3 chrome phosphate-bonded ramming mix (Lavalox X-8)	none				none		
99% Al_2O_3 brick (99AD)	none	none	none	none	none	none	none
90+% Al_2O_3 brick (Kricor)	none	none	none	none	none	none	none
90% Al_2O_3 brick (Arco-90)	none	none	none			none	none
60% Al_2O_3 tar-impregnated brick (Ufala TI)	none	none	none			none	none
45% Al_2O_3 fired super duty brick (KX-99)	+	none	none			none	none
78% Al_2O_3 calcium aluminate neat cement (CA-25)	none	none	none			none	none

* CA bond = calcium aluminate bond.

Table 2.2.2.3.2a

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castable (see Section B.2.2.22). Erosion (abrasion) ratios can be calculated for
the data in B.2.2.21 if the abrasion volume loss for the various CO-containing
gas exposures is divided by that of the nitrogen exposure. The data of B.2.2.21
summarized in Table A.2.2.2.3.2b shows that in general the 90 percent alumina
refractory was adversely affected by the presence of the steel fibers in the test
gas atmospheres. The 50 percent alumina exhibits a more erratic reaction, the
310 stainless steel reinforced specimens having less erosion loss and the other
reinforced specimens a greater loss in the test atmospheres as compared with
nitrogen exposure.

COMPARISON OF MATERIAL LOSS IN STEEL-REINFORCED CASTABLES WITH CO EXPOSURE

| Test Gas (vol%) | Fiber Type | Exposure Time, h | Abrasion Ratio | |
			90% Al_2O_3	50% Al_2O_3
CO	446	50	1.80	3.38
	30-20	50	10.	1.00
	310	50	1.23	0.89
40 CO-40 N_2-20 H_2O	446	50	1.73	2.58
	446	100	1.92	2.40
	30-20	50	2.76	1.06
	30-20	100	1.62	0.42
	310	50	1.75	0.41
80 CO-20 H_2O	310	50	6.83	0.32

Table A.2.2.2.3.2b

THE EFFECT OF ADDITION OF CERAMIC FIBER on the erosion resistance of a high-
alumina castable is given in Sections B.2.2.16 and B.2.2.24. Various quantities
of ceramic fibers (either Al_2O_3-B_2O_3-SiO_2 or Al_2O_3-SiO_2) were added to the cast-
able with varying amounts of water. The effect of varying the amount of water
added without fiber was also studied since more water was needed to add the
fibers to the castable mixture. Some samples were prepared with wetting agents
added to the mix to reduce the amount of water required. Samples prepared with
lower amounts of water (ten percent or less) had better erosion resistance than
samples with higher water content. If the water content is not more than ten
percent, small amounts (one or two percent) of ceramic fiber may improve the
erosion resistance but higher fiber content, even with only about ten percent
water, seems to lower the erosion resistance. Section B.3.2.29 shows correlation
of erosion loss with the modulus of rupture in that those specimens with higher
strength do show less erosion loss.

EROSION TESTING was conducted on a number of materials to screen those which
might be considered for valve use but some of these might also be considered as
useful in linings, or parts of linings, or as small internal component parts,
such as nozzles, etc. The materials were subjected to erosive attack by alumina
for three minutes at impingement angles of 20° and 90° at 20 °C and at an angle
of 90° at 700 °C. Not all materials were tested under all three conditions so

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that there are gaps in the data, as will be seen readily by glancing at any of
the part B sections listed in this text. When only one test at one angle is
performed, it is not possible to have a true picture of the erosion resistance of
the material. The results in terms of sample loss were compared to erosion loss
of samples of a cobalt-based alloy, Stellite 6B, arbitrarily chosen as a standard
and tested with each set of samples. The data consist of Relative Erosion Factors
(REF), values less than one indicating a more erosion resistant material than
Stellite 6B, values greater than one indicating a less erosion resistant mate-
rial. The reported values are the mean of five tests on a material. Although
the tests permit a ranking of materials with respect to erosion resistance, it
must be borne in mind that the test conditions are not comparable to those seen
by the linings or other components in coal gasifiers or other vessels in the
plants. The alumina erodent used is much harder than the coal, char, and ash
particles to which the materials are subjected in plants, and the tests did not
include any of the gaseous chemical constituents of gasifiers. Also, discussion
of performance of materials at 20 °C is not of very meaningful value if the
prospective material use is in vessels operating at high temperatures and pres-
sures (see operating requirements). Therefore, the high-temperature test results
are more important although there are data for only 90° impingement angle, the
angle at which brittle materials (refractories) are less erosion resistant. Any
discussion or ranking of materials using the high temperature data must be tem-
pered by the fact that the 700 °C (1292 °F) test temperature is much lower than
prospective gasifier operating temperatures, and the performance at the operating
temperatures may be rather different.

REFRACTORY MATERIALS, EITHER ALUMINA OR ALUMINA-BASED, were tested (see
B.2.2.1 and B.2.2.2). The data generally show the usual performance of brittle
materials in that the 90° impingement angle data indicate a greater loss of
material than the 20° angle data. Exceptions to this trend are a low alumina
SiAlON (13 percent Al_2O_3-87 percent Si_3N_4), an alumina-titanium carbide sample,
and experimental compositions of alumina with varying amounts of MgO, TiB_2, and
WC. These Al_2O_3-TiB_2-MgO-WC materials and the Al_2O_3-TiC material all had REF
values less than 0.4 for 90° angles at both 20 °C and 700 °C. The SiAlON mate-
rials were much less erosion resistant than the standard. None of the rest of
the alumina materials tested showed any outstanding erosion resistance, most of
them being much poorer than the Stellite 6B standard. Only a densified alumina
had REF values around 0.5 for the 90° impingement angle. Since the erodent was
alumina, further testing is called for to ascertain the usefulness of the mate-
rials in actual use.

SILICON CARBIDE REFRACTORIES of varying types were erosion tested (B.2.2.3).
The twenty-nine different materials from commercial sources included twenty-two
different SiC preparations, including hot-pressed, pressed and sintered, and
recrystallized materials. There were also two silicon carbides which were silica
bonded, two with silicon nitride-silica bond, and one with silicon nitride bond.
One material contained ZrB_2 and another had ZrB_2 and graphite. The erosion
performance with regard to impingement angle was, as expected, typical of brittle
materials, with less erosion for the 20° angle and greater for the 90° angle of
attack. The effect of temperature was mixed, 15 materials having less resistance
at 700 °C than at 20 °C, four samples showing little or no effect, and four
samples having increased resistance.

A.2.2.2.3.2   A.2.2 Refractory Linings and Components--Dry-Bottom Vessels
page 8 of 9   A.2.2.2 Performance Data
9/85   A.2.2.2.3 Materials Evaluation
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The plain SiC materials in general had good erosion resistance relative to the standard but with some very wide differences. Most of the plain samples had REF values less than one at both temperatures and both angles of impingement. The performance of SiC with either SiO_2 bond or Si_3N_4 bond was variable depending on the particular specimen but since the complete characterization of the various specimens is not given the reasons for the variability are unknown. SiC with ZrB_2 had good performance but that material with graphite added had very poor erosion resistance. Those samples which had REF values less than 0.5 for 90° impingement angles at both 20 °C and 700 °C are listed in the following.

Material	REF	
	20 °C	700 °C
SiC, hot-pressed	0.12	0.44
SiC, recrystallized (HD 430)	0.40	0.38
SiC, 98 percent dense	0.05	0.02
SiC (SDIP-1-3)	0.47	0.43
SiC (SDIP-1-4)	0.44	0.43
SiC-Si_3N_4, hot-pressed	0.20	0.42

Other materials showed good erosion resistance at 20 °C, but no data are available for 700 °C.

FOURTEEN CARBONITRIDES were tested at 20 °C and 700 °C at 90° impingement angle (see B.2.2.4). These were mostly experimental compositions having from 85 to 97 percent (Ti, Cr) or (Ti, Mo) carbonitrides and varying percentages of Ni and Mo. None of them showed an REF lower than 0.5, and in general their performance is not significantly better than Stellite 6B. Pressed and sintered TiCN and both pressed and sintered and cast MoTiCN yielded REF values close to one.

SILICON NITRIDE REFRACTORIES (see B.2.2.5) were also tested at both temperatures and both impingement angles. Those which showed good erosion resistance at the 90° angle are listed.

Material	REF	
	20 °C	700 °C
Si_3N_4, hot-pressed	0.18	0.57
Si_3N_4, hot-pressed	0.40	0.12

Reaction-bonded Si_3N_4 had a relative erosion factor of about six for all test conditions. None of the SiAlON materials (Si_3N_4-Al_2O_3) tested had good erosion resistance, nor did a Si_3N_4-SiC-SiO_2 refractory which had factors close to one.

MISCELLANEOUS MATERIALS were tested and the full results given in B.2.2.6. Those with better erosion resistance than the standard are listed for 90° impingement angle.

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Material	REF	
	20 °C	700 °C
B_4C, hot-pressed	0.38	0.21
B_4C, hot-pressed	0.0	0.38
$TiC-Al_2O_3$, pressed and sintered	0.19	0.30
Cubic BN	0.0	0.0

Materials which had poor erosion resistance compared with the standard are TiC with Ni or Ni-Mo binder and several $MgAl_2O_4$-based materials, all hot-pressed with varying amounts of MgO.

Many of the above materials which showed poor erosion resistance because they are brittle in nature and were tested using a 90° impingement angle may perform satisfactorily in design situations in which erodent material impinges on the material surface at low angles. See Section A.2.2.2.1, Plant Experience, and Section A.2.2.2.1.4 for erosion information obtained from a pilot plant refractory lining.

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A.2.2.2.3.3   EROSION/CORROSION

OVERVIEW

     Erosion/corrosion occurs where hot gas corrosion and abrasive particle
impingement can take place simultaneously. Chemical reaction is influenced
by gas composition, refractory composition, temperature and pressure. Some
variables which affect erosion include: type of erodent; erodent particle
velocity; shape and hardness of erodent; angle of impingement; temperature of
material impacted; condition (including hardness) of impacted material sur-
faces. Each of these variables is important in determining the rate at which
an erosion process takes place. Although erosion may not be a critical
problem for the gasifier lining generally, erosion/corrosion can be especial-
ly harmful to components of coal conversion plants because of the cyclic
nature of the processes. Chemical reactions may take place forming a layer
which can be eroded away to present fresh surface to the hot corrosive gases
which in time can form more of the new compound(s) which can erode away until
the design function can no longer be fulfilled.

LABORATORY TESTS

     EROSION/CORROSION EFFECTS ON REFRACTORIES under coal gasification condi-
tions have been studied in a testing program for screening candidate materials
(see B.2.2.18). Test samples of ten materials were subjected to erosion at a
90° impingement angle in a simulated coal gasification atmosphere with 1% H_2S
(see Section B.0) at 1800 °F for 100 hours under both atmospheric and 1000 psi
pressures using coarse (-20+24 mesh) FMC char at a particle velocity of
100 ft/s. One medium-alumina insulating castable was tested in several five
hour tests at 1800 °F with combinations of atmospheric and 1000 psi pressures
and 50 and 100 ft/s velocities in the coal gasification atmosphere. One test
was run in nitrogen at 1800 °F at atmospheric pressure for five hours at
100 ft/s velocity. The average corrosion loss of the refractories (in mils)
for the one side of the samples exposed to both erosion and corrosion was
calculated from thickness measurements of uneroded areas. The maximum ero-
sion/corrosion (Max E/C) loss (in mils) was calculated for the one side
exposed to both erosion and corrosion from thickness measurements made in
eroded areas and pits. In Section B.2.2.18 both average corrosion and max-
imum erosion/corrosion have been reported with visual observations. Ten
different refractory materials were tested and the results are summarized
below for the 2 in. x 2 in. x 3/4 in. bar samples. Values are for one spec-
imen per test.

     Only the crudest ranking of the materials is possible on the basis of
the very limited data. As might be expected, the better performance is
exhibited by brick and dense castables and the poorest performance by the one
insulating castable tested. In all the tests the insulating castable samples
lost large amounts of material. In plant use it is not to be expected that
insulating materials would be subject to erosion/corrosion effects. For the
rest of the materials tested the only parameter which varied was the pressure,
and there is a definite trend for most of the refractories showing an adverse
effect of increased pressure.

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Refractory	Max E/C, in mils	
	1 atmosphere	1000 psi
90% Al_2O_3 castable, calcium aluminate bond (Castolast G)	7.8	2.5
60% Al_2O_3 castable, calcium aluminate bond (Super Brikcast A)	34.4	23.6
54% Al_2O_3 castable, insulating, calcium aluminate bond (Litecast 75-28)	243.8	246.7
85% Al_2O_3 brick, phosphate bond (Chemal 85 B)	17.6	55.6
90% Al_2O_3 brick, self-bond (Kricor)	5.4	26.6
77% Al_2O_3 insulating brick, vitreous bond (B&W Insalcor)	7.0	49.2
70% Al_2O_3 brick, mullite (Alumex 70-HD)	13.9	45.7
60% Al_2O_3 brick, pitch impregnated (Ufala TI)	3.4	15.3
45% Al_2O_3 superduty fire brick (KX-99)	5.2	27.8
90% Al_2O_3-10% Cr_2O_3 brick (Ruby)	5.0	14.0

Another set of refractories was tested (see B.2.2.19) with the same char (FMC), temperature 1800 °F (980 °C), particle velocity 125 ft/s (39 m/s), pressure 35 psi (240 MPa), and the gas composition as in the previous tests. The weight change after 50 and 100 hours of testing was noted. The table below gives the total weight change for 100 hours. In all cases, the bulk of the change had occurred during the first 50 hour period (see B.2.2.19).

Refractory	Weight Change, g ($\pm$0.0005 g) for 100 h	% change
98% Al_2O_3 fused cast brick (Monofrax A2)	−0.001	0.001
99+% Al_2O_3 fused cast brick (Monofrax A1)	+0.029	0.03
99+% Al_2O_3 dense castable (Castolast G)	−0.476	0.05
90% Al_2O_3/10% Cr_2O_3 solid solution fired brick (CS 612)	−0.065	0.06

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~70% Si_2ON_2, ~20% Si_3N_4, ~10% SiC brick (SION)	+2.901	4.2
90% Al_2O_3 phosphate bond ramming mix (Brikram 90)	−0.009	0.04
90+% Al_2O_3 phosphate bonded castable (Resco Cast AA-22)	−0.370	0.4
72% Al_2O_3, 23% SiO_2 brick (Alumex 70)	−0.500	0.7
62% Al_2O_3 castable (AR 400)	−1.479	1.9
55% Al_2O_3 fire clay aggregate gunning mix (Lo-Abrade GR)	−1.075	1.7
50% Al_2O_3 fused cast brick (Monofrax R)	+0.048	0.04
~57% Al_2O_3 castable (Fraxcast ES)	−4.606 (No visible erosion. Experimental error is suspected to account for weight loss.)	6.2
46% Al_2O_3, 40% SiO_2 castable (B&W Kaocrete HS)	−2.327	4.0
79% SiC, ~18% Si_3N_4 bonded, dense fired brick (Refrax 20)	+1.852	2.0
73% SiC, 14% Al_2O_3, ~10% SiO_2 brick (Siltec)	+0.078	0.1

The weight loss data of these tests do not permit ranking of materials ac-
cording to performance although all materials show only moderate or no at-
tack. In general, the fused cast bricks and dense castables show smaller
material loss. The two SiC-based bricks and the silicon oxynitride-silicon
nitride brick all gained weight, possibly due to oxidation of some of the
SiC and Si_3N_4 according to the investigators [33]. See Section 10.2.2 for
oxidation data for these materials.

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A.2.2.2.3.4 MECHANICAL PROPERTIES

OVERVIEW

In most gasification processes the gasifier is planned to be a refractory-lined steel vessel with the steel providing most of the load-bearing and pressure-resisting strength. An intact refractory lining is necessary to insulate and protect the steel shell from the high-temperature erosive and corrosive gasification environment. A thorough knowledge of the various mechanical properties, such as tensile and compressive strengths, modulus of elasticity, creep, and crack resistance of the refractories, is needed to choose appropriate materials and construction designs for linings which will safely and reliably perform this protective function. Information on these properties at the operating conditions of various gasification processes and the response of the refractory to cycling between ambient and operating conditions is particularly important since there can be significant differences between ambient and high temperature measurements.

LABORATORY TESTS

HIGH-TEMPERATURE MEASUREMENTS OF SEVERAL MECHANICAL PROPERTIES of some alumina castables and ramming mixes have been made as part of a study on monolithic refractory concrete linings. Data on hot compressive strength (B.3.2.42 and B.3.2.50), hot modulus of rupture (B.3.2.43 and B.3.2.51), hot modulus of elasticity (B.3.2.44 and B.3.2.49), and fracture energy (B.3.2.39) are given for 50 and 90 percent alumina generic dense castables, a commercial 50 percent alumina dense castable with and without stainless steel fiber additions, and two commercial 50 percent alumina insulating castables. The compositions listed as "modified" have been coarsened by reducing the amount of the finest aggregate fraction (-325 mesh) with a corresponding increase in the amount of the intermediate and coarse fractions. These modified castables showed improvement in the mixing, casting, and working time characteristics relative to the original standard mixes. For all of the properties measured, the modified 90 percent generic composition seems to be slightly better than the standard 90 percent generic composition. While the ambient temperature values of these properties for the 90 percent composition tend to be significantly higher than comparable values for the 50 percent generic composition, they fall rapidly within a temperature rise of a few hundred degrees F. From 500 to 1800 °F, there is relatively little difference in the property values of the 90 percent and 50 percent castable compositions, and there generally seems to be a region toward the higher part of this temperature range where the 50 percent composition has the advantage. The addition of stainless steel fibers did not appreciably benefit the compressive strength or modulus of rupture of the commercial 50 percent alumina dense castable. For fracture energy (i.e., resistance to crack growth) (B.3.2.39), the 50 percent composition appears to be significantly better than the 90 percent composition over this whole temperature range. Beyond 1800 °F, the mechanical properties of the 50 percent composition deteriorate rapidly, and the 90 percent composition would become the more advantageous material at the highest temperature investigated (2000 °F). The insulating castable was the weakest material in all categories and, indeed, could not be tested beyond 1500 °F because of insufficient strength at higher temperatures.

The compressive strength of three phosphate-bonded ramming mixes, two generic (45 percent and 90 percent alumina) compositions and one high-alumina commercial mix, is given in B.3.2.37 and the modulus of rupture for these materials is

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given in B.3.2.60. Of the three, the 45 percent alumina composition shows the best higher temperature compressive strength, but none appears to have any advantages over the dense castable refractories. Young's modulus is given for these ramming mixes in Section B.3.2.61. All three ramming mixes show a sharp decrease in strength at the highest test temperatures in both compressive and bending testing modes.

The effect of heating on the cold flexural strength of six alumina refractories is given in B.3.2.66. Specimens were prepared with some variation in water content and then heated to various temperatures. Where available, the data are compared with the manufacturer's values. The castables all showed decreases in the cold strength with increased temperature of firing. The one ramming mix tested showed increased strength with temperature of heating.

CREEP DATA are given in B.3.2.38, B.3.2.40, B.3.2.41, B.3.2.62, and B.3.2.63 for a variety of the generic and commercial high- and medium-alumina calcium aluminate-bonded castables, both dense and insulating formulations, and for phosphate-bonded ramming mixes. The data suggest that a top temperature limit of 1800 °F should not be exceeded for any of the materials if creep problems are to be avoided. Ramming mixes have better creep properties below 1800 °F but worse above 1800 °F in comparison with the dense castables. The data for the 50 percent alumina insulating castable and the modified 90 percent alumina dense castable suggest that any excess water in the mix is detrimental to the creep resistance of the material. A prototype coarse-grain 50 percent alumina castable appears to have improved creep resistance over the standard generic 50 percent alumina castable. Stress-strain, stress-relaxation, and fracture strain data are given in Sections B.3.2.64, B.3.2.65, and B.3.2.59. The results indicate that creep is more temperature dependent than stress dependent. Stress-relaxation follows the same trends as the creep. The insulating castable showed no relaxation below 1000 °F, the dense castables showed none below 1500 °F.

Stress-strain data at ambient temperatures for the high-alumina and for one of the medium-alumina dense castables studied in the creep testing appear in Section B.3.2.136. The data were obtained in uniaxial compression tests. The project (reference [99]) was concerned with the development of thermo-mechanical models for the prediction of refractory liner-anchor interactions which included developing a creep model. Besides creep data collected in the project of reference [26], data from the project of reference [97] were used in the development of the creep model. Biaxial stress-strain data were also collected for a high-alumina dense generic and a medium-alumina commercial insulating castable. Sections B.3.2.137 through B.3.2.139 contain the results obtained in biaxial compression testing of flat square specimens. The stress ratio for a given test was kept constant. Ratios of the two stresses in the plane of the specimen were zero (uniaxial compression), 0.153, 0.484, and 1.0 (equal stress in both directions). The data show the maximum strength was obtained at the approximately 50 percent stress ratio for the dense high-alumina castable. The same effect in the insulating castable is much less noticeable. The initial elastic modulus for the two castables was obtained from the initial slope of the stress-strain curves (Section B.3.2.140). The temperature effect on the elasticity is very marked although the effect on the strength is not as marked from the stress-strain curves.

The effect of the initial heating rate on the creep of a high-alumina castable was tested (Section B.3.2.118) and found to be small. Creep testing was performed on 90 and 50 percent generic dense castables prepared with calcium

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aluminate cements of varying alumina content. Unit creep data are given in
B.3.2.117 and a composite creep curve in B.3.2.121 for the alumina castable. Data
are reported for constant temperature testing and constant stress testing and also
for cycling of these two parameters (see Sections B.3.2.123 and B.3.2.127). Some
specimens were prefired before testing, others were exposed to simulated coal gas-
ifier conditions. Constant stress-variable temperature data were found to have a
linear dependence on the inverse absolute temperature; constant temperature-
variable stress data were found to have a linear dependence on the ln of the
stress. An empirical model for steady-state strain rates was developed in an
Arrhenius form: $\dot{\varepsilon} = AS\sigma^n\exp(-\Delta H_c/RT)$ where $\dot{\varepsilon}$ is the creep rate, A is a constant,
S is a structure-dependent term, σ is stress to the power n, ΔH_c is the activation
energy for creep, R is the gas constant, and T is the absolute temperature (see
Sections B.3.2.124, B.3.2.125, B.3.2.126, B.3.2.128, and B.3.2.129). For cured
specimens, the activation energy for creep and the stress exponents were found to
be much lower upon initial heat-up than the values found on cycling. After cy-
cling, or prefiring, the materials were much more resistant to creep. The chemi-
cal changes occurring upon heating when the various hydrated compounds decompose
(see Section A.2.2.2.3.1) as well as crystallization changes, sintering, etc., all
have an effect on the creep resistance. The coal gasifier atmosphere had more
effect in promoting creep resistance for the 50 percent alumina and almost none on
the 90 percent refractory, in line with results found for strength testing of
these materials under coal gasifier conditions. The activation energies and the
stress exponents are summarized in Section B.3.2.120. Section B.3.2.131 shows, in
a graphical manner, the dependence of creep on the thermal/strain history of the
specimens.

     The effect of the alumina content of the various cements has been examined
(see Sections B.3.2.122, B.3.2.130, B.3.2.132, B.3.2.133, and B.3.2.134). Neat
high-alumina calcium aluminate cement has a lower creep activation energy and
stress exponent and exhibits greater strain for initial heat-up testing than the
90 percent alumina castable prepared with the same cement (see B.3.2.120 and
B.3.2.122). Comparison of creep data for several cements (see B.3.2.133) does not
include data for the same cement as in B.3.2.122, but the creep performance does
not appear to be directly related to the alumina content. The activation energies
and stress exponents (B.3.2.120) and the total strain data (B.3.2.130) show that
the creep of the refractories must be dependent on a number of factors. It is
difficult to see a direct dependence on the alumina content of the cement.

THE EFFECT OF EXPOSURE TO VARIOUS GASEOUS ENVIRONMENTS AT ELEVATED
TEMPERATURES ON THE COMPRESSIVE STRENGTH AND MODULUS OF RUPTURE of a number of
commercial refractories is given in B.3.2.11, B.3.2.17, B.3.2.18, B.3.2.19,
B.3.2.21, B.3.2.110 and B.3.2.111. A summary of the results is given in Tables
A.2.2.2.3.4a and A.2.2.2.3.4b in the form of the ratio of the strength after
exposure to the strength of a comparable air-fired sample. The tests reported in
Table A.2.2.2.3.4a were performed on fired specimens, those reported in Table
A.2.2.2.3.4b on dried but unfired specimens. The precise conditions and gaseous
compositions are given in the footnotes to the original sections. The most ob-
vious effect is shown by the dense intermediate-alumina castables in the presence
of steam or steam-containing gases. Insulating intermediate-alumina castables
show some strength improvement in the steam-containing environments, but it is
less marked. The difference in the low-Btu gas and high-Btu gas exposures for the
55 percent alumina dense castable is most likely due to the difference in steam
content (12 percent vs. 38 percent). The effect on the compressive strength of a
dense castable by exposure to a 45 percent steam--55 percent high-Btu gas is given

Ratio of Compressive Strength (CS) and Modulus of Rupture (MOR) of Prefired Refractories After Exposure to Various Gases to Air-Fired Values

	Steam		H_2		CO		CO_2		Low-Btu Gas	High-Btu Gas
	CS	MOR	CS	MOR	CS	MOR	CS	MOR	CS	CS
DENSE CASTABLE										
Calcium aluminate bond										
95% Al$_2$O$_3$ (Castolast G)	0.9	0.4	0.7	0.8	1.3	NT[a]	0.8	1.0	1.0	0.8
55% Al$_2$O$_3$ (Lo-Abrade)	3.5	3.1	1.3	1.0	1.4	NT	1.5	1.3	1.3	3.3
45% Al$_2$O$_3$ (H.S. Brikcast BF)	3.5	NT	NT	NT	NT	NT	0.8	0.8	2.3	2.6
Phosphate bond										
90% Al$_2$O$_3$ (Resco Cast AA-22)	0.7	NT	NT	NT	NT	NT	1.1	1.1	0.8	0.7
RAMMING MIX										
Phosphate Bond										
90% Al$_2$O$_3$ (Brikram 90R)	0.9	0.9	0.9	1.2	1.0	NT	0.9	1.0	0.8	1.0
INSULATING CASTABLE										
Calcium aluminate bond										
50% Al$_2$O$_3$ (Litecast 75-28)	1.6	2.2	1.6	1.4	NT	NT	0.9	1.0	1.9	1.3
CALCIUM ALUMINATE NEAT CEMENT										
78% Al$_2$O$_3$ (CA-25)	1.2	1.5	0.8	0.8	NT	NT	1.0	0.8	0.9	1.0
BRICK										
99% Al$_2$O$_3$ (99AD)	NT	0.8	1.4	1.2	1.3	NT	1.2	0.9	NT	NT
90% Al$_2$O$_3$ (Arco-90)	1.0	1.2	1.0	1.0	1.1	NT	0.9	1.0	1.4	0.8
60% Al$_2$O$_3$-tar impregnated (Ufala TI)	NT	1.4	0.5	0.9	1.0	NT	0.9	1.0	NT	NT
45% Al$_2$O$_3$ (KX-99)	1.7	1.6	0.6	1.1	1.1	NT	1.1	1.0	0.9	1.7

[a] NT = not tested

Table A.2.2.2.3.4a

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Ratio of Compressive Strength (CS) and Modulus of Rupture (MOR) of Refractories after Various Unsaturated CO/Steam Exposures to Air-Fired Values

	Steam		$CO/H_2O = 3.5$		$CO/H_2O = 7.0$	
DENSE CASTABLE	CS	MOR	CS	MOR	CS	MOR
Calcium aluminate bond						
94% Al_2O_3 (Greencast-94)	NT[a]	0.8	NT	0.8	NT	NT
High-Al_2O_3 (Plicast L-22-84)	0.9	0.5	1.3	1.0	1.2	NT
High-Al_2O_3 (Greenkon-33)	0.9	0.7	NT	0.4	0.5	NT
High-Al_2O_3 (H-W 9/79)	0.8	NT	1.1	NT	1.3	NT
High-Al_2O_3 (B&W Kaolite 3300)	NT	1.2	NT	NT	NT	NT
Intermediate-Al_2O_3 (Resco Cast RS 17E)	2.0	2.8	1.3	3.0	1.1	NT
Intermediate-Al_2O_3 (Blast Furnace Back-up)	NT	1.4	NT	1.2	NT	NT
Intermediate-Al_2O_3 (Lo-Erode)	NT	2.4	NT	2.9	NT	NT
Fireclay (Plicast HyMOR 3000)	1.4	1.1	NT	1.2	1.2	NT
Fireclay (H-W 8/80)	1.7	NT	NT	NT	0.9	NT
Phosphate bond						
93% Al_2O_3 (B&W KaoPhos 93)	NT	1.1	NT	1.1	NT	NT
RAMMING MIX						
Phosphate bond						
90% Al_2O_3 (Brikram 90R)	NT	1.0	NT	1.2	NT	NT
High-Al_2O_3 (Kritab)	NT	1.1	NT	1.2	NT	NT
High-Al_2O_3 chrome (Lavalox X-8)	1.2	1.0	0.9	NT	1.2	NT
GUNNING MIX						
Intermediate-Al_2O_3 (BF Castable 122-N)	NT	2.4	NT	2.6	NT	NT
INSULATING CASTABLE						
Calcium aluminate bond						
59% Al_2O_3 (Kast-O-Lite 30)	NT	2.3	NT	NT	NT	NT
50% Al_2O_3 (Litecast 75-28)	NT	1.4	NT	1.6	NT	NT
Intermediate-Al_2O_3 (Plicast LWI 206I)	NT	1.2	NT	NT	NT	NT
35% Al_2O_3 (VSL-50)	NT	2.8	NT	NT	NT	NT
29% Al_2O_3 (Greencast-29-L)	NT	1.0	NT	NT	NT	NT
BRICK						
90+% Al_2O_3 (Kricor)	2.2	1.2	2.3	1.1	2.4	NT

[a] Not tested

Table A.2.2.2.3.4b

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in B.3.2.9. The strengthening effect is seen to be completed in the first few hours and then remains essentially constant at the higher strength level (independent of time of exposure). Additional confirmation of the strengthening effect of exposure to a high-Btu gas containing a substantial proportion of steam (36 percent) on intermediate-alumina dense castables is seen in Section B.3.2.8. The two dense castables show increases in compressive strength by a factor greater than two, while the insulating castable and the spinel ramming mix show a much smaller effect.

The strengthening effect on the hot modulus of rupture for intermediate-alumina dense castables from exposure to steam-containing high-Btu gas may be influenced by the purity of calcium aluminate cement used. Data in B.3.2.13 suggest a greater increase in the hot modulus of rupture (relative to air-fired values) after exposure to a high-Btu atmosphere for an intermediate-alumina dense castable using intermediate-purity calcium aluminate cement compared to one using a similar high-purity cement. Different castables were used so it is not proven that the difference in cements is the primary source of the variation in hot modulus of rupture values. Unfortunately, this point does not seem to have been pursued further in this project, except for one experiment which compared data for a high-alumina castable prepared with barium aluminate cement and with calcium aluminate cement. The compressive strength of the calcium-cemented refractory was consistently greater than that of the barium-cemented under all conditions (see Section B.3.2.107). Strength was measured after drying, after firing, and after thermal cycling 24 times. The barium-cemented material strength diminished somewhat from the dried strength after firing and again after 24 thermal cycles. The castable with calcium showed a decrease after firing and about the same value after thermal cycling as for the dried material. For neither set of castables are the changes in strength very striking.

The effect of similar exposures to steam and steam-containing high-Btu gas on the compressive strengths of chromia-containing refractories (for possible slagging gasifier use) is given in B.3.2.20. One material, 73 SiC-21 Si_3N_4-3 Al_2O_3-SiO_2 sintered brick, disintegrated when exposed to steam and to the coal gas. For a chemically bonded ramming plastic (40 Cr_2O_3-23 Fe_2O_3-21 Al_2O_3-10 MgO) the compressive strength after exposure to steam (980 °C, 250 h, 1000 psi) decreased 60 percent from that for air-fired (980 °C, 250 h) and after exposure to the mixed gas (980 °C, 250 h, 1000 psi) decreased 40 percent from that for air-fired. Three of these materials (medium-alumina with varying compositions) increased slightly in compressive strength following both exposure conditions. The behavior of other materials tested was variable with only slight changes in compressive strength.

THE INFLUENCE OF ALKALI EXPOSURE on the compressive strength and hot modulus of rupture of a variety of refractories has been investigated in screening tests. All of the materials were initially fired in air at 980 °C for 24 hours and then subjected to various treatments. In B.3.2.16 are given the compressive strength measurements of eleven refractories after additional treatments of firing in a high-Btu gas (no alkali), firing in the same gas containing alkali vapor, and soaking in a strong alkali solution followed by either air firing or firing in the high-Btu alkali-containing atmosphere. All of these firings were for 200 hours at 980 °C. Six of the original eleven materials were measured for hot modulus of rupture values (B.3.2.12) after soaking in two strengths of alkali solutions followed by firing in either air or a high-Btu gas for 250 hours at 980 °C. The amount of alkali absorbed after the various treatments has been reported in B.1.2.4. It is clear that the soaking treatment introduced more alkali into the

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refractory than the vapor phase exposure and that the intermediate-alumina refrac-
tories absorb more alkali than the comparable high-alumina refractories. The
amount of alkali picked up in soaking is independent of firing in air or firing in
alkali-gas containing atmospheres. The presence of alkali appears to improve the
compressive strength of high-alumina compounds (with the possible exception of the
phosphate-bonded castable) and to lower the strength of intermediate-alumina
castables. Intermediate-alumina brick and ramming mixes are unchanged in strength.
The hot modulus of rupture data of B.3.2.12 is too sparse to draw any definite
conclusions. Generally, there is little difference between the air-fired and the
high-Btu values for all the materials. There are no strictly comparable MOR
values for these materials after exposure to an alkali-free environment which can
be used as a reference base. However, since the normal trend for intermediate-
alumina castables is a substantial increase in both MOR and compressive strength
after exposure to steam-containing gases, the data can be interpreted as support-
ing a deleterious effect by alkali on the strength behavior of such castables.

Four refractories were exposed to KOH vapor in a simulated catalytic coal
gasification atmosphere for 2000 hours at 730 °C and 500 psig. The data in
Section B.3.2.108 for compressive strength indicate degradation for the high-
alumina calcium aluminate-bonded castable and enhanced strength for the low-
alumina calcium aluminate-bonded castable both with and without added 310 SS
fibers. For a low-alumina fired brick and phosphate-bonded high-alumina ramming
mix the exposed values show a slight decrease but not to any significant level.
Since the gas environment contains about 32 percent steam and since the changes in
compressive strength for the refractories is consistent with those exhibited in
dry-ash gasification gases, it is not possible with such limited data to assign
the strength changes to the alkali content of the vapor. Descriptive information
given in Section B.1.2.33 for a shorter alkali exposure agrees with the data of
B.3.2.108. More data on similar materials plus longer exposure times are needed
to establish the\effect of alkali exposure on the strength of refractories.

THE EFFECT OF VARIOUS ADDITIVES ON THE STRENGTH of some castables has been
investigated in some preliminary experiments. Steel fiber reinforcement of five
castables (three commercial, two generic) shows no significant effect on the
compressive strength behavior (B.3.2.22). The strength values and the changes of
strength on firing in gasification atmospheres of the reinforced castables are
quite comparable to those of similar unreinforced refractories. Addition of
ceramic fibers (B.3.2.28) does not appear to be of any benefit in increasing the
modulus of rupture of a high-alumina castable. Excessive water in the mix is
detrimental to the modulus of rupture (B.3.2.28) and crushing strength (B.3.2.23
and B.3.2.24) for such castables. The addition of a plasticizer (polyvinyl alco-
hol, see B.3.2.24) also lowered the crushing strength. Attempts to add small
amounts of silica to a generic high-alumina (B.3.2.23), presumably with the hope
of getting the strengthening effects found for steam exposure of intermediate-
aluminas, did not produce any improvement. Overall, although these were all
preliminary experiments, it would appear that the use of additives is not a help-
ful technique to improve the strength of castable refractories.

THE EFFECT OF RELATIVELY LOW-TEMPERATURE STEAM/CO AND STEAM/CGA (COAL
GASIFICATION ATMOSPHERE) EXPOSURES ON THE MODULUS OF RUPTURE (MOR) of various
refractories has been investigated. The project was intended to explore possible
changes and reactions of refractory linings at the cold face (∿250 °C [482 °F]) of
gasifiers where conditions may be below the dew point of the atmosphere. These
same materials under the same exposure conditions have also been examined for

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changes in density, porosity, and phases (see Sections A.2.2.2.3.1 and A.2.2.2.3.5).
In Section B.3.2.57, values of MOR are given after 20 days exposure to three
ratios of a carbon monoxide/steam/hydrogen sulfide atmosphere at temperatures of
400-532 °F under steam-saturated conditions and at 1000 °F for unsaturated condi-
tions. Section B.3.2.58 shows the MOR values after 60 days exposure in an unsatu-
rated (with respect to steam) simulated coal gasification atmosphere (CGA) at
700 °F and in a comparable saturated CGA at 447 °F. The percentage difference in
MOR relative to controls air fired at 1000 °F (see Sections B.3.2.55 and B.3.2.56)
for samples tested in both of these exposures has been listed in Table A.2.2.2.3.4c
in the hope that any trends would be more obvious. The 1000 °F fired control
(rather than the 500 °F air-fired value) was chosen so that the phosphate-bonded
ramming mixes could be included and this choice was rationalized on the basis that
for most of these materials, there was very little difference in the MOR values of
air-fired samples at 500 or 1000 °F. Even with this limited data set, some trends
seem to be indicated. In the steam-saturated atmospheres, the specific carbon
monoxide-to-steam ratio does not appear to have a systematic effect for any of the
materials. The dense high-alumina castables using calcium aluminate cements tend
to show a marked increase in strength in saturated steam but not in the unsatu-
rated, higher temperature, atmospheres. There is a trend for castables with less
than 90 percent alumina to show some increase in strength after exposure to even
the unsaturated atmospheres. The intermediate-alumina castables generally show a
marked increase in their MOR when exposed to a steam-saturated atmosphere, although
the 54 percent alumina insulating castable seems to be an anomaly. The phosphate-
bonded ramming mixes show a marked deterioration in MOR upon exposure to the
steam-saturated conditions. In the phase change analysis (Section A.2.2.2.3.1),
the saturated exposures of calcium aluminate-bonded castables led to formation of
appreciable amounts of boehmite ($Al_2O_3 \cdot H_2O$) and some calcite ($CaCO_3$). These
compounds arise from the reaction of the calcium aluminate cement with the steam,
CO, and CO_2 and improve the bonding in the castables which is consistent with the
observation of higher MOR values after exposure relative to the air-fired values.

The indication that water vapor is the major reactant of the atmosphere, and
that boehmite formation is the major cause of improved MOR values in calcium
aluminate-bonded dense castables at temperatures below about 500 °C, is reinforced
by the data given in Sections B.3.2.55 and B.3.2.56. The data of Section B.3.2.55
are summarized in Table A.2.2.2.3.4d. The conditions of the exposures listed in
the table have the following meaning:

CGA = coal gasification atmosphere: 18% CO, 12% CO_2, 24% H_2, 41% H_2O, 5% CH_4

CGA No. 1: 500 °F, 500 psi, for 30 days

CGA No. 2: 1000 °F, 1000 psi, for 30 days

CGA, steam saturated: 447 °F, 1000 psi, for 30 days

CGA with 40% H_2O and 1% H_2S: 1000 °F, 1000 psi, for 30 days

CGA with 40% H_2O and 1% H_2S, steam saturated: 447 °F, 1000 psi, for 30 days

52.5% CO/47.5% H_2O No. 1: 500 °F, 465 psi, for ten days

52.5% CO/47.5% H_2O No. 2: 1000 °F, 465 psi, for ten days

52.5% CO/47.5% H_2O, steam saturated: 390 °F, 465 psi, for ten days

Percent Difference in Modulus of Rupture of Refractories Exposed to Various Atmospheres with respect to Air-Fired Values[a]

| | CO/H₂O/H₂S Atmospheres with CO/H₂O Ratios of | | | | | | Coal Gasification Atmospheres | |
| | 0.1 | | 1.0 | | 3.0 | | | |
	U[b]	S[b]	U	S	U	S	U	S
DENSE CASTABLES								
94% Alumina (Greencast-94)	not tested		not tested		not tested		- 35	+ 48(+ 71)
93% Alumina (UMR-1 generic)	- 31	+112(+110)	- 19	+ 32(+115)	- 24	+ 51(+ 63)	- 20	+110(+136)
91% Alumina (UMR-2 generic)	- 54	+ 27(+160)	- 2	+ 10(+108)	- 34	+ 7(+ 12)	- 13	+ 99(+ 34)
91% Alumina (UMR-3 generic)	not tested		not tested		not tested		+118	+ 72(+107)
87% Alumina (UMR-4 generic)	+ 46	+ 64(+ 17)	+ 20	+109(+140)	+ 45	- 5(+108)	not tested	
88% Alumina (UMR-6 generic)	not tested		+ 57	+102(+104)	+ 82	+145(+142)	not tested(+163)	
84% Alumina (UMR-7 generic)	not tested		+ 88	+166(+ 65)	+ 74	+199(+217)	not tested(+274)	
59% Alumina (UMR-5 generic)	+ 91	+190(+ 22)	+ 48	+154(+187)	+ 20	+204(+135)	NT[c]	+177(NT)
57% Alumina (RC-3)	+134	+222(+185)	+ 78	+232(+179)	+ 27	+192(+157)	+156	+156(+206)
INSULATING CASTABLES								
54% Alumina (Cer-Lite #75)	+ 5	- 48(- 73)	+ 7	+ 31(+ 27)	- 15	+ 17(+ 53)	+ 12	- 49(+ 43)
46% Alumina (Litecast 60-25)	+123	+148(+148)	+ 62	+376(+319)	+ 62	+274(+381)	NT	+143(NT)
35% Alumina (VSL-50)	- 50	+ 20(+ 70)	+ 10	+200(+320)	+ 10	+180(+290)	not tested	
RAMMING MIXES, PHOSPHATE BONDED								
96% Alumina (Greenpak-90P)	- 23	- 71(- 66)	- 9	- 40(- 40)	- 24	- 44(-51)	NT	- 50(NT)
90% Alumina (Brikram-90R)	not tested		not tested		not tested		NT	- 43(NT)

[a] Specimens were air fired at 1000 °F for 18 hours.

[b] U = unsaturated atmosphere, S = steam-saturated atmosphere; values in parentheses are for samples immersed in test vessel liquid.

[c] NT = not tested.

Table A.2.2.2.3.4c

Percent Difference in Modulus of Rupture Due to Various Exposures of Refractories as Compared with Air-Fired Values

DENSE CASTABLES

Exposures*	94% Alumina Greencast-94	93% Alumina Kao-Tab 93	90-95% Alumina Castolast G	90-95% Alumina Purotab	93% Alumina UMR-1 generic
CGA No. 1	+ 47	+ 89	+ 1.5	+ 35	+ 30
CGA No. 2	− 31	− 46	− 41	− 23	− 18
CGA, steam saturated	+126	+ 44	not tested	+119	+201
CGA with H_2S	− 36	− 32	− 36	− 18	− 8.2
CGA with H_2S, steam saturated	+ 88	+ 25	+ 57	+142	+121
CO/H_2O No. 1	+ 27	+ 62	+ 23	+ 59	+ 15
CO/H_2O No. 2	− 38	− 38	− 41	− 92	− 26
CO/H_2O, steam saturated	+138	+ 68	+ 84	+115	+120

DENSE CASTABLES

Exposures*	91% Alumina UMR-2 generic	91% Alumina UMR-3 generic	54-57% Alumina Lo-Abrade	57% Alumina RC-3
CGA No. 1	+ 72	+182	+ 53	+ 53
CGA No. 2	− 64	+ 29	+ 44	+ 76
CGA, steam saturated	not tested	+173	+101	+140
CGA with H_2S	− 43	+ 45	+ 5.1	+ 68
CGA with H_2S, steam saturated	+124	+119	+ 34	+182
CO/H_2O No. 1	+ 68	---	+ 30	+136
CO/H_2O No. 2	− 33	---	+ 15	+ 27
CO/H_2O, steam saturated	+ 83	---	+ 64	+136

	INSULATING CASTABLES		RAMMING MIXES		
Exposures*	59% Alumina Kast-O-Lite 30	54% Alumina Cer-Lite #75	96% Alumina Greenpak-90P	96% Alumina 90 Ram H.S.	90% Alumina Brikram 90R
CGA No. 1	− 4.2	− 24	+ 27	− 8.5	− 4.9
CGA No. 2	+ 35	+ 16	+ 13	− 12	− 39
CGA, steam saturated	+ 94	+ 2.1	− 49	− 86	− 83
CGA with H_2S	+ 8.3	− 4.3	− 30	− 29	− 20
CGA with H_2S, steam saturated	+ 65	− 40	− 66	− 82	− 66
CO/H_2O No. 1	+ 27	− 9.6	− 17	− 28	+ 5.7
CO/H_2O No. 2	+ 2.1	+ 16	− 7.2	− 33	− 21
CO/H_2O, steam saturated	+ 79	− 7.4	− 60	− 80	− 80

*See text for details of exposure conditions.

Table A.2.2.2.3.4d

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The table shows that lower-temperature (400-500 °F) steam-saturated atmospheres
produced the greatest increase in strength for calcium-aluminate bonded castables.

The chemical reactions are fairly rapid since exposures beyond ten days show
no trend of increasing MOR. Exposures at 1000 °F show decreased MOR values which
is consistent with the decomposition of boehmite expected at that temperature.
The higher-alumina castables tend to lose strength above the decomposition temper-
ature of boehmite but the lower-aluminas retain strength at higher temperatures
because of their silica content permitting other bonding phases to form (see
Sections A.2.2.2.1 and A.2.2.2.3.1). The effect of boehmite formation in steam-
saturated gases on the strength is also shown in Section B.3.2.105. Section
B.3.2.106 presents the effect of the degree of steam-saturation on the boehmite
content and the modulus of rupture for neat calcium aluminate cement. The strength
and the percent boehmite are directly related. Phosphate-bonded ramming mixes
(B.3.2.55) again show a marked reduction in the modulus of rupture when exposed to
steam-saturated conditions at 400-500 °F. The addition of one percent H_2S to the
atmosphere does not appear to have any effect on any of the tested refractories.

The set of refractories used for the cycling exposure testing given in B.3.2.56
is different from the group in B.3.2.55. The trends are the same as those exhib-
ited in the tests reported in B.3.2.55, however. The lower the alumina content,
at least for dense castables, the greater the increased strength upon steam exposure
which is retained at higher temperature. Repeated cycling between saturated
exposures at 465 °F and air firing at 1000 °F (B.3.2.56) does not indicate any
progressive changes in the modulus of rupture of calcium aluminate-bonded cast-
ables. Any significant changes generally occur in the first cycle, and then the
modulus of rupture remains essentially constant after subsequent cycles. The
major results from this project on the choice of refractories for cold wall con-
ditions would seem to be (1) phosphate-bonded refractories should not be used, (2)
either high- or medium-alumina calcium aluminate-bonded dense castables can be
used with probably a preference for the medium-alumina refractories on the basis
of cost and their improved strength at higher temperature exposures.

CARBON MONOXIDE DISINTEGRATION of metallic iron- and iron oxide-doped re-
fractories as indicated by compressive strength measurements has been investigated
on three generic compositions: a 50+ and a 90+ weight percent alumina castable,
and a 90+ weight percent alumina phosphate-bonded ramming mix (B.3.2.52 and B.3.2.54).
The objectives of the project were to (1) establish whether CO disintegration is
likely to occur in refractories used in coal gasifiers, and (2) define the con-
ditions in which it could be a serious problem. Primary variables were dopant
levels, gas compositions, and pressure.

The data in B.3.2.54 are the complete results of testing at ambient pressures
for 100 hours at 500 °C. The data are given in graphical form in Section B.3.2.67.
All samples were prefired for five hours in air at 1100 °C. Strength tests on
samples which were prefired but not exposed to the test gas environments showed
that the variations in strength of undoped samples after exposure to various gas
compositions are due to differences in raw materials, preparation techniques,
and/or environmental conditions at the time of preparation and not due to the
effects of the gas exposure. Therefore, in B.3.2.54, comparison of the actual
strengths as a function of dopant level is valid only within a given feed gas
composition. The ratio of the strengths of the doped to undoped materials is the
proper indicator for comparison between different gas exposures.

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In general, the two castables behave in a similar fashion for a given exposure condition with the 50+ percent alumina castable usually showing a greater deterioration in strength than the 90+ percent for a given Fe concentration. Neither castable appears to be sensitive to the presence of Fe_2O_3. After exposure to pure CO and several of the gas mixtures, the strength of the ramming mix doped with the higher levels of Fe_2O_3 is substantially reduced relative to the strength of the undoped material. The presence of metallic iron as a dopant degrades the strength of the ramming mix in pure CO exposures but not to the same extent as it does for the castables.

An attempt to depict the effect of the gaseous impurities has been made in Table A.2.2.2.3.4e where a strength index is listed for each dopant against the gas compositions used. This strength index is defined as the weighted average of the ratios of doped to undoped strengths for a given gaseous environment, where the average is taken over all levels of doping. In general, the addition of all of the various gases has some retarding effect on the strength losses relative to pure CO exposures caused by metallic iron doping of the castables. The retarding effect is fairly small at the five percent CO_2 and 0.2 percent H_2S levels for the 50 percent castable but appears to be more substantial at the higher concentrations of these gases. The addition of NH_3 to the gaseous environment has some retarding effect on the strength loss which does not seem to be sensitive to varying NH_3 concentration. The addition of 20 percent H_2O or 20 percent H_2 appears to improve the strength of the doped materials a good deal relative to the undoped materials, but a portion of this improvement disappears upon increasing the concentration of these gases to 40 percent.

The effect of the percentage of these additions on the strength index as compared with the strength index for pure CO exposure is shown graphically in Section B.3.2.71 for both the alumina castables doped with metallic iron and in Section B.3.2.72 for the ramming mix with both metallic iron and iron oxide dopants. The addition of CO_2 to the CO gas enhances the strength of both castables, but for the ramming mix the enhancement in 5 percent CO_2 is lost at 15 percent CO_2. Ammonia is apparently beneficial to the strength of both castables, especially the 90 percent alumina, whereas the effect on the ramming mix is a slight depression of strength at 0.2 percent NH_3 and a slight enhancement at 0.8 percent. Hydrogen sulfide has a greater strength enhancement at 0.2 percent for the high-alumina castable and at 0.8 percent for the low-alumina.

The effect of the various gases on the ramming mix is frequently quite different from that on the castables. With the exception of H_2S, the various gas additives have the same effect on the ramming mix, whether the dopant is Fe or Fe_2O_3. At the five percent CO_2 level, the strength index increases, but at 15 percent CO_2, it is at or below the level shown in pure CO. The addition of NH_3 to the gas environment has little effect. The addition of H_2S has a markedly different effect depending on whether the dopant is Fe or Fe_2O_3. For the Fe doped material, H_2S has no retarding effect at the 0.2 percent level but does have a strong effect at the 0.8 percent level. The Fe_2O_3 doped material shows a continuing decrease of the strength index as the H_2S concentration increases. The addition of H_2O or H_2 at the 20 percent level improves the strength index relative to the 100 percent CO exposure, but this improvement remains the same at 40 percent concentration levels for these gases.

A spalling index calculated by arbitrarily assigning numerical values to the degree of damage observed after exposure is presented with and related to the strength index in Sections B.3.2.68, B.3.2.69 and B.3.2.70. The higher the spal-

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Effect of Gas Composition on Strength Index of Doped Alumina
Refractories Exposed to CO-Containing Atmospheres

Gas Composition	DOE 90 Generic Castable doped with		DOE 50 Generic Castable doped with		DOE Generic Ramming Mix doped with	
	Fe	Fe_2O_3	Fe	Fe_2O_3	Fe	Fe_2O_3
100% CO	0.336	1.254	0.197	0.994	0.650	0.885
95% CO-5% CO_2	0.971	1.365	0.287	1.143	1.032	1.160
85% CO-15% CO_2	1.074	1.015	1.217	1.136	0.656	0.751
99.8% CO-0.2% NH_3	0.637	0.939	0.508	1.072	0.554	0.802
99.2% CO-0.8% NH_3	0.855	1.226	0.486	1.363	0.610	0.950
99.8% CO-0.2% H_2S	0.892	1.168	0.253	1.076	0.654	0.693
99.2% CO-0.8% H_2S	0.972	1.064	0.818	1.020	1.487	0.442
80% CO-20% H_2O	1.418	2.024	1.282	1.362	1.004	1.302
60% CO-40% H_2O	0.953	1.170	0.854	0.850	0.985	1.424
80% CO-20% H_2	1.540	1.721	1.953	2.051	0.929	1.067
60% CO-40% H_2	1.205	1.179	0.978	1.008	0.976	0.999

Table A.2.2.2.3.4e (see text for explanation)

ling index the less the degradation of the specimen and the data in these three
sections show that generally there is reasonably good correlation between the
strength index defined above and the spalling index.

Preliminary data for higher pressure exposures are given in B.3.2.52. These
materials were exposed to pure CO at 600 psi and 500 °C for 100 hours. All of the
samples doped with metallic iron at the 8.5 percent level were completely destroyed
under these conditions. This should be contrasted with the data from B.3.2.54,
where the same materials retained over 50 percent of the undoped strength at
atmospheric pressure exposure. The Fe_2O_3-doped materials were not affected by the
higher pressure exposure in terms of retained strength. These results indicate
that pressure is an extremely important variable and that metallic iron is the
more serious contaminant in the problem of carbon monoxide disintegration of
refractories.

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The effect of high-pressure exposure to carbon monoxide-containing atmo-
spheres is given in Sections B.3.2.74, B.3.2.75, B.3.2.76, B.3.2.77, and B.3.2.78.
Section B.3.2.74 shows that a 50 percent alumina castable with no iron dopant
retains strength in CO up to 10,000 hours at atmospheric pressure but at 1000 psi
the strength begins to fall off after about 50 hours. Adding iron to the castable
drastically reduces the time of exposure needed to degrade the refractory even at
atmospheric pressure (about 50 hours for 0.5 weight percent iron). Tests were
therefore run at 1000 psi and it was found that for the 90 percent alumina cast-
able some iron-doped specimens disintegrated completely in as little as three
hours (Section B.3.2.77). A series of tests were run on specimens of the two
alumina castables to which various stainless steel fibers had been added. Section
B.3.2.78 shows the data which indicate that there is a loss of strength in carbon
monoxide-containing atmospheres which varies for the specific steel fiber used,
the 310 stainless steel fiber-containing samples showed the largest strength loss
for all exposures for both castables. If refractories are to be reinforced with
stainless steel fibers, the specific choice of fiber appears very important to
avoid reaction with carbon monoxide. Four commercial insulating castables which
contain varying amounts of iron were tested in CO-containing atmospheres. All
tended to show loss of strength in varying degrees (see Sections B.3.8.79 and
B.3.2.80).

Carbon monoxide tests involving different particle sizes of iron oxide dopant
added to the 90 percent alumina castable are reported in Section B.3.2.73. The
total loss of strength at large particle sizes is explained by the investigators
[27] as being caused by the large cracks generated at the dopant sites. The large
iron oxide particles cause the deposition of large amounts of carbon which induce
strain in the castable matrix. The smaller dopant particle sizes are more dis-
persed providing more sites for carbon deposition but there is less carbon deposi-
tion at each site thereby producing less strain. As the particle sizes are re-
duced even more the cracks are even smaller but there are also more of them and
more of them are therefore able to intersect. The retained strength observed with
the first reduction in particle size is lost as the particle sizes are reduced
further.

Two dense castables, identified only as having 90 percent and 50 percent
alumina, were exposed to several high-Btu mixed gases and then tested for changes
in compressive strength with respect to the strength of air-fired specimens (see
Section B.3.2.109). Specimens of each either were doped with 0.1 percent iron, or
contained 310 stainless steel fibers, or were treated by soaking in an alkali
carbonate solution before air firing or gas exposure. Three insulating castables,
a 90 percent, a 55 percent, and a 40 percent alumina were tested in the same
gases. Specimens of each were doped with "rust". In general, both the dense and
the insulating high-alumina castables showed the same sort of reduction in strength
exhibited by such refractories exposed to other atmospheres with steam. The
medium-alumina castables, both dense and insulating, showed the increased strength
seen in other tests in the presence of steam. The role of the iron and the CO
component of the mixed gases is not well-defined in these limited tests.

REFRACTORY STRENGTH AFTER HYDROTHERMAL TREATMENT was tested since steam is a
major reactant with the component compounds in refractories (see Sections A.2.2.2.1
and A.2.2.2.3.1). The hydrothermal treatment consisted of sealing refractory
specimens in a pressure chamber with water, raising the temperature to produce
high-pressure steam, and holding for a set period of time. In some tests, the
amount of water varied, in others the temperature, and in others the time of

exposure. Refractories tested included high- and medium-alumina castables, both commercial and generic calcium aluminate-bonded preparations, and a high-alumina phosphate-bonded generic castable. Flexural strength was tested at ambient temperature by four-point bending after exposure and compared with control specimens which did not receive hydrothermal treatment. In one series of experiments, the effect of varying amounts of water at 510 °C was studied using a high- and a medium-alumina castable, both commercial materials. Different batches of both were tested as well as specimens which were only dried and specimens which were both dried and fired. The flexural strength after the various exposures is compared (see B.3.2.1) with the strength of untreated control samples, as well as samples treated in the pressure chamber with no water added, and samples merely soaked in water at ambient temperature for five hours. Firing or water soaking had relatively little effect on the strength of the high-alumina castable whether fired or merely dried. Heat treatment in the pressure chamber with no water present had little effect on fired specimens, but dried specimens dropped in flexural strength by a factor of about 3.5. The hydrothermal treatment had a drastic effect in reducing the strength of the high-alumina material. The medium-alumina castable showed a marked drop in strength of the fired materials as compared with the dried samples. Exposure to water, whether by soaking at ambient temperature or by the hydrothermal treatment, produced samples with strengths only slightly less than that of the dried samples.

The effect of temperatures of hydrothermal treatment from 10 to 510 °C on the high-alumina castable is seen in B.3.2.4. Whether the samples were merely dried or also prefired before the hydrothermal treatment did not affect the results significantly. The major change in strength was caused by the hydrothermal treatment above 310 °C. The strength was reduced dramatically. Included in B.3.2.4 are the results of tests performed after hydrothermal exposure and refiring in air to various temperatures. Refiring restores only some of the strength to the castable; the strength values of the untreated control samples are much greater.

Neat calcium aluminate cement was cast into samples, exposed to hydrothermal treatment at varying temperatures, and the flexural strength tested. The results given in B.3.2.5 show that for both dried and fired samples, the changes in the cement phase which lower the strength occur between 310 and 400 °C. These results are in agreement with the work on the high-alumina castable. Tests on sintered alumina were done to see the effect of the treatment on the aggregate (see B.3.2.6). There was a degradation of strength related to the density (or porosity), but there was no phase change detected by x-ray diffraction analysis. Tests were also run using a generic medium-alumina castable, calcium aluminate-bonded. Results given in B.3.2.2 for fired samples show that there are no drastic differences in flexural strength for specimens subjected to hydrothermal treatment at various temperatures as had occurred for high-alumina material tested the same way (see B.3.2.4). There are data given also in B.3.2.2 for a generic high-alumina phosphate-bonded refractory. Although the data are scanty, there seems not to be any large effect of hydrothermal treatment on the strength. There is only a relatively small drop in strength for the 310 to 400 °C region, which is a critical region for the calcium aluminate-bonded high-alumina refractory. Data reported for a commercial medium-alumina castable, calcium aluminate-bonded, tested after hydrothermal treatment for varying periods of time (see B.3.2.3) show that the refractory strength is not diminished by the treatment and some samples show somewhat increased strength after treatment at 510 °C. Comparison of strength data for commercial high- and medium-alumina castables after hydrothermal treatment (see B.3.2.6) shows directly the degradation of the strength of the high-

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alumina refractory in the 310 to 400 °C region compared with the relatively unaffected strength of the medium-alumina material.

Generic preparations of high- and medium-alumina calcium aluminate-bonded castables were strength tested after two modes of hydrothermal treatment, both different from the procedures used with the fixed-volume pressure chamber utilized in the rest of the treatments (see B.3.2.7). In the first mode, the test vessel was heated to the final test temperature and then steam was introduced to bring the vessel to operating pressure. Under these conditions, both the high-and medium-alumina castables showed no diminishment of flexural strength and both showed some increase over the strength of control samples. In the second mode, water was introduced to the test vessel and during heating the pressure and temperature followed the saturated vapor curve. When the operating pressure (1000 psi) was reached, steam was vented to maintain that pressure as heating was continued to the test temperature. Under this second mode, the medium-alumina refractory showed a constant increase in strength with the increased temperature. The high-alumina showed a drop in strength at about 410 °C, comparable to the results of the other sets of data, although at higher temperatures under these conditions the strength increases and at 910 °C the strength is close to that found for the first mode of operation. The second mode more closely approximates actual plant heat-up procedures. In Section B.3.2.47, data appear for exposures of medium-alumina generic castable to carbon dioxide, steam, and carbon dioxide-steam mixtures; in B.3.2.48, there are data for similar exposures of a high-alumina generic castable. These data show the degradation in strength caused by exposure to steam in the lower temperature range (here 410 and 510 °C). At higher temperature (910 °C), however, the strength was not degraded, but even enhanced. The data also show that exposure to a steam-carbon dioxide mixture or a simulated coal gasification atmosphere at even a temperature as low as 510 °C did not appreciably affect the strength. The procedures for these exposures are similar to the first mode described above in that the test chamber was heated containing argon at ambient pressure and temperature equilibrium was reached before any gases were put into the chamber.

One set of experiments was conducted testing the hot flexural strength of two medium-alumina generic preparations both before and after hydrothermal treatment. The temperature range of treatment and hot strength testing was 610 to 1310 °C. Over the entire temperature range, the hydrothermally treated specimens showed increased hot flexural strength compared to the hot strength of dried untreated samples (see B.3.2.14).

The differences in behavior of the high- and medium-alumina refractories is due to the chemical changes discussed in A.2.2.2.3.1 (see also A.2.2.2.1 and A.2.2.2.2). High-alumina refractories suffer degradation in strength in the region 300-400 °C because the calcium aluminate bonding phases break down as hydration occurs and hydrated alumina (boehmite, $Al_2O_3 \cdot H_2O$) and calcium aluminate hydrates form. As the calcium aluminate hydrates crystallize, they do not form good bonding phases. The boehmite does perform as a bonding phase initially, but at higher temperatures, it breaks down. These hydration-dehydration reactions account for the loss of flexural strength of the high-alumina refractory in the temperature region 300-400 °C. Although calcium aluminate bonding phases do seem to reform at higher temperatures, the original strength of the refractory is not restored. In contrast, medium-alumina refractories undergo reactions such that silica from the aggregate and calcium aluminate from the cement produce anorthite ($CaSi_2Al_2O_8$) which forms a strong continuous structure so that the refractory

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strength is enhanced. (See the earlier discussion in this section regarding
boehmite formation and degradation and the associated strength.)

THERMAL SHOCK EFFECTS ON THE BEHAVIOR AND STRENGTH of nine commercial high-
alumina refractories were investigated as part of a project to develop appropriate
test methods for measuring these properties at ambient and elevated temperatures
(∿2000 °F). Although these castables were tested as part of a ceramic valve
development program [2], they have also been tested for vessel liner use in other
programs. Section B.3.2.10 covers the modulus of rupture at ambient temperature
before and after thermal shock, and at 2000 °F. Section B.3.2.15 lists the am-
bient and 2000 °F values of compressive strength plus the ambient temperature
measurements for shear strength and mechanical shock. For the same materials,
visual observations are given as to their condition after thermal shock (B.3.2.31)
and the effect of thermal cycling on sample lengths is given (B.3.2.30). There
are no trends observable based on composition. As might be expected, the strength
values at high temperatures are generally lower than the ambient temperature
values. The thermal shock treatment provided the lowest MOR values and the poor-
est reproducibility of measurements. Length changes on cycling between ambient
temperatures and 2000 °F appear to be very minor relative to the fired condition.
No further conclusions can be drawn from this limited set of data.

Single and multiple quench thermal shock tests were performed on five generic
high-and medium-alumina dense castables and two commercial medium-alumina insu-
lating castables. The thermal shock damage was studied by testing the flexural
strength after heating specimens to a variety of elevated temperatures and quench-
ing into air. The results of a single heating and quenching are given in Sections
B.3.2.95 and B.3.2.96. The percent reduction in strength was least for the high-
alumina dense castable and highest for the two medium-alumina dense castables.
Multiple quench testing was performed by heating to temperature for 15 minutes,
cooling in air 15 minutes and then repeating this cycle for 3, 5, 7, and 10 times.
The results are reported in B.3.2.97 and B.3.2.98. The data show that the initial
cycle causes the greatest loss of strength which then continues with increasing
cycles at a much slower rate. After 10 cycles the high-alumina dense castable
still retained about 70 percent of the original flexural strength whereas the rest
of the castables, both dense and insulating types, had lost from 65 to 85 percent
of the original strength. Single and multiple quench testing was also performed
on a high-alumina dense generic castable prepared in different formulations so as
to provide specific aggregate particle size distributions and different amounts of
cement. The particle size distribution in the as-received aggregate is typical of
standard industrial products. Two other formulations were prepared by sizing the
aggregate so as to have maximum density. One formulation designated "continuous"
contained aggregate graded continuously so that there is a constant difference
between successive particle sizes. The second formulation designated "gap-sized"
provides sizes and proportions so that large voids created between large particles
are filled by smaller particles and new voids thus created are filled by even
smaller particles. Both formulations were prepared with varying amounts of cement:
20, 25, and 30 percent of calcium aluminate cement. Data for the thermal shock
testing are given in Sections B.3.2.99 and B.3.2.100. The gap-sized formulation
showed the least effect of the quenching of the three formulations, as-received,
continuous, and gap-sized. Apparently the gap-sized formulation provides the
greatest resistance to thermal shock and cracking damage. This resistance is in
agreement with the estimated damage resistance calculated from physical and mechan-
ical properties given in Section B.3.2.148 for all of the castables subjected to
thermal shock in the series of tests. The parameter R'''' which is intended to be

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inversely proportional to the cracking area per unit volume propagated by thermal shock is therefore a measure of the resistance to cracking due to thermal shock. The agreement of the flexural strength data with the calculated R'''' is shown in Section B.3.2.149. The figures show that as R'''' increases so does the retained strength.

FRACTURE CHARACTERISTICS OF REFRACTORY CONCRETES AS A FUNCTION OF TEMPERATURE were measured in one project with the intention of providing data for the engineering design of refractory liners. Eight different castables, all calcium aluminate bonded were studied. Six castables were generic dense formulations of which two were high-alumina and four were medium-alumina. The other two castables were commercial medium alumina insulating materials. Various cements of differing calcia-alumina content were used with the generic formulations.

Measurements of Young's modulus, shear modulus, fracture toughness, work of fracture, fracture surface energy, and flexural strength (MOR) made at ambient temperature (after firing at various temperatures) and at elevated temperatures up to 1200 °C are given in Sections B.3.2.81, B.3.2.82, and B.3.2.84. Graphical versions of the same data are given in Sections B.3.2.32 through B.3.2.36 and in B.3.2.83. These same materials were also examined for phase changes after firing at various temperatures (Sections A.2.2.2.3.1 and B.1.2.10). For some of the properties measured, a correlation with the phase changes can be seen, but the data are not completely consistent. The dependence of the properties on the different purity calcium aluminate cements is not always clear. Generally, the phase changes break into three regions: (1) from ambient temperature to ∿300 °C, formation of hydrated calcium aluminate bonding; (2) from ∿300 °C to 800 or 1000 °C, disappearance of hydrated bonding phases with no new bonding phases; and (3) 1000 °C and up, formation of anorthite ($CaAl_2Si_2O_8$) in silica-containing cements and higher-alumina calcium aluminates as bonding phases. The insulating castable follows this pattern but has additional iron-containing compounds which may be contributing to its usually more complicated behavior. For most of the properties and most of the materials, the values measured at temperature are not greatly different from, but are usually a little lower than, the corresponding ambient temperature values in the low and medium temperature range (i.e., up to 800-1000 °C). The trends are very roughly parallel with the possible exception of the insulating castable which has a more complicated behavior pattern, especially on the fracture toughness and fracture surface energy measurements. At the highest temperatures (> 1200 °C), the flexural strength and elasticity measurements at ambient and elevated temperature seem to be diverging rapidly for the dense castables. Unfortunately, the reality of this apparent trend, while physically reasonable because most materials can be expected to become weaker at high temperatures, is not proven by the data. This apparent high-temperature trend comes primarily from the single data point of ambient temperature measurements after firing at 1350 °C, whereas the high temperature measurements stopped at 1200 °C. More data on a variety of materials are needed before a consistent interpretation of these kind of fracture measurements can be made.

The same properties were measured for a high-alumina dense generic castable which was prepared with the varying aggregate particle size distributions and varying cement content as discussed above under thermal shock. The continuous formulation with 30 percent cement exhibited higher flexural strength at high temperature than the generic preparation but the gap-sized preparations at all cement contents showed only equal or less strength (Sections B.3.2.85 and B.3.2.86).

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The elastic properties are given in Sections B.3.2.87, B.3.2.88, and B.3.2.89.
The values for Young's modulus were greater for the gap-sized formulation for all
three cement contents than for the generic or continuous fomulations. Fracture
data are given in Sections B.3.2.90 through B.3.2.94. Fracture toughness and
notched beam test surface energy were not appreciably improved by the various
formulations but the work of fracture for the gap-sized systems was increased over
the generic and continuous formulated castables. In another project the modulus
of rupture was tested for a series of preparations of 90+ percent alumina generic
castable in which the water was held constant but the calcium aluminate cement
content varied from 7.5 to 32.5 percent (Section B.3.2.119). The maximum modulus
of rupture corresponded to a cement content of about 22 percent.

SLOW CRACK GROWTH IN CASTABLE REFRACTORIES occurs over a wide range of tem-
peratures and is a possible failure mode of reactor vessel monolithic linings.
Some studies have been made on a high- and a medium-alumina castable refractory
investigating the mechanism of and the effect of temperature on crack growth in
these materials. Initial data were obtained on a commercial 95 percent alumina
calcium aluminate-bonded castable using the as-received material (see B.3.2.25).
Controlled fracture in which the velocity can be measured properly was not ob-
tained with this material. The range of particle sizes of the as-received ag-
gregate with respect to total cast sample size was too large and interfered with
directed propagation of the crack. The calcium aluminate cement is the weaker
phase with respect to crack propagation. For these two reasons, it was necessary
to use castable with a higher cement-to-aggregate ratio by using only aggregate
particles smaller than #14 Tyler mesh from the castable mix. Such test samples
were cast and crack propagation data were obtained at 610, 810, 1010, and 1210 °C
(see B.3.2.25). Crack velocity data are plotted against the stress-intensity
factor, K_I. The stress-intensity factor is defined as a measure of the stress-
field intensity near the tip of the crack; it is directly proportional to the
applied load and depends on specimen geometry. The effect of temperature on the
relationship between crack velocity and the stress intensity for this material is
seen readily in B.3.2.25. At 610 °C the crack velocity increases rapidly at high
stress with only small increases in the stress intensity factor. The slope of the
line is a measure of crack growth, the inverse of the slope being related to crack
growth susceptibility; the larger the slope, the less susceptible to cracking. At
610 °C the crack growth at low stress is minutely slow, although at high stress
the crack velocity increases so rapidly with small increases in stress that fail-
ure occurs very rapidly. At higher temperatures (810 and 1010 °C), the data show
a region developing and expanding in which the crack velocity is not increasing
rapidly but which includes a larger range of the stress-intensity factor, involving
lower stresses. At 1210 °C, the relatively slow crack growth region covers the
entire range of the data, even to the lowest stress-intensity factor included.
While crack growth does not increase as rapidly at 1210 °C with increased stress
as it does at lower temperatures, such growth still has an appreciable velocity at
low stress. The high-temperature region then presents a potential for failure of
such refractory. (It should be noted that the two regions of large and small
slope were not observed in the as-cast material at 1010 °C. Only one region of
large slope occurs, indicating the possible importance of cement to aggregate
ratio on this region of slow crack growth.) The investigators [23] have provided
an example to illustrate the problems of the high-temperature region. Using the
data in B.3.2.25, they have calculated the time for a one mm crack subjected to a
constant tensile stress of one MNm^{-2} (one MPa, 145 psi) to grow to such a size in
a refractory component that catastrophic failure occurs. At 610 °C it would take

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10^{25} years for such failure due to cracking to take place, but at 1210 °C the same one mm crack would grow to critical size after 20 minutes.

The effect of firing temperature was studied as part of the crack growth testing of a commercial 55 percent alumina calcium aluminate-bonded castable. Samples were fired to 1010 °C or 1410 °C. Specimens fired to the lower temperature were tested at 610, 810, and 1010 °C. The crack velocity vs. stress-intensity factor data are given in B.3.2.26. There is little difference in the crack growth behavior for these samples at the three test temperatures, the slopes for all three sets of data being large. In contrast to the tests of the high-alumina castable with small particle aggregate, there is apparently no second region of crack growth, just as was true for the test on the cast-as-received high-alumina materials. Specimens of the medium-alumina castable fired at 1410 °C were tested at 1010, 1110, and 1210 °C (see B.3.2.26). The tests at 1010 °C for samples fired at the two firing temperatures show similar crack growth behavior in terms of the crack susceptibility (almost equal slopes) although the stress corresponding to a given crack velocity is much higher for the specimens fired at the higher temperature. The fracture toughness has increased by a factor of two, that is, for a crack to grow at a given velocity the stress must be approximately doubled. The data for tests at 1010, 1110, and 1210 °C show that even though the samples were fired to 1410 °C there is an increase in the crack susceptibility (the slope decreases in value) as the test temperature increases. This behavior is similar to that of the high-alumina material (increased cracking susceptibility with temperature) but without the presence of the two distinct regions of data exhibited by the high-alumina castable.

A series of castables were tested for slow crack growth, one high-alumina and four medium-alumina dense castables, and two commercial insulating castables. The data are given in Sections B.3.2.101, B.3.2.102, and B.3.2.103. The refractories were fired to various temperatures and tested for crack growth by a double torsion test in which the specimens which had a saw-cut center groove were loaded by four-point bending applied at one end. Tests were performed at ambient temperatures after firing and at the firing temperatures. The susceptibility to slow crack growth varied for the different firing temperatures. Comparison of the data for a given material at the different temperatures shows some probable correspondence of the change in the bonding phases with the susceptibility to slow crack growth. The slopes of the lines decrease with increasing temperature indicating increased susceptibility to cracking as the temperature increases. The temperature range of the tests corresponds to the dehydration of the hydrated bonding phases and the formation of the calcium aluminate bonding phases (see Section A.2.2.2.3.1). At whatever the curing or firing temperature of the castables, the material was more susceptible to cracking at the elevated temperature than at ambient temperature. Samples of a high-alumina refractory with various aggregate particle size distributions and varying cement content as outlined above in the thermal shock discussion were subjected to the same slow crack growth testing (see Section B.3.2.104). The data are consistent with the rest of the data but do not show any definite dependence of crack susceptibility on cement content or particle size distribution.

A.2.2.2.3.5 PHYSICAL PROPERTIES

OVERVIEW

Those properties which receive the greatest attention in this section are weight, dimensions, density, and porosity. Weight and dimensional changes may be indicators of chemical reactions occurring in the refractory. They are important in that major differences in them after installation of refractory materials in a vessel may produce changes, such as cracks, which would undermine the integrity and protective ability of the materials as a vessel liner. Changes in density and porosity might affect corrosion and/or erosion resistance in affecting the kinetics of any chemical reactions taking place. Thermal conductivity is directly affected by porosity, the better insulators having higher porosity. Higher porosity usually provides better resistance to thermal shock effects. A disadvantage of high porosity is the reduced strength and generally lower corrosion resistance.

LABORATORY TESTS

THE EFFECT OF HEATING to different temperatures on the dimensions, bulk density, and porosity of several alumina-based refractories was studied as part of a project to develop a computer model for predicting thermal profiles through refractory-lined vessel walls [88]. Length changes of the specimens after heating to various temperatures are given in B.4.2.22, bulk densities in B.4.2.23, and apparent porosities in B.4.2.24. As would be expected, the general trend in length change is a continued shrinkage as the heating temperature is increased. The bulk density shows a decrease with increased heating and the apparent porosity a corresponding increase. The density and porosity of the phosphate-bonded ramming mix was least affected by the heating. Further density and porosity data for two castables are given in B.4.2.26 and pore size distribution before and after firing for the same two castables in B.4.2.25. Small test panels (38-inch squares) simulating linings were prepared on steel plate using four different refractory designs. After a variety of heat treatments and gas exposures porosity measurements were made at various depths from the hot face. The data are in Section B.4.2.27. Section B.4.2.21 shows weight loss versus drying temperature for slabs of phosphate-bonded ramming mix tested in an effort to devise procedures to prevent delamination cracking. Bulk density data are given in Section B.4.2.28 for several refractories for various curing and firing temperatures. The effect of the same temperatures on the density of a high-alumina castable with various aggregate particle size distributions is given in Section B.4.2.29.

In Section B.4.2.18 are bulk density and shrinkage data for eleven dense refractories which include high- and low-alumina castables and ramming mixes. The castables include generic and commercial calcium aluminate-bonded mixes, some of which were prepared with different cements, with varied water content and with stainless steel fibers for reinforcement. The data are given for various curing and firing temperatures, but the data are not complete for every test condition.

THE EFFECT OF EXPOSURE TO VARIOUS ATMOSPHERES AT HIGH TEMPERATURE AND PRESSURE on the bulk density has been studied. The measurements of bulk density (and some apparent porosity measurements) were made in accordance with ASTM C20-73. For some tests, after curing and drying, the refractories were fired in air for 250 hours at 1100 °C and the properties measured. Air-fired samples were then exposed to the different atmospheres and the properties remeasured. The atmospheres were steam, hydrogen, carbon monoxide, carbon dioxide, and a mixed gas simulating a

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coal gasification environment: 24% H_2, 18% CO, 12% CO_2, 5% CH_4, 2% NH_3, 1% H_2S, and 38% H_2O. The full data with test conditions are given in B.4.2.1, B.4.2.2, B.4.2.3 and are summarized in Table A.2.2.2.3.5a. Not all refractories were tested under all conditions.

Percent Difference in Bulk Density of Prefired Refractories after Various Gas
Exposures with respect to Air-Fired Values

	Steam	H_2	CO	CO_2	Mixed Gas at 1000 °C	Mixed Gas at 760 °C
DENSE CASTABLES						
Calcium aluminate bond						
95% Al_2O_3 (Castolast G)	+3.7	+0.6	+1.8	−1.2	+1.2	−2.7
90% Al_2O_3 (DOE Generic)						−2.2
55% Al_2O_3 (Lo-Abrade)	+2.3	+0.8	+2.3	−2.3	−3.0	−5.8
45% Al_2O_3 (B&W Kaocrete D)						−1.0
45% Al_2O_3 (H.S. Brikcast BF)				−0.8	−0.8	+0.2
Phosphate bond						
90% Al_2O_3 (Resco Cast AA−22)				0	−0.6	−8.7
RAMMING MIX						
Phosphate bond						
90% Al_2O_3 (Brikram 90R)	−2.7	−3.2	+1.6	−1.1	−2.1	−1.2
60% Al_2O_3 (HW 23−75)						−4.5
INSULATING CASTABLE						
Calcium aluminate bond						
50% Al_2O_3 (Litecast 75−28)	−5.8	+1.2	+1.2	−2.3	+2.3	−2.4
CALCIUM ALUMINATE NEAT CEMENT						
78% Al_2O_3 (CA−25)	+2.7	+2.7	+1.8	−2.6	−4.4	−5.4
BRICK						
99% Al_2O_3 (99AD)	+1.6	+1.0	+2.6	−1.0	0	
90% Al_2O_3 (Arco-90)	−0.5	0	+0.5	−0.5	+1.6	+0.1
60% Al_2O_3 tar-impregnated (Ufala TI)	−3.1	−0.6	+2.5	−1.8	−0.6	
45% Al_2O_3 superduty (KX−99)	−1.4	0	+1.4	−0.7	−0.7	−1.0

Table A.2.2.2.3.5a

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Bulk density measurements were also made on cured and dried refractories exposed
to an unsaturated steam atmosphere and an unsaturated carbon monoxide/steam
atmosphere and to air firing at the same temperatue for the same time. The full
data are given in Section B.4.2.35 and summarized in Table A.2.2.2.3.5b. It can
be seen from the tables that the bulk density is affected very little by the test
exposures. There are no obvious trends indicating susceptibility of a group of
refractories to particular atmospheres as measured by density.

**Percent Difference in Bulk Density of Refractories after Unsaturated Steam and
CO/Steam Exposures with respect to Air-Fired Values**

	Steam	$CO/H_2O = 3.5$
DENSE CASTABLE		
Calcium aluminate bond		
94% Al_2O_3 (Greencast-94)	+2.5	+1.8
High-Al_2O_3 (Plicast L-22-84)	+0.5	+1.1
High-Al_2O_3 (Greenkon-33)	+0.5	+1.6
Intermediate-Al_2O_3 (Resco Cast RS-17E)	+0.7	+1.5
Intermediate-Al_2O_3 (Blast Furnace Back-up)	+3.8	+2.3
Intermediate-Al_2O_3 (Lo-Erode)	+0.8	+1.6
Fireclay (Plicast HyMOR 3000)	-3.5	-1.4
Phosphate bond		
93% Al_2O_3 (B&W KaoPhos 93)	+1.2	+2.4
RAMMING MIX		
Phosphate bond		
High-Al_2O_3 (Kritab)	-0.5	+0.5
High-Al_2O_3 (Lavalox X-8)	+0.5	+0.5
GUNNING MIX		
Intermediate-Al_2O_3 (BF Castable 122-N)	+0.8	0
INSULATING CASTABLE		
Calcium aluminate bond		
High-Al_2O_3 (B&W Kaolite 3300)	+4.7	
59% Al_2O_3 (Kast-O-Lite 30)	-1.2	
Intermediate-Al_2O_3 (Plicast LWI 206I)	-1.9	
35% Al_2O_3 (VSL-50)	0	
29% Al_2O_3 (Greencast-29-L)	0	
BRICK		
90+% Al_2O_3 (Kricor)	-1.1	-1.6

Table A.2.2.2.3.5b

A.2.2.2.3.5   A.2.2 Refractory Linings and Components--Dry-Bottom Vessels
page 4 of 22   A.2.2.2 Performance Data
9/85   A.2.2.2.3 Materials Evaluation

Percent Difference in Apparent Porosity of Refractories after the Indicated
Exposures with respect to Air-Fired Values

	Unfired		Prefired--Mixed Gas at 760 °C
	Steam	$CO/H_2O = 3.5$	
DENSE CASTABLE			
Calcium aluminate bond			
95% Al_2O_3 (Castolast G)			-2.3
94% Al_2O_3 (Greencast-94)	-30.	+10.	
90% Al_2O_3 (DOE Generic Preparation)			+2.7
High-Al_2O_3 (Plicast L-22-84)	-22.	0	
High-Al_2O_3 (Greenkon-33)	-13.	0	
55% Al_2O_3 (Lo-Abrade)			-17.
Intermediate-Al_2O_3 (Resco Cast RS-17E)	-64.	-16.	
Intermediate-Al_2O_3 (Blast Furnace Back-up)	-50.	-11.	
Intermediate-Al_2O_3 (Lo-Erode)	-17.	-3.4	
45% Al_2O_3 (B&W Kaocrete D)			-13.
45% Al_2O_3 (H.S. Brikcast BF)			-23.
Fireclay (Plicast HyMOR 3000)	-14.	+4.8	
Phosphate bond			
93% Al_2O_3 (B&W KaoPhos 93)	-29.	+4.2	
90% Al_2O_3 (Resco Cast AA-22)			-16.
RAMMING MIX			
Phosphate bond			
90% Al_2O_3 (Brikram 90R)	-13.	+13.	-5.0
High-Al_2O_3 (Kritab)	-19.	+6.3	
High-Al_2O_3 (Lavalox X-8)	-24.	+5.9	
60% Al_2O_3 (HW 23-75)			-13.
GUNNING MIX			
Intermediate-Al_2O_3 (BF Castable 122-N)	-22.	-9.4	
INSULATING CASTABLE			
Calcium aluminate bond			
High-Al_2O_3 (B&W Kaolite 3300)	-52.		
59% Al_2O_3 (Kast-O-Lite 30)	-33.		
50% Al_2O_3 (Litecast 75-28)	-30.	+3.6	-8.5
Intermediate-Al_2O_3 (Plicast LWI 206I)	-32.		
35% Al_2O_3 (VSL-50)	-32.		
29% Al_2O_3 (Greencast-29-L)	-28.		
BRICK			
90+% Al_2O_3 (Kricor)	-21.	+50.	
90% Al_2O_3 (Arco-90)			-19.
45% Al_2O_3 superduty (KX-99)			-4.6

Table A.2.2.2.3.5c

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Measurements of apparent porosity were made of air-fired samples before and after exposure to the mixed gas atmosphere at 760 °C (see Section B.4.2.3) and of unfired samples after exposure to the unsaturated steam and CO/steam atmospheres (see Section B.4.2.37). Data for these exposures compared to the porosities of comparable air-fired specimens are summarized in Table A.2.2.2.3.5c. All the unfired refractories showed a decrease in porosity after the steam exposure with respect to air-fired values in excess of ten percent, ranging from 13 to 64 percent. The effect of the CO/steam exposure was variable and of much smaller magnitude than the steam alone except for one high-alumina brick for which the porosity was 50 percent greater than that of the air-fired specimen. The prefired medium-alumina dense castables, the phosphate-bonded high-alumina castable and medium-alumina ramming mix, and the high-alumina brick showed decreases in apparent porosity in excess of ten percent after exposure to the mixed gas. It is difficult to relate this loss of apparent porosity with chemical and phase changes occurring in these refractories under these environmental conditions (see A.2.2.2.3.1). There is no measure of the effect of these porosity changes on the performance of the materials in actual use. In Section B.4.2.35 are the dimensional changes showing shrinkage or expansion of the refractories reported in B.4.2.36 and B.4.2.37 due to air-firing and the unsaturated steam and CO/steam exposures. The changes in dimensions are all small, the largest being 1.65 percent shrinkage for a low-alumina castable in steam.

THE EFFECT OF THE TIME OF EXPOSURE to a simulated coal gasification atmosphere on bulk density and apparent porosity is shown in Sections B.4.2.38 and B.4.2.39 for a high- and a low-alumina generic castable prepared with three different calcium aluminate cements. It is apparent that the initial dehydration reactions taking place during the first ten hours or so of exposure cause the greatest changes. The bulk densities do not change to any great extent with exposure time except for an apparent increase after 1000 hours for the high-alumina castable with the lowest-alumina content cement. The apparent porosities show more scatter in the data. The high-alumina castable with the two higher-alumina cements does not show much change with time but there is an apparent drop for the lowest-alumina cement after 1000 hours. The medium-alumina castable shows an increase in porosity after 1000 hours in combination with all three cements.

THE EFFECT OF CREEP TESTING on the density and porosity of the same high-alumina castable and the high-alumina neat cement is given in Section B.4.2.40. The bulk density for both castable and neat cement decreased from the as-cured value after creep testing and after heating to the creep test temperature but without load applied. The cement value, however, after creep testing, was greater than the temperature treatment without applied load. The apparent density showed exactly opposite effects for the materials, increasing after temperature treatment and creep testing. The apparent porosity increased from the as-cured valued for both castable and neat cement for both treatments. The porosity of the cement after temperature treatment was greater than after the load was applied in creep testing.

THE EFFECT OF SIMULATED COAL GASIFICATION ATMOSPHERE ON CASTABLES TO WHICH STEEL FIBER WAS ADDED for reinforcement was studied (see B.4.2.4). Two weight percent of various steel fibers were added to three castables as the samples were prepared. The bulk density and porosity were measured after air-firing at 980 °C for 250 hours and after exposure to a coal gasification environment at 980 °C and 1000 psi for 250 hours. The change in properties is summarized in the following tabulation.

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Refractory	Fiber	Percent Change in Bulk Density	Percent Change in Apparent Porosity
Dense 95% Al_2O_3 castable,	430 SS	-2.3	-16.7
calcium aluminate bonded	446 SS	0	- 8.3
(Castolast G)	446 SS, coated	-1.1	- 9.1
	310 SS, coated	0	- 7.7
Dense 90% Al_2O_3 castable,	430 SS	-1.1	- 9.5
calcium aluminate bonded	446 SS	-1.1	- 8.5
(DOE Generic Preparation)	446 SS, coated	-0.4	- 7.4
	310 SS, coated	+0.7	- 1.9
Dense 45% Al_2O_3 castable,	430 SS	+0.9	-15.0
calcium aluminate bonded	446 SS	+1.4	-10.2
(H.S. Brikcast BF)	446 SS, coated	+1.4	- 8.9
	310 SS, coated	+1.9	-12.1

   The data show that the presence of the fibers had no real effect on the
response of the refractories to the coal gas exposure as measured by the bulk
density changes. The apparent porosity decreased in all cases, but the effect of
the fibers is unclear.

   Bulk density data for air-fired and high-Btu gas-exposed specimens are given
in Section B.4.2.34 for two dense castables identified only as containing 90 and
50 percent alumina. Specimens of each were also either doped with 0.1 percent
iron, had 310 stainless steel fibers added, or were soaked in an alkali carbonate
solution before firing. The density differences between specimens of the same
refractory with the different additions, or between air-fired or gas-exposed
specimens of the same test samples, do not show any significant variations.

   THE EFFECT OF HEAT TREATMENT AND VARIOUS GASEOUS ENVIRONMENTS on seven high-
alumina and two medium-alumina dense castables, and two medium-alumina insulating
castables, all calcium aluminate bonded, and three ramming mixes was studied.
Samples were exposed in a simulated coal gasification atmosphere without H_2S for
periods of 10, 20, and 30 days, at 500 °F and 500 psi, and at 1000 °F and 1000 psi.
They were also exposed in the coal gasification atmosphere with one percent H_2S
at 1000 °F and 1000 psi and in a 52.5 percent carbon monoxide/47.5 percent steam
environment for ten days at 500 °F and 1000 °F at 465 psi. The materials were
also exposed to these atmospheres under steam-saturated conditions. Measurements
were made to determine weight and dimensional changes of the samples (see B.4.2.7
for full test conditions and data; values are averages for seven samples except
for saturated conditions where two samples were tested). The weight and dimension-
al changes were minimal, the largest percent weight change which occurred being
that for the difference between values for the air-dried samples and air-fired
control samples of the castables. A glance at B.4.2.7 will show that the maximum
weight change was 5.2 percent, and the maximum dimensional change was 1.2 percent
for all treatments, including the air firing of the control samples. The change
in weight was generally a weight loss, and the change in dimensions generally a
shrinkage.

   FIVE NEAT CALCIUM ALUMINATE CEMENTS were exposed to the same set of test
conditions given in the previous paragraph as well as to vapor and test vessel
liquid in steam-saturated conditions, and the weight and dimensional changes

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examined (see B.4.2.9). Data are given for the weight change experienced by the initially dry powders exposed to the various atmospheres. Appreciable weight gains were found, a not unexpected result, since various chemical reactions are expected upon mixing these powders with water, a major constituent of all the test atmospheres. Of more practical interest are the percent weight and percent dimensional changes experienced by the neat cement samples prepared by hydrating, casting, and drying. No data are given for air firing of these samples. The percent changes for the neat cements are somewhat greater than were the changes occurring for the calcium aluminate-bonded castables. The maximum weight gain was 16.5 percent and the maximum loss was 8.7 percent. The maximum shrinkage was 4.3 percent and the maximum expansion was 4.6 percent. Firm trends are difficult to find in the data because the data divide somewhat equally between positive and negative percent changes, as opposed to the castables which generally showed weight loss and shrinkage. This difference may be due to the presence of the aggregate in the castables. There is some indication that weight and dimensional gains correspond somewhat to the formation of hydrated compounds at lower temperatures and in atmospheres with higher water content and that the weight losses and shrinkage occur at higher temperatures. Cement specimens exposed to the saturated vapor and the liquid tended to show large weight gains and to expand. See A.2.2.2.3.1 and B.1.2.20 for the chemical changes which occurred in these tests.

DENSITY AND POROSITY MEASUREMENTS were also made on the same eleven castables mentioned above for which weight and dimensional changes were reported (see Section B.4.2.8). Tables A.2.2.2.3.5d and A.2.2.2.3.5e contain a summary of the percent changes in density and apparent porosity occurring after the various exposures which are reported in B.4.2.8. These data are average values of seven samples except in saturated conditions where two samples were exposed. All changes are with respect to values found for control samples air-fired at 1000 °F for 18 hours. The conditions of the exposures listed in Tables A.2.2.2.3.5d and A.2.2.2.3.5e have the following meaning:

CGA = coal gasification atmosphere: 18% CO, 12% CO_2, 24% H_2, 41% H_2O, 5% CH_4

CGA No. 1: 500 °F, 500 psi, for 30 days

CGA No. 2: 1000 °F, 1000 psi, for 30 days

CGA, steam saturated: 447 °F, 1000 psi, for 30 days

CGA with 40% H_2O and 1% H_2S: 1000 °F, 1000 psi, for 30 days

CGA with 40% H_2O and 1% H_2S, steam saturated: 447 °F, 1000 psi, for 30 days

52.5% CO/47.5% H_2O No. 1: 500 °F, 465 psi, for ten days

52.5% CO/47.5% H_2O No. 2: 1000 °F, 465 psi, for ten days

52.5% CO/47.5% H_2O, steam saturated: 390 °F, 465 psi, for ten days

Changes in density (Table A.2.2.2.3.5d) for the dense castables were almost always increases, the maximum value being +7.2 percent for the high-alumina refractories and +5.9 percent for the medium-aluminas. The insulating castables generally showed smaller changes, although the maximum value is +8.9 percent. The density changes for the ramming mixes were about evenly divided between increases and decreases with a maximum value of +4.1 percent. There are no obvious trends.

Percent Difference in Density Due to Various Exposures of Refractories as Compared with Air-Fired Values

DENSE CASTABLES

Exposures*	94% Alumina Greencast-94	93% Alumina Kao-Tab 93	90-95% Alumina Castolast G	90-95% Alumina Purotab	93% Alumina UMR-1 generic
CGA No. 1	+ 5.2	+ 5.6	+ 5.1	+ 4.3	+ 2.6
CGA No. 2	+ 3.0	+ 3.2	+ 4.0	+ 3.2	+ 1.9
CGA, steam saturated	+ 4.5	+ 1.6	not tested	+ 5.8	+ 2.6
CGA with H_2S	---	+ 2.4	+ 1.1	+ 4.0	+ 3.7
CGA with H_2S, steam saturated	+ 3.7	+ 0.8	+ 1.8	+ 3.2	+ 1.9
CO/H_2O No. 1	+ 6.0	+ 7.2	+ 5.5	+ 5.4	+ 2.2
CO/H_2O No. 2	+ 3.0	+ 2.8	+ 3.6	+ 2.5	+ 1.9
CO/H_2O, steam saturated	+ 4.1	+ 2.0	+ 4.7	+ 4.0	+ 1.5

DENSE CASTABLES

Exposures*	91% Alumina UMR-2 generic	91% Alumina UMR-3 generic	54-57% Alumina Lo-Abrade	57% Alumina RC-3
CGA No. 1	+ 6.3	+ 7.2	+ 2.7	+ 4.1
CGA No. 2	+ 2.8	+ 3.8	- 0.4	+ 1.8
CGA, steam saturated	not tested	+ 3.8	- 0.9	- 2.7
CGA with H_2S	+ 3.2	+ 4.3	- 1.3	+ 1.4
CGA with H_2S, steam saturated	+ 2.4	+ 3.8	0	+ 2.3
CO/H_2O No. 1	+ 7.1	---	+ 2.7	+ 5.9
CO/H_2O No. 2	+ 2.4	---	+ 0.4	+ 1.4
CO/H_2O, steam saturated	+ 3.6	---	- 0.9	+ 4.5

	INSULATING CASTABLES		RAMMING MIXES		
Exposures*	59% Alumina Kast-O-Lite 30	54% Alumina Cer-Lite #75	96% Alumina Greenpak-90P	96% Alumina 90 Ram H.S.	90% Alumina Brikram 90R
CGA No. 1	+ 2.7	- 1.3	+ 0.7	+ 0.7	- 0.7
CGA No. 2	+ 2.1	- 0.6	0	+ 1.4	+ 1.4
CGA, steam saturated	- 0.7	0	+ 0.3	+ 0.7	- 1.0
CGA with H_2S	+ 4.8	0	- 2.7	- 0.3	- 0.3
CGA with H_2S, steam saturated	+ 8.9	+ 3.2	- 1.7	+ 2.4	+ 0.3
CO/H_2O No. 1	+ 3.4	+ 0.6	- 2.7	+ 0.7	+ 4.1
CO/H_2O No. 2	+ 2.1	0	- 1.7	- 0.7	+ 2.7
CO/H_2O, steam saturated	+ 0.7	- 1.3	- 2.7	- 1.0	+ 2.0

*See text for details of exposure conditions.

Table A.2.2.2.3.5d

Percent Difference in Porosity Due to Various Exposures of Refractories as Compared with Air-Fired Values

DENSE CASTABLES

Exposures*	94% Alumina Greencast-94	93% Alumina Kao-Tab 93	90-95% Alumina Castolast G	90-95% Alumina Purotab	93% Alumina UMR-1 generic
CGA No. 1	-20	-14	- 5.0	-18	- 8.7
CGA No. 2	- 4.0	0	+ 5.0	- 4.5	+13
CGA, steam saturated	-48	+ 3.6	not tested	-73	-52
CGA with H_2S	- 4.0	0	+20	- 4.5	+ 4.3
CGA with H_2S, steam saturated	-36	+ 3.6	-20	-41	-35
CO/H_2O No. 1	-20	-18	-15	-18	- 4.3
CO/H_2O No. 2	- 4.0	- 3.6	+ 5.0	- 4.5	+ 4.3
CO/H_2O, steam saturated	-80	- 3.6	- 80	-82	-78

DENSE CASTABLES

Exposures*	91% Alumina UMR-2 generic	91% Alumina UMR-3 generic	54-57% Alumina Lo-Abrade	57% Alumina RC-3
CGA No. 1	-21	-13	-10	-13
CGA No. 2	- 3.6	+ 3.2	+ 5.0	- 4.3
CGA, steam saturated	not tested	- 9.7	-40	- 4.3
CGA with H_2S	- 3.6	0	+10	- 4.3
CGA with H_2S, steam saturated	-36	- 9.7	-20	-26
CO/H_2O No. 1	-25	---	- 5.0	-26
CO/H_2O No. 2	- 7.1	---	0	0
CO/H_2O, steam saturated	-75	---	-10	-52

INSULATING CASTABLES   **RAMMING MIXES**

Exposures*	59% Alumina Kast-O-Lite 30	54% Alumina Cer-Lite #75	96% Alumina Greenpak-90P	96% Alumina 90 Ram H.S.	90% Alumina Brikram 90R
CGA No. 1	- 2.0	+ 4.8	+ 5.9	0	+ 5.9
CGA No. 2	+ 6.1	+24	+ 5.9	- 5.0	- 5.9
CGA, steam saturated	+10	+24	+24	+ 5.0	+24
CGA with H_2S	+ 4.1	+21	+18	- 5.0	+ 5.9
CGA with H_2S, steam saturated	- 8.2	+ 7.1	+24	- 5.0	+12
CO/H_2O No. 1	0	0	+53	0	-18
CO/H_2O No. 2	+ 4.1	+12	+12	0	-12
CO/H_2O, steam saturated	+ 4.1	+14	+24	+ 5.0	0

*See text for details of exposure conditions.

Table A.2.2.2.3.5e

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Changes in apparent porosity (Table A.2.2.2.3.5e) varied greatly. For the dense castables, the porosity tended to decrease in most cases. There is a trend for a larger change in porosity (always a decrease) at lower temperatures and pressures for like atmospheres (compare data for CGA No. 1 and No. 2 and CO/H_2O No. 1 and No. 2). The changes for the higher temperatures and pressures (No. 2 exposures) are all either much smaller decreases in porosity or increases. There is some indication that for similar atmospheres, saturated steam conditions frequently decrease the porosity by large percentages (50-88 percent), with some exceptions. These data are consistent with the results discussed in A.2.2.2.3.1, indicating boehmite ($Al_2O_3 \cdot H_2O$) formation at lower temperatures and higher water concentrations which would reduce the apparent porosity due to the boehmite filling the pores. The apparent porosity of the insulating castables tended to increase upon exposure as did one of the ramming mixes. There are no consistent trends obvious for the three ramming mixes tested. The largest decrease in porosity of the intermediate-alumina dense castables is rather less than the largest decrease of the high-alumina castables. The tendency of the insulating castables to exhibit increased porosity upon exposure would enhance the insulating capability but could be detrimental if it indicated a breakdown of the integrity of the material (see A.2.2.2.1).

PRESSURE-TEMPERATURE CYCLING CAUSING BOEHMITE FORMATION AND DECOMPOSITION of alumina castables was studied in a series of experiments. Seven castables, four dense generic preparations containing alumina in percentages ranging from 93 to 58, and three commercial insulating castables containing 54 to 35 percent alumina were subjected to the same coal gasification atmosphere (CGA) with H_2S used in the tests of B.4.2.8 in a steam generator providing 85 percent steam saturation of the atmosphere. After this exposure, the samples were air-fired. The cycles, which caused boehmite to form in the saturated atmosphere and then decompose upon being air fired, were repeated with measurements being made after each cycle on seven samples to determine changes in weight, dimensions, density, and porosity. Tests were also run in a steam-saturated coal gasification atmosphere with H_2S and in a steam-only saturated atmosphere. Samples were exposed both in the vapor phase and the liquid phase to determine the effect of condensation which might occur in a coal gasifier during changes in temperature and/or pressure. The percent weight change and percent dimensional change measured after these exposures are given in Section B.4.2.10 along with the details of the test conditions. The changes are not of great magnitude, especially for the dense castables. The results of exposure in the liquid phase are not significantly different from the values for exposure in the vapor phase. The relatively consistent values found for the cycling experiments indicate little effect on the weight or dimensions of the complete cycling process. The insulating castables generally show a larger change in the weight or dimensions than do the dense castables. There seems to be a trend indicating that the lower the alumina content for the insulating castables, the larger the changes in the weight and dimensions upon exposure to the steam-saturated atmospheres.

For density and porosity changes for this last series of tests, see Section B.4.2.11. The densities of the dense castables seem relatively unaffected with the percent changes (most indicating density increases) being generally small. The largest change for the dense refractories is a five percent increase but most values are well below three percent. The insulating castables, especially the two lowest-alumina refractories, showed appreciably greater density changes in those exposures which apparently permitted large amounts of water to be retained

by the sample. The cycling tests indicated that reactions had occurred which had increased the density.

Table A.2.2.2.3.5f summarizes the changes in apparent porosity as compared with values for air-fired control samples. The exposure conditions listed in the table have the following meaning:

CGA = coal gasification atmosphere: 18% CO, 12% CO_2, 24% H_2, 41% H_2O, 5% CH_4

Steam No. 1: saturated steam, 447 °F, 410 psi for 10 days

Steam No. 2: saturated steam, 545 °F, 1000 psi for 10 days

CGA with H_2S No. 1: CGA with 40% H_2O, 1% H_2S, steam-saturated, 447 °F, 1000 psi, for 10 days

CGA with H_2S No. 2: CGA with 40% H_2O, 1% H_2S, steam-saturated, 447 °F, 1000 psi, for 44 days

CGA with H_2S, 85% saturated: CGA with 40% H_2O, 1% H_2S, 85% steam-saturated, 465 °F, 1000 psi, for 5 days

Air-firing: 1000 °F at ambient pressure for 18 hours

Correlating the porosity changes with the density changes is a bit difficult since some very large porosity changes have occurred with minimal density changes and vice versa. In general, the dense castables seemed to experience a large loss in apparent porosity upon exposure to the saturated test atmospheres with the relatively little change in density discussed above. The chemical and physical changes caused by the test atmospheres are filling the pores of the refractories. The formation of hydrated aluminas (especially boehmite) as discussed in A.2.2.2.3.1 is certainly occurring at the 447 °F saturated test temperature and even at 545 °F under the saturated conditions. The insulating castables tend to show an increase in porosity and that generally of a lower magnitude than the changes in the dense castables. Only one, the 54 percent alumina castable, exhibited any appreciable loss in porosity and that occurred both in the vapor and the liquid phases of the steam-saturated coal gasification atmosphere test after the 44-day tests. The boehmite formation-degradation cycling tests indicated that the dense castables retained some loss of porosity compared with air-fired materials upon continued cycling. This retention seems fairly constant if wide limits are accepted in some cases. Although the formation of boehmite seems to cause moderately large porosity loss as the pores are filled with the compound, and the air-firing definitely decomposes the boehmite, the porosity does not return quite to the level of the air-fired but otherwise unexposed material. See Section A.2.2.2.3.4 for a discussion of the strength of these refractories tested under these conditions.

The cycling of the insulating refractory appears to cause small increases in porosity generally. The exception is the 54 percent alumina castable which shows some slight trend toward increasing porosity with cycling. If the increase in porosity of the insulating castables does not coincide with a severe degradation of the material, then the insulating ability is merely enhanced by use. If the increased porosity is indeed caused by breakdown of bonding phases (see discussion in A.2.2.2.1 and A.2.2.2.3.1), then the change in this property may indicate the degree of degradation, and finding refractories exhibiting small changes will be desirable.

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THE EFFECT OF VARYING CARBON MONOXIDE-STEAM CONCENTRATIONS was studied in a series of tests since these are the apparently most important reactants in the coal gasification atmospheres at lower temperatures and pressures. A variety of refractories, dense high- and intermediate-alumina castables, insulating castables, and ramming mixes were tested. One series of tests exposed samples to gas

Percent Difference in Apparent Porosity after Saturated Vapor-Liquid Exposures
and Exposure/Air-Firing Cycles compared with Air-Fired Values

Exposures*		DENSE GENERIC PREPARATIONS			
		93% Alumina UMR-1	87% Alumina UMR-4	64% Alumina UMR-8	58% Alumina UMR-5
Steam No. 1	vapor	-32	- 5.6	-33	-10
	liquid	-28	- 5.6	-24	-14
Steam No. 2	vapor	-37	-19	-38	-15
	liquid	-61	-46	-34	-21
CGA with H_2S No. 1	vapor	-51	-26	-38	-46
	liquid	-50	-25	-40	-44
CGA with H_2S No. 2	vapor	-61	-86	-49	-34
	liquid	-68	not tested	-48	not tested
CGA with H_2S, 85% saturated		-23	-51	-41	-34
followed by air firing		- 5.7	-25	-19	-15
CGA with H_2S, 85% saturated followed by air firing	cycle 1	- 1.9	- 8.1	- 8.0	- 5.3
	cycle 2	- 3.4	- 9.3	- 4.5	- 5.3
	cycle 3	- 1.9	- 9.3	-12	- 6.3
	cycle 4	0	- 9.6	- 6.0	- 5.3
	cycle 5	- 4.2	- 8.9	- 2.5	- 6.3

		INSULATING CASTABLES		
		54% Alumina Cer-Lite #75	47% Alumina Litecast 60-25	35% Alumina VSL-50
Steam No. 1	vapor	+14	+ 6.3	+ 6.7
	liquid	+15	+ 9.9	+ 1.6
Steam No. 2	vapor	+12	+ 3.1	+ 3.8
	liquid	+16	+ 2.3	+ 1.7
CGA with H_2S No. 1	vapor	+ 6.1	- 5.5	- 4.1
	liquid	+ 5.0	- 4.8	+ 3.5
CGA with H_2S No. 2	vapor	-33	+ 3.9	+ 1.3
	liquid	-46	not tested	- 8.3
CGA with H_2S, 85% saturated		- 3.5	- 4.6	- 5.4
followed by air firing		+ 1.7	- 0.9	+ 2.5
CGA with H_2S, 85% saturated followed by air firing	cycle 1	+ 5.7	+ 3.7	+ 2.2
	cycle 2	+ 4.6	+ 2.4	+ 2.5
	cycle 3	+ 4.8	- 0.6	- 0.3
	cycle 4	+ 7.4	+ 2.9	+ 1.4
	cycle 5	+11	+ 4.6	+ 1.3

*See text for details of exposure conditions.

Table A.2.2.2.3.5f

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which contained CO/steam in ratios of 0.1, 1.0, and 3.0 with one volume percent
H_2S. The tests were run for 20 days at temperatures and pressures providing
these component ratios and also under conditions which exposed samples to both
vapor and liquid phases in a vessel in which the CO/steam atmosphere was steam
saturated. Section B.4.2.13 shows the weight changes which occurred (data are
averages for seven samples exposed in vapor, two samples exposed in liquid).
Values in parentheses are for samples exposed under liquid. The changes, either
losses or gains, are not generally meaningful for any of the refractories under
any of the conditions. Even the weight losses or gains experienced by samples
immersed in liquid did not exceed 9.5 percent. Density and porosity results for
these same tests are given in B.4.2.12 (data in parentheses are for samples
immersed in liquid). These density and porosity changes compared to values for
air-fired control samples are summarized in Tables A.2.2.2.3.5g and A.2.2.2.3.5h.
The density changes were generally greater for the samples immersed in liquid,
but these were not large for the dense castables or the ramming mixes tested, the
maximum loss for any castable in any test being 5.6 percent, the maximum gain
4.7 percent. The density changes occurring in vapor and liquid exposures tended
to be higher for the insulating castables, but the behavior seems erratic with
respect to gain or loss and CO/H_2O ratio, or vapor or liquid exposure. The
porosity changes for most of the dense castables consisted largely of appreciable
losses in porosity for most cases. Only the higher alumina materials showed
appreciable differences in behavior between vapor- and liquid-exposed samples.
The tests run in atmospheres of $CO/H_2O = 3.0$ tended to show larger differences
between vapor and liquid exposures for the dense castables. The insulating
castables tended to show increases in porosity but differences between exposures
to liquid as opposed to vapor phases do not seem meaningful.

THE EFFECT ON WEIGHT, DENSITY, AND POROSITY OF STEAM-SATURATED COAL
GASIFICATION ENVIRONMENTS was studied (see B.4.2.14). The density and porosity
data are summarized in Tables A.2.2.2.3.5g and A.2.2.2.3.5h. Examination of the
data for liquid exposures (values given in parentheses) shows a large effect of
decreasing porosity for some dense castables, but the densities are relatively
unaffected, the greatest increase being 7.7 percent.

THERMAL CHARACTERISTICS are also vital because these determine heat losses,
thermal stresses, and pressure vessel shell temperatures. A study was conducted
to generate a computer-programmed model for determining steady-state heat flow
and temperature profiles through multicomponent refractory-lined gasifier vessel
walls. Based on the effect of lining thickness, porosity, gaps, cracks, refractory
composition, physical form, anchor spacing and configuration, gas pressure and
composition, thermal conductivity, and other parameters controlling heat flow, it
is possible to predict the heat transfer through the refractory lining [88].
Another study [26] also developed computer models for heat transfer as well as
measurement techniques using high-temperature strain gauges and acoustic emission
monitoring. Thermal conductivity and heat transfer models were developed in a
third study [99] which modified the models in reference [88] and compared the
results obtained in the two studies. Comparison of the models of [99] was made
with experimental and modeling data for liner heat transfer given in the study
reported in reference [26].

As part of a study of the requirements and the conceptual design of pre-
stressed concrete pressure vessels [101], heat transfer requirements were con-
sidered. Thermal conductivity data for some castables were given (see Sections
B.4.2.43 and B.4.2.44). Thermal analysis for the design concepts included con-
sideration of anchor types and placement.

A.2.2.2.3.5
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A.2.2 Refractory Linings and Components--Dry-Bottom Vessels
A.2.2.2 Performance Data
A.2.2.2.3 Materials Evaluation

Percent Difference in Density of Refractories Exposed to Various Atmospheres with respect to Air-Fired Values[a]

| | CO/H2O/H2S Atmospheres with CO/H2O Ratios of | | | | | | Coal Gasification Atmospheres | |
| | 0.1 | | 1.0 | | 3.0 | | | |
	U[b]	S[b]	U	S	U	S	U	S
DENSE CASTABLES								
94% Alumina (Greencast-94)	not tested		not tested		not tested		+1.9	+0.7(+1.9)
93% Alumina (UMR-1 generic)	+1.1	+3.0(+3.4)	+1.5	-1.5(-0.4)	+1.1	+0.4(-5.6)	+1.1	+1.5(+3.7)
91% Alumina (UMR-2 generic)	+2.8	+5.2(+4.4)	+1.6	-0.4(+1.2)	+4.0	+3.6(-5.6)	+2.4	+0.8(+3.2)
91% Alumina (UMR-3 generic)	not tested		not tested		not tested		+3.8	0 (+7.7)
87% Alumina (UMR-4 generic)	+2.8	+2.4(+4.7)	+2.4	-0.8(+2.0)	+1.6	+1.6(-3.1)	not tested	
88% Alumina (UMR-6 generic)	not tested		+0.4	+0.4(-2.3)	+1.2	+2.3(-1.2)	not tested	(+6.9)
84% Alumina (UMR-7 generic)	not tested		-0.4	-0.4(+3.1)	+0.8	0 (-1.2)	not tested	(+7.1)
59% Alumina (UMR-5 generic)	-1.3	+0.8(+1.7)	0	+0.4(+0.4)	+0.4	+3.8(-1.7)	NT[c]	+1.3(NT)
57% Alumina (RC-3)	+2.7	+2.3(+3.6)	+3.6	+5.0(+2.3)	+1.4	+3.6(-2.7)	+5.0	+3.2(+7.7)
INSULATING CASTABLES								
54% Alumina (Cer-Lite #75)	-1.3	-1.9(-1.9)	+0.6	+1.9(+9.1)	-1.3	-0.6(-1.9)	-1.9	0 (+16)
46% Alumina (Litecast 60-25)	+3.5	+5.3(+16)	+4.4	+9.7(+18)	+2.7	+8.0(+0.9)	NT	+5.3(NT)
35% Alumina (VSL-50)	+1.1	-4.4(+3.3)	-1.1	-6.7(0)	0	-1.1(-3.3)	not tested	
RAMMING MIXES, PHOSPHATE BONDED								
96% Alumina (Greenpak-90P)	-2.4	-3.7(-3.7)	-1.4	-2.7(+0.7)	-2.7	-3.7(-2.4)	NT	-2.0(NT)
90% Alumina (Brikram-90R)	not tested		not tested		not tested		NT	-4.1(NT)

[a]Specimens were air fired at 1000 °F for 18 hours.

[b]U = unsaturated atmosphere, S = steam-saturated atmosphere; values in parentheses are for samples immersed in test vessel liquid.

[c]NT = not tested.

Table A.2.2.2.3.5g

Percent Difference in Apparent Porosity of Refractories Exposed to Various Atmospheres with respect to Air-Fired Values[a]

	CO/H2O/H2S Atmospheres with CO/H2O Ratios of						Coal Gasification Atmospheres	
	0.1		1.0		3.0			
	U[b]	S[b]	U	S	U	S	U	S
DENSE CASTABLES								
94% Alumina (Greencast-94)	not tested		not tested		not tested		+4.0	-8.0(-28)
93% Alumina (UMR-1 generic)	+13	-43 (-43)	+8.7	+13 (-13)	+8.7	-4.3(+13)	+17	-8.7(-74)
91% Alumina (UMR-2 generic)	0	-46 (-50)	-3.6	-21 (-21)	-11	-21 (+18)	-3.6	-18 (-14)
91% Alumina (UMR-3 generic)	not tested		not tested		not tested		0	+3.2(-23)
87% Alumina (UMR-4 generic)	-11	-30 (-37)	-7.4	-3.7(-19)	-3.7	-15 (+3.7)	not tested	
88% Alumina (UMR-6 generic)	not tested		-7.4	-15 (-33)	-7.4	-70 (-11)	not tested	(-48)
84% Alumina (UMR-7 generic)	not tested		-3.7	-33 (-26)	-7.4	-77 (-19)	not tested	(-55)
59% Alumina (UMR-5 generic)	+16	-16 (-26)	0	-11 (-16)	-5.3	-32 (-5.3)	NT[c]	-11 (NT)
57% Alumina (RC-3)	-4.3	-26 (-30)	-13	-22 (-13)	-4.3	-61 (-4.3)	-13	-17 (-43)
INSULATING CASTABLES								
54% Alumina (Cer-Lite #75)	+24	+24 (+24)	+16	+21 (-17)	+12	+21 (+24)	+24	+19 (-12)
46% Alumina (Litecast 60-25)	+13	+3.7(-5.6)	+7.4	+5.6(-17)	+1.9	+3.7(+11)	NT	+5.6(NT)
35% Alumina (VSL-50)	+7.9	+7.9(+1.6)	+6.3	+11 (+6.3)	+3.2	+6.3(+9.5)	not tested	
RAMMING MIXES, PHOSPHATE BONDED								
96% Alumina (Greenpak-90P)	+18	+35 (+29)	+12	+24 (+12)	+18	+29 (+29)	NT	+24 (NT)
90% Alumina (Brikram-90R)	not tested		not tested		not tested		NT	+29 (NT)

[a] Specimens were air fired at 1000 °F for 18 hours.

[b] U = unsaturated atmosphere, S = steam-saturated atmosphere; values in parentheses are for samples immersed in test vessel liquid.

[c] NT = not tested.

Table A.2.2.2.3.5h

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THERMAL CONDUCTIVITY data are given for three high-alumina dense castables, two generic preparations, and one commercial material. The data (see B.4.2.5) are consistent with the material with the lower bulk density having the lower thermal conductivity, as is to be expected. The one generic material with the lower density and lower thermal conductivity is unusual in that the aggregate portion of the refractory consists of sintered calcium aluminate cement. This material also shows the least change in thermal conductivity over the temperature range tested (ambient to 2500 °F). Thermal conductivity data are given for a medium alumina insulating castable and also for a number of coal gasification process gases in Section B.4.2.42. The data were obtained as part of the heat transfer modeling study of reference [99]. Further conductivity data at several temperatures are given for alumina-based and perlite-containing refractories (all commercial materials) in Section B.4.2.20. These data were collected as part of the heat-transfer modeling program [88] to characterize the materials. Conductivity data are also given in B.4.2.15 and B.4.2.17 for a variety of alumina castables and ramming mixes.

THERMAL EXPANSION data are given for four high-alumina castables in B.4.2.31. Linear thermal expansion curves for five alumina refractories, a high-alumina castable, a high-alumina ramming mix, two medium-alumina castables and one medium-alumina brick are shown in B.4.2.19 with coefficients of expansion calculated from cooling curves. Data are also plotted in B.4.2.32 for ten castables, three dense high-alumina, three dense medium-alumina, and four insulating medium- to low-alumina. The linear expansion was examined after a variety of exposures to a simulated coal gas atmosphere, steam-saturated and unsaturated. After exposure to saturated atmospheres there was generally an expansion between 500-600 °C which varied for the castables and was larger for the high-alumina than for the medium-alumina refractories. This effect is related to the dehydration of boehmite, $Al_2O_3 \cdot H_2O$ (see Section A.2.2.2.3.1) which forms in higher quantity in the high-alumina materials. See Section B.4.2.33 for data relating the thermal expansion and the boehmite content. Unsaturated atmospheres produced similar effects in the expansion curves for both high- and medium-alumina materials (B.4.2.32). Insulating castables showed distinct changes in thermal expansion rates over the temperature range of the tests after steam-saturated atmosphere exposure but had smoother curves after the unsaturated gas exposures.

The effect of firing temperature on the thermal expansion of a variety of calcium aluminate-bonded refractories is given in Section B.4.2.30. Data for several high- and low-alumina castables are given including different formulations for the aggregate particle size fractions of a high alumina castable. Further thermal expansion data for calcium aluminate-bonded alumina castables and some phosphate-bonded alumina ramming mixes after several different treatments are given in Section B.4.2.16.

PILOT PLANT TESTING

THE EFFECT OF EXPOSURE TO COAL GASIFICATION PILOT PLANT CONDITIONS on physical properties of a variety of refractories is given in Section B.4.2.6. Specimens of the refractories were placed on test racks in a total of nine locations within six pilot plants. Not all refractories were tested in all locations and for some plants there was only one exposure, in other plants there were two or three exposures. The full conditions to which the samples were exposed are not available. Although run conditions are given they cannot be directly related to the times of exposure of the refractories. Exposure times, when given, are

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approximate as are the concentrations of the chemical constituents of the atmo-
spheres, the temperatures, and the pressures. During plant operation, these
variables fluctuated widely, and the effect on the refractories is unknown.
Evaluation of the numerical data is difficult since percentage differences for
data for exposed and unexposed samples do not seem sufficiently reliable for all
of the data and multiplicate samples were not tested. The results for separate
exposures in the same location differ for many of the materials. In the light of
all of the uncertainties given above, the data must be viewed with caution. Four
measurements were made, the percent weight change, the apparent porosity, the
apparent specific gravity, and the bulk density. The porosity, specific gravity,
and density were measured by liquid immersion in kerosene using the ASTM C20-46
method.

Table A.2.2.2.3.5i contains a qualitative summary of the data in B.4.2.6 for
sample weight change. The plant locations are:

Synthane	1	gasifier off-gas
	2	gasifier fluidized bed
CO$_2$ Acceptor	1	gasifier off-gas
	2	dolomite regenerator off-gas
BI-GAS		gasifier off-gas
Battelle	1	gasifier off-gas
	2	combustor off-gas
U-GAS		gasifier off-gas
HYGAS		gasifier off-gas

The symbols used in the table have the following meaning:

nt   The material was not tested in that location.

-    The change in weight between unexposed and exposed samples is less than or
     about equal to five percent in either direction.

I   Increased--the gain in weight is greater than five percent.

D   Decreased--the loss in weight is greater than five percent.

The use of these symbols and the criteria used in assigning them are completely
arbitrary. It can be seen readily from Table A.2.2.2.3.5i that the weight change
for most samples was trivial. Only a few showed as much change as ten percent
and only one refractory had a change appreciably greater than ten percent (a
60 percent alumina dense castable, 38.5 percent change). This lack of major
weight change is consistent with the laboratory data reported above.

Qualitative data summaries for apparent porosity, apparent specific gravity,
and bulk density appear in Tables A.2.2.2.3.5j, k, and ℓ, respectively. The
symbols have the following meaning:

nt   Not tested.

-    The change in data between unexposed and exposed samples is less than or
     about equal to ten percent.

I   Increased--the increased value of the data between unexposed and exposed
     samples is greater than ten percent.

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Percent Weight Change of Refractory Coupons Due to Exposures in Pilot Plant Locations

Refractories	Synthane 1	Synthane 2	CO₂ Acceptor 1	CO₂ Acceptor 2	BI-GAS	Battelle 1	Battelle 2	U-GAS	HYGAS
CASTABLES, CALCIUM ALUMINATE-BONDED									
90+% Al₂O₃ dense (Purolite 30)	–	nt	–	D	nt	nt	nt	nt	nt
90+% Al₂O₃ dense (Greencast-97)	nt	nt	–	–	nt	nt	nt	nt	–
90+% Al₂O₃ light (Greencast-97L)	–	–	–	–	nt	nt	nt	nt	nt
90% Al₂O₃ dense (Castolast G)	–	D	–	–	nt	D	D	–	–
60% Al₂O₃ dense (Mizzou Castable)	–	–	–	–	nt	D	D	–	–
54% Al₂O₃ dense (Super Brikcast A)	–	–	–	–	D	nt	nt	–	–
54% Al₂O₃ lightweight insulating (Litecast 75-28)	D	–	-cr	–	D	nt	nt	nt	–
52% Al₂O₃ light (Litecast 80)	–	nt	–	–	nt	nt	nt	nt	nt
PHOSPHATE-BONDED REFRACTORIES									
90+% Al₂O₃ dense castable (Resco Cast AA-22)	–	–	–	–	–	–	–	–	nt
88% Al₂O₃ mortar (Chemal 111)	–	nt	nt	nt	nt	–	nt	nt	nt
90% Al₂O₃ ramming mix (Brikram 90)	–	–	-cr	–	nt	–	–	–	–
73% Al₂O₃ dense ramming mix (Blu Ram)	–	nt	nt	nt	nt	nt	nt	nt	nt
73% Al₂O₃ dense ramming mix (Blu Ram H.S.)	–	nt	nt	nt	nt	nt	nt	nt	nt
60% Al₂O₃ ramming mix (Wasp #60)	–	–	-f	–	nt	nt	nt	–	nt
85% Al₂O₃ dense fired brick (Chemal 85 B)	–	–	–	–	–	–	–	nt	–
85% Al₂O₃ fired brick (Altex 85-B)	–	–	–	–	nt	nt	nt	nt	–
80+% Al₂O₃ fired brick (Alumex P-8)	–	–	–	–	nt	–	–	nt	–
ALUMINA BRICKS									
99+% Al₂O₃ self-bonded dense fired (H-W Corundum)	–	–	–	–	nt	nt	nt	nt	–
99+% Al₂O₃ self-bonded fused-cast dense (Monofrax A)	–	–	–	–	nt	nt	nt	nt	nt
98+% Al₂O₃ self-bonded light insulating fired (Alfrax B 101)	–	–	–	I	nt	nt	nt	nt	–
90+% Al₂O₃ self-bonded dense fired (Kricor)	–	–	–	–	nt	nt	nt	nt	–
77% Al₂O₃ self-bonded light insulating fired (B&W Insalcor)	–	–	D	I	nt	nt	nt	nt	–
70% Al₂O₃ vitreous bond fired (Mul-8)	–	–	–	–	–	–	–	–	–
70+% Al₂O₃ vitreous fired (Hi Lumite 70 D)	–	–	D	–	nt	nt	nt	nt	–
70% Al₂O₃ vitreous dense fired (Alumex 70-HD)	nt	–	–	–	–	–	–	–	–
60% Al₂O₃ tar-impregnated vitreous fired (Ufala TI)	–	–	D	–	D	nt	D	nt	–
45-50% Al₂O₃ clay vitreous bond, high-fired super-duty (KX-99)	–	–	–	–	nt	nt	–	nt	–
MISCELLANEOUS BRICKS									
SiC silicon nitride-bonded dense fired (Refrax 20)	–	–	-c	–	nt	nt	nt	–	nt
SiC oxynitride bond (Crystolon 63)	–	–	D	–	nt	nt	nt	nt	nt
SiC clay-bonded (Harbide)	nt	nt	nt	nt	nt	–	–	nt	nt
Alumina-zirconia-silica fused cast (AZS)	–	–	nt	nt	nt	nt	nt	nt	nt
Zirconium silicate vitreous bond (Zircon)	nt	–	–	nt	nt	nt	nt	nt	nt
90% Al₂O₃/10% Cr₂O₃ solid solution fired (CS 612)	nt	nt	–	–	nt	nt	nt	nt	nt

Table A.2.2.2.3.5i (see text for definitions of symbols)

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D   Decreased--the decreased value of the data between unexposed and exposed
     samples is greater than ten percent.

c   One of the coupons from one or more of the plant exposures corroded.

cr   One of the coupons from one or more of the plant exposures cracked.

f   One of the coupons from one or more of the plant exposures fractured.

dis The coupon disintegrated during exposure.

?   Data are incomplete--data given only for the unexposed sample or only
     for the exposed sample.

Where both I and D appear, there are conflicting values for different exposures
of the same refractory in the same plant. The order I,D or D,I merely indicates
which has the greater numerical value. Where there is more than one exposure,
the qualitative rating is based on the worst result, the greatest difference for
exposed vs. unexposed values. The criteria used in the assignment of these
symbols are completely arbitrary.

    Table A.2.2.2.3.5j indicates that the apparent porosity generally increased
more than ten percent for most of the refractories for most of the tests. A
large number also show changes of less than ten percent. The number of large
decreases in porosity noted in the table is very small. Whether the increase in
apparent porosity for so many of the materials is disadvantageous or not is
difficult to assess. It is obvious that the bricks generally experienced an
increase in porosity upon exposure. Extremely large increases were measured for
fused cast materials which had developed a network of hairline cracks due to
thermal shock. Trends in this set of data are difficult to correlate with data
discussed in section A.2.2.2.3.1 and summarized in Table A.2.2.2.3.1b for the
same series of pilot plant exposures. Favorable performance with respect to the
stability of the bonding phase as discussed in A.2.2.2.3.1 seems to have little
relationship with any change or lack of change of the apparent porosity of the
material or indeed of the specific gravity (Table A.2.2.2.3.5k) or of the bulk
density (Table A.2.2.2.3.5ℓ). For most of the refractories tested, the changes
of these two properties, specific gravity and bulk density, were less than ten
percent.

    SILICON CARBIDE AND SILICON NITRIDE REFRACTORIES were among those refrac-
tories tested in the above described pilot plant exposures, and the data appear
in the tables summarizing the data. Silicon nitride-bonded and oxynitride-bonded
silicon carbides exhibited moderate to large increases in porosity with decreases
in the specific gravity and bulk density for the gasifier exposures. Data for
exposure in the dolomite regenerator shows a large decrease in porosity, with
some decrease in specific gravity and increase in density.

    IN GENERAL, DENSITY AND POROSITY, ALONG WITH WEIGHT AND DIMENSIONAL CHANGE
do not seem to be very sensitive measures of the effect of various atmospheres
and reactants on the performance of refractories for coal gasification use.
Mechanical properties (see A.2.2.2.3.2) form more important criteria for choosing
various refractories for different uses.

===

Percent Apparent Porosity Change of Refractory Coupons Due to Exposures in Pilot Plant Locations

Refractories	Synthane 1	Synthane 2	CO₂ Acceptor 1	CO₂ Acceptor 2	BI-GAS	Battelle 1	Battelle 2	U-GAS	HYGAS
CASTABLES, CALCIUM ALUMINATE-BONDED									
90+% Al_2O_3 dense (Purolite 30)	dis	nt	I	I	nt	nt	nt	nt	nt
90+% Al_2O_3 dense (Greencast-97)	nt	nt	-	D	nt	nt	nt	nt	-
90+% Al_2O_3 light (Greencast-97L)	-	I	D,I	I,D	nt	nt	nt	nt	nt
90% Al_2O_3 dense (Castolast G)	-	-	D,I	I,D	nt	nt	nt	D	-
60% Al_2O_3 dense (Mizzou Castable)	-	-	D,I	-	I	-	D	D	D
54% Al_2O_3 dense (Super Brikcast A)	-	-	-	-	-	nt	nt	-	D
54% Al_2O_3 lightweight insulating (Litecast 75-28)	I	I	I	I	I	nt	nt	nt	I
52% Al_2O_3 light (Litecast 80)	I	nt	Icr	I	nt	nt	nt	nt	nt
PHOSPHATE-BONDED REFRACTORIES									
90+% Al_2O_3 dense castable (Resco Cast AA-22)	I	D	-	I	-	-	-	-	nt
90% Al_2O_3 ramming mix (Brikram 90)	I	D	Dcr	D,I	-	-	-	-	I,D
73% Al_2O_3 dense ramming mix (Blu Ram)	I	nt	nt	nt	nt	nt	nt	nt	nt
73% Al_2O_3 dense ramming mix (Blu Ram H.S.)	-	nt	nt	nt	nt	nt	nt	nt	nt
60% Al_2O_3 ramming mix (Wasp #60)	-	I	Icr	I,D	-	-	nt	-	I
85% Al_2O_3 dense fired brick (Chemal 85 B)	I	I	-	I	nt	-	nt	nt	I
85% Al_2O_3 fired brick (Altex 85-B)	I	I	I	I	nt	nt	nt	nt	I
80+% Al_2O_3 fired brick (Alumex P-8)	I	I	I	I	I	-	nt	nt	I
ALUMINA BRICKS									
99+% Al_2O_3 self-bonded dense fired (H-W Corundum)	-	-	D,I	I	nt	nt	nt	nt	I
99+% Al_2O_3 self-bonded fused-cast dense (Monofrax A)	I	I	I,D	I,D	nt	nt	nt	nt	nt
98+% Al_2O_3 self-bonded light insulating fired (Alfrax B 101)	-	-	-	D	nt	nt	nt	nt	-
90+% Al_2O_3 self-bonded dense fired (Kricor)	I	I	-	-	-	nt	nt	nt	-
77% Al_2O_3 self-bonded light insulating fired (B&W Insalcor)	I	I	I	I	nt	nt	nt	nt	I
70% Al_2O_3 vitreous bond fired (Mul-8)	I	I	I	-	nt	-	nt	nt	I
70+% Al_2O_3 vitreous fired (Hi Lumite 70 D)	-	I	I	D,I	nt	nt	nt	nt	D,I
70% Al_2O_3 vitreous dense fired (Alumex 70-HD)	nt	nt	-	-	-	nt	nt	-	-
60% Al_2O_3 tar-impregnated vitreous fired (Ufala TI)	I	-	I	I	I	nt	I	I	I
45-50% Al_2O_3 clay vitreous bond, high-fired super-duty (KX-99)	I	I	I	-	I	-	-	nt	I
MISCELLANEOUS BRICKS									
SiC silicon nitride-bonded dense fired (Refrax 20)	I	I	I	D	nt	nt	nt	D	nt
SiC oxynitride bond (Crystolon 63)	I	I	I	D	nt	nt	nt	nt	nt
SiC clay-bonded (Harbide)	nt	nt	nt	nt	nt	-	I	nt	nt
Alumina-zirconia-silica fused cast (AZS)	I	nt	nt	nt	?	nt	nt	nt	nt
Zirconium silicate vitreous bond (Zircon)	nt	-	-	-	nt	nt	nt	nt	nt
90% Al_2O_3/10% Cr_2O_3 solid solution fired (CS 612)	nt	nt	-	-	nt	nt	nt	nt	nt

Table A.2.2.2.3.5j (see text for definitions of symbols)

Percent Specific Gravity Change of Refractory Coupons Due to Exposures in Pilot Plant Locations

Refractories	Synthane 1	Synthane 2	CO_2 Acceptor 1	CO_2 Acceptor 2	BI-GAS	Battelle 1	Battelle 2	U-GAS	HYGAS
CASTABLES, CALCIUM ALUMINATE-BONDED									
90+% Al_2O_3 dense (Purolite 30)	dis	nt	I	I	nt	nt	nt	nt	nt
90+% Al_2O_3 dense (Greencast-97)	nt	nt	-	-	nt	nt	nt	nt	-
90+% Al_2O_3 light (Greencast-97L)	I	I	I,D	I,D	nt	nt	nt	nt	nt
90% Al_2O_3 dense (Castolast G)	-	-	-	-	nt	nt	-	-	-
60% Al_2O_3 dense (Mizzou Castable)	-	-	-	-	-	-	nt	-	-
54% Al_2O_3 dense (Super Brikcast A)	I	I	I	I	I	nt	nt	-	I
54% Al_2O_3 lightweight insulating (Litecast 75-28)	I	I	Icr	I	I	nt	nt	nt	nt
52% Al_2O_3 light (Litecast 80)	I	nt	-	-	nt	nt	nt	nt	nt
PHOSPHATE-BONDED REFRACTORIES									
90+% Al_2O_3 dense castable (Resco Cast AA-22)	D,I	-	-	-	-	-	-	-	nt
90% Al_2O_3 ramming mix (Brikram 90)	-	-	-cr	D	-	-	-	-	-
73% Al_2O_3 dense ramming mix (Blu Ram)	-	nt	nt	nt	nt	nt	nt	nt	nt
73% Al_2O_3 dense ramming mix (Blu Ram H.S.)	-	nt	nt	nt	nt	nt	nt	nt	nt
60% Al_2O_3 ramming mix (Wasp #60)	-	-	-cr	-cr	nt	nt	nt	nt	nt
85% Al_2O_3 dense fired brick (Chemal 85 B)	-	-	-	-	-	-	-	-	-
85% Al_2O_3 fired brick (Altex 85-B)	-	-	-	-	nt	nt	nt	-	-
80+% Al_2O_3 fired brick (Alumex P-8)	-	-	-	-	nt	-	nt	nt	-
ALUMINA BRICKS									
99+% Al_2O_3 self-bonded dense fired (H-W Corundum)	-	-	-	-	nt	nt	nt	nt	-
99+% Al_2O_3 self-bonded fused-cast dense (Monofrax A)	-	-	-	D	nt	nt	nt	nt	nt
98+% Al_2O_3 self-bonded light insulating fired (Alfrax B 101)	D	-	D,I	D	nt	nt	nt	nt	-
90+% Al_2O_3 self-bonded dense fired (Kricor)	-	-	I	I	-	nt	nt	nt	-
77% Al_2O_3 self-bonded light insulating fired (B&W Insalcor)	D	I	I	I	nt	nt	nt	nt	-
70% Al_2O_3 vitreous bond fired (Mul-8)	-	-	-	-	nt	-	nt	-	-
70% Al_2O_3 vitreous fired (Hi Lumite 70 D)	-	-	I	D,I	nt	nt	nt	nt	-
70% Al_2O_3 vitreous dense fired (Alumex 70-HD)	nt	nt	-	-	-	nt	nt	-	-
60% Al_2O_3 tar-impregnated vitreous fired (Ufala TI)	-	-	-	-	-	nt	I	-	-
45-50% Al_2O_3 clay vitreous bond high-fired super-duty (KX-99)	-	-	-	-	-	nt	-	nt	-
MISCELLANEOUS BRICKS									
SiC silicon nitride-bonded dense fired (Refrax 20)	-	D	D	-	nt	nt	nt	D	nt
SiC oxynitride bond (Crystolon 63)	-	-	-	-	nt	nt	-	nt	nt
SiC clay-bonded (Harbide)	nt	nt	nt	nt	nt	-	nt	nt	nt
Alumina-zirconia-silica fused-cast (AZS)	-	nt	nt	nt	?	nt	nt	nt	nt
Zirconium silicate vitreous bond (Zircon)	nt	-	-	nt	nt	nt	nt	nt	nt
90% Al_2O_3/10% Cr_2O_3 solid solution fired (CS 612)	nt	nt	-	-	nt	nt	nt	nt	nt

Table A.2.2.2.3.5k (see text for definitions of symbols)

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Percent Bulk Density Change of Refractory Coupons Due to Exposures in Pilot Plant Locations

Refractories	Synthane 1	Synthane 2	CO_2 Acceptor 1	CO_2 Acceptor 2	BI-GAS	Battelle 1	Battelle 2	U-GAS	HYGAS
CASTABLES, CALCIUM ALUMINATE-BONDED									
90+% Al₂O₃ dense (Purolite 30)	dis	nt	–	–	nt	nt	nt	nt	nt
90+% Al₂O₃ dense (Greencast-97)	nt	nt	–	–	nt	nt	nt	nt	–
90+% Al₂O₃ light (Greencast-97L)	–	–	I	–	nt	nt	nt	nt	nt
90% Al₂O₃ dense (Castolast G)	–	–	–	–	nt	–	nt	–	–
60% Al₂O₃ dense (Mizzou Castable)	–	–	–	–	–	–	–	–	–
54% Al₂O₃ dense (Super Brikcast A)	–	–	–	–	–	nt	nt	–	–
54% Al₂O₃ lightweight insulating (Litecast 75–28)	D	–	–	I	D	nt	nt	nt	D
52% Al₂O₃ light (Litecast 80)	–	nt	–cr	–	nt	nt	nt	nt	nt
PHOSPHATE-BONDED REFRACTORIES									
90+% Al₂O₃ dense castable (Resco Cast AA-22)	D	–	–	–	–	–	–	–	nt
90% Al₂O₃ ramming mix (Brikram 90)	–	–	–cr	–	–	–	–	–	–
73% Al₂O₃ dense ramming mix (Blu Ram)	–	nt	nt	nt	nt	nt	nt	nt	nt
73% Al₂O₃ dense ramming mix (Blu Ram H.S.)	–	nt	nt	nt	nt	nt	nt	nt	nt
60% Al₂O₃ ramming mix (Wasp #60)	D	–	Dcr	Dcr	nt	nt	nt	nt	nt
85% Al₂O₃ dense fired brick (Chemal 85 B)	–	–	–	–	–	nt	nt	nt	–
85% Al₂O₃ fired brick (Altex 85–B)	–	–	–	–	nt	nt	nt	nt	–
80+% Al₂O₃ fired brick (Alumex P-8)	–	–	–	–	nt	–	nt	nt	–
ALUMINA BRICKS									
99+% Al₂O₃ self-bonded dense fired (H-W Corundum)	–	–	–	–	nt	nt	nt	nt	–
99+% Al₂O₃ self-bonded fused-cast dense (Monofrax A)	–	–	–	–	nt	nt	nt	nt	nt
98+% Al₂O₃ self-bonded light insulating fired (Alfrax B 101)	–	–	–	–	nt	nt	nt	nt	–
90+% Al₂O₃ self-bonded dense fired (Kricor)	–	–	–	–	–	nt	nt	nt	–
77% Al₂O₃ self-bonded light insulating fired (B&W Insalcor)	D	–	D	D	nt	nt	nt	nt	D
70% Al₂O₃ vitreous bond fired (Mul-8)	–	–	–	–	nt	–	nt	nt	–
70+% Al₂O₃ vitreous fired (Hi Lumite 70 D)	–	–	I	I,D	nt	nt	nt	nt	–
70% Al₂O₃ vitreous dense fired (Alumex 70-HD)	nt	nt	–	–	–	nt	nt	–	–
60% Al₂O₃ tar-impregnated vitreous fired (Ufala TI)	–	–	–	–	–	nt	nt	–	–
45–50% Al₂O₃ clay vitreous bond, high-fired super-duty (KX-99)	–	–	–	–	–	nt	–	nt	–
MISCELLANEOUS BRICKS									
SiC silicon nitride-bonded dense fired (Refrax 20)	D	D	D	–	nt	nt	nt	–	nt
SiC oxynitride bond (Crystolon 63)	–	D	–	–	nt	nt	–	nt	nt
SiC clay-bonded (Harbide);	nt	nt	nt	nt	nt	–	–	nt	nt
Alumina-zirconia-silica fused-cast (AZS)	–	nt	nt	nt	?	nt	nt	nt	nt
Zirconium silicate vitreous bond (Zircon)	nt	--	–	nt	nt	nt	nt	nt	nt
90% Al₂O₃/10% Cr₂O₃ solid solution fired (CS 612)	nt	nt	–	–	nt	nt	nt	nt	nt

Table A.2.2.2.3.5ℓ (see text for definitions of symbols)

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A.2.3.1 OPERATING REQUIREMENTS

All of the problems of erosion and corrosion noted in the previous section
for non-slagging vessels (A.2.2.1) exist for slagging vessels and are intensi-
fied in a portion of the gasifier by higher temperature requirements (up to
3,200 °F [2,033 K]) and the presence of liquid slag which is very corrosive.
Resistance to slag corrosion would be the major criterion for material selec-
tion in the hottest portion of slagging gasifiers. Slag compositions vary
greatly for different coals with Western coals generally more acidic and East-
ern coals more basic. This fact suggests that a single refractory composition
is unlikely to be equally effective against all slags and, once a choice is
made, a specific plant will be limited in the type of coal it can handle. The
corrosive effects of liquid slag can be substantially reduced by cold wall
designs which remove enough heat to freeze a protective layer of slag on the
walls. A substantial materials technology exists from coal combustors and
blast furnaces, but differences in operating conditions suggest that technology
transfer will not be straightforward.

Discussion of the materials and design requirements for refractories for
slagging gasifiers is to be found in reference [87]. These project reports
include: conceptual refractory liner designs; design and construction of a
system to test erosion and corrosion by slag of refractory linings and, to
evaluate total refractory materials and design performance; and development of
a model to predict long-term service life.

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A.2.3.2.2.1 SLAG CORROSION

THE RELATIVE CORROSION RESISTANCE of a number of refractories was studied
in a series of tests at about 1500 °C using a variety of slag compositions.
Specimen wedges were placed around the wall of a test vessel so as to act as a
vessel liner. Most of the wedges were manufacturer-supplied bricks but some
wedges were prepared from ramming mixes and castables. Synthetic slags were
added to the vessel and three burners on the surface of the melt provided some
rotary motion of the slag. Thirty-three different refractories were exposed in
a series of three tests for which the exposure time was either 200 or 500 hours
(see Section B.1.2.14). One slag was basic, the other two of two different
acidities. Thirty-eight refractories were exposed for periods of 500 or 1000
hours in a furnace modified so that the bricks experienced thermal gradients
(see B.1.2.15). Water cooling was arranged for the outermost portions of the
bricks and the bricks were of three different lengths. Additional charges of
slag were added during the exposures and the worst attack was observed at the
slag line. The data reported in B.1.2.15 are for the length specimen which
showed the worst attack (usually the longest brick, corresponding to the hottest
slag-brick interface). The slags used in the tests with water cooling were all
of differing acidities except for one basic slag. For both uncooled and water-
cooled tests the slag was analyzed at intervals during the tests and the compo-
sition was found to fluctuate widely. In Sections B.1.2.14 and B.1.2.15 average
values of slag constituents are given. It should be noted that the test condi-
tions effectively expose a brick wall composed of many kinds of bricks to a slag
liquid. Local variations in slag composition due to dissolution of some bricks
may influence the performance of neighboring bricks. The original investigators
also have pointed out that the slag velocity at the surface of the test specimens
is low so that there is minimum erosion by the slag and that the use of surface
burners produces a vertical temperature gradient in the slag. Therefore, these
tests do not simulate the behavior of a single composition wall in a real slag-
ging environment.

The effect of slag attack is reported as maximum depth of material removal
and maximum depth of slag penetration. Although the numerical values can not be
used for predicting lining performance in actual gasifier use, they are useful
for identifying classes of materials to be investigated further. Table A.2.3.2.2.1a
lists the materials from Section B.1.2.14 which had the least amount of material
removed (maximum depth of removal less than 10 mm). Table A.2.3.2.2.1b lists
the materials from Section B.1.2.15 with the least material removed (less than
10 mm depth) for the water-cooled refractories. In both tables the data are the
depths of material removed and the maximum penetration of slag, both in mm. In
Table A.2.3.2.2.1b the penetration value is the number in parentheses. Note
that in both tables the depth of slag penetration is often several times the
depth of removal. Although little material may have been removed the penetra-
tion of slag may seriously affect the properties of the remaining refractory
(see Section A.2.3.2.2.2).

Overall, the water-cooled refractories subjected to slag experienced some-
what less attack than those not cooled. Note that silicon carbide-based mate-
rials experienced extreme attack when not cooled compared with the one water-
cooled test to which they were subjected. Table A.2.3.2.2.1c lists materials
from both B.1.2.14 and B.1.2.15 which were subjected to more than one test and
gives the maximum depths of removal in mm. Note that for some refractories the

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Refractory	200 h B/A 0.45		500 h B/A 0.84		500 h B/A 1.35	
	Maximum Removal	Max. Pen.	Maximum Removal	Max. Pen.	Maximum Removal	Max. Pen.
$Al_2O_3(99)$, fused-cast brick	3	4				
$Al_2O_3(91)-SiO_2(8)$, sintered brick	7	12				
$Al_2O_3(84)-SiO_2(9)-P_2O_5(4)$, brick	7	25				
$Al_2O_3(89)-Cr_2O_3(10)$, sintered brick	5	20				
$Cr_2O_3(80)-MgO(8)-Fe_2O_3(6)$, fused-cast	0	0	2	5	0	0
$MgO(64)-Cr_2O_3(15)-Al_2O_3(13)-Fe_2O_3(6)$, s*	7	22				
$MgO(63)-Cr_2O_3(18)-Al_2O_3(5)-Fe_2O_3(12)$, s*			6	38		
$MgO(63)-Cr_2O_3(17)-Al_2O_3(10)-Fe_2O_3(9)$, s*			9	41		
$MgO(59)-Cr_2O_3(19)-Al_2O_3(13)-Fe_2O_3(7)$, s*	7	20	8	38	5	10
$MgO(53)-Cr_2O_3(20)-Al_2O_3(17)-Fe_2O_3(8)$, s*	8	21			5	10

*s = sintered brick
B/A = base to acid ratio

Table A.2.3.2.2.1a (see text for explanation of data)

amount of material removed fluctuated dramatically from test to test showing a dependence of reaction on the acidity of the slag. Certain conclusions can be drawn from these three tables. High-chromia refractories, magnesia-chromia refractories, and fused-cast materials exhibit good slag corrosion resistance in general whatever the slag composition. High-alumina refractories perform fairly well in acidic slags but poorly in basic slags.

Examination of slag-exposed specimens by metallography, scanning electron microscopy and electron probe analysis is reported in Sections B.1.2.22, B.1.2.23, B.1.2.24, B.1.2.25. The data indicate that the slag corrosion resistance of high-chromia refractories is due to the formation of relatively stable and non-reactive iron-rich mixed spinels. Spinels of varying compositions based on $(Mg, Fe)(Al, Cr, Fe)O_4$ seem to have formed at the refractory-slag interface in those specimens which had shown good slag corrosion resistance. More severe slag conditions were therefore imposed on magnesia-chromia spinel refractories in tests conducted as part of the same project. The tests provided for an erosion component in the slag attack. Cylindrical specimens were rotated in a slag bath at speeds from 50 rpm to 200 rpm. The results are given in B.1.2.26, showing somewhat variable but generally good response of many of the spinel-based refractories.

Equilibrium studies are being conducted [98] dealing with oxide combinations representing simplified coal-ash slags in combination with refractory oxides. Phase diagrams are being generated which show the effect of silicate slags on chromia-containing refractories.

Refractory	Base to Acid = 0.48 / 475 h	0.55 / 500 h	0.64 / 500 h	0.7 / 305 h	0.7 / 500 h	0.74 / 500 h	0.75 / 500 h	0.9 / 1000 h	1.4 / 500 h
$Al_2O_3(99)$, fused-cast	1 (60)								
$Al_2O_3(92)-SiO_2(8)$, sintered					9 (14)		9 (10)		
$Al_2O_3(90)-SiO_2(6)-P_2O_5(4)$, ram mix	4 (50)				9 (24)				
$Al_2O_3(70)-MgO(28)$, sintered	2 (23)								
$Al_2O_3(90)-MgO(3)-P_2O_5(6)$, ram mix									3 (8)
$Al_2O_3(90)-Cr_2O_3(10)$, sintered		7 (17)					4 (7)		
$Al_2O_3(81)-Cr_2O_3(17)$, sintered					2 (20)		2 (9)		
$Al_2O_3(67)-Cr_2O_3(32)$, sintered					4 (20)				
$Al_2O_3(85)-Cr_2O_3(10)-SiO_2(2)-$ $P_2O_5(2)$, ramming mix	3-5(50)	5 (7)							
$Al_2O_3(85)-Cr_2O_3(10)-SiO_2(2)-$ $P_2O_5(3)$, ramming mix		7 (9)							
$Al_2O_3(65)-Cr_2O_3(32)-FeO(1)$, fused	1 (0)			10 (12)				4 (-)*	1 (1)
$Al_2O_3(60)-Cr_2O_3(27)-MgO(6)-$ $Fe_2O_3(4)-SiO_2(2)$, fused-cast								2 (-)	1 (2)
$Cr_2O_3(80)-MgO(8)-Fe_2O_3(6)-$ $Al_2O_3(5)-SiO_2(1)$, fused-cast	0 (0)	3 (4)			2 (45)				
$Cr_2O_3(40)-Al_2O_3(21)-Fe_2O_3(23)-$ $MgO(10)-SiO_2(3)-P_2O_5(4)$, ram mix	2 (60)								
$MgO(95)-SiO_2(3)-CaO(1)$, ram mix	5,6(7)		8 (9)						
$MgO(63)-Cr_2O_3(18)-Al_2O_3(5)-$ $Fe_2O_3(12)-SiO_2(1)-CaO(1)$, sintered	1 (-)								4 (14)
$MgO(60)-Cr_2O_3(16)-Al_2O_3(15)-$ $Fe_2O_3(7)-SiO_2(2)$, sintered			8 (11)						
$MgO(59)-Cr_2O_3(19)-Al_2O_3(13)-$ $Fe_2O_3(7)-SiO_2(2)$, sintered	1 (40)								4 (16)
$MgO(55)-Cr_2O_3(20)-Al_2O_3(8)-FeO(11)$ $-SiO_2(3)-TiO_2(2)$, sintered			8 (11)						
$MgO(53)-Cr_2O_3(20)-Al_2O_3(17)-$ $Fe_2O_3(8)-SiO_2(2)-CaO(1)$sintered									5 (20)
$SiC(90)-Si_2ON_2(9)$						3-4(-)			
$SiC(73)-Al_2O_3(14)-SiO_2(11)$						4-7(-)			
$SiC(75)-Si_3N_4(23)$						4-5(-)			

*(-) means either value not given at all or it was stated that the value could not be determined.

Table A.2.3.2.2.1b (see text for explanation of data)

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Refractory	Water Cooled								Not Cooled		
Base to Acid =	1.4	0.9	0.75	0.74	0.7	0.64	0.55	0.48	1.35	0.84	0.45
$Al_2O_3(99)$, sintered	10				20		18	1	57		16
$Al_2O_3(99)$, fused-cast									22		3
$Al_2O_3(99)-Na_2O(0.5)$, fused-cast									19		
$Al_2O_3(92)-SiO_2(8)$, sintered		46	9						64	36	12
$Al_2O_3(85)-SiO_2(9)-P_2O_5(4)$			13								7
$Al_2O_3(92)-Cr_2O_3(8)$, sintered	21						18	2		11	
$Al_2O_3(90)-Cr_2O_3(10)$, sintered			4						10		
$Al_2O_3(89)-Cr_2O_3(10)$, sintered	10						17			15	5
$Al_2O_3(81)-Cr_2O_3(17)$, sintered	3		2				7				
$Al_2O_3(67)-Cr_2O_3(32)$, sintered	10				2		10				
$Al_2O_3(85)-Cr_2O_3(11)-P_2O_5(5)$, plastic	1						38				
$Al_2O_3(65)-Cr_2O_3(32)-FeO(1)$, fused cast							5				
$Al_2O_3(21)-Cr_2O_3(40)-Fe_2O_3(23)$, ram mix					2			2		11	21
$Al_2O_3(60)-Cr_2O_3(27)-MgO(6)-Fe_2O_3(4)-$ $SiO_2(2)$, fused-cast		4			10		7	1			
$Cr_2O_3(80)-MgO(8)-Fe_2O_3(6)$, fused-cast	1	2					3	0	0	2	0
$MgO(64)-Cr_2O_3(15)-Al_2O_3(13)-Fe_2O_3(6)$, sintered										11	7
$MgO(63)-Cr_2O_3(18)-Al_2O_3(5)-Fe_2O_3(12)-$ $SiO_2(1)-CaO(1)$, sintered						8		1		6	
$MgO(61)-Cr_2O_3(19)-Al_2O_3(6)-Fe_2O_3(11)-$ $SiO_2(1)-CaO(2)$, sintered	4				34		11				
$MgO(60)-Cr_2O_3(16)-Al_2O_3(15)-Fe_2O_3(7)-$ $SiO_2(2)-CaO(1)$						17					
$MgO(59)-Cr_2O_3(19)-Al_2O_3(13)-Fe_2O_3(7)$, sintered	4					8		1	5	8	7
$MgO(55)-Cr_2O_3(20)-Al_2O_3(8)-FeO(11)-$ $SiO_2(3)-TiO_2(2)$, sintered	5						23				
$MgO(53)-Cr_2O_3(20)-Al_2O_3(17)-Fe_2O_3(8)$, sintered						8			5	11	8
$SiC(90)-Si_2ON_2(9)$		32		3-4					64		11
$SiC(73)-Al_2O_3(14)-SiO_2(11)$		36		4-7					64		
$SiC(75)-Si_3N_4(23)$		38		4-5							

Table A.2.3.2.2.1c (see text for explanation of data)

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A.2.3.2.2.2 MECHANICAL PROPERTIES

THE EFFECT OF STEAM AND A SIMULATED COAL GASIFICATION ATMOSPHERE (25% H_2, 13% CO_2, 21% CO, 5% CH_4, 36% H_2O) [vs. air-fired] ON THE COMPRESSIVE STRENGTH of several refractories considered for possible use in slagging vessels is tabulated in B.3.2.20. One material, 73 SiC-21 Si_3N_4-3 Al_2O_3-3 SiO_2 sintered brick, disintegrated when exposed to steam and to the coal gas. For a chemically bonded ramming plastic (40 Cr_2O_3-23 Fe_2O_3-21 Al_2O_3-10 MgO) the compressive strength after exposure to steam (980 °C, 250 h, 1000 psi) decreased 60 percent from that for air-fired (980 °C, 250 h) and after exposure to the mixed gas (980 °C, 250 h, 1000 psi) decreased 40 percent from that for air-fired. Three of these materials (medium alumina with varying compositions) increased slightly in compressive strength following both exposure conditions. The behavior of other materials tested was variable with only slight changes in compressive strength. These test results have limited value for actually determining which materials to use for lining slagging vessels because the tests have been performed in the coal gasification environment with no slag present.

THE THERMAL SHOCK RESISTANCE of chromia-containing refractories is important. Although they have shown acceptable corrosion resistance to slag (see Section A.2.3.2.2.1), they tend to have poor thermal shock resistance. Low-porosity materials frequently exhibit poor thermal-shock resistance. The porosity of the chromia-containing refractories tends to be low. See Section B.4.2.41 where the porosity of the chromia-containing refractories varies from 3.50 to 19.20 percent compared with 25-35 percent for most dense alumina refractories. Thermal shock testing has been conducted for some commercial refractories by heating specimens to various high temperatures and quenching into boiling water at 100 °C. The tensile strength was determined on as-received material and then on the quenched specimens as a measure of the shock damage (see Sections B.3.2.146 and B.3.2.147). The data in B.3.2.146 shows that the greatest and most sudden loss of strength was by the refractory with the largest initial tensile strength. The materials with the lowest initial strength exhibited the least loss of strength. Variations in initial and quenched tensile strengths were large and the response to quenching also varied greatly.

Section B.3.2.147 shows the percent retained strength for quenching from 1000 °C to 100 °C for one, three, and five heating-quenching cycles. The data show the continuous drop in strength for all refractories tested as the number of cycles is increased. The final values after all quenches were less than 7 MPa for all refractories tested. Damage resistance parameters for thermal shock based on tensile strength, Young's modulus, thermal expansion coefficient, Poisson's ratio, and surface fracture energy were calculated and are given in Section B.3.2.145. Figure B of B.3.2.147 shows a correlation between increasing retained strength and an increase of the parameter R'''' which is intended to be inversely proportional to the cracking area per unit volume propagated by thermal shock. The general trend in the figure indicates that as R'''' increases so does the retained strength but there is no explanation for the drop off at higher R'''' values for multiple quench cycles. Of the eight refractories tested only data for five appear in the figure.

Studies are under way [100] to develop preparative methods based on variations of composition, powder preparation, particle size, sintering conditions, and binders to obtain refractories with suitable thermal and microstructural

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properties for good thermal-shock resistance. The model refractory base is
$MgCr_2O_4$ with second phase inclusions of ZrO_2, W, Mo, and $FeCr_2O_4$.

CREEP DATA for chromia-containing refractories appear in Section B.3.2.135.
Physical properties given in Section B.4.2.41 provide some characterization for
the refractories tested. The creep data were generated in air under compressive
loading. The tests were conducted in two different modes: temperature was
cycled with a constant applied stress, and stress was cycled under constant
temperature. The investigators found that generally the greatest strain at
constant temperature (usually 2500-2600 °F) was exhibited during the initial
load. It appeared that structural changes were occurring under the initial
load. The deformation under constant stress was greatest at the highest tem-
perature. It would appear from the data of B.3.2.135 that temperatures below
2100-2200 °F are necessary if the load is 500 psi or above. The refractories
failed in testing at much lower temperatures and stresses than expected by the
investigators.

Another project investigated the effect of slag penetration on the creep of
refractories. In penetrating refractories the slag is expected to affect the
viscosity and thickness of glass at grain boundaries in the refractory. An
increase in the glass (slag) thickness or a decrease in the viscosity is ex-
pected to increase the creep rate and enhance creep processes which reduce
refractory strength. An alumina-zirconia-silica fused-cast refractory with
glass in its microstructure was studied because its structure resembles that of
refractory penetrated by slag. Creep data obtained for this refractory in four-
point bending are given in Sections B.3.2.112 and B.3.2.113. The scatter in the
data were attributed to the fact that the specimens, cut from a fused-cast
block, did not exhibit a uniform microstructure throughout. The data are con-
sistent and typical of that of structural metals at high temperatures. The
time, stress, and temperature dependence exhibited by the creep data are shown
in Section B.3.2.114. The secondary strain rate is shown to be inversely propor-
tional to the time to failure. The plot of strain rate versus initial stress
given in B.3.2.114 indicates that the strain rate is a power function of the
stress with an exponent of 2.9. This exponent indicates that cavitation is
probably occurring in the glassy phase during creep deformation. The figure in
B.3.2.114 showing temperature dependence and activation energy is consistent
with data obtained for viscous creep of glass at elevated temperatures. To test
the role of the glassy phase in refractory creep, two aluminas with varying
glass contents of 4 and 15 percent were creep tested (see B.3.2.115 and B.3.2.116).
The stepped-temperature tests (B.3.2.115) indicated a higher temperature range
for creep testing for these materials (∿950-1100 °C) than could be used for the
alumina-zirconia-silica fused-cast refractory (600-800 °C). The creep data
given in B.3.2.116 for the 4%-glass content alumina showed far less scatter than
did the fused-cast specimens studied. Typical curves are given and the data
indicate that this material shows the inverse relationship of strain rate and
time to failure.

A program to study the failure mechanisms of refractory brick lined coal gas-
ification slagging vessels is underway [94]. Using mechanical and thermal prop-
erties at different temperature levels, models are being developed for bricks and
mortar to predict the stress and strain distribution during heat-up and cool-down
cycles and to detect crack formation and propagation during the cycles.

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For a discussion of corrosion/erosion and mineralogical changes caused by the reactor atmosphere and high-temperature and high-pressure effects on mechanical and physical properties, see Section A.2.2.2.3, Materials Evaluation for Dry-Bottom Vessels.

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* Sections included in SP-642, Supplement 1, and Supplement 2 combined.

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A.9.3.1 OPERATING REQUIREMENTS

The operating requirements for slurry and solids valves vary among the
several coal gasification and liquefaction processes, and also depend upon the
location of the valve within the process. Water and/or coal liquids are usually
the carrier fluids; the particulates include unreacted charcoal, char and ash.
Typical service conditions are high temperatures and pressures (maximums are
3000 °F (1922 K) and 1600 psi for gasification and 900 °F (755 K) and 4000 psi
for liquefaction) under cyclic loading and exposure to erosive and abrasive
particulates. Depending primarily upon temperature of service, slurry and
solids valves may be classified into four types. This classification is
summarized below.

DESIGN OPERATING TEMPERATURES[3]

Maximum Bulk Media Temperature	Maximum Interface Temperature
Type I - 350 °F	350 °F
Type II - 600 °F*	850 °F
Type III - 2000 °F	850 °F
Type IV - 600 °F (slurry & slag discharge)	600 °F

*The valve temperature may reach 850 °F on initial heat-up under maximum
pressure but without coal.

In addition to the above temperature requirements, valves must also be able
to operate at pressures from atmospheric to the process maximum, and to have
expected lifetimes of 30,000 cycles while exposed to highly erosive streams
containing corrosive gases, water and/or coal liquids. Construction materials
and design considerations must fulfill these requirements. Abrasive wear
associated with unlubricated materials contact often occurs, even in the presence
of purging, and often leads to more serious erosive failure. Materials com-
patibility is an important requirement in order to minimize contact welding,
spalling of protective surface coatings, and thermal distortion. Clogging of
valves by adhering solids or tarry substances can prevent complete closure. The
leak of gas-borne particles through the gap can result in erosion.

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A.9.3.2.1 PLANT EXPERIENCE

Plant experience has been reported for 22 different SLURRY AND SOLIDS VALVES (Section A.9.3.2.1.1). Sixteen different materials were in these valves. Service temperatures ranged from 180 to 2000 °F. Fourteen valves were removed from service due to wear and erosion. These valves were made of a wide variety of materials-- carbon steel, 316 stainless steel, Kennametal K701, Stellites 3 and 6, Teflon, Coors 999 ceramic and Stellite coated on 316 stainless steel. The material which lasted the longest under the most severe service conditions was Stellite 6 in the Synthane Plant (1.5 years/300 °F/600 psi/char-water-gas). Carbon steel lasted well. A valve with CVD-tungsten and plasma-sprayed Cr-Ni-B coated surfaces also performed well during about 18 months of service. The ceramic showed the shortest life at four days.

A 3-inch pinch valve of rubber was removed due to abuse by overload. A poor weld between the shaft and butterfly led to failure and removal of a valve made of RA 330 and Haynes 25 in service at 1,450 °F. An Incoloy 800 valve was removed after 9 months of service at 1,450 °F, but could have remained in service longer if misalignment had not caused severe erosion.

Inappropriate design leading to valve body distortion resulted in removal of three Type 316 stainless steel valves from service. Manufacturing defects over- looked by quality control resulted in two valves--one 440 stainless steel and one 316 stainless steel--being removed from service.

One RA 330 hinge pin was bent and galled due to excessive loading.

In some plant operations, there were indications that solids became lodged at the valve seats and prevented complete closure. The continuing leak of gas-borne particulates through the gap was identified as a source of considerable erosion in some cases. A method for removing solids buildup on mating surfaces could minimize this source of valve erosion.

Tungsten carbide trim material samples from slurry letdown valves from two liquefaction pilot plants were studied (see Section A.9.3.2.1.2). Erosion testing [103] was performed to investigate the reasons for inconsistent trim performance at the plants. Microhardness data were also reported [102] for these same specimens but are not given here since the specific test methods are not clearly stipulated and some inconsistency exists between the sets of data for the samples for the two pilot plants.

A.9.3 Slurry and Solids Valves
A.9.3.2 Performance Data
A.9.3.2.1 Plant Experience

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SOLIDS VALVES IN-SERVICE PERFORMANCE[5,70,104]

Trim Material	Valve Type (Plant ID)	Process	Service Life	Environment	Temp. °F	Pressure psig	Failure Mode
316 S.S. White Iron	6 in. full port ball (0612)	METC	297 cycles	Ash	900	125	Wear: scoring on ball, wear on seat
316 S.S. Stellite 6	10 in. full bore ball (1005)	METC	257 cycles	Coal	250	300(max)	Quality control: cracks in ball from a manufacturing defect
316 S.S.	(HV-719)	Westinghouse	200 hours	Coke breeze/recycle gas	500	200	Design: valve body distorted by impingement of fine particles
316 S.S.	(HV-718)	Westinghouse	200 hours	Coke breeze/recycle gas	500	200	Design: valve body distorted by impingement of fine particles
316 S.S.	(HV-1106)	Westinghouse	200 hours	Coke breeze/recycle gas	500	200	Design: valve body distorted by impingement of fine particles
S.S. Carbon Steel	Letdown (PCV-266)	Synthane	37 hours	Char/water/gas	800	600	Erosion: 50% erosion of trim/groove in downstream pipe
S.S. sleeve Ceramic seat	Letdown (LCV-405)	Synthane	2 months	Coal char	N.A.	1000	Erosion
440C S.S.	2 in. ball (XCV-26)	Synthane	∿200 cycles	Coal/CO_2	300	160	Quality control: surface defects and poor design led to failure of the stem
Stellite 3 (seat) 316 S.S. (ball)	10 in. ball (1003)	METC	1100 cycles	Coal gas/coal	250-350	0-10	Wear: deep scoring of ball, minor scratches on seat
Stellite 6 (seat) 316 S.S. (ball)	10 in. ball (1004)	METC	1475 cycles	Coal gas/coal	400-500	0-10	Wear: deep (1-5 mils) scoring over 4-in. band on ball surface/minor scuffing of seat
Stellite on 316 S.S.	Level control (LCV-201)	Synthane	1 month	Coal char fines in water	180-300	600	Erosion
Stellite 6	Letdown to flare (PCV-2205)	Synthane	1.5 years	Char/water/gas	300	600	Erosion: 100% erosion of trim/body unusable
RA 330	Hinge pin from solids transfer valve (LV 33C) in fluidized bed of gasifier	Hygas	N.A.	Steam/oxygen	1850-2000	1000	Pin was bent and galled due to excessive loading
RA 330 Haynes 25	High-temperature butterfly (LCV-2002)	CO_2 Acceptor	1200 hours	Char/inert gas	1450	N.A.	Fabrication: poor weld, butterfly came off shaft
Incoloy 800	High-temperature gate (XCV-2010)	CO_2 Acceptor	∿9 months	Dolomite/recycle gas	1450	150	Erosion: pipe liner was misaligned during installation leading to erosion of valve in line
Teflon Carbon Steel	6,10,12 in. ball (3 valves)	METC	100-200 hours	Coal/ash/gas/air/steam	200-700	N.A.	Wear: gouging and abrasion of seats
Carbon Steel	1 in., 800 lb. globe	Cresap	2400 hours	Carbonizer tar slurry, 3-28% solids	300	N.A.	Erosion: hole in bonnet/body and internals eroded
Carbon Steel	Plug (XV-271)	Synthane	1 year	Water/coal dust/coal char	300	600	Erosion: plug surface
Carbon Steel	Gate (in venturi scrubber bypass)	Synthane	6 months	Char/water/steam/CO_2/coal gas	N.A.	N.A.	Erosion: severe, body and gate
Kennametal K701 (WC with Co-Cr binder)	Pressure letdown (on product oil line)	Synthoil	915 hours	Product oil with 5-6% solids	257	4000	Erosion: plug tip, stem seat eroded/braze joint cracked

(Table Continued)

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A.9.3 Slurry and Solids Valves
A.9.3.2 Performance Data
A.9.3.2.1 Plant Experience

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SOLIDS VALVES IN-SERVICE PERFORMANCE[5,70,104], Continued

Trim Material	Valve Type (Plant ID)	Process	Service Life	Environment	Temp. °F	Pressure psig	Failure Mode
Coors 999 Ceramic	Coors Willis choke (LCV-405A)	Synthane	4 days	Char/water/dissolved gases	N.A.	N.A.	Erosion: holes in trim and downstream sleeve
CVD-tungsten coated on glass-reinforced TFE (seat) Plasma-sprayed METCO 19E (Cr-Ni-B) coated ball	10 in. ball full bore (1008)	METC	1230 cycles (∿18 months)	coal feed	250-300	100-160	None: little or no wear other than a lapping effect
Rubber	3 in. pinch (Series B3)	BIGAS	6 months	Coal slurry	Ambient	N.A.	Misuse, overstressing: tear in rubber sleeve from over-tightening hand wheel

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EROSION TEST RESULTS[a] OF TRIM MATERIALS FROM SLURRY LETDOWN VALVES[b] OF COAL LIQUEFACTION PILOT PLANTS [102,103]

Plant Valve Designation	Date Removed	Service Life (hours)	Material	Use Conditions	Reason for Removal	Erosion Crater[a] Depth (μm)	Length (μm)
FORT LEWIS SOLVENT REFINED COAL PLANT							
166A	8/81	878	WC-TaC-Co (<1.5% Co binder), K602[c]	8.5 MPa ΔP, 25% solids, 370-400 °C, Ireland coal	30° taper tip eroded	2.9	2.29x10³
175A	2/81	4093	WC-TaC-Co (<1.5% Co binder), K602[c]	4.1 MPa ΔP, <25% solids, <340 °C, Powhatan No. 6, & Kentucky No. 9 coal	Microform trim plug broke on startup	3.4	2.08x10³
166B	9/80	849	WC-10% Co-4% Cr, K701[c]	8.5 MPa ΔP (SRC II), 4.5 MPa ΔP (SRC I), 25% solids, 370-400 °C, Kentucky No. 9 (II), Powhatan No. 6 (I) coals	1/4-in. short tip eroded	3.9	2.21x10³
166B	3/81	218	WC-10% Co-4% Cr, K701[c]	Same as above	1/4-in. short tip eroded	2.7	2.08x10³
175B	2/81	1452	WC-10% Co-4% Cr, K701[c]	4.1 MPa ΔP, <25% solids, <340 °C, Powhatan No. 6 coal	Microform trim plug broke on startup	3.4	1.78x10³
166B	8/81	422	WC-6% Co-1% Cr, K703[c]	8.5 MPa ΔP, 25% solids, 370-400 °C, Ireland coal	30° taper tip eroded	2.3	2.41x10³
WILSONVILLE SOLVENT REFINED COAL PLANT							
LV 415 B	5/81	288	WC-6% Co-1% Cr, K703[d]	Not given	Loss of control, stem fractured near mid-length, only slight evidence of erosion[d]	—	Too deep to measure

(Table Continued)

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A.9.3 Slurry and Solids Valves
A.9.3.2 Performance Data
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EROSION TEST RESULTS[a] OF TRIM MATERIALS FROM SLURRY LETDOWN VALVES[b] OF COAL LIQUEFACTION PILOT PLANTS [102,103], Continued

Plant Valve Designation	Date Removed	Service Life (hours)	Material	Use Conditions	Reason for Removal	Erosion Crater[a] Depth (μm)	Erosion Crater[a] Length (μm)
LV 415 B	5/80	1200	WC-6% Co-1% Cr, K703[e]	Not given	Fabrication error, essentially no erosion observed	2.8	1.27×10^3
LV 415[f]	10/80	2000	Cemented WC, GEM-550[g]	Not given	Loss of control, relatively uniform erosion observed	3.8	1.28×10^3
LV 415 A	10/78	4000	Cemented WC, GEM-550[g]	Not given	Poor flow control, localized "wire drawing" erosion occurred	~5, inconclusive due to apparently rough surface finish	

[a] Tested by a jet of slurry consisting of 8% by weight of solids from the Wilsonville SRC coal liquefaction process suspended in anthracene oil. The impingement angle was 20°, the temperature 343 °C (650 °F), the particle velocity 100 m/s, and the test duration 1 hour. The surface of the test specimens was finished to 3.3×10^{-2} microns by grinding on a diamond wheel in the direction of slurry flow. The depth of the erosion crater was measured using a Talysurf Model 4 profilometer.

[b] All valves were Fisher DBAQ, 2.5-cm (1-in.) body, 9-mm (3/8-in.) orifice.

[c] Trim supplied by Alloy Carbide Co., Texas.

[d] Two regions of localized erosion on the inner surface of the seat observed.

[e] Supplied by McCartney Manufacturing Co., Kansas.

[f] Line designation unknown.

[g] Supplied by GEMOCO, Louisiana.

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A.9.3.2.2 COMPONENT TEST AND DEVELOPMENT

A project at Morgantown Energy Technology Center dealt with state-of-the-art lockhopper valve testing and development (see references [3], [4], [92]). Seat leakage test results (A.9.3.2.2.1) were obtained for 11 Lockhopper valve types, the components of which included 16 materials combinations. Valve design operating pressures varied from 200 psig to 1200 psig (13.8 bars to 82.7 bars). Operating temperatures varied from 37.8 °C (100 °F) to 454 °C (850 °F). Leak rates per number of cycles were determined in 3 different test categories for the various valve types and materials combinations: an acceptance test, a static test, and a dynamic test.

A typical range of values for leak rate per number of cycles is shown in the following table for those valves which performed satisfactorily.

LEAK RATE, m^3/min. ($ft.^3$/min.)/CYCLES

ACCEPTANCE TEST	STATIC TEST	DYNAMIC TEST
0.0008 (0.030)/0	0.0001 (0.005)/300	0.0618 (2.2)/3970
to	to	
0.1123 (4.0)/0	0.0730 (2.6)/370	

Valve types in the satisfactory category were a: (1) 6 in. Rotary Gate; (2) 6 in. Knife Gate; (3) 6 in. Y-Globe; (4) 8 in. Hybrid Ball; (5) 6 in. Hybrid Swing; and (6) 6 in. Ball. Respective component materials combinations for these were: (1) Colmonoy #5 on 440C SS with Stellite 6 on 440C SS; (2) 440A SS, R_c 50 to 52, with Stellite 6 on 304 SS; (3) 440 SS with PTFE asbestos fiber ring seals with 440 SS, R_c 55; (4) Stellite 6 on 316 SS with Stellite 6 on 316 SS; (5) Stellite 6 on carbon steel with Stellite 6 on 316 SS; and (6) Stellite 6 on 316 SS with cast Stellite 6.

Typical materials combinations for valves whose performance was not considered to be satisfactory because of galling and, in some cases, in combination with other failures such as valve stem failure, were: (1) 440 SS, R_c 58, with Stellite 6 on 316 SS; and (2) Kamog on cast Stellite 6 and Tribaloy 700.

Materials combinations of valves that failed due to external leakage problems or erratic performance (i.e., wide scatter in leakage rates) were: (1) Teflon (PTFE)-coated cast iron with cast iron integral with the body; (2) Colmonoy #5 on 440C SS with cast Stellite 6; (3) 304 SS with Thermalloy 400 on 304 SS; and (4) WC coating on 316 SS with Stellite 6 on 316 SS.

The Acceptance Test was used to determine conformance with the purchase agreement on leakage and dimensional requirements. Eleven valve types representing fifteen component material combinations were evaluated in this test. Of these tests, satisfactory performance resulted for 6 valve types.

The Static Test was designed to determine the initial leak rate, operating force or torque, response time, and the effects of dry cycling on these parameters. Valves were tested with air or inert gas at ambient temperatures

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and manufacturers' rated operating temperatures. Pressures varied according
to ANSI class. The same eleven valve types representing fifteen component
material combinations used in the Acceptance Test were also evaluated under
this test.

In the Dynamic Test the valve was required to handle solid materials at
ambient temperatures only. Only two dynamic tests were performed on two dif-
ferent valve types representing two materials combinations. Of these, only
one valve resulted in satisfactory performance. The short table above lists a
range of acceptable leak rates for each of these tests.

Design operating pressure for those valves which performed satisfactorily
ranged from 300 to 1000 psig (20.7 to 68.9 bars). Design operating tempera-
tures ranged from 149 °C (300 °F) to 454 °C (850 °F).

There were a wide range of failure types for those valves which did not
perform satisfactorily. Five valves were deleted from the test program.
Severe galling of the valve seat and ball; erratic performance, severe chip-
ping and spalling of the coating; unsatisfactory operation at design conditions
and valve stem failure were typical failure types for these valves. External
leakage and sleeve failures for three of the valves resulted in reworking the
valve but no further testing.

One of the valves locked up during dynamic tests which resulted in the
valve cracking. The valve was reworked, but there was no report of further
testing. A series of reports (see reference [92]) issued as part of the same
project provides detailed testing data for some of the valves listed in
A.9.3.2.2.1.

In Section A.9.3.2.2.2 are the results of seat-visor tests for twenty-two
materials combinations performed as part of another valve development project [6].
Nine different materials were involved. Sixteen tests were carried out in a
prototype valve test fixture and six tests were carried out in a seating
materials test fixture at 70 °F. Six tests were run at temperatures between
800 and 1050 °F--the other 16 were run at 70 °F. These 70 °F test results
have use for ranking materials for performance at 70 °F, but results of these
16 tests are of minimal value in evaluating materials performance for a typical
solids valve under conditions where temperatures range from 350-850 °F. The
number of cycles ranges from 22 to greater than 10,000.

Of the six high-temperature tests the materials which remained in ex-
cellent condition after severe testing included: Inconel 718 inlaid with
Stellite 1016 or Stellite 6, and uncoated Inconel 718. Boron carbide per-
formed well in some tests but poorly in others. The performance of boron
carbide did not seem to be related to temperature.

Of the sixteen 70 °F tests, the materials which remained in excellent
condition after severe testing included: boron carbide, Stellite 6B, silicon
nitride, and TiCN coated on Ti-6Al-4V. Twelve of the 16 tests involved a TiCN
coating on the seat (two tests) or the visor substrate (ten tests). The TiCN
coating performed well on a Ti-6Al-4V substrate, but spoiled and cracked on an
Inconel 718 or titanium substrate. Borided TY502 tended to chip in one test
out of the two conducted with it as a seat material. Among the 16 tests at 70
°F, there were three test malfunctions, including one test with significant

misalignment, which invalidated those test results.

A third valve development program dealt with design, fabrication, and testing of valves utilizing ceramics and castable refractories for high-wear and high-temperature areas (see reference [2]).

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A.9.3.2.3 MATERIALS EVALUATION

A survey of valve materials for coal conversion use containing data gen-
erated by DoE-sponsored projects and by materials manufacturers is to be found in
reference [93].

EROSION--OVERVIEW

Erosion occurs in various parts of coal conversion systems as abrasive
particles of coal, ash, char, or dolomite are driven by fluid pressures against
surface scales and metal or refractory surfaces. Some variables which affect
erosion include: type of erodent, erodent particle velocity, shape and hardness,
angle of impingement, temperature of material impacted, condition (including
hardness) of impacted material surface. Each of these variables is important in
determining the rate at which an erosion process takes place. It is important
that any testing methodology which is used to evaluate and rank material perfor-
mance takes into account all of these variables. Attention to each of these
testing variables has been considered in the testing methodology which is re-
ported in Sections B.2.1.1-.14, B.2.1.21-.22, and B.2.1.46-.51. Highlights of
this testing methodology include the following:

1. Erosion weight loss shows a mixed dependence on angle of impingement. In
    considering the effect of impingement angle on erosion, the character of the
    materials being subjected to the erosion must be considered, whether ductile
    (most metallic materials) or brittle (ceramic materials). For ductile
    (metallic) materials, weight loss shows a peak in the angular range 10-20°,
    usually followed by a systematic decrease as impingement angle approaches
    90° (see Section B.2.1.47). For brittle (ceramic) materials, the maximum
    erosion occurs at 90°. Examples of such performance are the data in Sections
    B.2.1.11 and B.2.1.14, which show erosion data typical of that for ductile
    materials, and Sections B.2.2.10 and B.2.2.13 (data at 20 °C), which show
    data typical of brittle materials. (Some variations of brittle materials
    behavior are to be seen at high temperatures because of phase changes in the
    refractories.)

2. Erosion weight loss is dependent upon the type of erodent. The rate of
    erosion tends to decrease as the hardness of the erodent decreases. Since
    char, ash, and coal particles are not as hard as silicon carbide or aluminum
    oxide, test results from the latter erodents will be conservative relative
    to a coal conversion environment. Most erosion test results reported in
    Sections B.2.1.1-.14, B.2.1.21-.22, and B.2.1.46-.51 were obtained with
    Al_2O_3 as the erodent. Sometimes SiC, SiO_2 or pumice was used as an erodent.
    Erosion due to char, ash, and coal particles is yet to be thoroughly evaluated.

3. Erosion weight loss increases with increasing erodent velocity in the range
    10 to 100 m/s. Data in B.2.1.5, B.2.1.13, B.2.2.7, B.2.2.11, B.2.2.12, and
    B.2.1.49 show the trend for both ductile and brittle materials. Some of the
    erosion data included in this book have been normalized by dividing the
    weight of sample lost by the weight of erodent used. Generally, a greater
    amount of erodent creates a greater material loss, although when very large
    amounts of erodent are involved there is not a corresponding increased
    erosion effect, probably because erodent particles are acting against each
    other. Section B.2.2.8 contains data showing an initial decrease in erosion
    at larger particle flux and then the approach of a steady state.

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4. Erosion weight loss increases with increasing particle size in the range five to 50 μm. Particle size and erosion are generally directly proportional. In Sections B.2.1.9 and B.2.1.13 there are data showing increased material loss with increased particle size.

5. Erosion weight loss depends upon the temperature of the material impacted, and may increase or decrease with increasing temperature, depending upon the material. Data for alloys in Sections B.2.1.5, B.2.1.9, B.2.1.10, B.2.1.11, and B.2.1.12 do not indicate a definite trend. Conflicting results are also indicated for refractory materials in B.2.2.8, B.2.2.11, B.2.2.12, and B.2.2.13. Although data in B.2.2.8 and B.2.2.13 show increased erosion loss with increasing temperature, the results in B.2.2.11 and B.2.2.12 are conflicting. For both alloys and refractories, the results seem more dependent on the response of individual materials to increased temperature in terms of possible changes in the properties rather than a direct effect of temperature as a parameter on the phenomenon of erosion.

6. Erosion weight loss depends upon the hardness of the material impacted and tends to decrease slightly as the material hardness increases.

EROSION--LABORATORY TESTS

EROSION TESTING was conducted on a large number of materials. The materials were subjected to erosive attack by alumina for three minutes at impingement angles of 20° and 90° at 20 °C and at an angle of 90° at 700 °C. Not all materials were tested under all three conditions so that there are gaps in the data, as will be seen readily by glancing at any of the Part B sections listed in this text. When only one test at one angle is performed, it is not possible to have a true picture of the erosion resistance of the material. The results in terms of sample loss were compared to erosion loss of samples of a cobalt-based alloy, Stellite 6B, arbitrarily chosen as a standard and tested with each set of samples. The data consist of Relative Erosion Factors (REF); values less than one indicate a more erosion resistant material than Stellite 6B, values greater than one indicate a less erosion resistant material. The reported values are the mean of five tests on a material. Although the tests permit a ranking of materials with respect to erosion resistance, it must be borne in mind that the test conditions are not comparable to those seen by components in coal gasification plants. The alumina erodent used is much harder than the coal, char, and ash particles to which gasifier internals are subjected in the plants and the tests did not include any of the gaseous chemical constituents to be found with the char and ash particles. Also, discussion of performance of materials at 20 °C is not of very significant value if the prospective material use is in gasifier vessels at high temperatures and pressures (see operating requirements). Therefore, the high-temperature test results are more important although there are data only for the 90° impingement angle, the angle at which ductile materials (alloys) are generally more erosion resistant and brittle materials (refractories) less erosion resistant. Any discussion or ranking of materials using the high-temperature data must be tempered by the fact that the 700 °C (1292 °F) test temperature is much lower than many prospective gasifier operating temperatures, and the performance at the higher operating temperatures may be rather different. Further data are required for definitive choices to be made.

EROSION TESTING OF METALS AND ALLOYS was performed on fifteen alloys, including mild steel, tool steel, several stainless steels and superalloys (B.2.1.1). The same tests were performed on twenty-three materials which included tungsten,

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molybdenum, and tantalum, seven cobalt-based alloys, two titanium alloys, high nickel-chromium alloys, and miscellaneous alloys (B.2.1.2).

The effect of high temperature on the erosion results are mixed. Nineteen materials for which there are both 20 °C and 700 °C data exhibited increased erosion resistance, while six showed less resistance and five appeared little affected by temperature. The effect of angle of impingement for the various steels and the superalloys is generally what is expected for ductile materials, a greater material loss (less erosion resistance) at lower angle. For the cobalt-based alloys, and for tungsten and molybdenum, the performance is more like that expected of brittle materials in that they are less erosion resistant at the higher angle. The following list of materials are less than, approximately, or equally erosion resistant with respect to the Stellite 6B standard at both test angles at 20 °C:

> Aluminized 304 SS
> 316 SS
> Incoloy 800 and 800H
> HK-40
> RA 330
> HC-250
> Graph-air tool steel
> Mild steel
> Ti-6Al-4V
> RA 333
> Inconel 671
> 00025 copper alloy
> SPA (proprietary alloy)

The following materials show greater erosion resistance than the standard at both angles at 20 °C:

> Tungsten, plain and with diffused boron
> Molybdenum, plain and with diffused boron
> Mo with Ti, Zr, C, and diffused boron
> Tantalum nitride
> Tungsten alloy, 90W-10(Ni, Cu, Fe)

At the higher temperature (700 °C), twenty of the samples tested exhibit erosion resistance better than the standard material and these are ranked in the following.

Relative Erosion Factor	Material
0-0.20	Wrought Tungsten, Molybdenum alloy with diffused B
0.21-0.60	Molybdenum with diffused B
0.41-0.60	Tungsten with diffused B, Ti-6Al-4V, Titanium alloy Beta III, Tungsten alloy 90W-10(Ni, Cu, Fe), 316 SS, Incoloy 800 and 800H
0.61-0.80	RA 333, Inconel 600 and 671, 304 SS, 430 SS, HK-40, RA 330

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Any judgment of the value of these numbers and the ranking of materials must be tempered by the fact that these data are for 90° impingement angle, that angle for which ductile materials are expected to be most erosion resistant.

Those materials which are very little better than, approximately equal to, or worse than the standard at 700 °C are listed below.

Relative Erosion Factor	Material
0.81-1.00	Stellite 3, Haynes 188, Haynes 25, SPA (proprietary alloy)
1.01-1.20	Stellite 6K, Haynes 93, 25 Cr iron
1.21-1.40	Wrought Molybdenum, Stellite 31 with diffused B, Stellite 6 with diffused B
1.41-1.61	Stellite 3, HC-250, HR-37

WELD OVERLAYS were erosion tested (see B.2.1.3), but data are present only for a 90° impingement angle at 20 °C. There were six cobalt-based weld alloys, four iron-based chromium alloys, one nickel-based and three composite weld alloys. All were only equal to or worse than the standard.

SEVEN MARTENSITIC STEEL-BONDED CARBIDES were tested under conditions which varied somewhat from those of all the other tests (Section B.2.1.15). The impingement angle was 90° but there were three temperatures for testing, 20 °C, 350 °C, and 550 °C. Four of the samples were TiC in steels, two were TiC in (Fe, Ni, Cr, Mo) carbide, and one was an iron tungsten carbide material. The last sample was the only one exhibiting any better erosion resistance than the standard, the REF values being 0.73 at 20 °C, 0.86 at 350 °C, and 1.01 at 550 °C. All of the others had REFs greater than one.

TWENTY-FIVE TUNGSTEN CARBIDE commercially-supplied materials of various kinds (see B.2.1.16) were tested, eight with varied amounts of cobalt binder, six with undesignated binders, two with cobalt-chromium binder, one with nickel binder, six with a varying amount of other elements, one with diffused boron and one dispersion strengthened. There are few data values for 20 °C tests at 20° impingement angle. Those values which are included when compared with 20 °C, 90° impingement angle data indicate that the erosion behavior is indeed that of brittle materials. Most of the materials are more erosion resistant than the standard to varying degrees.

The effect of the elevated temperature is either almost nil or there is a decrease in the erosion resistance. Those materials for which the effect is almost nil are a tungsten carbide with 1.5 percent Co binder, one with 5.8 percent Co, one with 6 percent Co-1 percent Cr, and one with a 7.8 percent unspecified binder. For the several materials listed in the following the temperature effect is either extreme and/or the change ranks the materials as less erosion resistant than the Stellite 6B standard.

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Material	REF (20 °C–700 °C)
with 6% Co binder	0.38–1.53
with 6% Co binder	0.19–1.32
with 8.8% Co	0.78–1.03
with 11.5% binder (?)	0.57–0.84
with unknown bond	0.31–0.72
with diffused B	0.02–0.72

One material, with 25 percent unspecified binder, has REF values at one or above for both temperatures.

Those samples which had REF values below 0.5 for both temperatures are:

Material	REF 20 °C	REF 700 °C
<1.5% Co binder	0.11	0.13
5.8% binder	0.43	0.50
6% Co binder	0.25	0.47
6% Co binder	0.23	0.36
6% Co binder	0.33	0.48
6% Co–1% Cr	0.26	0.25
6% Ni	0.32	0.46
7% Co	0.32	0.50
10% Co–4% Cr	0.25	0.47
7.8% binder	0.42	0.47

See Section B.2.2.23 for the effect of varying particle velocity and impingement angle on the material with 5.8% cobalt binder.

REFRACTORY MATERIALS, EITHER ALUMINA OR ALUMINA-BASED were tested in the same way (see B.2.2.1 and B.2.2.2). The data generally show the usual performance of brittle materials in that the 90° impingement angle data indicate a greater loss of material than the 20° angle data. Exceptions to this trend are a low-alumina SiAlON (13 percent Al_2O_3–87 percent Si_3N_4), an alumina-titanium carbide sample, and experimental compositions of alumina with varying amounts of MgO, TiB_2, and WC. These Al_2O_3–TiB_2–MgO–WC materials and the Al_2O_3–TiC material all had REF values less than 0.4 for 90° angles at both 20 °C and 700 °C. The SiAlON materials were much less erosion resistant than the standard. None of the rest of the alumina materials tested showed any outstanding erosion resistance, most of them being much poorer than the Stellite 6B standard. Only a densified alumina had REF values around 0.5 for the 90° impingement angle. Since the erodent was alumina, further testing is called for to ascertain the usefulness of the materials in actual use.

SILICON CARBIDE REFRACTORIES of varying types were erosion tested (B.2.2.3). The twenty-nine different materials from commercial sources included twenty-two different SiC preparations, including hot-pressed, pressed and sintered, and recrystallized materials. There were also two SiC materials which were silica bonded, two with silicon nitride-silica bond, and one with silicon nitride bond. One material contained ZrB_2 and another ZrB_2 and graphite. The erosion performance with regard to impingement angle was, as expected, typical of brittle materials, with less erosion for the 20° angle and greater for the 90° angle of

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attack. The effect of temperature was mixed, 15 materials having less resistance at 700 °C than at 20 °C, four samples showing little or no effect, and four samples having increased resistance.

The plain SiC materials (i.e., with no additives) in general had good erosion resistance relative to the standard but with some very wide differences. Most of the plain samples had REF values less than one at both temperatures and both angles of impingement. The performance of SiC with either SiO_2 bond or Si_3N_4 bond was variable depending on the particular specimen but since the complete characterization of the various specimens is not given the reasons for the variability are unknown. SiC with ZrB_2 had good performance but that material with graphite added had very poor erosion resistance. Those samples which had REF values less than 0.5 for 90° impingement angles at both 20 °C and 700 °C are listed below.

Material	REF	
	20 °C	700 °C
SiC, hot-pressed	0.12	0.44
SiC, recrystallized (HD 430)	0.40	0.38
SiC, 98% dense	0.05	0.02
SiC (SDIP-1-3)	0.47	0.43
SiC (SDIP-1-4)	0.44	0.43
SiC-Si_3N_4, hot pressed	0.20	0.42

Other materials showed good erosion resistance at 20 °C but no data are available for 700 °C.

FOURTEEN CARBONITRIDES were tested at 20 °C and 700 °C at 90° impingement angle (see B.2.2.4). These were mostly experimental compositions having from 85 to 97 percent (Ti, Cr) or (Ti, Mo) carbonitrides and varying percentages of Ni and Mo. None of them showed an REF lower than 0.5 and in general their performance is not significantly better than Stellite 6B. Pressed and sintered TiCN, and both pressed and sintered and cast MoTiCN were tested and showed REF values close to one.

SILICON NITRIDE REFRACTORIES (see B.2.2.5) were also tested at both temperatures and both impingement angles. Those which showed good erosion resistance at 90° angle are listed below.

Material	REF	
	20 °C	700 °C
Si_3N_4, hot-pressed	0.18	0.57
Si_3N_4, hot-pressed	0.40	0.12

Reaction-bonded Si_3N_4 had a relative erosion factor of about six for all test conditions. None of the SiAlON materials (Si_3N_4-Al_2O_3) tested had good erosion resistance, nor did a Si_3N_4-SiC-SiO_2 refractory which had factors close to one.

MISCELLANEOUS MATERIALS were tested and the full results given in B.2.2.6. Those with better erosion resistance than the standard are listed for 90° impingement angle.

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Material	REF	
	20 °C	700 °C
B_4C, hot-pressed	0.38	0.21
B_4C, hot-pressed	0	0.38
$TiC-Al_2O_3$, pressed and sintered	0.19	0.30
Cubic BN	0	0

Hot-pressed TiB_2 materials with varying amounts of Ni binder generally showed
good erosion resistance at 20 °C, 90° angle (0.036-0.60) but no 700 °C data were
reported. Materials which had poor erosion resistance compared with the standard
are TiC with Ni or Ni-Mo binder, and several $MgAl_2O_4$-based materials, all hot-
pressed with varying amounts of MgO. The TiB_2-Ni materials were prepared at Oak
Ridge National Laboratory as part of a program to synthesize and study erosion-
resistant materials (reference [105]). Data for these same materials tested with
coal slurry are discussed below under slurry erosion.

Many of the above materials which showed poor erosion resistance because
they are brittle in nature and were tested using a 90° impingement angle may
perform satisfactorily in design situations in which erodent material impinges on
the material surface at low angles.

EROSION RESISTANCE WAS MEASURED FOR ELEVEN ALLOYS and is reported in Sections
B.2.1.4-.14, B.2.1.21-.22, and B.2.1.46-.51. At 25 °C and an impingement angle
of 90°, Type 310 stainless steel erodes at a slightly lower rate than does Type
304 stainless steel (compare B.2.1.14 and B.2.1.21). At 25 °C, Type 304 erodes
about twice as fast as Type 310 for all angles of impingement. At angles above
30° and for increased temperature above 25 °C, Type 310 erodes faster than the
25 °C rate, whereas Type 304 erodes more slowly. The effect of impingement angle
between 15 and 90° on the erosion rate of six alloys appears in Section B.2.1.47;
in general, 1015 carbon steel shows the lowest erosion rate, whereas Type 310
stainless steel shows the highest erosion rate. A chrome plate on steel tended
to erode more slowly than Types 304 and 310 at 25 °C and at 500 °C (see B.2.1.5).
The chrome plate on steel tends to erode more slowly than Types 304 and 310 and
Inconel 671, for impingement angles of 10 to 90°. The erosion rate for 250 MS
tends to decrease slightly as hardness increases from HRC 37 to HRC 52. Chromium
metal showed slightly better than average resistance to erosion, whereas chromium
plate on steel consistently showed much better than average erosion resistance.
For a variety of test conditions, Inconel 671 showed better erosion resistance
than any of the other ten materials tested.

COATINGS FOR EROSION PROTECTION were investigated by testing some twenty-
five coating materials for erosion resistance (B.2.3.1). Substrates were vari-
able, in some cases unknown. Testing was also performed on the pure coating
material. The materials were subjected to erosive attack by alumina for three
minutes at impingement angles of 20° and 90° at 20 °C and at an angle of 90° at
700 °C. A few materials were tested at 30°, 45° and 90° angles. The results in
terms of sample loss were compared to erosion loss of samples of Stellite 6B
arbitrarily chosen as a standard tested with each set of samples. The data
consist of Relative Erosion Factors, values less than one indicating a more
erosion resistant material. The reported values are the mean of five tests on a
material.

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For twenty-one of the test samples the coating was readily penetrated and the indications were that the coating was too thin. Those coatings which had erosion resistance greater than Stellite 6B are given in Table A.9.3.2.3a. It is possible that some of the coatings which were readily penetrated by the alumina might survive erosion by char or ash which are much softer materials.

Nickel-base alloys for coating and cladding use have been erosion tested at 20 °C with SiC at 90° impingement angle. The coatings were applied to a low carbon steel substrate by three different methods. The data are reported in Section B.2.3.4. Further testing of ball and seat materials and of laser-processed coatings was reported in reference [49].

SLURRY EROSION--LABORATORY TESTS

SLURRY EROSION TESTING was performed on a number of materials under consideration for slurry let-down valves in coal liquefaction plants. Two slurries were used, both comparable with respect to weight percent of solids and the viscosity of the fluid medium. The slurries were a coal-anthracene oil slurry and a synthetic slurry using silica in a machine oil. The silica is harder and more erosive than the suspended insoluble particles of the coal-derived slurry. Experimental conditions were adjusted for the difference in erosivity and also the difference in the aging effects in tests using these slurries.

Differences in the two erosive slurries and the effect of various parameters are shown in Section B.2.2.29. Tests which fed a constantly fresh supply of slurry showed a linear variation of the crater depth with the duration of the test for both slurries for test times up to 18 minutes. Although the silica slurry showed a deviation from linearity for a 30-minute test it was suggested that this deviation is a statistical scatter rather than the effect of decreasing angle of attack with development of the crater profile. The silica particles erode the specimens at about 2.5 times the rate of the coal-derived slurry (Figure A, B.2.2.29).

To examine the effect of repeated use of the same batch of slurry which can cause a reduction of the solid particle size or a blunting or rounding of irregular particle shapes, experiments were run in which the same slurry was reused several times (Figure B, B.2.2.29). For both slurries the erosion rates decreased with each use of the same slurry although there is indication of a decrease in the decreasing rates for the silica slurry and a total loss of erosivity of the coal slurry after sufficient reuse (about 6 uses). In order to use slurry more economically, the effect of refreshing the used slurry by periodic additions of new slurry was tested. Slurry was recycled with substitution of 25% by weight of slurry after every three hours of use. For coal slurry, after two cycles of slurry addition the condition of the slurry stabilized, but for unknown reasons the periodic substitution did not restore erosion rates for the silica slurry, and the aging process seemed to continue (Figure C, B.2.2.29). For most of the tests in the following discussion, those with coal slurry utilized the recycling method with periodic replenishment and those with silica slurry utilized a once-through procedure.

For the series of tests of materials using coal slurry, the impingement angle of the slurry jet upon the specimen surface was 20°, for the series using silica slurry, the angle was 90°. Since most of the materials in this series of tests would be expected to respond in a brittle way to erosion conditions, the 90° angle would provide maximum erosion. The test times for the silica slurry

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Material	Relative Erosion Factor			Remarks
	20 °C		700 °C	
	20°	90°	90°	
TiB_2 electrodeposited on nickel	0	0	0	
TiB_2 electrodeposited on 310 SS	0	0	0	spalling occurred at 700 °C
TiB_2 sputtered on 410 SS	0	0	0	
TiB_2 (controlled nucleation thermochemical deposition, substrate not given)	0	0	0	some spalling on retesting at 20 °C after 700 °C
TiB_2 electrodeposited on Kovar	0	0	0	
SiC chemically vapor deposited pure coating	0.20	0.05	0	weight loss noted but no visible crater
SiC chem. vap. dep. on C converted to SiC	0.13	0.06	0	
SiC chem. vap. dep. on SiC and graphite	0	0	0	
SiC (controlled nucleation thermochemical deposition) on Ni over Cu	0.006*†	0.009*		
Si_3N_4 (controlled nucleation thermochemical deposition) on graphite	0.011*†	0.070†		
W chem. vap. dep. pure coating		0.53	0.25	
W chem. vap. dep. on mild steel		0.57	0.34	
W chem. vap. dep. on mild steel		0.48	0.16	
TiC on WC		0.31	0.72	

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*after 15 minutes testing   †for 30° angle

Table A.9.3.2.3a

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were only five or 10 minutes compared with one hour for most coal-slurry tests.
The particle velocity for coal-slurry tests was usually 100 m/s but other veloc-
ities were used, the lowest being 55 m/s, the highest 185 m/s. Velocities around
140 m/s were used for the silica slurry, various values from 130 to 145 m/s being
reported.   The coal-slurry temperature varied from 288 °C to 343 °C, the silica-
slurry temperature was usually 177 °C with one test run as high as 250 °C and two
at 163 and 165 °C.

The erosion effect was usually reported as a maximum erosion crater depth
but in a few instances the crater volume or the erosion rate (crater volume
divided by volume of slurry used) was given. In almost every one of the series
of materials tests a reference standard (a cemented tungsten carbide) was tested
to check test procedures. It is possible, therefore, to compare, at least in a
qualitative way, the results of the various series of tests by calculating a
relative erosion value for each material tested. This relative erosion figure is
equal to the ratio of the test specimen crater depth (or volume, or erosion rate)
to the reference standard crater depth (or volume, or erosion rate). A value
less than one indicates greater erosion resistance than the standard, greater
than one, less erosion resistance. In the discussion which follows, only the
relative erosion is used. It should also be kept in mind that multiple samples
of the materials were generally not run and that most of the data are for one
test on one specimen.

In Section B.2.2.28, comparison tests using both slurries are reported.

Material	Relative erosion	
	Coal slurry	Silica slurry
Cemented WC (reference standard)	1.0	1.0
Sintered α-SiC	0.11	0.80
Hot-pressed SiC	5.1	14.
Sintered B$_4$C, I	0.69	0.80
Sintered B$_4$C, II	0.51	0.92

It is clear that at least for this comparison the relative ranking of materials
with respect to the standard is qualitatively the same with some differences for
the order of magnitude of the relative values for the two slurries.

VARIOUS CEMENTED TUNGSTEN CARBIDES were studied in a series of tests using
both coal and silica slurry. Most of the WC materials contained varying amounts
of cobalt binder but other specimens with various binders were included. [The
Co-Cr binder for the reference standard was given in part of the original reports
as 10% Co-4% Cr and in other parts as 8.5% Co-4.5% Cr.] For tungsten carbides
reported with Co only as a binder, the Co content varied from <1.0% to 36.9%.
The relative erosion values for most of these materials were close to one for Co-
containing compositions in the range of <1.5% to about 9%. Examination of Sec-
tions B.2.2.30, B.2.2.35, B.2.2.37, B.2.2.40, and B.2.2.41 indicates that none of
the materials was greatly superior to the reference standard and only a few were
somewhat more erosion resistant. Materials for which the binder was a combina-
tion of cobalt and chromium were in general more erosion resistant, the relative
erosion values varying from 0.41 to 0.74. A binderless tungsten carbide as well
as several WC materials with other binders were tested, nickel, nickel-molybdenum,
nickel-copper-iron, cobalt-multicarbide, TiCN, and a proprietary binder. All of
these are much less erosion-resistant than the reference standard according to
these test results (relative erosion greater than one). The effect of impingement

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angle on cemented WC materials with varying cobalt binder content has been stud-
ied (Section B.2.2.37). The behavior of all of these materials indicates a
general decrease in erosion resistance with increasing binder content. The
response to a change in impingement angle is that of brittle materials in that
much more material loss occurred at the 90° angle that at the 20° angle. Data
reported in Sections B.2.2.35 and B.2.2.40 for varying cobalt binder content
agrees well in general with that in B.2.2.37 but few compositions show an in-
crease in erosion resistance over the reference standard.

In Sections B.2.2.39 and B.2.2.41 are reported data for tests on specimens
with different surface finishes to determine the effect of surface stress on
erosion resistance. In general, it would appear that the absence of surface
stresses tends to decrease the erosion resistance somewhat from the results of
B.2.2.41, although from the minimal data of B.2.2.39 it would appear that the
cobalt binder content influences the results.

Data in B.3.2.141 and B.3.2.144 compare hardness and fracture toughness with
Co binder content. The data show an inverse relationship between hardness and
fracture toughness, the hardness decreasing and the fracture toughness increasing
with increasing cobalt content. See also B.3.2.142 for cobalt binder and frac-
ture toughness data. The relationship between hardness and erosion loss for the
cemented tungsten carbides studied is shown in Sections B.2.2.33 and B.2.2.42.
The cobalt content of the binder in weight percent labels the data points. Both
sets of data generally indicate minimum values for erosion loss (maximum erosion
resistance) for hardness values in the range of 16-18 GPa corresponding to cobalt
content of 3-6 weight percent depending on the specific series of binder and
probably on the manufacturers' procedures. It is apparent that very low and very
high cobalt binder content materials both have low erosion resistance. The orig-
inal project reports state that the erosion craters in low binder content (high
hardness) materials indicated that the erosion was characteristic of brittle hot-
pressed ceramics, but in the high binder content (low hardness) materials the
erosion mechanism seemed to be that suggestive of ductile materials.

A NUMBER OF MISCELLANEOUS MATERIALS were subjected to coal and silica
slurry testing in the same program (see Sections B.2.1.78, B.2.2.27, B.2.2.31,
B.2.2.43 for coal slurry data and Sections B.2.2.34 and B.2.2.36 for silica
slurry data). The materials tested included a nickel-titanium alloy with boron,
several aluminas, a SiAlON, silicon carbides, aluminum nitride, silicon nitride,
several boron carbides, a soda-lime glass, a variety of chromium cermets and
chromium boride cermets, several titanium borides with nickel, and diamond/
silicon-silicon carbide developmental materials. Very few of these materials
exhibited better erosion resistance than the reference standard. In fact the
CrB cermets could only be tested under less severe test conditions than the rest
of the materials in order to obtain erosion craters which were not too deep to be
measured (see Section B.2.2.31). Those materials with a relative erosion value
less than one are B_4C, a B_4C with 8% Co, a sintered β-SiC, AlN, and one hot-
pressed alumina. Another hot-pressed alumina and a sintered alumina had relative
erosion values of 8.3 and 3.6, and hot-pressed SiC lost from 5 to 16 times more
material than did the reference standard. The original reports stated that the
hot-pressed SiC eroded by intergranular failure and the loss of whole grains.
The sintered SiC material showed smooth uniform wear over the microstructure in a
way suggestive of a ductile mechanism for the erosion.

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The developmental diamond/Si-SiC materials did not have better erosion resistance than the standard but a commercial sintered diamond showed no erosion at all. Partially stabilized zirconia was tested both with coal slurry (Section B.2.2.32) and with silica slurry (Section B.2.2.38) but in neither set of tests did this material appear to have superior erosion resistance.

A research project at another laboratory was conducted which studied methods of producing erosion-resistant materials with TiB_2 as a major phase (reference [105]). Hot-pressing techniques were tested varying temperatures, pressures, starting compositions, and particle sizes of the starting powders. The materials were studied for erosion-resistance, hardness, fracture toughness, and crack growth. These materials were dry erosion tested (see Section B.2.2.6, ORNL-supplied materials) and slurry erosion tested (see B.2.2.31 and B.2.2.43 for TiB_2-Ni materials). Although these materials had good dry erosion resistance (B.2.2.6) the results in coal slurry tests were poor with material loss generally several times that of the reference standard.

TITANIUM DIBORIDE COATINGS were studied using coal slurry and varying the type of coating deposition, coating thickness, deposition temperature and sub-strates (see Sections B.2.3.5 and B.2.3.9). Overall the TiB_2 coatings were not superior in erosion resistance to the reference standard. Coatings which were chemically vapor deposited (CVD) on a TiB_2 or TiB_2-Ni substrate and on a cemented tungsten carbide (WC-Co-TaC-TiC) tended to have good erosion resistance (relative erosion value less than one).

OTHER COATINGS were also tested with coal slurry (Section B.2.3.6) but very few exhibited an erosion resistance superior to the reference standard. Those which exhibited good erosion resistance, the CVD SiC coatings on graphite, and some TiC and WC-Co coatings are not well-enough identified to permit drawing of firm conclusions. Some laser-processed coatings were prepared by fusing powder mixtures to substrates with high-power laser radiation (see Sections B.2.3.7 and B.2.3.8). Since the test times were short for coal slurry use and the crater depths reported are generally large for coal slurry at a 20° impingement angle, it would appear that neither the Stellite series of coatings nor the tungsten carbide coatings of these two series are effective performers with respect to slurry erosion. From the data in B.2.3.8 it would appear that low carbide volume fractions do not enhance the erosion resistance. The data in both B.2.3.7 and B.2.3.8 have no reference standard values included for comparison.

From the results of the slurry erosion tests it would appear that further testing is necessary to determine the behavior of all these materials more exactly. The very few duplicate specimens which were run (see Sections B.2.2.35 and B.2.2.37) would indicate that the scatter in the data may not be too large for qualitative purposes, but further testing as well as complete characteriza-tion (composition, preparation, etc.) is needed to determine the reasons for differences in performance between seemingly similar materials.

ABRASION--LABORATORY TESTS

ABRASION TESTING was performed by thrust washer wear testing for a large number of materials (see B.2.1.17, B.2.1.18, B.2.1.19, and B.2.1.20). Material loss was measured after rotating two washer samples against each other under constant load and rotation rate for four minutes. In some tests the two washers were of the same material, in others the two washers were different materials. An abrasive, either alumina, fly ash, or coal was introduced between the washers

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for some of the tests but most were run without abrasive. The measured material
loss for two mild steel washer samples tested with alumina was used as the base
line standard and the testing results are reported as a factor showing the
improvement over the abrasion resistance of mild steel with alumina abrasive.

ABRASION TESTING OF A MATERIAL AGAINST ITSELF IN THRUST WASHER WEAR is re-
ported in B.2.1.17 where the washer pairs were of the same materials. The per-
formance of mild steel actually degraded to the same extent with no alumina
abrasive and with abrasive, with heat and steam. Diffused boron, tungsten
carbide and Tribaloy 800 coatings on mild steel improved performance by factors
of 2 to 3.5 but this is a minimal effect. Among materials tested without alumina
abrasive the following are materials which showed improvement factors over mild
steel of at least ten.

Material	Improvement Factor (approximate)
LW-IN40 coating material	900
LC-4 coating material	150
316 SS with diffused B	100
440C SS with diffused B	70
Graph-air tool steel	45
HC-250	40
24% Cr white iron	20

With abrasive, only two materials had improvement factors greater than ten.

Material	Abrasive	Improvement Factor
Graph-air tool steel	-14 mesh coal	150
Graph-air tool steel	-14 mesh ash	50
Graph-air tool steel	alumina	25
24% Cr white iron	-14 mesh coal	65
24% Cr white iron	-14 mesh ash	35
24% Cr white iron	alumina	20

Materials which showed less than ten times the abrasion resistance of mild steel
or equal to mild steel with or without abrasive used are 316 SS, AISI 4140
steel, Stellite 6 with and without diffused boron and SPA (a proprietary alloy).

ABRASION TESTING OF PAIRS OF DIFFERENT MATERIALS was performed in the same
way with the material loss measured being the total loss for both materials.
Several miscellaneous materials and a large number of cobalt- and iron-based and
composite weld overlay alloys were tested against both 316 SS (see B.2.1.18) and
440C SS (see B.2.1.19) with no abrasive added. A few tests were performed with
abrasive material present. The weld alloys were overlaid by two different
methods, tungsten-inert gas overlay (Tig) and arc-welded overlay (Arc).

In tests of the weld overlays with 316 SS (B.2.1.18) only one overlay (40%
Haynes 47-60% Stellite 954) with 316 SS showed an improvement factor over mild
steel of ten or more (∿12) with no abrasive. All other combinations were less
than ten and the overlay method did not appear to make a great difference in
those cases where both overlay methods were used for the same alloy. In tests of
the overlays against 440C SS (B.2.1.19) a few values indicated that the weld
overlay method may make a difference. The improvement over mild steel for the
weld alloys paired with 440C SS were quite large in a number of cases.

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Materials Against 440C SS	Improvement Factor (approximate)
Comp. 4E (Haynes 47–Stellite 954)	32
Comp. 40E (Ni-based)	32
No. 94 (Fe-based) Tig	29
No. 94 (Fe-based) Arc	25
Comp. 2 (WC–Stellite 6) Tig	24
Comp. 2 (WC–Stellite 6) Arc	19
No. 90 (Fe-based) Tig	21
No. 1 (Co-based) Arc	13
1016 (Co-based) Arc	12

No. 90 Arc-welded, No. 21 Tig overlay, No. 1 Tig overlay, and 1016 Tig with 440C SS all had improvement factors of about seven.

Two chromium oxide coating materials were tested against 316 SS with no abrasive. One combination (LW-IN40/316 SS) had an improvement factor over mild steel of 37 whereas another combination (LC-4/316 SS) had a factor of only 1.5. Graph-air tool steel tested with 316 SS had an improvement factor over 20 when tested with no abrasive but only a factor of ten when alumina and steam were introduced and the system was subjected to externally applied heat (250 °C).

Several combinations of materials were tested without abrasive, with abrasive, and with abrasive and steam in the system, and externally applied heat. In the case of a soft material such as polytetrafluoroethylene paired with 316 SS the absence or presence of abrasive seems to make little difference in the performance of the pair but in the case of tool steel (with 316 SS), Stellite 6 (with 316 SS or 440C SS), and 24 percent chromium iron (with 316 SS) the results were mixed. For the tool steel (with 316 SS) the presence of alumina cut the improvement factor back to nine from the value of over 20 without abrasive. For the 24 percent chromium iron there is a trend indicating that the presence of abrasive (and also heat and steam) may serve to lubricate the abrading surfaces slightly since less material was lost and the improvement factor showed some increase. The same trend appears for Stellite 6 with 440C SS with fly ash abrasive. For Stellite 6 with 316 SS the fly ash appears to lower the material loss but alumina, heat, and steam have an adverse effect. It is clear that further testing is required to find answers to the questions raised by the data.

Miscellaneous pairs of materials were tested, most of which showed large improvement over mild steel factors when tested with no abrasive present.

Material Pairs	Improvement over mild steel
LW-IN40/LC-4 (chromium oxide coatings)	104
Tool steel/LW-IN40	100
316 SS borided/Stellite 6 borided	44
Tool steel/LC-4	31
440C SS borided/Stellite 6	26
316 SS borided/Stellite 6	14
440C SS borided/Stellite 6 borided	9

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     The preceding abrasion data are of limited use and further testing is re-
quired to help choose materials which will perform well in valves where two
surfaces must meet, will probably trap solid particles between them, and will
also be subjected to temperatures and gaseous atmospheres which may affect the
abrasion resistance.

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* Sections included in SP-642, Supplement 1, and Supplement 2 combined.

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A.10.1 OPERATING REQUIREMENTS

The primary objective of modern direct coal combustion is to use high-sulfur and high-ash coals efficiently and economically in an environmentally acceptable way. Many research efforts have been concentrated on atmospheric and pressurized fluidized bed combustion (AFBC and PFBC) with major emphasis on the atmospheric type.

Direct combustion systems can be considered as consisting of three major areas: 1) solids handling system, 2) combustor (including containment shells, heat transfer tubes, baffles and air distributor plates), and 3) peripheral items (including cyclones, carbon burn-up cell, and various heat exchangers. Most potential problem areas with respect to alloys have already been discussed in other sections of this book.

Major differences from, for instance, gasification processes, will occur in the composition of the gaseous environment, e.g., flue gases would be higher in CO and CO_2 and lower in H_2 and H_2O than in a gasification process. Also, erosion/corrosion reaction of in-bed tubes in a fluidized bed combustor would have no direct parallel in a gasification process. Temperatures in a fluidized bed combustor are limited to 1600-1700 °F (1144-1200 K) and pressures range from atmospheric in AFBCs to 10 atmospheres in PFBCs. Temperatures may go to 2000 °F (1366 K) in carbon burn-up cells, but these units normally do not have in-bed tubes.

In summary, materials used in coal fluidized bed combustors will be subjected to most of the potential thermal, mechanical and chemical stresses outlined for metal internal gasification needs (Section A.2.4.1) albeit at somewhat lower temperature and pressure limits. In-bed erosion/corrosion problems provide an additional potential complication.

Combustors burning coal-oil slurries and other fossil fuel combinations in the higher temperature range of 1800-2500 °F (1250-~1700 K) would provide both economy and greater efficiency in generating power and driving engines. These temperatures are, however, well above the useful operating temperatures of metallic materials. Structural ceramics can generally withstand such temperatures but must possess other properties also to be useful in heat exchangers or recuperators. Ceramics must 1) resist the corrosive environment of the combustion gases and slag and possible gas-particulate erosion, 2) contain high-pressure gases while supporting pressure differentials between inside and outside tube pressures, 3) have good heat transfer properties as a function of time and temperature, 4) resist large thermal stresses.

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A.10.2.2 MATERIALS EVALUATION

ALLOYS

Corrosion of structural metals for heat exchanger tubes was determined following exposures in flue gases from coal-fired steam generators and in atmospheric fluidized-bed combustion facilities. Corrosion and erosion-corrosion rates were measured and are reported in Sections B.1.1.8-.11, .13, .84, .85, .87, .88, .188-.191, and B.2.1.35. Results of metallographic examination and scale analysis appear in Sections B.1.1.7, .14, .15, .86, .89, .90, .91, .97, and B.2.1.36-.39. Some hardness measurements were reported on test specimens after exposures in flue gases (B.3.1.28-.30) and in a fluidized bed coal combustor (Section B.3.1.51). For the same alloys and similar exposure times, corrosion rates were considerably lower than rates reported for exposures to a standard coal gasification atmosphere in Section A.2.4.2.2.1. Similar corrosion products formed, regardless of the exposure medium.

FLUE GAS EXPOSURE of seven alloys in a coal fired steam generator resulted in the corrosion losses reported in Sections B.1.1.11 and .13. The graphs in Section B.1.1.11 show that the type of coal feedstock influences the weight loss. For example, all seven alloys showed rather substantial losses in Lignite A coal, but not in HV bituminous A coal. Inconel 617 showed the least loss in all four of the coals used in the tests. On the other hand, Alloy 12R72 tended to show rather high losses in all four coals. Some welds involving combinations of Type 316 stainless steel, Incoloy 800, and Haynes 188 joined with Inconel 82 and Inconel 617 filler metal were studied after exposure to flue gas (Section B.1.1.7). Exposures were at 800-1280 °F for 3552 to 8081 hours. Although the welds showed lack of penetration, porosity and cold shuts, very little corrosive attack occurred during exposure. Hardness tended to show a slight decrease with exposure (Section B.3.1.30). Microstructure changes of six alloys and two coated alloys exposed to flue gases from four coal feed stocks are reported in Section B.1.1.15. Exposure times and temperatures ranged from 300-7368 hours and 700-1710 °F, respectively. Observations generally included carbide precipitation (inter- and intra-granular), oxidation, and chromium depletion.

CORROSION RATES AND EROSION/CORROSION DATA IN AN ATMOSPHERIC FLUIDIZED BED COAL COMBUSTOR are reported for fourteen alloys in Sections B.1.1.8-.10, .84, .85, .87, .88, .188-.191 and B.2.1.35, .36, .38 and .39. Graphs of metal loss (wastage) vs. temperature for ten alloys (Sections B.1.1.85 and B.1.1.87) show that the metal loss generally increases with increasing temperature for exposures of 1500 hours. The time dependence of the average corrosion rate for exposures of up to 1500 hours at temperatures between 1200 and 1500 °C are tabulated in Section B.2.1.35 and graphed in Sections B.1.1.84 and B.1.1.88. Average rates for Types 304 and 310 SS, and for P9, IN 671 and FSX 414 were comparable, and did not exceed 0.08 inches per year for long exposure times. Rates for exposures in the beds were higher than for exposures in the freeboard position. The influence of salt additions on penetration and scale formation for fourteen alloys is reported in Sections B.1.1.188-.191. Exposures were for 100 hours at temperatures near 1571 °F. Salt additions generally increased the depth of penetration and scale thickness (Sections B.1.1.188 and .190). Air cooling the test specimens tended to decrease the depth of penetration (compare results in Sections B.1.1.189 and .191). Tubular specimens of six alloys exposed during 144 hour exposures at 400 to 890 °F showed notable weight losses (Section B.2.1.36). Test specimens of

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four alloys showed significant metal loss during a 1080 hour exposure at 1620 °F.
Losses were much higher in the lower bed than in the upper bed (Section B.2.1.38).
A comparison of scale thickness formed on three alloys exposed in air and in a
fluidized bed coal combustor at temperatures between 610 and 1068 °F for 144
hours showed that much higher scale thicknesses resulted from exposure in the
fluidized bed than in air (Section B.2.1.39).

Fireside corrosion measurements (Section B.1.1.8) showed comparable maximum
oxide scale thicknesses on Types 304, 310 and 316 stainless steels and Incoloy
800H. The maximum penetration of intergranular corrosion was also comparable.
A comparison of fireside and airside corrosion generally showed higher oxide
scale thicknesses for airside exposures (Section B.1.1.9). Type 304 stainless
steel generally showed better oxidation resistance and resistance to inter-
granular penetration than did 2 1/4 Cr-1 Mo steel (Section B.3.1.10).

SCALE FORMATION AND MICROSTRUCTURE CHANGES following exposure in an at-
mospheric fluidized bed coal combustor are reported in Sections B.1.1.14, .86,
.89, .90 and B.2.1.37. Surface appearance and phase identification after air
exposures at elevated temperatures are reported in Sections B.1.1.91 and .97 to
facilitate comparisons with scale formation after exposure in a fluidized bed.
The principal phases detected on Alloy P9, E-Brite (26-1) and Type 316 stainless
steel after air exposures for 144 hours at 1600 °F were Fe_2O_3, $(Cr,Fe)_2O_3$ and Cr_2O_3
(Section B.1.1.91). Fe_2O_3 was also detected after exposure in a fluidized bed
(Section B.2.1.37). Corrosion products formed after air exposures at tem-
peratures between 215 and 925 °F for up to 144 hours included gold and yellow
powder, red-rust scale and heavy black scale, depending upon the alloy (Section
B.1.1.97). Intensity profiles for Fe, Cr, Ni and Ca in the scale formed on Type
316 stainless steel, E-Brite, and P9 exposed for 144 hours at 685-910 °F general-
ly show lower intensities in the scales than in the base metal, which indicates
depletion (Section B.1.1.86). Phases identified in the scales included Fe_2O_3,
Fe_3O_4, $CaSO_4$, $K_3Fe(SO_4)_3$, and $Ca_3Fe_2Si_3O_{12}$ (Section B.2.1.37). Microstructural
observations made on 12 alloys and two coated alloys exposed in various locations
at temperatures between 1440 and 1620 °F for up to 1500 hours are reported in
Sections B.1.1.14, .89 and .90. Results show indications of carbon pick-up,
intergranular oxide penetration, and carbide precipitation. Insufficient in-
formation is presented for making a meaningful assessment of the role of coat-
ings in the corrosion process.

HARDNESS MEASUREMENTS were made on seven alloys exposed to flue gases from
four coals (Section B.3.1.28-.30) and in a fluidized bed coal combustor (Section
B.3.1.51). Exposure conditions ranged between temperatures of 1100 and 1700 °F
for up to 1500 hours. Hardness tended to show a slight decrease in some cases
(Sections B.3.1.28, .30, and .51), no trend in other cases (Section B.3.1.29),
and an increase in some cases (Section B.3.1.51).

CERAMICS

The use of ceramics for heat exchangers in combustion environments has been
investigated in several projects. Although screening tests included alumina,
zirconia, and several aluminosilicates, most of the testing has been concerned
with various silicon carbides and silicon nitrides. Limited data are available
for chemical and phase changes occurring under various high temperature atmospheres,
Sections B.1.2.37-.44, and B.4.2.48. Strength data are given in Sections B.3.2.45,
.46, .53, and B.3.2.150-.156. Fracture toughness and crack growth data are given in

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B.3.2.157-.166, .168, and .169. Gas permeability data are given in B.4.2.45 and
thermal expansion data in B.4.2.46-.47.

The fabrication of composite ceramics with ceramic-fiber reinforcement,
processing procedures to produce optimum properties of SiC including powder
synthesis with various additives, and ceramic fabrication with controlled de-
velopment of the microstructure are being studied and are reported in references
[106] and [108]. Another project dealing with short fiber reinforced structural
ceramics is reported in reference [109]. Reference [107] includes thermochemical
studies of various Si_3N_4 preparations and SiAlON compositions. Another project
(reference [15]) has dealt with materials studies, engineering analyses, and
computer modeling for the design, construction, and testing of a ceramic tube
heat exchanger. One feature of this project was the design of a relaxing joint
to relieve thermal stresses for ceramic-ceramic and metal-ceramic seals using
glass adhesives.

CHEMICAL AND PHASE CHANGES in a simulated heat exchanger environment were
investigated. Ceramic tubes were subjected to the flue gases and hot partic-
ulates in the gas stream of a combustor. In the first test a No. 6 fuel oil with
a high vanadium content was burned. The second test burned a No. 6 oil with 10
weight percent of a powdered coal which had an acidic slag (base/acid = 0.29).
The third combustion used a No. 6 fuel oil with 20 weight percent of coal with a
basic slag (base/acid = 1.14). The fourth combustion fuel was a 20% coal-80% oil
mixture in which the coal was acidic (base/acid = 0.09) and the ash was very
refractory.

During the first combustion, with high-vanadium fuel oil, five different
oxide-based ceramics and nine different silicon carbide ceramics were exposed.
Visual and x-ray diffraction examination results are given in Section B.1.2.37.
Nodules formed on SiC tubes on the upstream side and generally contained, besides
original component phases of the tube, silica and iron aluminate. The investi-
gators [106] stated that elemental species present were consistent with impurities
in the fuel oil. Metallographic and electron probe examination showed that an
Fe-Ni-S alloy formed in siliconized SiC tube walls. Fuel oil with both iron and
nickel impurities would be more destructive to siliconized SiC than oil with only
an iron impurity. Oxide-based tubes all had dark deposits, probably primarily
iron aluminate. Mullite, cordierite, and zirconia-mullite tubes all cracked
extensively.

In Section B.1.2.38 is given a typical differential thermal analysis figure
for nodules found on SiC tubes after exposure. All nodule materials showed a
glass transition point. The temperatures of these transition points are all
below the approximately 1200 °C temperature of combustion so that materials in
tubes and nodules were above their glass transition points during combustion.

Silicon-based ceramics exposed to the flue gases and acidic slag of Combus-
tion Test Exposure 2 were examined for chemical changes. Besides SiO_2, which was
expected to form on the surface, other oxides were found (see Section B.1.2.39).
The densification aids were found to affect both the identity and the quantity of
the phases. Within both the ceramic and oxide layer the Si_3N_4 containing Y_2O_3 as
a densification aid was found to undergo complex reactions. The material also
cracked extensively and was penetrated by slag during various exposures. These
exposure effects of exposure are associated with the formation of α-cristabolite
and complex yttria-silica phases listed in Section B.1.2.39. These compounds
form with net volume expansions. Formation of the SiO_2 is responsible for strength

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losses exhibited by these materials in combustion exposures and oxidation tests
(see Sections B.3.2.150 through B.3.2.156) See also B.1.2.43 for the formation
of SiO_2 on SiC in pure O_2 over time at various temperatures. Three materials
(two SiC and one Si_3N_4) were examined by x-ray diffraction after oxidation test-
ing cycles to check the phases forming in the oxide surface layer (Section
B.1.2.40). There was continuous oxide formation although in diminishing amounts
with continued oxidation. Densification aids and impurities are depleted as the
oxidation continues, according to the investigators [106]. These materials were
also strength tested (Section B.3.2.154) showing greater strength retention when
the oxide layers are removed between oxidation cycles. Other investigators [107]
studying the effects of oxidation on silicon-based ceramics stated that sintered
α-SiC showed negligible oxidation up to 1500 °C and good resistance to slow crack
growth. For SiAlONs of the series $Si_{6-z}Al_zO_zN_{8-z}$, the higher the value of z, the
lower the susceptibility to crack growth and to oxidation.

The corrosive nature of the basic slag in Combustion Test Exposure 3 is
shown by the thinning of tube walls (Section B.4.2.48). Tube dimensions did not
change in the other combustion tests. The larger attack occurred on the up-
stream side. Removal of material was also dependent on the position along the
tube; at the cooler end (air inlet) less material was removed. The SiAlONs were
most severely attacked. Limited data are given in Section B.1.2.44 on the effect
of a gas turbine environment on three SiC materials. A combustor burned diesel
fuel with V_2O_5 and with fly ash and sea salt contaminants to simulate the gas
turbine environment.

TENSILE AND FRACTURE STRENGTH TESTING was performed on the materials exposed
in the four combustion environments given above. Not all test materials were
exposed in all four combustion tests.

The tensile strength of exposed tube materials was compared with strength
before exposure (Section B.3.2.150). Tubes were sectioned to provide half-ring
(C-ring) specimens for compression testing. Silicon nitride-bonded silicon
carbide, siliconized silicon carbides, and clay-bonded silicon carbide showed
increases in strength after testing in the high-vanadium fuel oil and the two
acidic-ash coal-oil fuels. The extreme increase in strength of the Si_3N_4-
bonded SiC in Test 4 was attributed by the investigators [106] to the glazelike
formation of the very acidic slag on the tube. The SiC porosity was considered
responsible for the low as-received strength. The slag glaze on the tube would
have greatly reduced the porosity.

Tubes of four silicate materials tested in the high-vanadium fuel oil
cracked extensively. The SiAlON material subjected to the acidic and the basic
ash coal-oil fuels also cracked extensively. Pure alumina does not seem a
promising material for heat exchangers. The basic-ash coal-oil fuel had a det-
rimental effect on all materials exposed to it. The basic slag was used in
exposure of sintered silicon carbide at high-temperature with and without applied
stress and the strength was tested at high temperature (see Section B.3.2.151).
The data show a strength increase occurring in both air and slag environment
after 24 hours at 1250 °C and a very slight decrease of that higher strength
after 168 hours exposure. The effect of applied stress during exposure to air
and slag at 1175 °C is apparently not significant. It was reported that the
value for the slag exposure with no applied stress is probably in error and is
too high. Note that the SiC tested in B.3.2.151 is the one showing the least
strength loss (about 3%) due to basic slag in combustion testing (B.3.2.150).

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Flexure bars were also exposed in the combustor during Test Exposures 2, 3, and 4 above. The flexure bars were stressed to fracture in four-point bending. The percent strength retained after exposure is reported in Section B.3.2.152. The coal-oil mixture with the basic slag (Exposure 3) was found to be highly corrosive and most detrimental to the specimens in agreement with the results of the tube tests. Of the materials exposed (the same set as in Exposure 2) strengths are reported for only two SiC materials. The rest of the specimen bars thinned to such an extent that they fell from the supports (Section B.4.2.48). In Exposure 2, bars strength tested as-exposed with adhering slag were found to have lost appreciable strength, but the interior material tested after grinding to remove affected surfaces appeared unchanged. Si_3N_4 and SiC bar specimens were also coated with a slurry of the basic coal slag and then exposed to flowing oxygen at 1200 °C. The data (Section B.3.2.156) show that the exposed specimens did not, in general, retain strength to resist fracture (sintered α-SiC bar specimens were an exception). The rest of the materials experienced slag and corrosion penetration and the Si_3N_4 materials also cracked. Data from B.3.2.152 and B.3.2.156 are shown graphically in B.3.2.153.

The effect of oxidation of SiC and Si_3N_4 ceramics and of the oxide additives and impurities on the fracture strength is shown in Sections B.3.2.154 and B.3.2.155. Air oxidation was performed on the specimens in B.3.2.154. Removal of oxide impurities provides for strength retention, shown by results of tests in which the oxidized surface was periodically removed. After long-term (2000 and 5000 hour) oxidation in flowing oxygen (B.3.2.155) the strengths of siliconized SiC and sintered α-SiC were enhanced but the strength of the hot-pressed SiC containing alumina was seriously degraded.

GAS PERMEABILITY of tubes exposed in the four combustion tests was tested. Heat exchanger tubes must transfer only heat from combustor flue gases to the air or other gas passing through the tubes and not permit transfer of gases through tube walls. Section B.4.2.45 contains the results of permeability testing of as-received and exposed tubes. Most of the data are for helium permeability since the applicability of these ceramics to closed-cycle systems is reduced if helium leakage is high. The data indicate a substantial increase in permeability after exposure when tested at high pressure differentials. Differences in permeabilities from exposure to exposure would reflect the effect of slag attack and other changes in ceramic microstructure. Some leak rates too high to be measured were attributed to possible flaws in sealing the tubes in the test fixture and to flaws in tube materials. Clay-bonded and silicon nitride-bonded silicon carbides showed a decrease in air permeability after exposure, but these tests were all at atmospheric pressure.

THERMAL EXPANSION measurements before and after exposure to Combustion Test Exposure 1 are given in Section B.4.2.46. The change in expansion coefficient of the siliconized SiC materials, the two clay-bonded silicon carbides, and the two magnesium aluminosilicates is not uniform. The chemically-vapor-deposited SiC showed no effect of exposure but the same material deposited on reaction-sintered SiC had a large increase in thermal expansion after exposure. If exposure to flue gases provides for large changes in thermal expansion with respect to as-received materials, such tubes would, upon cycled use, experience stresses causing cracking, breaking of seals, etc. rendering the tubes useless. Materials which showed little or no change in thermal expansion were two of the siliconized silicon carbides, the chemically vapor deposited SiC, one clay-bonded SiC, the Si_3N_4-bonded SiC, and the high-purity alumina.

There are thermal expansion data (Section B.4.2.47) for tubes exposed in only one of the coal-oil mixtures, in Test Exposure 2 (acidic slag, base to acid ratio = 0.29). The data for exposed alumina tubing show an almost unchanged linear expansion for the downstream side but increased linear expansion for the upstream side. The SiAlON data exhibited considerable hysteresis for the as-received material. The extensive cracking exhibited by this material upon combustion exposure is attributed to this considerable difference in expansion and contraction of the SiAlON.

THE EFFECT OF TIME AT HIGH TEMPERATURE (1400 °C) in a nitrogen plus 1000 ppm sulfur dioxide atmosphere on the phases present in several SiC materials is shown in Section B.1.2.42. The formation of SiO_2, Si_3N_4, and Si_2N_2O were followed at various intervals. The investigators [107] also studied the dependence of nitridation of SiC ceramics on the presence of SO_2. The reactions are individual for each material; some reactions take place within a fairly short time period (4 or 8 hours) and the products (SiO_2, Si_2N_2O) then tend to decrease as Si_3N_4 is formed.

FRACTURE PROPERTIES have been studied and critical stress intensity values for some silicon nitrides are given in Section B.3.2.158. Although none show a strong temperature dependence there is a distinct variation in the fracture toughness of the various Si_3N_4 ceramics. The hot-pressed Y_2O_3-doped Si_3N_4 shows a much higher toughness than the other materials and a strong dependence on the strain rate.

Fracture toughness data in B.3.2.159 for Si_3N_4 doped with yttrium aluminum garnet (YAG) indicate very high critical stress intensity factor values for some materials. These high values indicate the occurrence of rapid creep deformation which masks the true toughness values. See Section B.1.2.41 for the effect of high temperature exposure on the phase composition of these materials. New compounds formed on the surface at the test temperature (1300 °C) but there was little apparent correlation of the oxidation products with the preparation and processing methods. Examination of the toughness data does not reveal any obvious trends dependent on fabrication or phase content.

Some fracture data for several SiC materials and hot-pressed Si_3N_4 are given in Section B.3.2.163. The effect of a simulated combustion gas on the fracture toughness of two SiC materials is shown in Section B.3.2.165. There is little effect on sintered material but some possible effect on the siliconized SiC. The data for reaction-bonded SiC in B.3.2.166 and limited data for a siliconized SiC in B.3.2.169 show no significant effect of the combustion gas.

In fracture studies of Si_3N_4 and a SiAlON it was found that slow crack growth preceded fracture. Rapid crack growth then started from a crack formed naturally in the slow growth phase. Critical stress intensity factors determined for rapid crack growth in four-point bending are given in Section B.3.2.160 for Si_3N_4 and a SiAlON. The data show dependence on temperature and displacement rate for Si_3N_4 but no such dependence for the SiAlON. The dependence of the Si_3N_4 data on displacement rate is attributed to creep at the crack tip. The SiAlON is creep resistant (see Section B.3.2.161). See Section B.3.2.162 for creep crack growth data for a SiAlON. Data for siliconized SiC in Section B.3.2.164 shows that the critical stress intensity factor tends to increase with increasing temperature and decrease with increasing displacement rate. These trends indicate some creep deformation at the crack tip.

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CRACK GROWTH maps were constructed from stress intensity factors versus crack velocity and stress intensity factors versus temperature (Section B.3.2.157). The maps show regions of varying cracking mechanisms. The stress intensity factor for Si_3N_4 changes rapidly in the 1200-1400 °C temperature range. More data are needed above 1400 °C for heat exchanger application. In B.3.2.168 data for elevated temperature crack growth in sintered α-SiC measured in double torsion show a deceleration in crack growth when a crack is unloaded after formation and reloaded a second time without any sharpening of the crack tip.

In flexure strength tests sintered α-SiC exhibits a temperature- and stress-dependent static fatigue limit at short exposure times. This limit is shown in Section B.3.2.167; below the limit specimens show delayed failure and above it strength generally increases with longer exposure time. The data show a strengthening regime independent of temperature and applied stress, possibly attributable to blunting or healing of the crack. At still longer times there is a rapid strength retrogression of the strengthening region.

COMPRESSIVE STRENGTH data for silicon carbide and silicon nitride are given in Sections B.3.2.45 and B.3.2.46. Extruded tubes were radially loaded, so as to prevent point loading, at ambient temperatures and at 1500, 1700, 1900, 2300, and 2500 °F. The effect of temperature on the flexural strength of several silicon-containing refractories is shown in B.3.2.53. All the materials tested but one showed a decrease in flexural strength at about 1371 °C (2500 °F). The one material which did not decrease in flexural strength up to 1700 °C is a sintered α-SiC. The variation of both compressive and flexural strength with temperature appears fairly constant within experimental error, falling off at temperatures at the high end of the expected use temperature range (2500 °F). Flexural strength as a function of probability of failure for SiC and Si_3N_4 are given in B.3.2.170.

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INTRODUCTION

The purpose of Part B is twofold. The first is to present the data on which
the "Materials Evaluation" portions of Part A of this book are based. The second
is to make available, in one place, the information generated by the wide variety
of projects which are sponsored by the Department of Energy in the field of
materials for fossil energy applications. The contents of this section, there-
fore, are data summaries abstracted from the reports of materials research con-
tractors to the Department of Energy. Some tables and graphs have been generated
by the compilers using the data given in the reports, others are reproduced from
the reports with little or no modification. The units appearing in the tables or
graphs are those used by the authors of the various reports, the compilers not
having converted all data to a common system of units. Although this practice
results in a wide variation in the reporting of the data, and requires the user
to exercise great care in comparing data from section to section, conversion to a
common system of units for all the data in Part B would have been a very costly
effort.

The original sources of the data are identified by the number in square
brackets following the title of each table or other data summary. References to
the source documents may be found by looking for that number in Part C, Refer-
ences. In order to condense the information and to bring related data together,
data from more than one individual report may appear in a given summary. The
same data may appear in more than one of the source reports. The references,
therefore, are to the series of reports for a given project and, in some cases,
to related publications by the same authors. It was considered unnecessary, and
possibly confusing, to attempt to identify the specific report(s) of a series
from which a given data value was taken. Those readers who wish to check the
original reports would do well to examine the entire series in any event. For
alloy data, the project reports are covered through the reporting periods ending
December 31, 1982. For refractories data the project reports are covered through
the reporting periods ending December 31, 1983. A number of the same materials
research projects are continuing and new projects have begun sponsored by the
Department of Energy since the cut-off dates for this compilation. The reports
of both continuing and new projects are being published in the Oak Ridge National
Laboratory Advanced Research and Technology Development Fossil Energy Materials
Program Quarterly Progress Reports available from the National Technical Infor-
mation Service.

The great majority of the programs generating the data presented in Part B
have one or more of the following purposes:

1. To expose materials to one or more conditions typical of a coal conversion
    process; to examine the performance of the materials; and to test the effect
    of exposure on the various properties. Materials have been exposed in
    laboratory vessels under simulated coal conversion and combustion condi-
    tions and also in various locations in coal conversion pilot plants.

2. To develop materials with specific resistance to the effects of coal con-
    version conditions.

3. To provide understanding of the basic phenomena affecting materials under
    the abusive conditions of coal conversion in order to provide criteria for
    development of materials and for design use of existing materials.

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Most test exposures involved the use of small test specimens or coupons rather than very large samples or actual component pieces. In presenting the data we have attempted to include experimental details such as the test methods, sample size, and number of samples to help the reader judge the value of the data for his application. The source documents are not all equally explicit about such details and the information is, therefore, often missing from the footnotes to tables and graphs in Part B. Conditions for the exposures are given much the same as they are in the source documents, with simplification and abbreviation. Laboratory conditions could be specified by the original authors but in the case of pilot plant exposures, the complete conditions with all fluctuations for the full time the samples were in pilot plant test locations were not available to the authors, and the conditions stated are therefore incomplete. Much of the laboratory testing for which the data are discussed in Part B was performed utilizing a "typical" or "simulated" coal gasification atmosphere. The composition was given as 18 percent CO, 12 percent CO_2, 24 percent H_2, 5 percent CH_4, 1 percent NH_3, with varying amounts of H_2S (0.1 to 1.5 percent), and the balance H_2O. In many reports, it is clearly indicated that the above was an input composition and equilibrium compositions at temperature and pressure were often given. Some reports indicated that the above composition was the equilibrium one and others did not make any clear indication at all. The compilers have included the composition in the footnotes to tables as given in the reports.

Sources of materials, preparations, thermomechanical histories, etc., are indicated if they were given in the original reports. Material identification follows that of the source reports for the most part. The materials are usually given the designation the authors of the original reports assigned although this practice causes some inconsistency in the book. This inconsistency is especially noted for alloys for which the designations given may or may not follow any one of the standard systems such as AISI, ASTM, or ANSI. In the ASTM system for designating alloys, the type or grade refer to an alloy manufactured by a specific producer.

Brand names and manufacturers, where included, are meant only as aids to identification of similar test samples from section to section, and inclusion of these is not intended either as an endorsement or recommendation of any brand name material or manufacturer, nor, conversely, is it intended to prejudice users against the use of any specific product.

The numerical data reported should be viewed with caution. Many of the tests were conducted to screen materials, and the numerical values cannot be considered definitive. Since in many tests the number of samples per material per test is few, often only one, no statistical significance is attached to these values. In most cases, complete characterization of the materials with preparative and thermomechanical history is lacking. The user, therefore, must bear in mind that the data should be used for guidance only and to support the "Materials Evaluations" portions of Part A of this book and are not suitable for inclusion in design calculations. Such use of the data is at the sole risk of the user, and no responsibility for such use can be taken either by the compilers of the data or by the sponsors of this compilation project.

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EFFECT OF EXPOSURE TO VARIOUS GASES AND CONDITIONS ON THE MINERAL
PHASES PRESENT IN A MEDIUM-ALUMINA REFRACTORY[a] [23]

Gas Environment	Temperature °C	Pressure MPa (psi)	Time h	Mineral Phases Present[b]
Control, fired for 5 hr at 1000°C				S_C, A_3S_2, α-A, CA_2, CA, CAS_2(T)
Carbon dioxide	610	(1000)	65	S_C, A_3S_2, α-A, CA_2, CA, CAS_2(T)
50% CO_2 and 50% H_2O	610	(1000)	20	S_C, A_3S_2, α-A, CAS_2(T), $C\overline{C}$(tr), S_t(tr)
50% CO_2 and 50% H_2O	610	(1000)	165	S_C, A_3S_2, α-A, CAS_2(T), $C\overline{C}$(tr), S_t(tr)
Control				A_3S_2, S_C, CA, α-A, CAS_2(T,tr), CA_2(tr)
Steam[c]	{400, 620	2.07, 4.14	17, 100}	A_3S_2, S_C, CAS_2(T)[d], α-A, CA(tr)
Control				A_3S_2, S_C, α-A, CA_2, CAS_2
70% H_2O and 30% CO_2[e]	510	7.5	90	A_3S_2, S_C, α-A, CAS_2, CA_2
	510	15.0	90	A_3S_2, S_C, CAS_2, α-A, CA_2
	710	7.5	90	A_3S_2, S_C, CAS_2, α-A, CA_2(tr), S_t(tr)
	710	15.0	90	A_3S_2, S_C, CAS_2, α-A, CA_2(tr), S_t(tr)
	910	7.5	90	A_3S_2, S_C, CAS_2, CA_2(tr), S_t(tr)
	910	15.0	90	A_3S_2, S_C, CAS_2, α-A, CA_2(tr), S_t(tr)

[a] An NBS-prepared medium-alumina castable refractory bonded with high-purity calcium aluminate cement: 55.6 wt% Al_2O_3, 4.5 wt% CaO, 37.0 wt% SiO_2, 0.8 wt% Fe_2O_3, 0.2 wt% Na_2O, 1.9 wt% others and loss on ignition.

[b] Phases identified by x-ray diffraction; cement notation used, A = Al_2O_3, C = CaO, $\overline{C}$ = CO_2, S = SiO_2, S_C = cristobalite, S_t = tridymite, T = triclinic, tr = trace.

[c] The pressure chamber was initially evacuated and 150 ml water was metered into the chamber; the chamber was then heated and held at the temperatures and pressures for the times given which are bracketed together; phases determined after the total 117 hour exposure.

[d] This compound only moderately crystallized.

[e] Specimens were first heated in argon at ambient pressure; when temperature equilibrium reached water and carbon dioxide were metered into the chamber; after 90 hour exposure the specimens were cooled to 500°C and the gases slowly vented.

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EFFECT OF HEAT TREATMENT ON THE MINERAL PHASES OF SEVERAL ALUMINA REFRACTORIES[a][16]

MATERIAL / Firing Temperature	α-A	S	AS	A_3S_2	CA	CA_2	CA_6	C_3A_5	CAS_2	C_2AS	AH_3	CAH_{10}	C_2AH_8	C_3AH_6	$3CaO\cdot Al_2O_3\cdot CaCO_3\cdot 11H_2O$
Tabular alumina/calcium aluminate cement[c]															
25 °C	S				W						W	W		T	
100	S				W						W			W	
200	S				W						W			W	
400	S				W										
600	S				W										
800	S				W										
1000	S				T	M	W								
1200	S					M	W								
1350	S					W	M								
Tabular alumina/calcium aluminate cement[d]															
25 °C	S				W	W							T		
100	S				T	T						W		W	
200	S				T	T						W		W	
400	S				T	T									
600	S				T	T									
800	S				T	T									
1000	S				W	W	T								
1200	S				T	T	T								
1350	S					T	W								
Calcined bauxite/calcium aluminate cement[e]															
25 °C	M	S[f]			T	T							T		
100	M	S		S	T	T						W		W	
200	M	S		S	T	T						W		W	
400	M	S		S	T	T									
600	M	S		S	T	T									
800	M	S		S	T	T									
1000	M	S		S	W	W									
1200	M	S		S	T	W	T								
1350	M	S		S	T	T	T								
Calcined bauxite/calcium aluminate cement[g]															
25 °C	M	S[f]		S	T	T							T		
100	M	S		S	T	T						W		W	
200	M	S		S	T	T						W		W	
400	M	S		S	T	T									
600	M	S		S	T	T									
800	M	S		S	W	T									
1000	M	S		S	W	W									
1200	M	S		S	T	W	T								
1350	M	S		S	T	T	T								

(Table Continued)

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EFFECT OF HEAT TREATMENT ON THE MINERAL PHASES OF SEVERAL ALUMINA
REFRACTORIES[a][16], Continued

Material / Firing Temperature	α-A	S	AS	A_3S_2	CA	CA_2	CA_6	C_3A_5	CAS_2	C_2AS	AH_3	CAH_{10}	C_2AH_8	C_3AH_6	$3CaO \cdot Al_2O_3 \cdot CaCO_3 \cdot 11H_2O$
Calcined kaolin/calcium aluminate cement[h]															
25 °C	M	S[f]		S	T							T	T		
100	M	S		S	T							T		W	
200	M	S		S	T							W		W	
400	M	S		S	T										
600	M	S		S	T										
800	M	S		S	T				T						
1000	M	S		S	T				W	T					
1200	M	S		S	T	T	T		M	T					
1350	M	S		S		T	T		M	T					
Calcined kaolin/calcium aluminate cement[i]															
25 °C		S[f]		S	T			T					T		
100		S		S								W		W	
200		S		S								W		W	
400		S		S											
600		S		S											
800		S		S	T				T	T					
1000		S		S	T				T	T					
1200		S		S	T				W	W					
1350		S		S	T		T		W	W					
57% alumina insulating castable[j]															
25 °C	M	W[k]	M	S	T								T		
100	M	W	M	S								T		T	
200	M	W	M	S								T		T	
400	M	W	M	S											
600	M	W	M	S											
800	M	W	M	S						T					
1000	M	W	M	S						W					
1200	M	W	M	S						W					
50% alumina insulating castable[l]															
25 °C	M	W[k]	M	S	T										T
100	M	W	M	S	T							T		T	T
200	M	W	M	S	T							T		T	
400	M	W	M	S	T										
600	M	W	M	S	T										
800	M	W	M	S	T	T				T					
1000	M	W	M	S	T	T	T			T					
1200	M	W	M	S	T	T				T					

(Table Continued)

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EFFECT OF HEAT TREATMENT ON THE MINERAL PHASES OF SEVERAL ALUMINA
REFRACTORIES[a][16], Continued

Footnotes
_____

[a] Samples were cast, vibrated one minute, cured sealed in plastic 24 hours at ambient temperature, cured in air 24 hours at ambient temperature, dried in air 24 hours at 100 °C, fired at 60 °C/h to 200, 400, 600, 800, 1000, 1200, and 1350 °C, and soaked at temperature 5 hours, and furnace cooled to ambient temperature.

[b] Identity and relative quantity of compounds present were determined by x-ray diffraction analysis. Cement notation used: $A = Al_2O_3$, $C = CaO$, $H = H_2O$, $S = SiO_2$. Relative quantity indicated by S = strong, M = medium, W = weak, T = trace.

[c] 75% T-61 alumina (Alcoa) aggregate, 25% Casting Grade CA-25 cement (Alcoa), water to ball-in-hand consistency 10.4% of aggregate and cement.

[d] 75% T-61 alumina (Alcoa) aggregate, 25% Secar 71 cement (Lone Star Lafarge), water to ball-in-hand consistency 12.7% of aggregate and cement.

[e] 75% Mulcoa M-60 (C-E Minerals) aggregate, 25% Casting Grade CA-25 (Alcoa) cement, water to ball-in-hand consistency 10.6% of aggregate and cement.

[f] Silica is in the form of cristobalite at all temperatures.

[g] 75% Mulcoa M-60 (C-E Minerals) aggregate, 25% Secar 71 cement (Lone Star Lafarge), water to ball-in-hand consistency 14.6% of aggregate and cement.

[h] 75% Mulcoa M-47 (C-E Minerals) aggregate, 25% Casting Grade CA-25 (Alcoa) cement, water to ball-in-hand consistency 11.3% of aggregate and cement.

[i] 75% Mulcoa M-47 (C-E Minerals) aggregate, 25% Refcon cement (Universal Atlas), water to ball-in-hand consistency 13.5% of aggregate and cement.

[j] A commercial lightweight insulating castable (Cer-Lite #75, C-E Refractories), water to ball-in-hand consistency 22% of mix.

[k] Silica is in the form of quartz at all temperatures.

[l] A commercial lightweight insulating castable (Litecast 75-28, General Refractories), water to ball-in-hand consistency 24% of mix.

EFFECT OF HEAT TREATMENT ON THE STABILITY[a] OF CHEMICAL PHASES PRESENT IN SEVERAL ALUMINA REFRACTORIES[b][16]

(Data Continued)

EFFECT OF HEAT TREATMENT ON THE STABILITY[a] OF CHEMICAL PHASES PRESENT IN SEVERAL ALUMINA REFRACTORIES[b][16], Continued

50% ALUMINA INSULATING CASTABLE [g]

PURE Al_2O_3 STANDARD

CURED AT RT

CURED AT 100°C

FIRED AT 200°C

FIRED AT 300°C

FIRED AT 400°C

DIFFERENTIAL TEMPERATURE
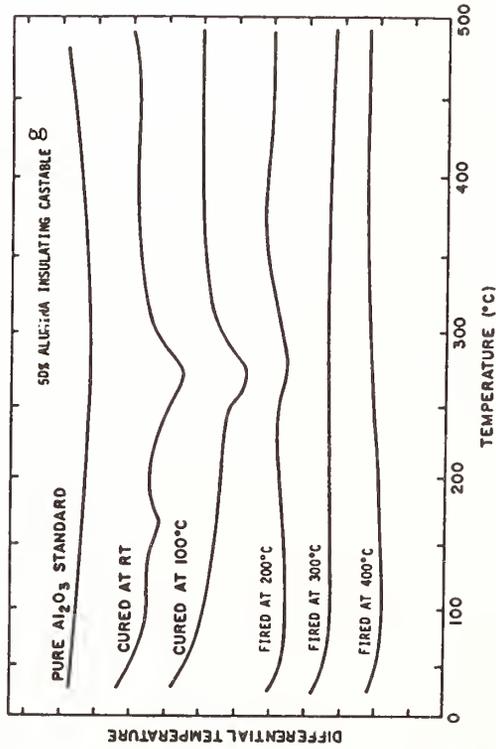
TEMPERATURE (°C)

[a] Determined by differential thermal analysis, samples heated at 5 °C/minute.

[b] Samples were cast, vibrated one minute, cured sealed in plastic 24 hours at ambient temperature, cured in air 24 hours at ambient temperature, dried in air 24 hours at 100 °C, fired at 60 °C/h to 200, 300, and 400 °C, and soaked at temperature 5 hours, and furnace cooled to ambient temperature.

[c] 75% T-61 alumina (Alcoa) aggregate, 25% Casting Grade CA-25 cement (Alcoa), water to ball-in-hand consistency 10.4% of aggregate and cement.

[d] 75% Mulcoa M-47 (C-E Minerals) aggregate, 25% Casting Grade CA-25 (Alcoa) cement, water to ball-in-hand consistency 11.3% of aggregate and cement.

[e] 75% Mulcoa M-47 (C-E Minerals) aggregate, 25% Refcon cement (Universal Atlas), water to ball-in-hand consistency 13.5% of aggregate and cement.

[f] A commercial lightweight insulating castable (Cer-Lite #75, C-E Refractories), water to ball-in-hand consistency 22% of mix.

[g] A commercial lightweight insulating castable (Litecast 75-28, General Refractories), water to ball-in-hand consistency 24% of mix.

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EFFECT OF STEAM ON THE MINERAL PHASES[a] OF A CALCIUM ALUMINATE CEMENT[b][24]

Temperature °C	Pressure psig	Time h	Mineral Phases Present[a]
25	ambient	0	CA_2, small amounts of α-A, CA
93	10	20	CA_2 (intensities greatly reduced), C_3AH_6 and AH_3 present as minor phases
110	15	46	C_3AH_6, AH_3, AH, α-A, CA_2(tr)
210	200	4	C_3AH_6, AH, α-A(tr)
210	150	3	C_3AH_6, AH (crystallinity of AH improved)
210	150	15	C_3AH_6, AH (AH well crystallized)
210	150	7	C_3AH_6, AH (both major phases), α-A (minor phase)
100	15	3	no change
365	1100	15	$C_4A_3H_3$, AH (both major phases), α-A (minor phase)
310	970	6	no change
410	1130	2	no change
410	1120	14	$C_4A_3H_3$, α-A, AH (all major phases; AH dissociating)
410	1120	8	$C_4A_3H_3$, α-A, AH (all major phases; AH dissociating, α-A increasing)
450	1130	1	no change
450	1130	1	no change
450	1130	1	no change
475	1130	1	CA_2, CA, $C_4A_3H_3$, α-A (all major phases), AH(tr)
475	1130	15	CA_2, CA, $C_4A_3H_3$, α-A (all major phases), CA_2 increasing
500	1075	3	no change
550	1100	4	CA_2, CA, α-A (all major phases), CA_2 increasing
575	1100	22	CA_2, CA, α-A
600	1030	1	CA_2, CA, α-A (CA_2 increasing; α-A, CA decreasing)
600	1030	3	no change
600	1050	15	CA_2 (major phase), α-A (minor phase)
700	1000	3	no change
800	1000	16	CA_2, α-A(tr)
900	1060	1	no change
1000	1000	1	no change

[a] Specimen was subjected to steam in a pressure vessel constructed so as to permit _in situ_ examination of specimens and identification of compounds by energy dispersive x-ray diffraction; EDXD patterns were taken at the various pressures and temperatures without removing the specimen from the environment.

[b] Neat high-purity calcium aluminate cement (CA-25 Calcium Aluminate Cement, Alcoa) dried at 110 °C and fired at 1010 °C for 5 hours.

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DISINTEGRATION OF SOME Fe-DOPED[a] REFRACTORIES DUE TO EXPOSURE TO CARBON MONOXIDE-CONTAINING ATMOSPHERES
UNDER VARYING PRESSURES[27]

Exposure Conditions	90% Alumina Castable[b]	50% Alumina Castable[c]	90% Alumina Ramming Mix[d]

Pressure: 600 psi
Gas: CO
Time: 100 h
Temperature: 500 °C
	Undoped--
	  Color changed to gray
	  from carbon deposition
	  (not observed at 1 atm.)
	Fe dopant--
	  3 of 4 samples with 0.5
	  wt% Fe totally disinte-
	  grated. 3 samples with
	  2.0% Fe remained intact.
	Fe_2O_3 dopant--
	  Turned very dark gray,
	  large amounts of C in
	  surface pores.
		Undoped--
		  No color change. Very
		  small amounts of edge
		  spalling seen in all 4
		  samples. Small portion
		  of one face spalled to
		  reveal a carbon ball.
		Fe dopant--
		  Samples with 0.5% Fe com-
		  pletely disintegrated,
		  with 2.0% Fe they remained
		  intact.
		Fe_2O_3 dopant--
		  No spalling occurred.
		  Color was lighter than the
		  90% castable.
			Undoped--
			  No spalling damage observed.
			  No apparent accelerated C
			  deposition from the 1 atm.
			  test.
			Fe dopant--
			  Samples with 0.5 wt% Fe
			  completely disintegrated.
			Fe_2O_3 dopant--
			  Samples with 1.0 wt% Fe
			  turned medium gray but on-
			  ly spalled slightly on
			  corners and edges.

Pressure: 200 psi
Gas: CO
Time: 100 h
Temperature: 500 °C
	Undoped--
	  Carbon deposition at
	  the surface, not as
	  great as at 600 psi.
	Doped--
	  2 samples with 0.5 wt%
	  Fe completely disinte-
	  grated.
		Undoped--
		  Carbon deposition at the
		  surface, not as great as
		  at 600 psi.
		Doped--
		  2 samples with 0.5 wt%
		  Fe completely disinte-
		  grated.
			These samples behaved the
			same as similarly doped
			samples of the castable
			refractories.

Pressure: 1000 psi
Gas: CO
Time: 50 h
Temperature: 550 °C
	Undoped (6 samples)--
	  No disintegration oc-
	  curred. Color changed
	  to light gray. Slight
	  surface carbon deposi-
	  tion. Thin cracks ap-
	  peared.
	0.1 wt% Fe (2 samples)--
	  Completely disintegrated.
		Undoped (6 samples)--
		  No disintegration oc-
		  curred. Color changed to
		  gray. Thin surface cracks
		  appeared. One sample had
		  one pop-out area.
		0.1 wt% Fe (2 samples)--
		  Completely disintegrated.
			Not tested.

Pressure: 1000 psi
Gas: CO
Time: 50 h
Temperature: 500 °C
	Undoped (9 samples)--
	  Did not disintegrate.
	  Color changed white to
	  gray. Thin cracks and
	  small pop-out areas ap-
	  peared.
	Doped--
	  4 samples with 0.1 wt% Fe
	  and 3 with 0.25 wt% com-
	  pletely disintegrated.
		Undoped (3 samples)--
		  Did not disintegrate. Con-
		  siderable carbon deposition
		  occurred. Crack lines and
		  edge flaking observed.
		Doped--
		  3 samples with 0.1 wt% Fe
		  and 3 with 0.25 wt% com-
		  pletely disintegrated.
			Not tested.

Pressure: 1000 psi
Gas: CO
Time: 50 h
Temperature: 450 °C
	Undoped (6 samples)--
	  Did not disintegrate.
	  Slight discoloration oc-
	  curred. Light carbon
	  deposition, thin cracks,
	  edge spalling, and several
	  "spots" of carbon or pop-
	  outs observed.
	Doped--
	  2 samples with 0.1 wt% Fe
	  completely disintegrated.
		Undoped (3 samples)--
		  Did not disintegrate.
		  Heavy carbon deposition,
		  distinct pop-outs, and
		  flaking observed.
		Doped--
		  3 samples each with 0.1 wt%
		  and 0.25 wt% Fe completely
		  disintegrated.
			Not tested.

Pressure: 1000 psi
Gas: CO
Time: 50 h
Temperature: 400 °C
	Undoped (6 samples)--
	  Did not disintegrate. No
	  spalling observed. Very
	  slight discoloration.
	Doped (0.1 wt% Fe, 2 samples)
	  Completely disintegrated.
		Undoped (6 samples)--
		  Did not disintegrate.
		  Color changed white to light
		  gray. Light carbon deposi-
		  tion observed and thin cracks.
		Doped (0.1 wt% Fe, 2 samples)
		  Completely disintegrated.
			Not tested.

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(Table Continued)

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DISINTEGRATION OF SOME Fe-DOPED[a] REFRACTORIES DUE TO EXPOSURE TO CARBON MONOXIDE-CONTAINING ATMOSPHERES

UNDER VARYING PRESSURES[27], Continued

Exposure Conditions	90% Alumina Castable[b]	50% Alumina Castable[c]	90% Alumina Ramming Mix[d]
Pressure: 200 psi Gas: 80% CO-20% H_2O Time: 100 h Temperature: 500 °C	3 samples, all doped with 0.5 wt% Fe, completely disintegrated.	3 samples, all doped with 0.5 wt% Fe, completely disintegrated.	3 samples, all doped with 0.5 wt% Fe, showed little or no damage.
Pressure: 500 psi Gas: 80% CO-20% H_2O Time: 100 h Temperature: 500 °C	2 of 3 samples with 0.5 wt% Fe completely disintegrated. The 3rd spalled extensively (over 30% weight loss).	All 3 samples with 0.5 wt% Fe completely disintegrated.	All 3 samples with 0.5 wt% Fe completely disintegrated.

Pressure: 1 atm
Gas: coal gas mix[e]
Time: 1000 h
Temperature: 500 °C
	Undoped--
	  No disintegration.
	Fe dopant--
	  Samples with 0.5, and 1.0 wt% Fe showed no signs of disintegration.
	Fe_2O_3 dopant--
	  Samples with 2.0 wt% Fe showed no signs of disintegration. Color turned medium gray.
		Undoped--
		  No disintegration.
		Fe dopant--
		  Samples with 0.5, and 1.0 wt% Fe showed no signs of disintegration.
		Fe_2O_3 dopant--
		  Samples with 2.0 wt% Fe showed no signs of disintegration. Color turned medium gray.
			Undoped--
			  No disintegration.
			Fe dopant--
			  Samples with 0.5, and 1.0 wt% Fe showed no signs of disintegration.
			Fe_2O_3 dopant--
			  Samples with 2.0 wt% Fe showed no signs of disintegration. Color turned black.

Pressure: 600 psi
Gas: coal gas mix[f]
Time: 400 h
Temperature: 500 °C
	Undoped--
	  Samples showed minor signs of disintegration. Considerable carbon deposition seen on the outsides and all samples were discolored throughout the interiors. Some edge and corner weakness observed.
		Undoped--
		  Samples showed minor signs of disintegration. Considerable carbon deposition seen on the outsides and all samples were discolored throughout the interiors. Some edge and corner weakness observed.
			Not tested

[a] Refractories were doped with varying weight percent of iron either in the form of metallic iron (Fe) or hematite (Fe_2O_3).

[b] The DOE generic castable was prepared from 65 wt% tabular alumina (25 wt% 6 to 10 mesh, 20 wt% 10 to 20 mesh, 20 wt% less than 20 mesh), 10 wt% calcined alumina (less than 325 mesh), 25 wt% calcium aluminate cement (Casting Grade CA-25 cement), all Alcoa materials. Materials were dry mixed for 2 min, then water added with continued mixing to ball-in-hand consistency ($\sim$605 ml); refractory was then poured into 2 in. x 2 in. x 2 in. aluminum molds, vibrated for 5 min, sealed in plastic for 24 h, then fired for 5 h in air at 1100 °C.

[c] The DOE generic castable was prepared from 75 wt% calcined kaolin (25 wt% 6 to 10 mesh, 20 wt% 10 to 20 mesh, 15 wt% less than 20 mesh, 20 wt% less than 200 mesh), 25 wt% calcium aluminate cement (Casting Grade CA-25, Alcoa). Samples were prepared as described in footnote b except that the water used was $\sim$700 ml.

[d] The DOE generic ramming mix was prepared from 82 wt% tabular alumina (30 wt% 6 to 10 mesh, 20 wt% 10 to 20 mesh, 15 wt% less than 20 mesh, 17 wt% less than 48 mesh), 15 wt% calcined alumina (less than 325 mesh), 3 wt% bentonite; to this was added 1 wt% hydrated alumina, 6 wt% phosphoric acid (85%), and 0.25 wt% water. Dry materials were mixed and then hand mixed in plastic bags with the liquids; mixture was aged for 18 h; samples were formed by die pressing at 500 psi, cured at 250 °C in air for 12 h, then fired at 1100 °C for 5 h.

[e] Simulated coal gasification feed gas composition: 14% CO, 5% CO_2, 45% H_2, 17% H_2O, 17% CH_4, 0.1% H_2S.

[f] Simulated coal gasification feed gas composition: 18.5% CO, 10.4% CO_2, 22.8% H_2, 40% H_2O, 4.6% CH_4, 0.69% H_2S, 1.0% NH_3.

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RELATIVE CORROSION RESISTANCE OF REFRACTORIES EXPOSED[a] TO SLAG ATTACK[32]

Refractory Material	Brand Name/Manufacturer	Maximum Depth[b] of Removal, mm	Maximum Depth[b] of Penetration, mm

Average Slag Composition During Test, weight percent   Exposure Time 200 hours

SiO_2	Al_2O_3	CaO	Fe_2O_3	MgO	Na_2O/K_2O	TiO_2	SO_3	P_2O_5	V_2O_5	Base/Acid
46.4	23.0	12.8	9.4	4.3	3-6	0.7	0.01	0.10	0.04	0.45

Refractory Material	Brand Name/Manufacturer	Max Depth of Removal, mm	Max Depth of Penetration, mm
Al_2O_3(99), sintered brick	99AD/A. P. Green	16	49
Al_2O_3(99), fused-cast brick	Monofrax A2/Carborundum	3	4
Al_2O_3(95)-CaO(5), castable	B&W Kao-Tab CS/Babcock & Wilcox	27	78
Al_2O_3(94)-CaO(5), castable	Purotab/Kaiser Refractories	24	57
Al_2O_3(93)-P_2O_5(6), castable	B&W Kao Phos 30/Babcock & Wilcox	20	50
Al_2O_3(90)-SiO_2(9), sintered brick	Greenal-90/A. P. Green	10	12
Al_2O_3(91)-SiO_2(8), sintered brick	Kricor/Kaiser Refractories	12	20
Al_2O_3(91)-SiO_2(8), sintered brick	Korundal XD/Harbison-Walker	7	12
Al_2O_3(89)-Cr_2O_3(10), sintered brick	CS 612/Kaiser Refractories	5	20
Al_2O_3(84)-SiO_2(9)-P_2O_5(4), brick	Chemal 85 B/C-E Refractories	7	25
Cr_2O_3(40)-Fe_2O_3(23)-Al_2O_3(21)-MgO(10), ramming mix	Kemram/C-E Refractories	21	--[c]
MgO(53)-Cr_2O_3(20)-Al_2O_3(17)-Fe_2O_3(8), sintered brick	Dibond 50/General Refractories	8	21
MgO(59)-Cr_2O_3(19)-Al_2O_3(13)-Fe_2O_3(7), sintered brick	GR-200/General Refractories	7	20
MgO(64)-Cr_2O_3(15)-Al_2O_3(13)-Fe_2O_3(6), sintered brick	Nucon 60/Harbison-Walker	7	22
Cr_2O_3(80)-MgO(8)-Fe_2O_3(6), fused-cast brick	Monofrax E/Carborundum	∿0	∿0

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Average Slag Composition During Test, weight percent   Exposure Time 500 hours

SiO_2	Al_2O_3	CaO	Fe_2O_3	Cr_2O_3	MgO	Na_2O/K_2O	TiO_2	SO_3	P_2O_5	V_2O_5	Base/Acid
27.0	15.5	37.3	9.0	0.12	8.0	0.5-1.5	0.34	0.04	0.86	0.03	1.35

Refractory Material	Brand Name/Manufacturer	Max Depth of Removal, mm	Max Depth of Penetration, mm
Al_2O_3(99), sintered brick	99AD/A. P. Green	57	64
Al_2O_3(99.5), sintered brick	Alundum/Norton	∿25	76
Al_2O_3(90)-Cr_2O_3(10), sintered brick	CS 612/Kaiser Refractories	∿10	∿25
MgO(53)-Cr_2O_3(20)-Al_2O_3(17)-Fe_2O_3(8), sintered brick	Dibond 50/General Refractories	∿5	∿10
MgO(59)-Cr_2O_3(19)-Al_2O_3(13)-Fe_2O_3(7), sintered brick	GR-200/General Refractories	∿5	∿10
SiC(79)-Si_3N_4(18.5), sintered brick	Refrax 20/Carborundum	64	64
SiC(73)-Al_2O_3(14)-SiO_2(11), sintered brick	Harbide/Harbison-Walker	64	64
Al_2O_3(85)-SiO_2(9)-P_2O_5(4), brick	Chemal 85 B/C-E Refractories	64	76
Al_2O_3(93)-P_2O_5(1), castable	---/Pryor-Giggey	33	64
Al_2O_3(81)-Cr_2O_3(10)-P_2O_5(7), ramming mix	I-75/Harbison-Walker	18	43
Al_2O_3(Winnofos Bond), brick	ICI-B/Imperial Chemical	56	76
Al_2O_3(99)-Na_2O(0.5), fused-cast brick	Monofrax A1/Carborundum	19	20
Al_2O_3(99)-B_2O_3(0.5), fused-cast brick	Monofrax A2/Carborundum	22	24
Cr_2O_3(80)-MgO(8)-Fe_2O_3(6), fused-cast brick	Monofrax E/Carborundum	∿0	∿0
Al_2O_3(80), fused-cast brick	SR 504C/Corhart Refractories	17	∿18

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Average Slag Composition During Test, weight percent   Exposure Time 500 hours

SiO_2	Al_2O_3	CaO	Fe_2O_3	Cr_2O_3	MgO	Na_2O	K_2O	TiO_2	SO_3	P_2O_5	V_2O_5	Residue	Base/Acid
25.9	27.0	30.7	8.7	0.4	3.0	0.5	0.5	0.4	0.03	0.04	<0.01	2.2	0.84

Refractory Material	Brand Name/Manufacturer	Max Depth of Removal, mm	Max Depth of Penetration, mm
MgO(53)-Cr_2O_3(20)-Al_2O_3(17)-Fe_2O_3(8), sintered brick	Dibond 50/General Refractories	11.4	35.6
MgO(59)-Cr_2O_3(19)-Al_2O_3(13)-Fe_2O_3(7), sintered brick	GR-200/General Refractories	8.1	38.1
MgO(64)-Cr_2O_3(15)-Al_2O_3(13)-Fe_2O_3(6), sintered brick	Nucon 60/Harbison-Walker	11.4	40.6
MgO(63)-Cr_2O_3(18)-Al_2O_3(5)-Fe_2O_3(12), sintered brick	Guidon/Harbison-Walker	5.8	38.1
MgO(63)-Cr_2O_3(17)-Al_2O_3(10)-Fe_2O_3(9), sintered brick	Krilex 621/Kaiser Refractories	8.9	40.6
Al_2O_3(90)-Cr_2O_3(10), sintered brick	Ruby/Harbison-Walker	10.7	48.3

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(Table Continued)

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RELATIVE CORROSION RESISTANCE OF REFRACTORIES EXPOSED[a] TO SLAG ATTACK[32], Continued

Refractory Material	Brand Name/Manufacturer	Maximum Depth[b] of Removal, mm	Maximum Depth[b] of Penetration, mm
$Al_2O_3(81)-Cr_2O_3(17)$, sintered brick	852Z (Serv-M)/Taylor Refractories	14.5	27.9
$Al_2O_3(68)-Cr_2O_3(32)$, sintered brick	TS 1290/Union Carbide	16.0	58.4
$Si_2ON_2(70)-Si_3N_4(20)-SiC(10)$, sintered brick	Sioxyn/Norton	62.7[c]	62.7[c]
$Al_2O_3(92)-SiO_2(8)$, sintered brick	Kricor/Kaiser Refractories	35.6	45.7
$Al_2O_3(56)-SiO_2(27)-ZrO_2(16)$, sintered brick	ZRX/Taylor Refractories	73.7[c]	73.7[c]
MgO, Al_2O_3, sintered brick	Experimental Spinel/Taylor	22.9	73.7[c]
$Al_2O_3(21)-Cr_2O_3(40)-Fe_2O_3(23)$, ramming mix	Kemram/C-E Refractories	11.4	53.3
$Al_2O_3(98)$, fused-cast brick	SR 504C/Corhart Refractories	19.6	22.9
$Cr_2O_3(80)-MgO(8)-Fe_2O_3(6)$, fused-cast brick	Monofrax E/Carborundum	1.9	5.1
$Al_2O_3(50)-ZrO_2(34)-SiO_2(14)$, fused-cast brick	Monofrax R/Carborundum	73.7[c]	73.7[c]

[a]Exposure to slag attack took place in a 24-in. diameter, 13-in. high furnace. Test specimens were 9 in. x 4.5 in. x 3.2 in. brick wedges mounted around the circumference of the vessel with the small end of the brick exposed to the slag. Most specimens were manufacturer-supplied bricks. Some ramming mixes and castables were tested; these were formed in a mold, dried, and prefired in situ to 1500 °C prior to adding the slag. The furnace was fired with natural gas, air, and oxygen to a temperature around 1500 °C. Oxygen partial pressure was maintained at 10^{-2} to 10^{-3} Pa by adjusting the ratio of the three component gases. The furnace cavity was charged with up to 75 kg of synthetic slag. Some rotary motion of the slag was achieved by the tangential impingement of the flames from three burners on the surface of the melt. Wet chemical analyses were performed on samples of slag drawn at intervals of 100 hours or less during the course of the tests. The slag compositions given in the table are the averages of all the analyses.

[b]Depth is measured from the original hot face.

[c]Complete attack.

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RELATIVE CORROSION RESISTANCE OF WATER-COOLED REFRACTORIES EXPOSED[a] TO SLAG ATTACK[32]

Refractory Material	Brand Name/Manufacturer	Maximum Depth[b] of Removal, mm	Maximum Depth[b] of Penetration, mm

Average Slag Composition During Test, weight percent   Exposure Time 500 hours

SiO$_2$	Al$_2$O$_3$	CaO	Fe$_2$O$_3$	FeO	MgO	Na$_2$O	K$_2$O	TiO$_2$	Base/Acid
39.2	18.3	22.4	2.1	9.4	5.4	1.2	0.9	0.9	0.74

Refractory Material	Brand Name/Manufacturer	Maximum Depth of Removal, mm	Maximum Depth of Penetration, mm
SiC(90)-Si$_2$ON$_2$(9), brick	Crystolon 63/Norton	3-4	not given
SiC(73)-Al$_2$O$_3$(14)-SiO$_2$(11), brick	Harbide/Harbison-Walker	4-7	not given
SiC(75)-Si$_3$N$_4$(23), brick	Refrax 20/Carborundum	4-5	not given
Si$_2$ON$_2$(70)-Si$_3$N$_4$(20)-SiC(10), brick	Sioxyn/Norton	47-52	not given

Average Slag Composition During Test, weight percent   Exposure Time 500 hours

SiO$_2$	Al$_2$O$_3$	CaO	Fe$_2$O$_3$	FeO	MgO	Na$_2$O	K$_2$O	TiO$_2$	Cr$_2$O$_3$	Base/Acid
39.5	20.0	24.0	0.4	6.6	6.4	1.4	0.8	0.9	0.3	0.64

Refractory Material	Brand Name/Manufacturer	Maximum Depth of Removal, mm	Maximum Depth of Penetration, mm
MgO(53)-Cr$_2$O$_3$(20)-Al$_2$O$_3$(17)-Fe$_2$O$_3$(8)-SiO$_2$(2)-CaO(1), brick	Dibond 50/General Refractories	8	11[c]
MgO(59)-Cr$_2$O$_3$(19)-Al$_2$O$_3$(13)-Fe$_2$O$_3$(7)-SiO$_2$(1.5)-CaO(0.5), brick	Gr-200/General Refractories	8	11[c]
MgO(63)-Cr$_2$O$_3$(18)-Al$_2$O$_3$(5)-Fe$_2$O$_3$(12)-SiO$_2$(1)-CaO(1), brick	Guidon/Harbison-Walker	8	9[c]
MgO(60)-Cr$_2$O$_3$(15.5)-Al$_2$O$_3$(15)-Fe$_2$O$_3$(7)-SiO$_2$(1.5)-CaO(1), brick	Krilex 911/Kaiser Refractories	17	21[c]

Average Slag Composition During Test, weight percent   Exposure Time 305 hours[d]

SiO$_2$	Al$_2$O$_3$	CaO	Fe$_2$O$_3$	FeO	MgO	Na$_2$O	K$_2$O	TiO$_2$	Base/Acid
38.7	20.2	24.3	1.4	5.6	6.9	0.9	0.7	1.0	0.7

Refractory Material	Brand Name/Manufacturer	Maximum Depth of Removal, mm	Maximum Depth of Penetration, mm
Al$_2$O$_3$(99.3)-Na$_2$O(0.4)-CaO(0.1), fused cast brick	Monofrax Al/Carborundum	20	52
Al$_2$O$_3$(60.4)-Cr$_2$O$_3$(27.3)-MgO(6.0)-Fe$_2$O$_3$(4.2)-SiO$_2$(1.8), fused-cast brick	Monofrax K-3/Carborundum	10	~12
MgO(56.5)-Cr$_2$O$_3$(20.0)-Fe$_2$O$_3$(10.5)-Al$_2$O$_3$(8.0)-SiO$_2$(2.5)-TiO$_2$(1.5)-CaO(0.5), fused-cast brick	Corhart C-104/Corhart Refractories	28	~70
MgO(60.8)-Cr$_2$O$_3$(18.6)-Fe$_2$O$_3$(11.0)-Al$_2$O$_3$(6.6)-CaO(1.9)-SiO$_2$(1.1), sintered brick	GR-224/General Refractories	34	44

Average Slag Composition During Test, weight percent   Exposure Time 500 hours

SiO$_2$	Al$_2$O$_3$	CaO	Fe$_2$O$_3$	FeO	MgO	Na$_2$O	K$_2$O	TiO$_2$	Other	Base/Acid
39.4	17.1	25.8	2.8	4.9	6.4	1.3	1.0	1.1	0.2	0.75

Refractory Material	Brand Name/Manufacturer	Maximum Depth of Removal, mm	Maximum Depth of Penetration, mm
Al$_2$O$_3$(91.6)-SiO$_2$(8.0)-Fe$_2$O$_3$(0.15)-Alkali(0.15), sintered brick	Kricor/Kaiser Refractories	9	10
Al$_2$O$_3$(83.9)-SiO$_2$(9.0)-Fe$_2$O$_3$(1.0)-TiO$_2$(2.3)-P$_2$O$_5$(3.6), brick	Chemal 85B/C-E Refractories	13	17
Al$_2$O$_3$(89.7)-Cr$_2$O$_3$(10.0)-SiO$_2$(0.1)-Fe$_2$O$_3$(0.1)-Alkali(0.1), sintered brick	Ruby/Harbison-Walker	4	7
Al$_2$O$_3$(81.1)-Cr$_2$O$_3$(16.6)-P$_2$O$_5$(0.8)-SiO$_2$(0.5)-Fe$_2$O$_3$(0.5)-Na$_2$O(0.5), sintered brick	852Z (Serv-M)/Taylor Refractories	2	9

Average Slag Composition During Test, weight percent   Exposure Time 500 hours

SiO$_2$	Al$_2$O$_3$	CaO	Fe$_2$O$_3$	FeO	Fe	MgO	Na$_2$O	K$_2$O	TiO$_2$	Other	Base/Acid
38.4	20.4	28.8	1.3	4.9	1.0	2.8	0.6	0.4	1.0	0.4	0.7

Refractory Material	Brand Name/Manufacturer	Maximum Depth of Removal, mm	Maximum Depth of Penetration, mm
Cr$_2$O$_3$(39.6)-Al$_2$O$_3$(21.4)-Fe$_2$O$_3$(22.6)-MgO(9.6)-SiO$_2$(2.5)-TiO$_2$(0.6)-P$_2$O$_5$(3.7), ramming mix	Kemram/C-E Refractories	2	45[e]
Al$_2$O$_3$(85.2)-Cr$_2$O$_3$(9.7)-SiO$_2$(1.9)-Fe$_2$O$_3$(0.5)-Na$_2$O(0.4)-P$_2$O$_5$(2.3), ramming mix	Shamrock 888/Taylor Refractories	4	20
Al$_2$O$_3$(89.5)-SiO$_2$(6.0)-Alkali(0.2)-P$_2$O$_5$(3.7)-other(0.6), ramming mix	Brikram 90R/General Refractories	9	14
Al$_2$O$_3$(89.6)-MgO(3.4)-SiO$_2$(0.7)-P$_2$O$_5$(6.2)-Alkali(0.1), ramming mix	Resco Cast AA-22/Resco	9	24
Al$_2$O$_3$(95)-SiO$_2$(0.2)-Na$_2$O(0.2)-Fe$_2$O$_3$(0.05)-P$_2$O$_5$(4.55), ramming mix	Lavalox X-95/Lava Crucible	10	42
Al$_2$O$_3$(67)-Cr$_2$O$_3$(32), sintered brick	LT5/Union Carbide	2	20[e]

(Table Continued)

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RELATIVE CORROSION RESISTANCE OF WATER-COOLED REFRACTORIES EXPOSED[a] TO SLAG ATTACK[32], Continued

Refractory Material	Brand Name/Manufacturer	Maximum Depth[b] of Removal, mm	Maximum Depth[b] of Penetration, mm

Average Slag Composition During Test, weight percent — Exposure Time 1000 hours

SiO$_2$	Al$_2$O$_3$	CaO	Fe$_2$O$_3$	FeO	Fe	MgO	Na$_2$O	K$_2$O	TiO$_2$	Base/Acid
36.9	17.4	29.2	2.8	3.2	0.4	7.3	1.4	0.8	0.6	0.9

Refractory Material	Brand Name/Manufacturer	Removal	Penetration
Al$_2$O$_3$(60)-Cr$_2$O$_3$(27)-MgO(6)-Fe$_2$O$_3$(4)-SiO$_2$(2), fused-cast brick	Monofrax K-3/Carborundum	4	not given
Cr$_2$O$_3$(80)-MgO(8)-Fe$_2$O$_3$(6)-Al$_2$O$_3$(5)-SiO$_2$(1), fused-cast brick	Monofrax E/Carborundum	2	not given
SiC(75)-Si$_3$N$_4$(23), sintered brick	Refrax 20/Carborundum	38	not given
SiC(73)-Al$_2$O$_3$(14)-SiO$_2$(11), sintered brick	Harbide/Harbison-Walker	36	not given
SiC(90)-Si$_2$ON$_2$(9), sintered brick	Crystolon 63/Norton	32	not given
Al$_2$O$_3$(91.6)-SiO$_2$(8.0)-Fe$_2$O$_3$(0.15)-Alkali(0.15), sintered brick	Kricor/Kaiser Refractories	46	not given

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Average Slag Composition During Test, weight percent — Exposure Time 475 hours

SiO$_2$	Al$_2$O$_3$	CaO	Fe$_2$O$_3$	FeO	Fe	MgO	Na$_2$O	K$_2$O	TiO$_2$	Cr$_2$O$_3$	Base/Acid
38.0	28.9	17.4	1.3	1.6	0.7	4.2	0.14	8.2	0.25	<0.1	0.48

Refractory Material	Brand Name/Manufacturer	Removal	Penetration
Al$_2$O$_3$(99)-Na$_2$O(0.4), fused-cast brick	Monofrax Al/Carborundum	1	60
Cr$_2$O$_3$(80)-MgO(8)-Fe$_2$O$_3$(6), fused-cast brick	Monofrax E/Carborundum	0	0
Al$_2$O$_3$(60.4)-Cr$_2$O$_3$(27.3)-MgO(6.0)-Fe$_2$O$_3$(4.2)-SiO$_2$(1.8), fused-cast brick	Monofrax K-3/Carborundum	1	0
MgO(59)-Cr$_2$O$_3$(19)-Al$_2$O$_3$(13)-Fe$_2$O$_3$(7)-SiO$_2$(1.5)-CaO(0.5), sintered brick	GR-200/General Refractories	1	40[f]
Al$_2$O$_3$(89.7)-Cr$_2$O$_3$(10), sintered brick	Ruby/Harbison-Walker	2	23
MgO(63)-Cr$_2$O$_3$(19)-Al$_2$O$_3$(5)-Fe$_2$O$_3$(12)-SiO$_2$(1)-CaO(1), sintered brick	Guidon/Harbison-Walker	1	--[f]
Al$_2$O$_3$(70)-MgO(28), sintered brick	---/---	4	50
Cr$_2$O$_3$(39.6)-Al$_2$O$_3$(21.4)-Fe$_2$O$_3$(22.6)-MgO(9.6)-SiO$_2$(2.5)-TiO$_2$(0.6)-P$_2$O$_5$(3.7), ramming mix	Kemram/C-E Refractories	2[g]	60
Al$_2$O$_3$(85)-Cr$_2$O$_3$(10)-SiO$_2$(2)-P$_2$O$_5$(3), ramming mix	---/---	3-5	50
MgO(95)-SiO$_2$(3)-CaO(1), ramming mix	---/---	5,6	7
Cr$_2$O$_3$(33)-Al$_2$O$_3$(28)-MgO(19)-FeO(13)-SiO$_2$(5), ramming mix	---/---	20[g]	70

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Average Slag Composition During Test, weight percent — Exposure Time 500 hours

SiO$_2$	Al$_2$O$_3$	CaO	Fe$_2$O$_3$	FeO	Fe	MgO	Na$_2$O	K$_2$O	TiO$_2$	Base/Acid
24.4	15.5	38.9	4.2	9.0	1.0	5.7	<0.1	<0.1	1.0	1.4

Refractory Material	Brand Name/Manufacturer	Removal	Penetration
Al$_2$O$_3$(89.7)-Cr$_2$O$_3$(10), sintered brick	CS 612/Kaiser Refractories	10	18
Al$_2$O$_3$(81.1)-Cr$_2$O$_3$(16.6)-P$_2$O$_5$(0.8)-SiO$_2$(0.5)-Fe$_2$O$_3$(0.5)-Na$_2$O(0.5), sintered brick	852Z (Serv-M)/Taylor Refractories	3	8
Al$_2$O$_3$(92)-Cr$_2$O$_3$(7.5)-SiO$_2$(0.5), sintered brick	AR90/Findlay	21	31
Al$_2$O$_3$(84.5)-Cr$_2$O$_3$(10.5)-P$_2$O$_5$(4.5)-SiO$_2$(0.2), plastic	86B/Lava Crucible	10	16
MgO(63)-Cr$_2$O$_3$(18)-Fe$_2$O$_3$(12)-Al$_2$O$_3$(5)-SiO$_2$(1)-CaO(1), sintered brick	Guidon/Harbison-Walker	4	14
MgO(60)-Cr$_2$O$_3$(15.5)-Al$_2$O$_3$(15)-Fe$_2$O$_3$(7)-SiO$_2$(1.5)-CaO(1), sintered brick	Krilex 911/Kaiser Refractories	4	16
MgO(55)-Cr$_2$O$_3$(20)-Al$_2$O$_3$(8)-FeO(11)-SiO$_2$(2.5)-CaO(0.5)-TiO$_2$(1.5), sintered brick	Corhart RFG/Corhart Refractories	5	20
Al$_2$O$_3$(99)-Na$_2$O(0.5), fused-cast brick	Monofrax Al/Carborundum	10	13
Al$_2$O$_3$(65)-Cr$_2$O$_3$(32)-FeO(1)-CaO(0.6)-MgO(0.6), fused-cast brick	Corhart AC-28/Corhart Refractories	1	1
Cr$_2$O$_3$(80)-MgO(8)-Fe$_2$O$_3$(6)-Al$_2$O$_3$(5)-SiO$_2$(1), fused-cast brick	Monofrax E/Carborundum	1	2
MgO(65)-Al$_2$O$_3$(35), fused-cast brick	X-317/Corhart Refractories	15	18

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(Table Continued)

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RELATIVE CORROSION RESISTANCE OF WATER-COOLED REFRACTORIES EXPOSED[a] TO SLAG ATTACK[32], Continued

Refractory Material	Brand Name/Manufacturer	Maximum Depth[b] of Removal, mm	Maximum Depth[b] of Penetration, mm
Average Slag Composition During Test, weight percent	Exposure Time 493 hours (at 1575 °C)		

Average Slag Composition During Test, weight percent:

SiO_2	Al_2O_3	CaO	Fe_2O_3	FeO	Fe	MgO	Na_2O	K_2O	TiO_2	Other	Base/Acid
44.6	18.7	10.1	1.2	18.0	0.5	3.0	1.2	1.4	0.9	0.3	0.55

Refractory Material	Brand Name/Manufacturer	Maximum Depth of Removal, mm	Maximum Depth of Penetration, mm
$Al_2O_3(84.5)$-$Cr_2O_3(10.5)$-$SiO_2(0.2)$-$P_2O_5(4.6)$, plastic	86B/Lava Crucible	38	63
$Al_2O_3(92)$-$Cr_2O_3(7.5)$-$SiO_2(0.5)$, sintered brick	AR90/Findlay	18	28
$Al_2O_3(89.7)$-$Cr_2O_3(10)$, sintered brick	CS-612/Kaiser Refractories	17	28
$Al_2O_3(89)$-$Cr_2O_3(10)$, sintered brick	---/---	16	27
$Al_2O_3(81.1)$-$Cr_2O_3(16.6)$-$P_2O_5(0.8)$-$SiO_2(0.5)$-$Fe_2O_3(0.5)$-$Na_2O(0.5)$, sintered brick	852Z (Serv-M)/Taylor Refractories	7	17
$Al_2O_3(67)$-$Cr_2O_3(32)$-$P_2O_5(1)$, plastic	---/---	10	30
$Al_2O_3(67)$-$Cr_2O_3(32)$, sintered brick	LT5/Union Carbide	10	--[f]
$MgO(61)$-$Cr_2O_3(19)$-$Fe_2O_3(11)$-$Al_2O_3(6)$-$SiO_2(1)$-$CaO(2)$, sintered brick	GR-224/General Refractories	11	14
$MgO(55)$-$Cr_2O_3(20)$-$Al_2O_3(8)$-$FeO(11)$-$SiO_2(2.5)$-$CaO(0.5)$-$TiO_2(1.5)$, sintered brick	Corhart RFG/Corhart Refractories	23	27
$MgO(41.6)$-$Cr_2O_3(27.1)$-$Al_2O_3(13.3)$-$Fe_2O_3(15.9)$-$CaO(0.5)$-$SiO_2(1.2)$, sintered brick	---/---	14	18
$Al_2O_3(52.5)$-$Cr_2O_3(20.6)$-$C(9.1)$-$Si(9.6)$-$P_2O_5(2.8)$-$SiO_2(4.1)$, carbon-impregnated brick	---/---	--[h]	--[f]
$Al_2O_3(38.1)$-$Cr_2O_3(25.2)$-$C(17.9)$-$Si(9.4)$-$SiO_2(8.2)$, carbon-impregnated brick	---/---	--[h]	--[f]
$Al_2O_3(99)$-$Na_2O(0.5)$, fused-cast brick	Monofrax A1/Carborundum	18	24
$Cr_2O_3(80)$-$MgO(8)$-$Fe_2O_3(6)$-$Al_2O_3(5)$-$SiO_2(1)$, fused-cast brick	Monofrax E/Carborundum	3	4
$Al_2O_3(60)$-$Cr_2O_3(27)$-$MgO(6)$-$Fe_2O_3(4)$-$SiO_2(2)$, fused-cast brick	Monofrax K-3/Carborundum	7	9
$Al_2O_3(65)$-$Cr_2O_3(32)$-$FeO(1)$-$CaO(0.6)$-$MgO(0.6)$, fused-cast brick	Corhart AC-28/Corhart Refractories	5	7

[a] Exposure to slag attack took place in the 24-in. diameter, 13-in. high furnace used in the tests in Section B.1.2.14. The furnace was modified to produce prototypic thermal gradients within the refractory bricks through the use of water-cooled chills. The basic brick test specimens were 9-in. x 4.5-in. x 3.2-in. wedges mounted around the circumference of the vessel with the small end of the brick exposed to the slag. Most specimens were manufacturer-supplied brick. Some ramming mixes and castables were tested; these were formed in a mold, dried, and prefired in situ to 1500 °C prior to adding the slag. In performing these tests in which the bricks were water-cooled, the bricks were cut so as to provide three different lengths so that there were different thermal gradients operating. The furnace was fired with natural gas, air, and oxygen to a temperature ∿1500 °C. Oxygen partial pressure was maintained at 10^{-2} to 10^{-3} Pa by adjusting the ratio of the three component gases. Some rotary motion of the slag was achieved by the tangential impingement of the flames from three burners on the surface of the melt. Since the slag was heated from the top, there was a strong vertical temperature gradient and most of the slag attack occurred at the slag line. The furnace cavity was charged with up to 75 kg of synthetic slag. For some tests the slag was added in two or three charges at different times during the test period giving more than one slag attack line. The removal and penetration values in the table are the maximum values of all attack lines of all length bricks of a material. Wet chemical analyses were performed on samples of slag drawn at intervals of 100 hours or less during the course of the tests. The slag compositions given above are the averages of all the analyses.

[b] Depth is measured from the original hot face; the values given are the maximum values reported for each material, the maximum for all length bricks for all slag attack lines. The values are frequently those for the longest bricks, those specimens with the least cooling effect of the chills.

[c] These values are from a table heading "Depth of Obvious Penetration" instead of "Maximum Depth of Penetration".

[d] Run terminated prematurely due to a temperature excursion resulting from a thermocouple failure.

[e] Visual evaluation was uncertain.

[f] Measurement is in doubt or was impossible.

[g] Measurement on one specimen was impossible because the refractory fractured on removal from the furnace.

[h] Specimens were removed after 36 hours because of extreme corrosion.

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EFFECT OF EXPOSURE TO COAL GASIFICATION PILOT PLANT CONDITIONS[a] ON THE BONDING OF REFRACTORIES[b]
AS MEASURED BY ABRASION TESTING[c][12,90]

Refractory (Brand Name)	Exposure[d]	Ambient Temp. Volume Loss[e]	% Change	1400 °F Volume Loss[e]	% Change	Ambient Temp. Rod Penetration[f]	% Change	1400 °F Rod Penetration[f]	% Change
Two exposures in gasifier off-gas (SYNTHANE): reducing gas, H_2O, CO_2, CO, H_2, H_2S, ash and char (small amounts); 800-1500 °F (average 1290 °F), 600 psi, operational time 781.8 hours (first exposure), 181 hours (second exposure).									
90+% Al_2O_3 dense castable, CA bond[g] (Castolast G)	U	0.50		0.20		2.0			
	E1	0.85	+70.	0.33	+65.				
	E2	0.79	+58.			1.7	-15.		
60% Al_2O_3 dense castable, CA bond (Mizzou Castable)	U	1.54		0.63		2.9			
	E1	0.68	-56.	0.45	-29.				
	E2	0.80	-48.			1.02	-65.		
90+% Al_2O_3 light castable, CA bond (Purolite 30)	U	17.9		24.7					
	E1	disintegrated, could not be tested							
90+% Al_2O_3 light castable, CA bond (Greencast-97L)	U	6.48		4.18					
	E1	7.04	+8.6(+74)[h]	5.01	+20.				
54% Al_2O_3 dense castable, CA bond (Super Brikcast A)	U	2.67				4.6			
	E2	0.58	-78.			1.14	-75.		
54% Al_2O_3 lightweight insulating castable, CA bond (Litecast 75-28)	U	4.11		4.42		7.9			
	E1	7.13	+73.	7.07	+60.				
	E2	12.44	+203.			19.9	+152.		
70% Al_2O_3 (mullite), vitreous bond high-fired brick (Mul-8)	U	0.70		0.56		1.5			
	E1	0.68	- 2.9	0.61	+ 8.9				
	E2	0.59	-15.			0.69	-54.		
85% Al_2O_3 phosphate bond, dense fired brick (Chemal 85 B)	U	0.28		0.14		0.9			
	E1	0.33	+18.	0.24	+71.				
	E2	0.37	+32.			0.71	-21.		
90+% Al_2O_3 self-bonded dense fired brick (Kricor)	U	0.61		0.38		1.5			
	E1	0.79	+30.	0.56	+47.				
	E2	0.80	+31.			1.44	- 4.0		
99+% Al_2O_3 self-bonded dense fired brick (H-W Corundum)	U	0.94		0.45		2.4			
	E1	0.78	-17.	0.65	+44.				
	E2	1.13	+20.			1.54	-36.		
77% Al_2O_3 self-bonded light insulating fired brick (B&W Insalcor)	U	4.83		4.03					
	E1	14.2	+194.[i]	10.4	+158.				
98+% Al_2O_3 self-bonded light insulating fired brick (Alfrax B 101)	U	4.37		3.12					
	E1	9.09	+108.[i]	5.12	+64.				
99+% Al_2O_3 self-bonded dense fused-cast brick (Monofrax A)	U	0.36		0.17		2.4			
	E1	0.33	- 8.3	0.10	-41.				
	E2	0.22	-39.			0.77	-68.		
SiC, silicon nitride bonded, dense fired brick (Refrax 20)	U	0.13		0.14		0.9			
	E1	8.98	+6808.	---[j]					
	E2	2.86	+2200.[k]			4.3	+378.		
Alumina-zirconia-silica fused-cast brick (AZS)	U	0.04				0.7			
	E2	0.50	+1150.			0.89	+27.		
60% Al_2O_3 tar-impregnated vitreous fired brick (Ufala TI)	U	0.48				2.1			
	E2	0.66	+38.			0.71	-66.		
refired Ufala TI[l]	E2	0.57	+19.			1.22	-42.		
45-50% Al_2O_3 clay vitreous bond, high-fired super-duty brick (KX-99)	U	0.70				1.9			
	E2	0.79	+13.			0.97	-49.		
refired KX-99[l]	E2	0.67	- 4.3			2.0	+ 5.3		
60% Al_2O_3 phosphate-bonded ramming mix (Wasp #60)	U	2.69		1.01					
	E1	2.00	-26.	1.76	+68.[k]				

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(Table Continued)

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EFFECT OF EXPOSURE TO COAL GASIFICATION PILOT PLANT CONDITIONS[a] ON THE BONDING OF REFRACTORIES[b]
AS MEASURED BY ABRASION TESTING[c][12,90], Continued

Refractory (Brand Name)	Expo-sure[d]	Ambient Temp. Volume Loss[e]	% Change	1400 °F Volume Loss[e]	% Change	Ambient Temp. Rod Pene-tration[f]	% Change	1400 °F Rod Pene-tration[f]	% Change
Two exposures in gasifier off-gas (SYNTHANE), continued									
90% Al_2O_3 phosphate-bonded ramming mix (Brikram 90)	U	1.19		0.34		2.6			
	E1	1.05	-12.	0.35	+ 2.9				
	E2	0.79	-34.			1.9	-27.		
52% Al_2O_3 light castable, CA bond (Litecast 80)	U	7.38		4.18					
	E1	14.6	+98.	13.9	+233.				
70+% Al_2O_3 vitreous fired brick (Hi Lumite 70 D)	U	1.98[j]		1.07[j]					
	E1	---[j]		---[j]					
85% Al_2O_3 phosphate-bonded fired brick (Altex 85-B)	U	0.22		---		1.1			
	E1	0.45	+105.	0.31					
	E2	0.42	+91.			2.1	+91.		
80+% Al_2O_3 phosphate-bonded fired brick (Alumex P-8)	U	0.33		---		1.1			
	E1	0.66	+100.	0.41					
	E2	0.51	+55.			1.2	+ 9.1		
90+% Al_2O_3 phosphate-bonded castable (Resco Cast AA-22)	U	1.00		0.35		2.2			
	E1	0.94	- 6.0	0.45	+29.				
	E2	1.32	+32.			2.2	0		
SiC, oxynitride bond, coupons/bricks (Crystolon 63)	U	0.31				1.2			
	E2	3.78	+1119.			1.80	+50.		
73% Al_2O_3 chemical bond ramming mix (Blu Ram)	U	1.32				2.06			
	E2	0.79	-53.[k]			1.25	-39.		
73% Al_2O_3 chemical bond ramming mix (Blu Ram H.S.)	U	1.34				1.74			
	E2	1.68	+25.			2.44	+40.		
One exposure in the fluidized bed of the gasifier (SYNTHANE): reducing gas as in above, plus coal fines, ash and char; 800-1650 °F (average 1434 °F), 600 psi, operational time 181 hours.									
90+% Al_2O_3 dense castable, CA bond (Castolast G)	U	0.50				2.0			
	E	0.53	+ 6.0			1.0	-50.		
60% Al_2O_3 dense castable, CA bond (Mizzou Castable)	U	1.54				2.9			
	E	0.33	-79.			0.9	-69.		
90+% Al_2O_3 light castable, CA bond (Greencast-97L)	U	6.48				11.2			
	E	3.31	-49.			9.2	-18.		
54% Al_2O_3 dense castable, CA bond (Super Brikcast A)	U	2.67				4.6			
	E	0.49	-82.			0.7	-85.		
54% Al_2O_3 lightweight insu-lating castable, CA bond (Litecast 75-28)	U	4.11				7.9			
	E	3.36	-18.			21.6	+173.		
70% Al_2O_3 (mullite), vitre-ous bond high-fired brick (Mul-8)	U	0.70				1.5			
	E	0.47	-33.			0.7	-53.		
85% Al_2O_3 phosphate bond, dense fired brick (Chemal 85 B)	U	0.28				0.9			
	E	0.32	+14.			0.6	-33.		
90+% Al_2O_3 self-bonded dense fired brick (Kricor)	U	0.61				1.5			
	E	0.57	- 6.7			0.9	-40.		
99+% Al_2O_3 self-bonded dense fired brick (H-W Corundum)	U	0.94				2.4			
	E	1.04	+11.			1.1	-54.		
77% Al_2O_3 self-bonded light insulating fired brick (B&W Insalcor)	U	4.83				8.8			
	E	7.36	+52.			11.8	+34.		
98+% Al_2O_3 self-bonded light insulating fired brick (Alfrax B 101)	U	4.37				7.2			
	E	4.67	+ 6.9			7.4	+ 2.8		

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(Table Continued)

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EFFECT OF EXPOSURE TO COAL GASIFICATION PILOT PLANT CONDITIONS[a] ON THE BONDING OF REFRACTORIES[b]
AS MEASURED BY ABRASION TESTING[c][12,90], Continued

Refractory (Brand Name)	Exposure[d]	Ambient Temp. Volume Loss[e]	% Change	1400 °F Volume Loss[e]	% Change	Ambient Temp. Rod Penetration[f]	% Change	1400 °F Rod Penetration[f]	% Change
One exposure in the fluidized bed of the gasifier (SYNTHANE), continued									
99+% Al_2O_3 self-bonded dense fused-cast brick (Monofrax A)	U	0.36				1.3			
	E	0.26	−28.			0.4	−69.		
SiC, silicon nitride bonded, dense fired brick (Refrax 20)	U	0.13				0.9			
	E	crumbled				poor specimen			
Zirconium silicate, vitreous bond coupon (Zircon)	U	1.24				2.6			
	E	1.79	+44.			2.7	+ 3.8		
60% Al_2O_3 tar-impregnated vitreous fired brick (Ufala TI)	U	0.48				2.1			
	E	0.63	+31.			0.7	−67.		
45-50% Al_2O_3 clay vitreous bond, high-fired super-duty brick (KX-99)	U	0.70				1.9			
	E	0.66	− 5.7			0.9	−53.		
60% Al_2O_3 phosphate-bonded ramming mix (Wasp #60)	U	2.69				4.1			
	E	2.24	−17.			2.4	−41.		
90% Al_2O_3 phosphate-bonded ramming mix (Brikram 90)	U	1.19				2.6			
	E	3.03	+155.			0.7	−73.		
70+% Al_2O_3 vitreous fired brick (Hi Lumite 70 D)	U	1.98				5.0			
	E	3.11	+57.			4.0	−20.		
85% Al_2O_3 phosphate-bonded fired brick (Altex 85-B)	U	0.22				1.1			
	E	0.44	+100.			0.4	−64.		
80+% Al_2O_3 phosphate-bonded fired brick (Alumex P-8)	U	0.33				1.1			
	E	0.53	+61.			0.5	−55.		
90+% Al_2O_3 phosphate-bonded castable (Resco Cast AA-22)	U	1.00				2.2			
	E	1.25	+25.			1.4	−36.		
SiC, oxynitride bond, coupons/bricks (Crystolon 63)	U	0.31				1.2			
	E	poor specimen				poor specimen			

Three exposures in gasifier off-gas (Conoco, CO₂ Acceptor): 48 H_2, 23 H_2O, 12 CH_4, 8.5 CO, 6 CO_2, 2.5 N_2 (all vol. %); 1500 °F, 150 psig, ~800 hours (exposure 1), 1000-2400 hours (exposure 2), 740-1450 hours (exposure 3).

Refractory (Brand Name)	Exposure	Ambient Temp. Volume Loss	% Change	1400 °F Volume Loss	% Change	Ambient Temp. Rod Penetration	% Change	1400 °F Rod Penetration	% Change
90+% Al_2O_3 dense castable, CA bond (Castolast G)	U	0.50		0.20		0.20		0.17	
	E1	0.56	+12.	0.24	+20.	0.12	−40.	0.07	−59.
	E2	0.68	+36.	0.47	+135.				
	E3	0.89	+78.	0.37	+85.				
60% Al_2O_3 dense castable, CA bond (Mizzou Castable)	U	1.54		0.63		0.29		0.13	
	E2	0.76	−51.	0.45	−29.				
	U	1.54		0.29[m]					
	E3	1.21	−22.	0.40	+38.				
90+% Al_2O_3 light castable, CA bond (Purolite 30)	U	17.9		24.7		1.87		3.48	
	E1	15.9	−11.	47.1	+91.	2.36	+26.	6.67	+92.
	E2	31.2	+74.	26.9	− 8.9				
90+% Al_2O_3 light castable, CA bond (Greencast-97L)	U	6.48		4.18		1.12		0.88	
	E1	3.69	−43.	2.20	−47.	0.76	−32.	0.45	−49.
	E2	3.93	−39.	3.18	−24.				
	E3	6.72	− 3.8	6.83	+63.				
54% Al_2O_3 dense castable, CA bond (Super Brikcast A)	U	2.67		1.00		0.46		0.39	
	E3	1.01	−58.[k]	0.38	−62.				
54% Al_2O_3 lightweight insulating castable, CA bond (Litecast 75-28)	U	4.11		4.42		0.79		1.00	
	E2	7.29	+77.	6.39	+45.				
	E3	19.3	+370.	15.0	+240.				

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(Table Continued)

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EFFECT OF EXPOSURE TO COAL GASIFICATION PILOT PLANT CONDITIONS[a] ON THE BONDING OF REFRACTORIES[b]
AS MEASURED BY ABRASION TESTING[c][12,90], Continued

Refractory (Brand Name)	Expo-sure[d]	Ambient Temp. Volume Loss[e]	% Change	1400 °F Volume Loss[e]	% Change	Ambient Temp. Rod Pene-tration[f]	% Change	1400 °F Rod Pene-tration[f]	% Change
Three exposures in gasifier off-gas (Conoco, CO_2 Acceptor), continued									
70% Al_2O_3 (mullite), vitre-ous bond high-fired brick (Mul-8)	U	0.70		0.56		0.15		0.10	
	E1	0.97	+39.	0.52	− 7.1	0.24	+60.	0.13	+30.
	E2	0.65	− 7.1	0.30	−46.				
	E3	0.80	+14.	0.55	− 1.8				
85% Al_2O_3 phosphate bond, dense fired brick (Chemal 85 B)	U	0.28		0.14		0.09		0.05	
	E1	0.32	+14.	0.17	+21.(+2.1)[h]	0.09	0.	0.06	+20.
	E2	0.57	+104.	0.39	+180.				
	E3	0.31	+11.	0.20	+43.				
90+% Al_2O_3 self-bonded dense fired brick (Kricor)	U	0.61		0.38		0.15		0.14	
	E1	0.76	+24.	0.36	− 5.3	0.19	+27.	0.09	−36.
	E2	0.59	− 3.3	0.33	−15.				
99+% Al_2O_3 self-bonded dense fired brick (H-W Corundum)	U	0.94		0.45		0.24		0.10	
	E1	1.14	+21.	0.63	+40.	0.27	+13.	0.13	+30.
	E2	1.09	+16.	0.70	+56.				
	E3	1.69	+80.	0.84	+87.				
77% Al_2O_3 self-bonded light insulating fired brick (B&W Insalcor)	U	4.83		4.03		0.88		0.64	
	E1	4.80 (6.15)[h]	− 0.6 (−27.)[h]	9.19 (7.86)[h]	+128. (+95.)[h]	1.05	+19.	1.30	+103.
	E2	7.60	+57.	10.1	+150.				
	U	4.83		0.88[m]					
	E3	8.13	+68.	6.76	+670.				
98+% Al_2O_3 self-bonded light insulating fired brick (Alfrax B 101)	U	4.37		3.12		0.72		0.54	
	E1	4.75	+ 8.7	3.71	+19.	0.80	+11.	1.03	+91.
	E2	5.86	+57.[k]	6.22	+99.				
	E3	4.49	+ 2.7	2.42	−22.				
99+% Al_2O_3 self-bonded dense fused-cast brick (Monofrax A)	U	0.36		0.17		0.13		0.11	
	E1	0.18	−50.	0.19	+12.	0.10	−23.	0.04	−64.
	E2	0.33	− 8.3	0.13	−24.				
	E3	0.22	−39.	0.16	− 0.6[k]				
SiC, silicon nitride bonded, dense fired brick (Refrax 20)	U	0.13		0.14		0.09		0.16	
	E1	0.28	+115.	0.14	0.[k]	0.13	+44.	0.08	−50.
	E2	1.89	+1350.[k]	1.62	+1150.[k]				
	E3	1.03	+760.[k]	--					
70% Al_2O_3 (mullite), vitre-ous dense fired brick (Alumex 70-HD)	U	1.02		0.48		0.20		0.10	
	E3	1.53	+50.	0.55	+16.[k]				
Zirconium silicate, vitre-ous bond coupon (Zircon)	U	1.24		0.70		0.26		0.15	
	E3	2.21	+79.	1.52	+120.				
60% Al_2O_3 tar-impregnated vitreous fired brick (Ufala TI)	U	0.48		--		0.21		--	
	E3	0.65	+35.	0.47	--				
45–50% Al_2O_3 clay vitreous bond, high-fired super-duty brick (KX-99)	U	0.70		0.35		0.19		0.10	
	E3	0.69	− 1.4	0.43	+23.				
60% Al_2O_3 phosphate-bonded ramming mix (Wasp #60)	U	2.69		1.01		0.41		0.16	
	E1	specimens cracked							
	E2	1.78	−34.	1.47	+75.[k]				
	E3	2.38	−12.	1.75	+73.				
90% Al_2O_3 phosphate-bonded ramming mix (Brikram 90)	U	1.19		0.34		0.26		0.10	
	E1	specimens cracked							
	E2	0.66	−45.	0.51	+50.				
	E3	0.84	−29.	0.52	+53.				
52% Al_2O_3 light castable, CA bond (Litecast 80)	U	7.38		4.18		1.19		0.61	
	E1	specimens cracked							
	E2	16.1	+118.	36.1	+164.[k]				

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(Table Continued)

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EFFECT OF EXPOSURE TO COAL GASIFICATION PILOT PLANT CONDITIONS[a] ON THE BONDING OF REFRACTORIES[b]
AS MEASURED BY ABRASION TESTING[c][12,90], Continued

Refractory (Brand Name)	Expo-sure[d]	Ambient Temp. Volume Loss[e]	% Change	1400 °F Volume Loss[e]	% Change	Ambient Temp. Rod Penetration[f]	% Change	1400 °F Rod Penetration[f]	% Change
Three exposures in gasifier off-gas (Conoco, CO_2 Acceptor), continued									
90+% Al_2O_3 dense castable, CA bond (Greencast-97)	U	0.60		0.60		0.24		0.06	
	E1	1.98	+230.	1.07	+78.	0.31	+29.	0.15	+150.
70+% Al_2O_3 vitreous fired brick (Hi Lumite 70 D)	U	1.98		1.07		0.50		0.16	
	E1	3.43	+173.[k]	3.41	+220.	0.57	+14.	0.43	+169.
	E2	1.85	− 6.6	0.76	−29.				
85% Al_2O_3 phosphate-bonded fired brick (Altex 85-B)	U	0.22		0.26		0.11		0.06	
	E1	0.33	+50.	0.23	−12.	0.11	0.	0.09	+50.
80+% Al_2O_3 phosphate-bonded fired brick (Alumex P-8)	U	0.33		0.29		0.11		0.08	
	E1	0.38	+15.	0.21	−28.	0.11	0.	--	
	U	0.33		0.30					
	E2	0.55	+67.	0.29	− 3.3				
90% Al_2O_3/10% Cr_2O_3 solid solution fired brick (CS 612)	U	0.49		0.37		0.13		--	
	E1	0.71	+45.	0.44	+18.	0.14	+ 7.7		
90+% Al_2O_3 phosphate-bonded castable (Resco Cast AA-22)	U	1.00		0.35		0.22		0.11	
	E3	2.55	+155.	1.81	+420.				
SiC, oxynitride bond, coupons/bricks (Crystolon 63)	U	0.31		0.21		0.12		0.09	
	E3	1.42	+360.	--					
Three exposures in dolomite regenerator off-gas (Conoco, CO_2 Acceptor): 70 N_2, 27 CO_2, 3 CO, H_2S (trace) (in vol. %); 1850 °F, 150 psi, ∿800 hours (exposure 1), 1000-2400 hours (exposure 2), 740-1450 hours (exposure 3).									
90+% Al_2O_3 dense castable, CA bond (Castolast G)	U	0.50		0.20		0.20		0.17	
	E1	0.43	−14.	0.40	+100.	0.16	−20.	0.09	−47.
	E2	0.56	+12.	0.35	+75.	0.15	−25.	0.08	−53.
	E3	0.66	+32.	0.24	+20.				
60% Al_2O_3 dense castable, CA bond (Mizzou Castable)	U	1.54		0.63		0.29		0.13	
	E2	4.52	+193.	2.52	+300.	0.62	+114.	0.37	+185.
	U	1.54		0.29[m]					
	E3	1.63	+ 5.8	0.76	+160.				
90+% Al_2O_3 light castable, CA bond (Purolite 30)	U	17.9		24.7		1.87		3.48	
	E1	95.7	+430.	fractured		10.7	+472.	broke up	
90+% Al_2O_3 light castable, CA bond (Greencast-97L)	U	6.48		4.18		1.12		0.88	
	E1	4.11	−37.	3.09	−26.	0.73	−35.	0.52	−41.
	E2	6.05	− 6.6	4.19	+ 0.2	0.95	−15.	0.73	−17.
	E3	13.0	+101.	14.4	+240.				
54% Al_2O_3 dense castable, CA bond (Super Brikcast A)	U	2.67		1.00		0.46		0.39	
	E3	1.34	−50.	0.87	−13.				
54% Al_2O_3 lightweight insulating castable, CA bond (Litecast 75-28)	U	4.11		4.42		0.79		1.00	
	E2	15.6	+280.	13.1	+196.	3.25	+311.	2.71	+171.
	E3	16.3	+300.	19.8	+350.				
70% Al_2O_3 (mullite), vitreous bond high-fired brick (Mul-8)	U	0.70		0.56		0.15		0.10	
	E1	0.61	−13.	0.24	−57.	0.18	+20.	0.07	−30.
	E2	0.49	−30.	0.32	−43.	0.11	−27.	0.07	−30.
85% Al_2O_3 phosphate bond, dense fired brick (Chemal 85 B)	U	0.28		0.14		0.09		0.05	
	E1	0.33	+18.	0.12	−14.	0.08	−11.	0.02	−60.
	E2	1.08[n]	+290.	0.42[n]	+200.	0.17[n]	+89.	0.09[n]	+80.
	E3	0.48	+71.	0.47	+240.				
90+% Al_2O_3 self-bonded dense fired brick (Kricor)	U	0.61		0.38		0.15		0.14	
	E1	0.64	+ 5.0	0.28	−26.	0.15	0.	0.08	−43.
	E3	0.47	−23.	0.19	+50.				

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(Table Continued)

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EFFECT OF EXPOSURE TO COAL GASIFICATION PILOT PLANT CONDITIONS[a] ON THE BONDING OF REFRACTORIES[b]
AS MEASURED BY ABRASION TESTING[c][12,90], Continued

Refractory (Brand Name)	Expo-sure[d]	Ambient Temp. Volume Loss[e]	% Change	1400 °F Volume Loss[e]	% Change	Ambient Temp. Rod Pene-tration[f]	% Change	1400 °F Rod Pene-tration[f]	% Change
Three exposures in dolomite regenerator off-gas (Conoco, CO_2 Acceptor), continued									
99+% Al_2O_3 self-bonded dense fired brick (H-W Corundum)	U	0.94		0.45		0.24		0.10	
	E1	1.43 (1.14)[h]	+52. (+21.)[h]	0.77 (0.63)[h]	+71. (+40.)[h]	0.28	+17.	0.18	+80.
	E2	1.02	+ 8.5	0.27	-40.	0.24	0.	0.08	-20.
	E3	1.24	+32.	0.56	+24.				
77% Al_2O_3 self-bonded light insulating fired brick (B&W Insalcor)	U	4.83		4.03		0.88		0.64	
	E1	4.80 (6.15)[h]	- 0.62 (+27.)[h]	9.19 (7.86)[h]	+128. (+95.)[h]	0.79	-10.	0.68	+ 6.3
	E2	8.98	+86.	4.00	- 0.7	1.48	+68.	0.59	- 7.8
	U	4.83		0.88[m]					
	E3	4.04	-16.	1.77	+360.[k]				
98+% Al_2O_3 self-bonded light insulating fired brick (Alfrax B 101)	U	4.37		3.12		0.72		0.54	
	E1	3.85	-12.	3.58	+15.	0.72	0.	0.66	+22.
	E2	6.42	+47.	5.09	+63.	1.13	+57.	1.07	+98.
	E3	4.37	0.	4.65	+49.				
99+% Al_2O_3 self-bonded dense fused-cast brick (Monofrax A)	U	0.36		0.17		0.13		0.11	
	E1	0.22	-39.	0.14	-18.	0.13	0.	0.06	-45.
	E2	0.14	-61.	0.13	-24.	0.07	-46.	0.04	-64.
	E3	0.19	-47.	0.08	-53.				
SiC, silicon nitride bonded, dense fired brick (Refrax 20)	U	0.13		0.14		0.09		0.16	
	E1	0.12	- 7.7	0.09	-36.	0.09	0.	0.05	-69.
	E2	0.15	+15.	0.08	-43.	0.05	-44.	0.06	-63.
	E3	0.15	+15.	specimen crumbled					
70% Al_2O_3 (mullite), vitre-ous dense fired brick (Alumex 70-HD)	U	1.02		0.48		0.20		0.10	
	E3	1.09	+6.9(+69.)[h]	0.24	-50.				
60% Al_2O_3 tar-impregnated vitreous fired brick (Ufala TI)	U	0.48		--		0.21		--	
	E3	0.38	-21.	0.16					
45-50% Al_2O_3 clay vitreous bond, high-fired super-duty brick (KX-99)	U	0.70		0.35		0.19		0.10	
	E3	0.54	-23.	0.26	-26.				
60% Al_2O_3 phosphate-bonded ramming mix (Wasp #60)	U	2.69		1.01		0.41		0.16	
	E1	specimen cracked							
	E2	0.77	-71.	0.88	-13.	0.15	-63.	0.14	-13.
	E3	0.89	-67.	0.47	-54.				
90% Al_2O_3 phosphate-bonded ramming mix (Brikram 90)	U	1.19		0.34		0.26		0.10	
	E1	0.59	-50.	0.28	-18.	0.17	-35.	0.07	-30.
	E2	0.64 (0.34)[h]	-46. (-71.)[h]	0.55 (0.19)[h]	+62. (-44.)[h]	0.09	-65.	0.08	-20.
	E3	0.25	-79.	0.19	-44.				
52% Al_2O_3 light castable, CA bond (Litecast 80)	U	7.38		4.18		1.19		0.61	
	E1	specimen fractured							
	E2	24.0	+225.	45.3	+984.	2.0	+68.	5.92	+870.
90+% Al_2O_3 dense castable, CA bond (Greencast-97)	U	0.60		0.60		0.24		0.06	
	E1	2.15	+260.	1.42	+137.	0.40	+67.	0.26	+333.
70+% Al_2O_3 vitreous fired brick (Hi Lumite 70 D)	U	1.98		1.07		0.50		0.16	
	E1	1.89	- 4.5	0.59	-45.	0.29	-42.	0.08	-50.
	E2	2.20	+11.(+1.1)[h]	0.78	-27.	0.37	-26.	0.15	- 6.3
85% Al_2O_3 phosphate-bonded fired brick (Altex 85-B)	U	0.22		0.26		0.11		0.06	
	E1	0.28	+27.	0.12	-54.	0.10	- 9.	0.05	-17.
80+% Al_2O_3 phosphate-bonded fired brick (Alumex P-8)	U	0.33		0.29		0.11		0.08	
	E1	0.25	-25.	0.17	-41.	0.10	- 9.		
	U	0.33		0.30[m]					
	E2	0.53	+60.	0.30	0.	0.12		0.09	

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(Table Continued)

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EFFECT OF EXPOSURE TO COAL GASIFICATION PILOT PLANT CONDITIONS[a] ON THE BONDING OF REFRACTORIES[b]
AS MEASURED BY ABRASION TESTING[c][12,90], Continued

Refractory (Brand Name)	Exposure[d]	Ambient Temp. Volume Loss[e]	% Change	1400 °F Volume Loss[e]	% Change	Ambient Temp. Rod Penetration[f]	% Change	1400 °F Rod Penetration[f]	% Change
Three exposures in dolomite regenerator off-gas (Conoco, CO_2 Acceptor), continued									
90% Al_2O_3/10% Cr_2O_3 solid solution fired brick (CS 612)	U	0.49		0.37		0.13		--	
	E1	0.55	+12.	0.32	-14.	0.14	+ 7.7		
90+% Al_2O_3 phosphate-bonded castable (Resco Cast AA-22)	U	1.00		0.35		0.22		0.11	
	E3	2.02	+102.	1.99	+470.				
SiC, oxynitride bond, coupons/bricks (Crystolon 63)	U	0.31		0.21		0.12		0.09	
	E3	0.30	- 3.2	0.02	-90.				
Two exposures in gasifier off-gas (Bi-Gas): H_2O, CO_2, CO, H_2, N_2, CH_4, H_2S, ash and char (entrained); 1800 °F, 750 psi, exposure time not given (exposure 1); 1525-1750 °F, 750 psig, 1100 hours in operating conditions (exposure 2).									
54% Al_2O_3 dense castable, CA bond (Super Brikcast A)	U	2.67		--		4.6		--	
	E1	0.90	-66.	0.30		1.9	-59.	0.7	
	U	1.30[m]				2.16[m]			
	E2	1.16	-10.7			2.44	+13.0		
54% Al_2O_3 lightweight insulating castable, CA bond (Litecast 75-28)	U	4.11		--		7.9		--	
	E1	22.1	+438.	9.0		23.2	+194.	9.2	
85% Al_2O_3 phosphate bond, dense fired brick (Chemal 85 B)	U	0.28		--		0.9		--	
	E1	0.39	+39.	0.21		0.7	-22.	0.25	
	U	0.28				0.83[m]			
	E2	0.59	+110.7			1.27	+53.0		
70% Al_2O_3 (mullite), vitreous dense fired brick (Alumex 70-HD)	U	1.06		--		2.1		--	
	E1	1.43	+35.	0.66		2.2	+ 4.8	1.5	
60% Al_2O_3 tar-impregnated vitreous fired brick (Ufala TI)	U	--		--		--		--	
	E1	0.81		0.58		3.1		0.9	
90% Al_2O_3 phosphate-bonded ramming mix (Brikram 90)	U	--		--		--		--	
	E1	1.01		0.40		1.8		0.5	
	U	0.55				1.21			
	E2	0.82	+49.1			0.89	-26.4		
90+% Al_2O_3 phosphate-bonded castable (Resco Cast AA-22)	U	1.00		--		7.2		--	
	E1	2.84	+184.	1.67		3.4	-53.	2.5	
	U	1.13[m]				1.98[m]			
	E2	16.97[o]	+1401.			21.28[o]	+975.		
60% Al_2O_3 dense castable, CA bond, (Mizzou Castable)	U	1.91				2.77			
	E2	2.03	+ 6.2			3.65	+31.8		
90+% Al_2O_3 self-bonded dense fired brick (Kricor)	U	0.61				1.50			
	E2	0.65	+ 6.6			1.38	- 8.0		
Alumina-zirconia-silica fused-cast brick (AZS)	U	0.04				0.7			
	E2	specimen not recovered							
45-50% Al_2O_3 clay vitreous bond, high-fired super-duty brick (KX-99)	U	0.55				1.17			
	E2	0.67	+21.8			1.19	+ 1.7		
One exposure in gasifier off-gas (Battelle): 1800 °F, 100 psi, ∿50 hour exposure.									
60% Al_2O_3 dense castable, CA bond (Mizzou Castable)	U	0.50[p]				0.20[p]			
	E	--				--			
70% Al_2O_3 (mullite), vitreous bond high-fired brick (Mul-8)	U	0.70[p]				0.15			
	E	0.79	+13.			0.15	0.		
85% Al_2O_3 phosphate bond, dense fired brick (Chemal 85 B)	U	0.28[p]				0.09[p]			
	E	0.24	-14.			0.07	-22.		

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(Table Continued)

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EFFECT OF EXPOSURE TO COAL GASIFICATION PILOT PLANT CONDITIONS[a] ON THE BONDING OF REFRACTORIES[b]
AS MEASURED BY ABRASION TESTING[c][12,90], Continued

Refractory (Brand Name)	Expo-sure[d]	Ambient Temp. Volume Loss[e]	% Change	1400 °F Volume Loss[e]	% Change	Ambient Temp. Rod Pene-tration[f]	% Change	1400 °F Rod Pene-tration[f]	% Change
One exposure in gasifier off-gas (Battelle), continued									
SiC, clay-bonded (Harbide)	U	--				--			
	E	0.43				0.01			
90% Al₂O₃ phosphate-bonded ramming mix (Brikram 90)	U	1.19[p]				0.26[p]			
	E	0.55	-54.			0.10	-62.		
80+% Al₂O₃ phosphate-bonded fired brick (Alumex P-8)	U	0.33[p]				0.11[p]			
	E	0.42	+27.			0.08	-27.		
90+% Al₂O₃ phosphate-bonded castable (Resco Cast AA-22)	U	1.00[p]				0.22[p]			
	E	5.44	+444.			0.85	+286.		
One exposure in combustor off-gas (Battelle): 2000 °F, >100 psi, ∿50 hour exposure.									
60% Al₂O₃ dense castable, CA bond (Mizzou Castable)	U	--				--			
	E	0.50[p]				0.20[p]			
SiC, clay-bonded (Harbide)	U	--				--			
	E	0.43				0.06			
60% Al₂O₃ tar-impregnated vitreous fired brick (Ufala TI)	U	0.43[p]				0.17[p]			
	E	0.50	+16.			--			
45-50% Al₂O₃ clay vitreous bond, high-fired super-duty brick (KX-99)	U	0.62[p]				0.16[p]			
	E	0.54	-13.			0.14	-13.		
90% Al₂O₃ phosphate-bonded ramming mix (Brikram 90)	U	1.19[p]				0.26			
	E	0.45	-62.			0.14	-46.		
90+% Al₂O₃ phosphate-bonded castable (Resco Cast AA-22)	U	1.00[p]				0.72[p]			
	E	4.02	+302.			0.63	-13.		
Two exposures in gasifier off-gas (U-GAS): reducing gas, H₂O, CO₂, CO, H₂, CH₄, N₂, H₂S, ash and char (entrained); 1803-1930 °F, 5.5-43 psig, exposure times not given.									
90+% Al₂O₃ dense castable, CA bond (Castolast G)	U	0.50				2.0			
	E1	1.36	+172.			2.17	+ 8.5		
	U	1.31[m]				2.41[m]			
	E2	1.28	- 2.			2.28	- 5.		
60% Al₂O₃ dense castable, CA bond (Mizzou Castable)	U	1.54				2.9			
	E1	1.88	+22.			2.49	-14.1		
	U	0.99[m]				1.74[m]			
	E2	0.81	-18.			1.54	-12.		
54% Al₂O₃ dense castable, CA bond (Super Brikcast A)	U	2.67				4.6			
	E1	1.98	-25.8			3.26	-29.1		
	U	3.01[m]				5.69[m]			
	E2	1.48	-51.			2.81	-51.		
85% Al₂O₃ phosphate bond, dense fired brick (Chemal 85 B)	U	0.28				0.83			
	E1	0.31	+10.			0.84	+ 1.2		
	U	0.34[m]				0.76[m]			
	E2	0.31	- 9.			0.85	+12.		
SiC, silicon nitride bonded, dense fired brick (Refrax 20)	U	0.13				0.9			
	E1	0.25	+92.			0.54	-40.0		
	U	0.23[m]				0.41[m]			
	E2	0.22	- 4.			0.52	+27.		
70% Al₂O₃ (mullite), vitreous dense fired brick (Alumex 70-HD)	U	1.02				2.0			
	E1	1.07	+ 4.9			1.81	- 9.5		
60% Al₂O₃ tar-impregnated vitreous fired brick (Ufala TI)	U	0.39				0.85			
	E2	0.53	+36.			1.25	+47.		

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(Table Continued)

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EFFECT OF EXPOSURE TO COAL GASIFICATION PILOT PLANT CONDITIONS[a] ON THE BONDING OF REFRACTORIES[b]
AS MEASURED BY ABRASION TESTING[c][12,90], Continued

Refractory (Brand Name)	Expo-sure[d]	Site[q]	Ambient Temp. Volume Loss[e]	% Change	1400 °F Volume Loss[e]	% Change	Ambient Temp. Rod Pene-tration[f]	% Change	1400 °F Rod Pene-tration[f]	% Change
Two exposures in gasifier off-gas (U-GAS), continued										
90% Al_2O_3 phosphate-bonded ramming mix (Brikram 90)	U		1.19				2.6			
	E1		0.46	−61.3			1.07	−59.0		
	U		0.95[m]				1.67[m]			
	E2		0.53	−44.			1.22	−27.		
90+% Al_2O_3 phosphate-bonded castable (Resco Cast AA-22)	U		1.00				3.8			
	E1		1.73	+73.			2.86	−24.7		
	U		0.66[m]				1.37[m]			
	E2		1.11	+68.			2.03	+48.		

Three exposures in gasifier off-gas of high-temperature reactor (HYGAS): H_2O, CO, CO_2, H_2, H_2S, CH_4, char; ~1250 °F, 900–1100 psi, exposure times for 1 and 2 not given, exposure 3 >1355 hours.

Refractory(Brand Name)	Expo-sure[d]	Site[q]	Ambient Temp. Volume Loss[e]	% Change	1400 °F Volume Loss[e]	% Change	Ambient Temp. Rod Pene-tration[f]	% Change	1400 °F Rod Pene-tration[f]	% Change
90+% Al_2O_3 dense cast-able, CA bond (Castolast G)	U		0.50		0.20		0.20		0.17	
	E1	0	1.83	+266.	1.38	+590.	0.29	+45.	0.19	+12.
		9	1.24	+148.	0.82	+310.	0.23	+15.	0.09	−47.
	E3	0	0.91	+82.			0.19	− 5.0		
		1	0.68	+36.			0.16	−20.		
		8	0.81	+62.			0.18	−10.		
		9	1.02	+104.			0.16	−20.		
60% Al_2O_3 dense cast-able, CA bond (Mizzou Castable) (2 specimens in E2)	U		1.54		0.63		0.29		0.13	
	E2	0	0.71	−54.	0.36	−43.	0.21	−28.	0.13	0.
		9	1.00	−35.	0.71	+13.	0.24	−17.	0.17	+31.
		0	0.80	−48.	0.29	−54.	0.18	−38.	0.10	−23.
		9	0.70	−55.	0.18	−71.	0.16	−45.	0.09	−31.
	E3	0	0.73	−53.			0.18	−38.		
		1	0.50	−68.			0.15	−48.		
		8	0.68	−56.			0.19	−34.		
		9	0.70	−55.			0.22	−24.		
54% Al_2O_3 dense cast-able, CA bond (Super Brikcast A) (2 specimens in E3)	U		2.67		1.00		0.46		0.39	
	E3	0	0.62	−77.			0.15	−69.		
		1	0.41	−85.			0.14	−70.		
		8	0.53	−80.			0.15	−69.		
		9	0.53	−80.			0.14	−70.		
		0	0.64	−76.			0.18	−61.		
		1	0.69	−74.			0.17	−63.		
		8	0.57	−79.			0.14	−70.		
		9	0.66	−75.			0.15	−69.		
54% Al_2O_3 lightweight insulating castable, CA bond (Litecast 75-28)	U		4.11		4.42		0.79		1.00	
	E3	0	17.2	+318.			2.37	+200.		
		1	11.1	+170.			1.75	+122.		
		8	14.3	+248.			2.24	+184.		
		9	10.8	+163.			1.74	+120.		
70% Al_2O_3 (mullite), vitreous bond high-fired brick (Mul-8)	U		0.70		0.56		0.15		0.10	
	E1	0	0.66	− 5.7	1.05	+88.	0.18	+20.	0.22	+120.
		9	0.82	+17.	0.70	+25.	0.16	+ 6.7	0.16	+60.
	E2	0	0.87	+24.	0.67	+20.	0.24	+60.	0.16	+60.
		9	0.77	+10.	0.58	+ 3.6	0.19	+27.	0.13	+30.
	E3	0	1.04	+49.			0.21	+40.		
		1	0.74	+ 5.7			0.18	+20.		
		8	0.58	−17.			0.15	0.		
		9	0.59	−16.			0.14	− 6.7		

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(Table Continued)

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EFFECT OF EXPOSURE TO COAL GASIFICATION PILOT PLANT CONDITIONS[a] ON THE BONDING OF REFRACTORIES[b]

AS MEASURED BY ABRASION TESTING[c][12,90], Continued

Refractory(Brand Name)	Expo- sure[d]	Site[q]	Ambient Temp. Volume Loss[e]	% Change	1400 °F Volume Loss[e]	% Change	Ambient Temp. Rod Penetration[f]	% Change	1400 °F Rod Penetration[f]	% Change
Three exposures in gasifier off-gas of high-temperature reactor (HYGAS), continued										
85% Al$_2$O$_3$ phosphate	U		0.28		0.14		0.09		0.05	
bond, dense fired brick	E1	0	0.42	+50.	0.19	+36.	0.12	+33.	0.06	+20.
(Chemal 85 B)		9	0.39	+39.	0.22	+57.	0.12	+33.	0.06	+20.
	E2	0	0.39	+39.	0.22	+57.	0.12	+33.	0.04	-20.
		9	0.28	0.	0.21	+50.	0.11	+22.	0.06	+20.
	E3	0	0.37	+32.			0.11	+22.		
		1	0.35	+25.			0.11	+22.		
		8	0.31	+11.			0.10	+11.		
		9	0.36	+29.			0.10	+11.		
90+% Al$_2$O$_3$ self-	U		0.61		0.38		0.15		0.14	
bonded dense fired	E1	0	0.69	+13.	0.51	+34.	0.17	+13.	0.16	+14.
brick (Kricor)		9	0.47	-23.	0.21	-45.	0.11	-27.	0.07	-50.
	E2	0	0.78	+28.	0.38	0.	0.17	+13.	0.09	-36.
		9	0.77	+26.	0.47	+24.	0.18	+20.	0.09	-36.
	E3	0	0.82	+34.			0.19	+27.		
		1	0.73	+20.			0.17	+13.		
		8	0.60	- 1.6			0.17	+13.		
		9	0.61	0.			0.14	- 6.7		
99+% Al$_2$O$_3$ self-	U		0.94		0.45		0.24		0.10	
bonded dense fired	E1	0	0.93	- 1.1	0.77	+71.	0.18	-25.	0.12	+20.
brick (H-W Corundum)		9	0.87	- 7.4	0.43	- 4.4	0.20	-17.	0.13	+30.
	E2	0	0.82	-13.	0.25	-44.	0.17	-29.	0.08	-20.
		9	1.20	+28.	0.75	+67.	0.21	-13.	0.13	+30.
	E3	0	1.22	+30.			0.24	0.		
		1	1.08	+15.			0.22	- 8.3		
		8	1.09	+16.			0.22	- 8.3		
		9	0.95	+ 1.1			0.20	-17.		
77% Al$_2$O$_3$ self-	U		4.83		4.03		0.88		0.64	
bonded light insulat-	E1	0	7.63	+58.	6.23	+55.	1.26	+43.	0.91	+42.
ing fired brick (B&W		9	6.75	+40.	8.18	+103.	1.16	+32.	1.29	+102.
Insalcor)										
98+% Al$_2$O$_3$ self-	U		4.37		3.12		0.72		0.54	
bonded light insulat-	E1	0	4.50	+ 3.0	3.68	+18.	0.77	+ 6.9	0.66	+22.
ing fired brick		9	4.44	+ 1.6	3.89	+25.	0.82	+14.	0.66	+22.
(Alfrax B 101)										
70% Al$_2$O$_3$ (mullite),	U		1.02		0.48		0.20		0.10	
vitreous dense fired	E3	0	0.93	- 8.8			0.19	- 5.0		
brick (Alumex 70-HD)		1	0.72	-29.			0.16	-20.		
		8	0.93	- 8.8			0.19	- 5.0		
		9	0.99	- 2.9			0.21	+ 5.0		
60% Al$_2$O$_3$ tar-impreg-	U		0.48				0.21			
nated vitreous fired	E3	0	0.43	-10.			0.12	-43.		
brick (Ufala TI)		1	0.49	+ 2.1			0.14	-33.		
		8	0.39	-19.			0.12	-43.		
		9	0.47	- 2.1			0.13	-38.		
45-50% Al$_2$O$_3$ clay	U		0.70		0.35		0.19		0.10	
vitreous bond, high-	E3	0	0.82	+17.			0.18	- 5.3		
fired super-duty		1	0.71	+ 1.4			0.16	-16.		
brick (KX-99)		8	0.71	+ 1.4			0.17	-11.		
		9	0.76	+ 8.6			0.19	0.		
90% Al$_2$O$_3$ phosphate-	U		1.19		0.34		0.26		0.10	
bonded ramming mix	E2	0	0.91	-24.	0.49	+44.	0.20	-23.	0.12	+20.
(Brikram 90)		9	0.67	-44.	0.39	+15.	0.16	-38.	0.11	+10.
(2 specimens		0	1.10	- 7.6	0.62	+82.	0.25	- 3.8	0.10	0.
in E2)		9	0.64	-46.	0.55	+62.	0.15	-42.	0.09	-10.
	E3	0	0.56	-53.			0.14	-46.		
		1	1.05	-12.			0.21	-19.		
		8	0.79	-34.			0.17	-35.		
		9	0.54	-55.			0.13	-50.		

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(Table Continued)

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EFFECT OF EXPOSURE TO COAL GASIFICATION PILOT PLANT CONDITIONS[a] ON THE BONDING OF REFRACTORIES[b]

AS MEASURED BY ABRASION TESTING[c][12,90], Continued

Refractory(Brand Name)	Expo-sure[d]	Site[q]	Ambient Temp. Volume Loss[e]	% Change	1400 °F Volume Loss[e]	% Change	Ambient Temp. Rod Penetration[f]	% Change	1400 °F Rod Penetration[f]	% Change
Three exposures in gasifier off-gas of high-temperature reactor (HYGAS), continued										
90+% Al_2O_3 dense	U		0.60		0.60		0.24		0.06	
castable, CA bond	E2	0	3.83	+538.	broke up		0.50	+108.	broke up	
(Greencast-97)		9	5.48	+813.	4.40	+633.	0.79	+229.	0.69	+1050.
70+% Al_2O_3 vitreous	U		1.98		1.07		0.50		0.16	
fired brick (Hi	E1	0	2.77	+40.	1.76	+64.	0.47	- 6.0	0.24	+50.
Lumite 70 D)		9	1.51	-24.	0.84	-21.	0.29	-42.	0.17	+ 6.3
	E2	0	0.87	-56.	0.67	-37.	0.24	-52.	0.16	0.
		9	0.77	-61.	0.58	-46.	0.19	-62.	0.13	-19.
85% Al_2O_3 phosphate-	U		0.22		0.26		0.11		0.06	
bonded fired brick	E1	0	0.42	+91.	0.37	+42.	0.11	0.	0.09	+50.
(Altex 85-B)		9	0.21	- 4.5	0.27	+ 3.8	0.08	-27.	0.08	+33.
	E2	0	0.28	+27.	0.20	-23.	0.10	- 9.1	0.05	-17.
		9	0.41	+86.	0.28	+ 7.7	0.11	0.	0.07	+17.
80+% Al_2O_3 phosphate-	U		0.33		0.30		0.11		0.08	
bonded fired brick	E1	0	0.49	+48.			0.12	+ 9.1		
(Alumex P-8)		9	0.39	+18.			0.14	+27.		
	E2	0	0.42	+27.	0.32	+ 6.7	0.14	+27.	0.19	+138.
		9	0.58	+76.	0.36	+20.	0.13	+18.	0.14	+75.

[a] Specimens were exposed in pilot plants in gaseous phase atmospheres. Full conditions to which the refractories were subjected were not available in direct relation to the exposure times. Conditions fluctuated during the exposures. Time periods, when given, are only approximate.

[b] Brick specimens were prepared by diamond saw sectioning of commercial refractories supplied by manufacturers. Castables were mixed with amount of distilled water recommended by manufacturers, poured into 9 in. straights in wooden molds, cured in 100% humidity for 48 h, then saw cut, fired to 1500 °F and cooled. All refractories were fired to 1500 °F for 4 h in air before all testing both before and after exposure in the plants. Sample sizes varied for different pilot plants: Synthane and Conoco, 4 1/2 x 2 x 3/4 in.; HYGAS, 2 1/2 x 4 1/2 x 9 in.; Bi-Gas, 4 1/2 x 2 1/2 x 3/4 in.; Battelle 1.6 x 1.6 x 1 in.; U-GAS, 4 1/2 x 2 1/2 x 3/4 in.

[c] Rotating Rod Abrasion Tests were performed to measure any changes in the bond of the refractories due to exposure; test results do not represent abrasion or erosion resistance of refractories under gasification conditions. Procedure consists of rotating the end of a weighted (20 lb.) rod (1 in. diameter, 10 in. long KT-SiC) on the face of a specimen at a determined rpm (36 rpm) for a determined time; abrasive grain (250 grams of SiC, -20 + 35 mesh ASTM) is applied between rod and specimen to intensify the abrasive effect; high temperature tests are performed by heating the specimen to the test temperature, attaining equilibrium in ∿one hour and then rotating the rod.

[d] U = unexposed, E1 = first exposure, E2 = second exposure, etc.

[e] Volume loss calculated from weight loss; expressed as cc per 1000 revolutions of the rod.

[f] Rod penetration is measured by a dial gauge resting on the rod and weight assembly; expressed as dial movement in mm per 1000 revolutions.

[g] CA bond = calcium aluminate bond.

[h] Values in parentheses are conflicting values for the same data reported in different tables of the set of reports.

[i] An incorrect value for this percent change is given in the project final report (obviously confused with data for another material).

[j] Fractured during testing.

[k] Value reported is not consistent with the unexposed and exposed values but it is not possible to determine which of the three values, unexposed, exposed, or percent change, is incorrect.

[l] Specimens were refired to 2640 °F for 4 hours before exposure.

[m] Although the same values are given in the tables for most unexposed samples, these values differed.

[n] A "skin" had formed on the refractory; the values given are for tests with the "skin" still on. After removing the skin, tests were repeated--ambient temp. volume loss 0.64, at 1400 °F, 0.20; ambient temp. rod penetration 0.14, at 1400 °F, 0.07.

[o] Calculated from 550 revolutions, the limit of penetration of the apparatus (0.5 in.).

[p] Average properties of all unexposed specimens evaluated in this exposure.

[q] Hygas samples, 9 x 2 1/2 x 4 1/2 in. bricks, were placed so that the 2 1/2 x 4 1/2 in. face was exposed to the gasifier conditions. Slices, one inch thick, were cut from each end and both sides of each slice were tested after exposure three. Test site is the distance in inches from the hot face of the brick.

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EFFECT OF HEAT TREATMENT AND GASEOUS ENVIRONMENT[a] ON THE PHASES PRESENT IN VARIOUS HIGH-ALUMINA DENSE CASTABLE REFRACTORIES[b][39]

Treatment Conditions				XRD Analysis Results[c]								DTA-TGA Analyses			
Atmosphere[a]	Temperature °C / °F	Pressure MPa/psi	Time days	Alumina	Boehmite	Calcite	Gibbsite	CA	CA₂	C₃AH₆	Aragonite	% Weight Loss	% Boehmite	% Calcite	% CaO, Dry Basis
— 94% ALUMINA[d] —															
Air (dried)	110/ 230	ambient	1	M			tr	tr		tr					
CGA exposure	260/ 500	3.4/500	10	M	M	M					tr	6.0	16.3	8.1	4.8
			20									4.1	7.8	6.7	3.9
			30	M	m	M						5.1	13.3	7.1	4.2
CGA exposure	538/1000	6.9/1000	10	M		M				tr					
			20	M		M									
			30	M		M				tr		3.4	0	6.8	3.9
CGA with H₂S	538/1000	6.9/1000	10	M		M									
			20	M		M									
			30	M		M						3.6	0	7.3	4.3
CGA, H₂O saturated	231/ 447	6.9/1000	30	M	M	M						6.6	22.3	5.5	3.3
CGA with H₂S, H₂O satd.	231/ 447	6.9/1000	30	M	M	M						6.7	22.8	5.0	3.0
CO/H₂O, H₂O saturated	199/ 390	3.2/465	10	M	M	m						5.5	16.1	3.6	2.1
CO/H₂O	260/ 500	3.2/465	10	M	M	M						5.8	14.3	7.8	4.6
	538/1000	3.2/465	10	M		m						3.3	0	6.3	3.6
— 93% ALUMINA[e] —															
Air (dried)	110/ 230	ambient	1	M				m							
CGA exposure	260/ 500	3.4/500	10	M	M	M					m	6.2	20.6	7.0	4.2
			20									4.9	14.3	6.1	3.6
			30	M	m	M					m	5.7	17.7	6.9	4.1
CGA exposure	538/1000	6.9/1000	10	M		M		tr							
			20	M		M									
			30	M		M						3.2	0	6.4	3.7
CGA with H₂S	538/1000	6.9/1000	10	M		M		m							
			20	M		M		tr							
			30	M		M		m				3.4	0	6.4	3.7
CGA, H₂O saturated	231/ 447	6.9/1000	30	M	M	M						5.6	20.3	5.9	3.5
CGA with H₂S, H₂O satd.	231/ 447	6.9/1000	30	M	M	M									
CO/H₂O, H₂O saturated	199/ 390	3.2/465	10	M	M	m						6.2	18.6	4.4	2.6
CO/H₂O	260/ 500	3.2/465	10	M	M	M						6.3	20.7	7.4	4.4
	538/1000	3.2/465	10	M		M						2.3	0	4.1	2.3
— 90-95% ALUMINA[f] —															
Air (dried)	110/ 230	ambient	1	M			m								
CGA exposure	260/ 500	3.4/500	10	M	M	M					tr	5.0	13.5	6.7	3.9
			20									3.8	9.2	5.5	3.2
			30	M	M	M					tr	4.7	11.4	6.7	3.9
CGA exposure	538/1000	6.9/1000	10	M		M									
			20	M		M									
			30									3.1	0	6.4	3.7
CGA with H₂S	538/1000	6.9/1000	10	M		M									
			20	M		M									
			30	M		M						3.5	0	6.9	4.0
CGA, H₂O saturated	231/ 447	6.9/1000	30	not tested											
CGA with H₂S, H₂O satd.	231/ 447	6.9/1000	30	M	M	M									
CO/H₂O, H₂O saturated	199/ 390	3.2/465	10	M	M	m						5.5	17.6	2.8	1.6
CO/H₂O	260/ 500	3.2/465	10	M	M	M						5.3	13.4	7.1	4.2
	538/1000	3.2/465	10	M		M						2.6	0	5.1	3.0

(Table Continued)

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EFFECT OF HEAT TREATMENT AND GASEOUS ENVIRONMENT[a] ON THE PHASES PRESENT IN VARIOUS HIGH-ALUMINA DENSE CASTABLE REFRACTORIES[b][39], Continued

Atmosphere[a]	Temperature °C / °F	Pressure MPa/psi	Time days	Alumina	Boehmite	Calcite	Gibbsite	CA	CA2	C3AH6	Aragonite	% Weight Loss	% Boehmite	% Calcite	% CaO, Dry Basis
— — — — — — — — — — — — — — — 90-95% ALUMINA[g] — — — — — — — — — — — — — — —															
Air (dried)	110/ 230	ambient	1	M					m						
CGA exposure	260/ 500	3.4/500	10	M	M	M						5.7	16.0	7.4	4.3
			20									4.1	9.8	6.0	3.4
			30	M	M	M						5.0	13.2	6.9	4.1
CGA exposure	538/1000	6.9/1000	10	M		M									
			30									3.7	0	7.6	4.5
CGA with H2S	538/1000	6.9/1000	10	M		M									
			20	M		M									
			30	M		M						4.4	0	8.4	4.9
CGA, H2O saturated	231/ 447	6.9/1000	30	M	M	m						6.4	23.9	3.5	2.1
CGA with H2S, H2O satd.	231/ 447	6.9/1000	30	M	M	m									
CO/H2O, H2O saturated	199/ 390	3.2/465	10	M	M	m						5.8	16.7	2.8	1.7
CO/H2O	260/ 500	3.2/465	10	M	M	M						5.6	13.8	7.7	4.6
	538/1000	3.2/465	10	M		M						3.3	0	6.5	3.7
— — — — — — — — — — — — — — — 93% ALUMINA[h] — — — — — — — — — — — — — — —															
Air (dried)	110/ 230	ambient	1	M			m								
CGA exposure	260/ 500	3.4/500	10	M	M	M						5.5	16.5	9.1	5.4
			20	M	m	M						4.4	7.9	7.2	4.2
			30	M	M	M					tr	5.7	13.3	8.3	4.9
CGA exposure	538/1000	6.9/1000	10	M		M						3.9	0	9.0	5.3
			30									3.9	0	8.0	4.5
CGA with H2S	538/1000	6.9/1000	10	M		M						3.7	0	7.5	4.3
			20	M		M						4.1	0	9.4	5.5
			30	M		M						3.7	0	7.8	4.5
CGA, H2O saturated	231/ 447	6.9/1000	30	M	M	M						7.5	29.1	5.2	2.9
CGA with H2S, H2O satd.	231/ 447	6.9/1000	30	M	M	M						6.6	29.1	5.1	3.1
CO/H2O, H2O saturated	199/ 390	3.2/465	10	M	M	m						6.4	19.1	3.5	2.1
CO/H2O	260/ 500	3.2/465	10	M	M	M						6.2	14.4	8.7	5.2
	538/1000	3.2/465	10	M		M						3.6	0	7.1	4.1
— — — — — — — — — — — — — — — 91% ALUMINA[i] — — — — — — — — — — — — — — —															
Air (dried)	110/ 230	ambient	1	M			tr	m		tr					
CGA exposure	260/ 500	3.4/500	10	M	M	M					m	8.4	24.5	10.8	6.7
			20									6.5	15.9	9.3	5.5
			30	M	M	M					tr	8.3	24.1	10.5	6.3
CGA exposure	538/1000	6.9/1000	10	M		M					tr	4.6	0	10.3	6.1
			20	M		M									
			30									4.9	0	10.1	6.0
CGA with H2S	538/1000	6.9/1000	10	M		M						2.6	0	4.3	2.5
			20	M		M						5.5	0	10.8	6.4
			30	M		M						5.4	0	10.94	6.5
CGA, H2O saturated	231/ 447	6.9/1000	30	not tested											
CGA with H2S, H2O satd.	231/ 447	6.9/1000	30	M	M	M						8.3	37.3	5.2	3.2
CO/H2O, H2O saturated	199/ 390	3.2/465	10	M	M	m						7.8	20.0	3.8	2.3
CO/H2O	260/ 500	3.2/465	10	M	M	M						8.9	24.7	11.7	7.2
	538/1000	3.2/465	10	M		M						4.2	0	7.3	4.3

(Table Continued)

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EFFECT OF HEAT TREATMENT AND GASEOUS ENVIRONMENT[a] ON THE PHASES PRESENT IN VARIOUS HIGH-ALUMINA DENSE CASTABLE REFRACTORIES[b][39], Continued

Treatment Conditions				XRD Analysis Results[c]								DTA-TGA Analyses			
Atmosphere[a]	Temperature °C / °F	Pressure MPa/psi	Time days	Alumina	Boehmite	Calcite	Gibbsite	CA	CA2	C3AH6	Aragonite	% Weight Loss	% Boehmite	% Calcite	% CaO, Dry Basis
91% ALUMINA[j]															
Air (dried)	110/ 230	ambient	1	M				m							
CGA exposure	260/ 500	3.4/ 500	10	M	M	M					m	7.5	24.9	8.5	5.2
			20									5.1	13.7	6.9	4.1
			30	M	M	M					tr	6.9	22.3	8.0	4.8
CGA exposure	538/1000	6.9/1000	10	M		M				m		3.8	0	8.5	5.0
			20	M		M									
			30									4.0	0	7.7	4.5
CGA with H2S	538/1000	6.9/1000	10	M		M		m							
			20	M		M		m							
			30	M		M		m				3.4	0	6.1	3.5
CGA, H2O saturated	231/ 447	6.9/1000	30	M	M	M						8.1	27.9	8.8	5.3
CGA with H2S, H2O satd.	231/ 447	6.9/1000	30	M	M	M									
CO/H2O, H2O saturated	199/ 390	3.2/ 465	10	M	M	m						7.8	18.7	4.7	2.8
CO/H2O	250/ 500	3.2/ 465	10	M	M	M						7.8	25.6	9.1	5.5
	538/1000	3.2/ 465	10	M	m							2.8	0	4.7	2.7

[a]Environments included a simulated coal gasification atmosphere (CGA) with an overall composition (in vol %) of 18 CO, 12 CO2, 24 H2, 5 CH4, and 41 H2O; another CGA which has 1% H2S added; and a CO-H2O atmosphere which is 52.5 CO and 46.5 H2O (vol %). Samples were exposed in two pressure vessels. One vessel was a steam generator and tests specifying saturated conditions occurred in this vessel. After placing samples, the vessel was heated to the desired steam pressure and the other gases were then added so as to obtain the concentrations specified above. The second vessel was connected to the steam generator and after samples were placed and the vessel heated to the test temperature, the vessel was pressurized with the steam/gas mixture from the steam generator. A gaseous flow rate for both vessels was maintained so as to provide one complete change in atmosphere every 24 hours.

[b]All these cement-bonded castables were cast in molds, cured 24 hours in air at 100% humidity at ambient temperatures, and dried at 230 °F for 24 hours. All specimens were stored at 230 °F until tested.

[c]Phases were identified by x-ray diffraction analysis; M = major phase, m = minor phase, tr = trace. Cement notation used (C = CaO, A = Al2O3, H = H2O) to specify some compounds. No data are given for the phases present after firing in air.

[d]94% alumina dense castable, calcium aluminate (79% alumina, 18% calcia) bonded (Greencast-94, A.P. Green; CA-25 Calcium Aluminate Cement, Alcoa).

[e]93% alumina dense castable, calcium aluminate (72% alumina, 25% calcia) bonded (B&W Kao-Tab 93, Babcock & Wilcox; C-3 cement, Babcock & Wilcox).

[f]90-95% alumina dense castable, calcium aluminate (79% alumina, 18% calcia) bonded (Castolast G, Harbison-Walker; CA-25 Calcium Aluminate Cement, Alcoa).

[g]90-95% alumina dense castable, calcium aluminate (79% alumina, 18% calcia) bonded (Purotab, Kaiser Refractories; CA-25 Calcium Aluminate Cement, Alcoa).

[h]93% alumina dense castable, UMR-1 generic preparation (70% tabular alumina + 30% calcium aluminate cement) (T-61 alumina, Alcoa; CA-25 Calcium Aluminate Cement, Alcoa).

[i]91% alumina dense castable, UMR-2 generic preparation (70% tabular alumina + 30% calcium aluminate cement) (T-61 alumina, Alcoa: Secar 71(250), a 72% alumina-26% calcia cement, Lone Star Lafarge).

[j]91% alumina dense castable, UMR-3 generic preparation (70% tabular alumina + 30% calcium aluminate cement) (T-61 alumina, Alcoa; C-3, a 72% alumina-25% calcia cement, Babcock & Wilcox).

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EFFECT OF HEAT TREATMENT AND GASEOUS ENVIRONMENT[a] ON THE PHASES PRESENT IN VARIOUS INTERMEDIATE-ALUMINA REFRACTORIES[b][39]

| Treatment Conditions | | | | XRD Analysis Results[c] | | | | | | | | | DTA-TGA Analyses | | | |
Atmosphere[a]	Temperature °C / °F	Pressure MPa/psi	Time days	Kyanite	Mullite	α-Quartz	β-Cristobalite	Alumina	Boehmite	Calcite	CaAl₂O₄	Bayerite	% Weight Loss	Boehmite %	Calcite %	CaO, Dry Basis %
— — — — — — — —	— — — 54-57% ALUMINA DENSE CASTABLE[d] — — —															
Air (dried)	110/ 230	ambient	1		M		M	m		m						
CGA exposure	260/ 500	3.4/500	10		M		M	M	m	m			4.2	16.0	4.2	2.4
			20		M		M	m		m			3.1	7.9	3.7	2.2
			30		M		M	m	tr	m			4.5	13.4	5.5	3.2
CGA exposure	538/1000	6.9/1000	10		M		M	m		m						
			20		M		M	m		m						
			30										1.6	0	3.1	1.8
CGA with H₂S	538/1000	6.9/1000	10		M		M	m			tr					
			20		M		M	m			tr					
			30		M		M	m			tr		1.8	0	2.4	1.4
CGA, H₂O saturated	231/ 447	6.9/1000	30		M		M	m	tr	m		tr	4.2	8.2	2.7	1.6
CGA with H₂S, H₂O satd.	231/ 447	6.9/1000	30		M		M	m	tr	m						
CO/H₂O, H₂O saturated	199/ 390	3.2/465	10		M		M	m	tr	m			4.9	9.0	3.4	2.0
CO/H₂O	260/ 500	3.2/465	10		M		M	m	m	m			4.9	16.6	5.5	3.2
	538/1000	3.2/465	10		M		M	m		m			2.4	0	3.9	2.2
— — — — — — —	— — — 57% ALUMINA DENSE CASTABLE[e] — — —															
Air (dried)	110/ 230	ambient	1		M		M	m		m						
CGA exposure	260/ 500	3.4/500	10		M		M	m	m	m			5.7	17.2	7.0	4.1
			20										3.6	7.5	5.7	3.3
			30		M		M	m	m	m			5.3	14.1	7.2	4.2
CGA exposure	538/1000	6.9/1000	10		M		M	m		m						
			20		M		M	m		m						
			30										2.3	0	4.4	2.6
CGA with H₂S	538/1000	6.9/1000	10		M		M	m		m			2.0	0	3.9	2.3
			20		M		M	m		m			2.6	0	5.0	2.8
			30		M		M	m		m			2.7	0	4.4	2.5
CGA, H₂O saturated	231/ 447	6.9/1000	30		M		M	m	m	m		m	5.3	13.5	4.1	2.4
CGA with H₂S, H₂O satd.	231/ 447	6.9/1000	30		M		M	m								
CO/H₂O, H₂O saturated	199/ 390	3.2/465	10		M		M	m	m	tr			5.0	27.2	2.0	1.2
CO/H₂O	260/ 500	3.2/465	10		M		M	m	m	m			6.3	11.7	7.4	4.4
	538/1000	3.2/465	10		M		M	m		m			2.0	0	3.43	2.0
— — — — — — —	— — — 59% ALUMINA INSULATING CASTABLE[f] — — —															
Air (dried)	110/230	ambient	1	M	M	m	m	m		m						
CGA exposure	260/ 500	3.4/500	10	M	M		m	m	tr	m			4.9	12.1	6.2	3.7
			20										4.2	9.8	6.1	3.5
			30	M	M		m	m	m	m			5.2	15.0	6.7	3.9
CGA exposure	538/1000	6.9/1000	10	M	M		m	m		m						
			20	M	M			m								
			30	M	M	m	m	m		m			1.9	0	3.4	1.9
CGA with H₂S	538/1000	6.9/1000	10	M	M			m		tr	m					
			20	M	M			m		tr	tr					
			30	M	M			m		tr	m		2.0	0	3.2	1.8
CGA, H₂O saturated	231/ 447	6.9/1000	30	M	m	m		m			m	m	5.3	11.5	1.5	0.9
CGA with H₂S, H₂O satd.	231/ 447	6.9/1000	30	M	m	tr		m	m	tr	tr					
CO/H₂O, H₂O saturated	199/ 390	3.2/465	10	M	M	m		m	tr	tr			6.8	9.9	3.4	2.0
CO/H₂O	260/ 500	3.2/465	10										4.5	12.8	5.8	3.4
	538/1000	3.2/465	10										2.3	0	3.8	2.2

(Table Continued)

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EFFECT OF HEAT TREATMENT AND GASEOUS ENVIRONMENT[a] ON THE PHASES PRESENT IN VARIOUS INTERMEDIATE-ALUMINA REFRACTORIES[b][39], Continued

Atmosphere[a]	Temperature °C / °F	Pressure MPa/psi	Time days	XRD Analysis Results[c]									DTA-TGA Analyses			
				Kyanite	Mullite	α-Quartz	β-Cristobalite	Alumina	Boehmite	Calcite	CaAl₂O₄	Bayerite	% Weight Loss	% Boehmite	% Calcite	% CaO, Dry Basis
--- 54% ALUMINA INSULATING CASTABLE[g] ---																
Air (dried)	110/ 230	ambient	1	M	M	M			m							
CGA exposure	260/ 500	3.4/500	10	M	M	M	tr	M					3.9	13.0	4.3	2.5
			20										3.2	10.4	3.8	2.2
			30	M	M	M	tr	m					4.1	13.3	4.4	2.6
CGA exposure	538/1000	6.9/1000	10	M	M	M		m								
			20	M	M	M										
			30	M	M	M							1.4	0	2.2	1.3
CGA with H₂S	538/1000	6.9/1000	10	M	M	M		m			m		1.0	0	1.7	1.0
			20	M	M	M		m			m		1.3	0	2.2	1.3
			30	M	M	M		m			m		1.3	0	1.9	1.1
CGA, H₂O saturated	231/ 447	6.9/1000	30	M	M	M			m			m	5.3	9.1	2.8	1.7
CGA with H₂S, H₂O satd.	231/ 447	6.9/1000	30	M	M	M	tr	tr	m				7.3	6.4	3.3	2.0
CO/H₂O, H₂O satd.	199/ 390	3.2/465	10	m	M	m	tr						4.2	14.1	1.0	0.6
CO/H₂O	260/ 500	3.2/465	10	m	M	m	tr	tr					3.5	12.2	3.8	2.2
	538/1000	3.2/465	10										1.4	0	2.3	1.3

[a]Environments included a simulated coal gasification atmosphere (CGA) with an overall composition (in vol %) of 18 CO, 12 CO₂, 24 H₂, 5 CH₄, and 41 H₂O; another CGA which has 1% H₂S added; and a CO-H₂O atmosphere which is 52.5 CO and 46.5 H₂O (vol %). Samples were exposed in two pressure vessels. One vessel was a steam generator and tests specifying saturated conditions occurred in this vessel. After placing samples, the vessel was heated to the desired steam pressure and the other gases were then added so as to obtain the concentrations specified above. The second vessel was connected to the steam generator and after samples were placed and the vessel heated to the test temperature, the vessel was pressurized with the steam/gas mixture from the steam generator. A gaseous flow rate for both vessels was maintained so as to provide one complete change in atmosphere every 24 hours.

[b]After casting, the specimens were cured 24 hours in air at 100% humidity at ambient temperature, and dried at 230 °F for 24 hours. All specimens were stored at 230 °F until tested.

[c]Phases were identified by x-ray diffraction analysis; M = major phase, m = minor, tr = trace. No data are given for the phases present in the fired condition.

[d]54-57% alumina, 34-37% silica castable, calcium aluminate (79% alumina, 18% calcia) bonded (Lo-Abrade, A.P. Green; CA-25 Calcium Aluminate Cement, Alcoa).

[e]57% alumina, 34% silica castable, calcium aluminate (79% alumina, 18% calcia) bonded (RC-3, General Refractories; CA-25 Calcium Aluminate Cement, Alcoa).

[f]59% alumina, 33% silica insulating castable, calcium aluminate bonded (Kast-O-Lite 30, A.P. Green; CA-25 cement, Alcoa).

[g]54% alumina, 40% silica insulating castable, calcium aluminate bonded (Cer-Lite #75, C-E Refractories; CA-25 cement, Alcoa).

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EFFECT OF HEAT TREATMENT AND GASEOUS ENVIRONMENT[a] ON THE PHASES PRESENT IN VARIOUS PHOSPHATE-BONDED REFRACTORIES[b][39]

| Environment | Treatment Conditions[a] | | | XRD Analysis Results[d] | | | |
	Temperature °F	Pressure psi	Time[c]	Alumina	AlPO$_4$ (Berlinite)	AlPO$_4$ (orthorhombic)	Unidentified
— — — — — — —	— — — — —	— —96% ALUMINA RAMMING MIX[e]—	— — — — —	— — — —	— — — —		
Air (fired)	1000	ambient	18 h	M	tr	tr	
CGA exposure	500	500	10 d	M	m		
	500	500	20 d	M	m		
	500	500	30 d	M	m		
CGA exposure	1000	1000	10 d	M	M		
	1000	1000	20 d	M	m	tr	
	1000	1000	30 d	M	M		
CGA, H$_2$O saturated	447	1000	30 d	M	m		m
— — — — — — —	— — — —	— — — —96% ALUMINA RAMMING MIX[f]—	— — — — —	— — — —	—		
Air (fired)	1000	ambient	18 h	M		tr	
CGA exposure	500	500	10 d	M	tr	tr	
CGA exposure	1000	1000	10 d	M	tr	m	
CGA, H$_2$O saturated	447	1000	30 d	M			m
— — — — — — —	— — — —	— — — —90% ALUMINA RAMMING MIX[g]—	— — — — —	— — — —	—		
Air (fired)	1000	ambient	18 h	M	tr		
CGA exposure	500	500	10 d	M	m		
CGA exposure	1000	1000	10 d	M	M	tr	
CGA, H$_2$O saturated	447	1000	30 d	M	M		tr

[a] Environments included a simulated coal gasification atmosphere (CGA) with an overall composition (in vol %) of 18 CO, 12 CO$_2$, 24 H$_2$, 41 H$_2$O and 5 CH$_4$. Samples were exposed in two pressure vessels. One vessel was a steam generator and tests which specify saturated conditions occurred in this vessel. After placing samples, the vessel was heated to the desired steam pressure and the other gases were then added so as to obtain the concentrations specified above. The second vessel was connected to the steam generator and after samples were placed and the vessel heated to the test temperature, the vessel was pressurized with the steam/gas mixture from the steam generator. A gaseous flow rate for both vessels was maintained so as to provide one complete change in atmosphere every 24 hours.

[b] Ramming mix specimens were prepared by hand ramming in 3 x 3/4 x 3/4 in molds, immediately drying for 24 hr at 230 °F and then firing at 1000 °F for 18 hr. All specimens were stored at 230 °F until tested.

[c] Time units are given, either h for hours or d for days.

[d] Phases identified by x-ray diffraction analysis; M = major phase, m = minor phase, tr = trace.

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(Continued)

EFFECT OF HEAT TREATMENT AND GASEOUS ENVIRONMENT[a] ON THE PHASES
PRESENT IN VARIOUS PHOSPHATE-BONDED REFRACTORIES[b][39], Continued

[e]96% Alumina dense phosphate-bonded ramming mix (Greenpak-90P, A.P. Green).

[f]96% Alumina dense phosphate-bonded ramming mix (90 Ram H.S., C-E Refractories).

[g]90% Alumina dense phosphate-bonded ramming mix (Brikram 90R, General Refractories).

EFFECT OF HEAT TREATMENT, GASEOUS ENVIRONMENT, AND SATURATED CONDITIONS[a] ON THE PHASES PRESENT IN CALCIUM ALUMINATE CEMENTS[b][39]

XRD Analysis Results, Initially Dry[b] / Hydrated[b]

Atmosphere[a]	Temp °C/°F	Pressure MPa/psi	Time days	Alumina	Boehmite	Calcite	Aragonite	CA	CA$_2$	C$_2$AS	C$_3$AH$_6$	C$_{12}$A$_7$	C$_4$A$_3$H$_3$	Ca$_8$Al$_6$O$_{12}$SO$_4$	Calcium Formate
79% ALUMINA, 18% CALCIA CEMENT[d]															
Air (dried)	110/230	ambient	1	M/				M/							
CGA exposure	260/500	3.4/500	10	M/	M/	M/	m/								
			20	M/m	M/m	M/m									
			30	M/	M/	M/	m/								
CGA exposure	538/1000	6.9/1000	10	M/	M/	M/									
			20	M/M	M/M	M/M									
			30	M/M	M/M	M/M									
CGA with H$_2$S	538/1000	6.9/1000	10	m/m	M/M	M/m		/tr							
			20	M/M	M/M	M/m									
			30	m/m	M/M	M/M									
CGA, H$_2$O saturated	231/447	6.9/1000	30	m/tr		m/tr		M/							
CO/H$_2$O, H$_2$O saturated	199/390	3.2/465	10	M/		M/		m/							
CO/H$_2$O	260/500	3.2/465	10	M/		M/		m/							
	538/1000	3.2/465	10	M/		/M		/M						m/	
Saturated steam {vapor	231/447	2.8/410	10	tr/m	M/M	m/tr					m/m	m/			
liquid}	231/447	2.8/410	10	/m	/M	/tr					/m				
Saturated steam {vapor	285/545	6.9/1000	10	/m	M/M	m/m		tr/tr			tr/m		/m		
liquid}	285/545	6.9/1000	10	/m	/M	/m		tr/m			/m		/m		
CGA with H$_2$S, {vapor	231/447	6.9/1000	10	tr/tr	M/M	M/M		M/m							
H$_2$O saturated {liquid}	231/447	6.9/1000	10	/m	/M	/m		tr/m			/tr				
72% ALUMINA, 26% CALCIA CEMENT[f]															
Air (dried)	110/230	ambient	1	m/				M/	m/						
CGA exposure	260/500	3.4/500	10	tr/m	m/	m/	M/	m/m	m/						
			20	/m	/M	M/M									
			30	tr/	m/	m/	m/	m/	tr/						
CGA exposure	538/1000	6.9/1000	20	/M	/M	/M		/M							
			30	/M	/M	/M									
CGA with H$_2$S	538/1000	6.9/1000	10	m/tr	M/M	M/M		tr/m			m/m		m/m		
			10	/tr	/M	/M		tr/m			/m		/m		
CGA, H$_2$O saturated	231/447	6.9/1000	30	M/		M/		M/			/m		m/		
CO/H$_2$O, H$_2$O saturated	199/390	3.2/465	10	tr/	m/	m/	M/	M/							
CO/H$_2$O	260/500	3.2/465	10	/m	M/M	tr/m	/m	/m			/m		/m		
	538/1000	3.2/465	10	tr/	M/	m/	m/	M/							
Saturated steam {vapor	231/447	2.8/410	10	m/tr	M/M	M/M		m/			m/m		m/m		
liquid}	231/447	2.8/410	10	/tr	/M	/M					/m		/m		
Saturated steam {vapor	285/545	6.9/1000	10	M/	M/M	M/M		M/M			m/m				
liquid}	285/545	6.9/1000	10		/M	/M		M/M			/m				
CGA with H$_2$S, {vapor	231/447	6.9/1000	10	m/	M/M	M/M		m/			/m				
H$_2$O saturated {liquid}	231/447	6.9/1000	10	/m	/M	/m		M/			/m		m/		
72% ALUMINA, 25% CALCIA CEMENT[g]															
Air (dried)	110/230	ambient	1	m/				m/	M/						
CGA exposure	260/500	3.4/500	10	tr/m	m/	m/	M/	m/							
			20	/m	M/M	M/M	tr/m	/m							
			30	M/	M/	m/	m/	M/							
CGA exposure	538/1000	6.9/1000	10	m/M	m/M	M/M		M/M							
			20	M/M	/M	M/M		M/M							
			30	M/	M/	M/		m/							
CGA with H$_2$S	538/1000	6.9/1000	10	m/m	m/	M/M		m/M			m/m				
			20	m/m	/M	M/M		M/M			/m				
			30	m/		M/m		m/			/m				
CGA, H$_2$O saturated	231/447	6.9/1000	30	m/		/m		m/			/m		m/		
CO/H$_2$O, H$_2$O saturated	199/390	3.2/465	10	m/	m/	M/		m/							
CO/H$_2$O	260/500	3.2/465	10	tr/m	M/	m/		m/							
	538/1000	3.2/465	10	m/	M/	M/		M/							

DTA–TGA Analyses, Initially Dry Cement[b] / Hydrated Cement[b] (%)

Atmosphere[a]	Temp °C/°F	Time days	Weight Loss	Boehmite	Calcite	CaO, Dry Basis	C$_3$AH$_6$	Calcium Formate
79% ALUMINA, 18% CALCIA CEMENT[d]								
CGA exposure	260/500	10–30	17.6/	46.5/	24.3/	16.5/		
			8.9/14.5	23.3/39.8	12.2/19.4	7.4/12.7		
CGA exposure	538/1000	10–30	11.7/11.7	0/0	24.5/24.5	15.6/15.6[e]		
			11.8/11.8	0/0	25.0/25.0	15.9/15.9[e]		
CGA with H$_2$S	538/1000	10–30	12.6/	0/	26.2/	16.8/		
			/19.4	/73.4	/11.4	/7.9		
CGA, H$_2$O saturated	231/447	30	14.6/	62.4/	9.2/	6.0/		0/
CO/H$_2$O, H$_2$O saturated	199/390	10	19.5/	39.4/	18.3/	12.7/		6.8/
CO/H$_2$O	260/500, 538/1000	10	6.7/	0	13.7/	8.2/		0/
Saturated steam	231/447	10		58.1/51.4			25.6/25.3	
				/50.2			/26.6	
Saturated steam	285/545	10	17.8/19.3	63.0/67.4	12.9/ 9.4			
			/17.1	/59.3	/ 6.8			
CGA with H$_2$S, H$_2$O saturated	231/447	10	23.5/20.5	55.3/60.4	25.9/20.8			
			/20.7	/61.8	/13.4			
72% ALUMINA, 26% CALCIA CEMENT[f]								
CGA exposure	260/500	10–30	24.5/19.4	64.7/56.1	32.8/24.9	24.4/17.3		
			13.5/	19.0/	12.2/	7.9/		
CGA exposure	538/1000	10–30	14.7/14.7	0/0	31.3/31.3	20.5/20.5[e]		
			15.0/15.0	0/0	32.5/32.5	21.4/21.4[e]		
CGA with H$_2$S	538/1000	10	10.0/	0/	19.7/	12.2/		
			/21.7	/57.5	/12.8	/ 9.1		
CGA, H$_2$O saturated	231/447	30	25.7/	58.3/	19.9/	15.0/		0/
CO/H$_2$O, H$_2$O saturated	199/390	10	29.5/	52.4/	21.1/	16.8/		15.2/
			4.2/	0	7.3/	4.3/		0
Saturated steam	231/447	10		37.9/47.9			24.8/31.2	
				/32.6			/35.5	
Saturated steam	285/545	10	19.2/22.6	43.0/57.3	15.7/ 9.3			
			/20.9	/53.9	/11.7			
CGA with H$_2$S, H$_2$O saturated	231/447	10	25.6/23.3	52.9/57.9	33.9/12.3			
			/24.	/52.5	/14.			
72% ALUMINA, 25% CALCIA CEMENT[g]								
CGA exposure	260/500	10–30	21.4/	67.0/	24.9/	17.8/		
			9.9/	28.9/	12.1/	7.5/		
CGA exposure	538/1000	10–30	11.2/11.2	0/0	21.0/21.0	13.3/13.3[e]		
			12.1/12.1	0/0	24.0/24.0	15.2/15.2[e]		
CGA with H$_2$S	538/1000	10–30	12.7/	0/	24.2/	15.6/		
CGA, H$_2$O saturated	231/447	30	16.7/	82.9/	7.0/	4.7/		0/
CO/H$_2$O, H$_2$O saturated	199/390	10	25.4/	56.8/	18.4/	13.8/		10.8/
			1.4/	0	2.5/	1.4/		0

(Table Continued)

EFFECT OF HEAT TREATMENT, GASEOUS ENVIRONMENT, AND SATURATED CONDITIONS[a] ON THE PHASES PRESENT IN CALCIUM ALUMINATE CEMENTS[b][39], Continued

XRD Analysis Results, Initially Dry[b] / Hydrated[b]; DTA-TGA Analyses, Initially Dry Cement[b] / Hydrated Cement[b]

Atmosphere[a]	Temperature °C / °F	Pressure MPa/psi	Time days	Alumina	Boehmite	Calcite	Aragonite	CA	CA2	C2AS	C3AH6	C12A7	C4A3H3	Ca4Al6O12SO4	Calcium Formate	Weight Loss %	Boehmite %	Calcite %	CaO, Dry Basis %	C3AH6 %	Calcium Formate %
58% ALUMINA, 33% CALCIA CEMENT[h]																					
Air (dried)	110/ 230	ambient	1	m/				M/	M/	M/											
CGA exposure	260/ 500	3.4/500	20		M/M	M/M		M/m	M/	M/M						5.4/15.2	14.3/44.0	7.4/19.6	4.4/13.0		
CGA exposure	538/1000	6.9/1000	20			M/M		m/m	m/m	M/M						11.7/11.7	0 / 0	22.8/22.8	14.5/14.5[e]		
	538/1000	6.9/1000	30													13.8/13.8	0 / 0	28.6/28.6	18.6/18.6[e]		
CGA with H2S	538/1000	6.9/1000	10		M/M	M/M		m/m	m/m	m/m											
			20		M/M	M/M		tr/tr	m/m	m/m											
			30		m/m	m/m		m/m	m/m	M/m						4.7/	0 /	9.3/	5.5/		0 /
CGA, H2O saturated	231/ 447	6.9/1000	30		/M	/m				/m						/18.9	/44.2	/12.7	/ 8.8		0 /
CO/H2O, H2O saturated	199/ 390	3.2/465	10		M/	m/		m/	m/							20.4/	62.3/	15.6/	11.0/		0 /
CO/H2O	260/ 500	3.2/465	10		M/	M/			M/						m/	26.8/	48.4/	16.2/	12.4/		15.2/
	538/1000	3.2/465	10					M/	m/			m/				2.0/	0 /	4.3/	2.5/		0 /
Saturated steam { vapor	231/ 447	2.8/410	10		/M	m/m				/tr	/m		/m			/18.8	/43.9	/ 6.6			
liquid }	231/ 447	2.8/410	10		/M	/m				/tr	/m		/m			/17.6	/40.6	/ 5.4			
Saturated steam { vapor	285/ 545	6.9/1000	10	/tr	M/M	M/M	m/m	m/m		/m	m/m										
liquid }	285/ 545	6.9/1000	10	/tr	/M	/m	m/tr	/m			/m										
CGA with H2S,	231/ 447	6.9/1000	10		M/M	M/M			M/M		/m					26.7/22.7	55.7/41.5	28.7/24.7			
H2O saturated { liquid }	231/ 447	6.9/1000	10		/M	/tr			M/m		/m					/18.9	/36.8	/ 8.3			
44% ALUMINA, 36% CALCIA CEMENT[i]																					
Air (dried)	110/ 230	ambient	1	m/				M/	m/	M/											
CGA exposure	260/ 500	3.4/500	20		m/m	M/M		m/m	m/m	M/m						5.6/15.5	12.3/39.8	8.6/21.6	5.1/14.3		
CGA exposure	538/1000	6.9/1000	20			M/M		m/m	m/m	M/M						13.0/13.0	0 / 0	27.5/27.5	17.7/17.7[e]		
	538/1000	6.9/1000	30													11.6/11.6	0 / 0	23.5/23.5	14.9/14.9[e]		
CGA with H2S	538/1000	6.9/1000	10			m/M		m/m	M/m	m/m											
			20			m/M		m/tr	M/m	m/m											
			30			m/m		/tr	m/m	M/m	/m					2.8/	0 /	5.8/	3.3/		0 /
CGA, H2O saturated	231/ 447	6.9/1000	30		/M	/m			m/							/18.9	/34.7	/17.6	/12.2		0 /
CO/H2O, H2O saturated	199/ 390	3.2/465	10	m/	M/	M/			M/							18.7/	54.9/	15.0/	10.4/		0 /
CO/H2O	260/ 500	3.2/465	10	m/	M/	M/		M/	m/							20.8/	51.2/	29.8/	21.1/		0 /
	538/1000	3.2/465	10		M/	m/		M/	tr/	M/						2.0/	0 /	4.5/	2.6/		0 /

[a] Environments included a simulated coal gasification atmosphere (CGA) with an overall composition (in vol %) of 18 CO, 12 CO2, 24 H2, 5 CH4, and 41 H2O; another CGA which has 1% H2S added; and a CO-H2O atmosphere which is 52.5 CO and 46.5 H2O (vol %). Samples were exposed in two pressure vessels. One vessel was a steam generator and tests specifying saturated conditions occurred in this vessel. For some of the tests samples were placed both in the vapor and in the liquid in the bottom of the vessel. After placing samples, the vessel was heated to the desired steam pressure and the other gases were then added so as to obtain the concentrations specified above. The second vessel was connected to the steam generator and after samples were placed and the vessel heated to the test temperature, the vessel was pressurized with the steam/gas mixture from the steam generator. A gaseous flow rate for both vessels was maintained so as to provide one complete change in atmosphere every 24 hours.

[b] Cements were tested as initially dry powders and as neat cement specimens. Hydrated neat cement samples (water/cement ratio of ~0.3) were cast as 1/2-inch cubes, cured in air at 100% humidity for 24 hours, and dried at 230 °F for 24 hours. Samples were stored at 230 °F until tested.

[c] Phases identified by x-ray diffraction analyses; M = major phase, m = minor phase, tr = trace. Cement notation used (C = CaO, A = Al2O3, H = H2O, S = SiO2) to specify some compounds. No data are given for the phases present after firing in air.

[d] 79% Alumina, 18% calcia cement (CA-25 Calcium Aluminate Cement, Alcoa).

[e] Identical values are given in different reports of the series for both the dry powder and the hydrated cement for all the analyses of this test.

[f] 72% Alumina, 26% calcia cement (Secar 71(250), Lone Star Lafarge).

[g] 71.5-72.5% Alumina, 24-25% calcia cement (C-3, Babcock & Wilcox).

[h] 58% Alumina, 33% calcia cement (Refcon, Universal Atlas Cement).

[i] 44% Alumina, 36% calcia cement (Lumnite, Universal Atlas Cement).

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EFFECT OF SATURATED VAPOR AND LIQUID AND EXPOSURE[a] TO PRESSURE-TEMPERATURE CYCLINC CAUSINC BOEHMITE FORMATION AND DECOMPOSITION[b]
ON THE PHASES PRESENT IN ALUMINA CASTABLE REFRACTORIES[c][39]

DTA-TCA Analyaea

Atmosphere[a]		Treatment Conditiona Temperature °C / °F	Preaure MPa/pai	Time days	XRD Analyaia Reaulta[d]							% Weight Loss	% Boehmite	% Calcite	% CaO, Dry Basis

- 93% ALUMINA DENSE CASTABLE[e] -

					α-Al₂O₃	Boehmite	Calcite	C₃AH₆	CA						
Air (dried)		110/ 230	ambient	1	M				m						
Saturated steam	vapor	231/ 447	2.8/410	10	M	M	tr	m			6.5	20.2	5.4	
	liquid	231/ 447	2.8/410	10	M	M	tr	m			6.7	22.0	7.9	
Saturated ateam	vapor	285/ 545	6.9/1000	10	M	M	m				6.2	22.5	5.2	
	liquid	285/ 545	6.9/1000	10	M	M	m				6.8	23.5	2.3	
CCA with H₂S, H₂O saturated	vapor	231/ 447	6.9/1000	10	M	M	m				7.6	22.3	7.6	
	liquid	231/ 447	6.9/1000	10	M	M	tr				7.5	21.8	4.7	
CGA with H₂S, H₂O aaturated	vapor	231/ 447	6.9/1000	44	M	M	m				8.1	24.9	7.8	4.8
	liquid	231/ 447	6.9/1000	44	M	M	m	tr			8.0	25.5	5.1	3.1
CCA with H₂S, saturated[f] followed by firing in air[g]		240/ 465 538/1000	6.9/1000 ambient	5 3/4	M M	m	m m				6.5 3.8	12.4 0	7.8 7.3	4.7 4.3

Cycling teata--repeat of preceding exposure followed by the same air firing

				1 cycle	M		m				4.0	0	8.3	4.9
				2 cycles	M		m				4.2	0	8.7	5.1
				3 cyclea	M		m				4.1	0	8.5	5.0
				4 cyclea	M	tr	m				3.9	0	8.0	5.0
				5 cyclea	M		m	tr			4.5	0	9.5	5.6

- 87% ALUMINA DENSE CASTABLE[h] -

					α-Al₂O₃	Boehmite	Calcite	C₃AH₆	CA₂	C₂AS					
Air (dried)		110/ 230	ambient	1	M			m		m				
Saturated steam	vapor	231/ 447	2.8/410	10	M	M		m	tr		6.4	15.1	10.5	
	liquid	231/ 447	2.8/410	10	M	M		m	tr		6.5	14.9	12.6	
Saturated ateam	vapor	285/ 545	6.9/1000	10	M	M	m				7.1	20.0	5.7	
	liquid	285/ 447	6.9/1000	10	M	M	tr				7.0	18.3	3.9	
CCA with H₂S, H₂O saturated	vapor	231/ 447	6.9/1000	10	M	M	m				11.3	18.4	11.0	
	liquid	231/ 447	6.9/1000	10	M	M	M				8.9	19.6	10.5	
CCA with H₂S, H₂O saturated	vapor	231/ 447	6.9/1000	44	M	M	m				8.1	18.9	6.2	3.8
	liquid	231/ 447	6.9/1000	44	not tested									
CCA with H₂S, saturated[f] followed by firing in air[g]		240/ 465 528/1000	6.9/1000 ambient	5 3/4	M M	M	m m				8.5	15.2	8.5	5.2

Cycling tests--repeat of preceding exposure followed by the same air firing

				1 cycle	M		m				5.9	0	11.6	6.9
				2 cycles	M		m				6.5	0	12.4	7.4
				3 cycles	M		m				5.5	0	8.4	4.9
				4 cyclea	M		m				5.4	0	8.0	4.6
				5 cycles	M		m				5.9	0	12.2	7.2

- 64% ALUMINA-28% SILICA DENSE CASTABLE[i] -

					Mullite	α-Al₂O₃	Boehmite	β-Cristobalite	Calcite						
Saturated ateam	vapor	231/ 447	2.8/410	10	M	m	m	tr			3.7	9.5		
	liquid	231/ 447	2.8/410	10	M	m	m	tr			4.2	11.0		
Saturated steam	vapor	285/ 545	6.9/1000	10	M	m	m		m		3.7	11.4	1.7	
	liquid	285/ 545	6.9/1000	10	M	m	m		m		3.4	10.8	1.5	
CCA with H₂S, H₂O saturated	vapor	231/ 447	6.9/1000	10	M	m	m	tr	m		5.2	11.1	5.8	
	liquid	231/ 447	6.9/1000	10	M	m	m	tr	m		5.	14.	3.2	
CGA with H₂S, H₂O saturated	vapor	231/ 447	6.9/1000	44	M	m	m	tr	m		5.0	10.1	3.9	2.3
	liquid	231/ 447	6.9/1000	44	M	m	m	tr	m		5.8	14.0	5.7	3.4
CGA with H₂S, aaturated[f] followed by firing in air[g]		240/ 465 538/1000	6.9/1000 ambient	5 3/4	M M	m m	m	tr m	m m		6.1 4.6	10.9 0	7.0 7.9	4.2 4.6

Cycling tests--repeat of preceding exposure followed by the aame air firing

				1 cycle	M	m		m	m		3.7	0	7.4	4.3
				2 cycles	M	m		tr	m		3.9	0	8.1	4.7
				3 cycles	M	m		tr	m		2.7	0	6.2	3.6
				4 cycles	M	m		tr	m		2.8	0	5.7	3.3
				5 cycles	M	m		tr	m		3.0	0	5.8	3.4

- 58% ALUMINA-30% SILICA DENSE CASTABLE[j] -

					Mullite	α-Al₂O₃	Boehmite	C₃AH₆	C₂AH₁₈	Calcite	α-Cristo-balite	C₂AS			
Air (dried)		110/ 230	ambient	1	M	tr	tr								
Saturated steam	vapor	231/ 447	2.8/410	10	M	m	m	tr					4.4	8.7	7.3
	liquid	231/ 447	2.8/410	10	M	m							7.0	10.1	
Saturated steam	vapor	285/ 545	6.9/1000	10	M	m			tr	tr			5.2	11.1	3.2
	liquid	285/ 545	6.9/1000	10	M	m			tr	m			4.6	10.3	3.0
CCA with H₂S H₂O saturated	vapor	231/ 447	6.9/1000	10	M	m		m	tr				7.5	14.6	7.8
	liquid	231/ 447	6.9/1000	10	M	m		tr	tr				7.2	13.2	8.0

(Table Continued)

EFFECT OF SATURATED VAPOR AND LIQUID AND EXPOSURE[a] TO PRESSURE-TEMPERATURE CYCLING CAUSING BOEHMITE FORMATION AND DECOMPOSITION[b] ON THE PHASES PRESENT IN ALUMINA CASTABLE REFRACTORIES[c][39], Continued

58% ALUMINA-30% SILICA DENSE CASTABLE, Continued

Atmosphere[a]		Temperature °C / °F	Pressure MPa/psi	Time days	XRD Analysis Results[d] ($Mullite$, α-Al_2O_3, $Boehmite$, C_3AH_6, C_2AH_{18}, $Calcite$, α-$Cristobalite$, C_2AS)								DTA-TGA: % Weight Loss	% Boehmite	% Calcite	% CaO Dry Basis
CGA with H_2S, H_2O saturated	vapor	231/ 447	6.9/1000	44	M	tr	m			tr	tr	tr	8.1	19.8	6.2	3.8
	liquid	231/ 447	6.9/1000	44	not tested											
CGA with H_2S, saturated[f] followed by firing in air[g]		240/ 465	6.9/1000	5	M	tr	m			tr	tr		6.7	13.2	6.6	4.0
		538/1000	ambient	3/4	not tested											
Cycling tests--repeat of preceding exposure followed by the same air firing				1 cycle	M	tr				m	tr		4.2	0	8.3	4.9
				2 cycles	M	tr				m	tr		4.8	0	9.4	5.6
				3 cycles	M	tr				m	tr		3.0	0	6.2	3.6
				4 cycles	M	tr				m	tr	tr	3.1	0	6.5	3.8
				5 cycles	M	tr				m	tr		4.1	0	8.8	5.1

54% ALUMINA-40% SILICA INSULATING CASTABLE[k]

Atmosphere[a]		Temperature °C / °F	Pressure MPa/psi	Time days	XRD (α-$Quartz$, $Kyanite$, α-Al_2O_3, $Boehmite$, CA, $Calcite$, CAS_4H_2)							DTA-TGA: % Weight Loss	% Boehmite	% Calcite
Air (dried)		110/ 230	ambient	1	M	M	m		m					
Saturated steam	vapor	231/ 447	2.8/410	10	M	m	m					4.1	4.0	
	liquid	231/ 447	2.8/410	10	M	m	m	tr				3.7	4.0	
Saturated steam	vapor	285/ 545	6.9/1000	10	M	m	m					3.8	3.6	
	liquid	285/ 545	6.9/1000	10	M	m	m			tr		6.2	6.1	
CGA with H_2S, H_2O saturated	vapor	231/ 447	6.9/1000	10	M	m	m	tr	tr	m		4.5	6.5	3.5
	liquid	231/ 447	6.9/1000	10	M	m	m	tr	tr	m		6.7	6.0	2.8
CGA with H_2S, H_2O saturated	vapor	231/ 447	6.9/1000	44	M	m	tr	tr	m					
	liquid	231/ 447	6.9/1000	44	M	m	tr	tr	m					
CGA with H_2S, saturated[f] followed by firing in air[g]		240/ 465	6.9/1000	5	M	m	m	tr	m					
		538/1000	ambient	3/4	M	m	m		m					
Cycling tests--repeat of preceding exposure followed by the same air firing				1 cycle	M	m	m		m					
				2 cycles	M	m	m		m					
				3 cycles	M	m	m		m					
				4 cycles	M	m	m		m					
				5 cycles	M	m	m		m					

47% ALUMINA-40% SILICA INSULATING CASTABLE[l]

Atmosphere[a]		Temperature °C / °F	Pressure MPa/psi	Time days	XRD ($Mullite$, α-Al_2O_3, β-$Cristobalite$, $Boehmite$, $Kyanite$, α-$Quartz$, CAS_4H_2, $Calcite$, CA)									DTA-TGA: % Weight Loss	% Boehmite	% Calcite
Saturated steam	vapor	231/ 447	2.8/410	10	M	tr	tr	m						7.6	13.5	
	liquid	231/ 447	2.8/410	10	M	tr	tr	m						4.7	10.1	
Saturated steam	vapor	285/ 545	6.9/1000	10	M			m	m					6.2	12.8	
	liquid	285/ 545	6.9/1000	10				tr	m	M	m			5.5	13.0	
CGA with H_2S, H_2O saturated	vapor	231/ 447	6.9/1000	10		m		tr	m	M	m	tr		10.8	16.2	6.1
	liquid	231/ 447	6.9/1000	10		m		tr	m	M	m	tr		8.4	16.5	6.6

35% ALUMINA-53% SILICA INSULATING CASTABLE[m]

Atmosphere[a]		Temperature °C / °F	Pressure MPa/psi	Time days	XRD (α-$Quartz$, α-Al_2O_3, C_2AS, $Bayerite$, CAS_4H_2, $Boehmite$, $Calcite$)							DTA-TGA: % Weight Loss	% Boehmite	% Calcite
Saturated steam	vapor	231/ 447	2.8/410	10	M	m	m	tr				5.7	5.4	
	liquid	231/ 447	2.8/410	10	M	m	m	tr				5.8	5.5	
Saturated steam	vapor	285/ 545	6.9/1000	10	M	m			m			4.0	8.2	
	liquid	285/ 545	6.9/1000	10	M	m		m	m	tr		4.7	9.5	
CGA with H_2S, H_2O saturated	vapor	231/ 447	6.9/1000	10	M	m				m		10.	9.5	6.5
	liquid	231/ 447	6.9/1000	10	M	m				m		10.8	10.8	7.3

[a] For testing the effect of water-saturated vapor and of liquid, samples were exposed in a steam generator, both in the vapor and in the liquid in the bottom of the vessel. After placement of samples, the vessel was heated to the desired steam pressure and where tests included other gases, these were then added so as to obtain the desired overall concentrations. The overall composition of the coal gasification atmosphere (CGA) is (in vol %) 18 CO, 12 CO_2, 24 H_2, 40 H_2O, 5 CH_4, and 1 H_2S. A gaseous flow rate was maintained so as to provide one complete change in atmosphere every 24 hours.

[b] Other tests showed that boehmite formation occurred in steam-saturated atmospheres and resulted in very large increases in the flexural strength. These cycling tests were designed to investigate the effect of repetitive formation and decomposition of boehmite on the properties of the refractories.

[c] After casting, specimens were cured 24 hours in air at 100% humidity at ambient temperatures, and dried at 230 °F for 24 hours. Specimens were stored at 230 °F until tested.

[d] Phases were identified by x-ray diffraction analysis; M = major phase, m = minor, tr = trace. Cement notation used to specify some compounds (C = CaO, A = Al_2O_3, H = H_2O, S = SiO_2). No data are given for the phases present after firing in air.

[e] 93% Alumina dense castable, UMR-1 generic preparation (70% tabular alumina + 30% calcium aluminate cement) (T-61 alumina, Alcoa; CA-25 Calcium Aluminate Cement, Alcoa).

(Table Continued)

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EFFECT OF SATURATED VAPOR AND LIQUID AND EXPOSURE[a] TO PRESSURE-TEMPERATURE CYCLING CAUSING BOEHMITE FORMATION AND DECOMPOSITION[b]
ON THE PHASES PRESENT IN ALUMINA CASTABLE REFRACTORIES[c][39], Continued

Footnotes continued

[f]85% steam saturated; this exposure is the one forming the first step in the cycling tests, forming boehmite.

[g]The firing step, following the saturated CGA exposure completed one cycle, decomposing the boehmite.

[h]87.4% Alumina dense castable, UMR-4 generic preparation (70% tabular alumina + 30% calcium aluminate cement) (T-61 alumina, Alcoa; Refcon, 58% alumina-33% calcia cement, Universal Atlas).

[i]63.8% Alumina, 28.3% silica castable, UMR-8 generic preparation (75% calcined kaolin aggregate + 25% calcium aluminate cement) (Mulcoa M60 and Mulcoa M47, C-E Minerals; CA-25 Calcium Aluminate Cement, 79% alumina-18% calcia, Alcoa).

[j]58.6% Alumina, 29.6% silica castable, UMR-5 generic preparation (75% calcined kaolin aggregate + 25% calcium aluminate cement) (Mulcoa M60 and Mulcoa M47, C-E Minerals; Refcon, 58% alumina-33% calcia cement, Universal Atlas).

[k]54% Alumina, 40% silica insulating castable, calcium aluminate (79% alumina-18% calcia) bonded (Cer-Lite #75, C-E Refractories; CA-25 Calcium Aluminate Cement, Alcoa).

[l]46.7% Alumina, 40.2% silica insulating castable (Litecast 60-25, General Refractories).

[m]34.5% Alumina, 52.5% silica insulating castable (VSL-50, A.P. Green).

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ANALYSIS OF REACTION PRODUCTS[a] OF REFRACTORIES EXPOSED[b] TO AN
ACIDIC HIGH-IRON COAL SLAG[c][32]

Refractory	Results of Exposure
General: all alumina-chromia refractories reacted with the iron oxide in the slag to form complex intermediate spinel layers, $(Fe,Mg)(Cr,Al)_2O_4$.	
$Al_2O_3(99)-Na_2O(0.5)$, fused-cast brick (Monofrax Al, Carborundum)	Reaction produced a reasonably coherent layer of spinel (80-200 μm), mostly Fe and Al with some Mg and Cr. Minimal intergranular attack occurred. Fe and Mg penetrated ∿200 μm into refractory. $(Fe,Mg)Al_2O_4$ spinel crystallites precipitated in slag on cooling.
$Al_2O_3(89.7)-Cr_2O_3(10)$, sintered brick (CS-612, Kaiser Refractories)	Coherent spinel layer formed ∿100 μm thick containing Cr in addition to Fe, Al and some Mg, $(Fe,Mg)(Cr,Al)_2O_4$. Intergranular attack was significant.
$Al_2O_3(67)-Cr_2O_3(32)-P_2O_5(1)$, plastic (---/---)	Coherent spinel layer formed ∿200-400 μm thick, $(Fe,Mg)(Cr,Al)_2O_4$. Intergranular attack was significant. Thin layer at slag-refractory interface was highly enriched with Fe^{+2}.
$Al_2O_3(60)-Cr_2O_3(27)-MgO(6)-Fe_2O_3(4)-SiO_2(2)$, fused-cast brick (Monofrax K-3, Carborundum)	As-received refractory is $(Fe,Mg)(Cr,Al)_2O_4$ spinel in $(Al,Cr)_2O_3$ matrix. No intergranular attack occurred. Matrix reacted with iron oxide to form 50-100 μm thick spinel layer. Highly Fe^{+2}-rich layer formed at slag-refractory interface.
$Al_2O_3(65)-Cr_2O_3(32)-Fe_2O_3(1)-CaO(0.6)-MgO(0.6)$, fused-cast brick (Corhart AC-28, Corhart Refractories)	As-received refractory is a sesquioxide solid solution. Penetration of iron oxide to form spinel reaction product was limited to only 50-75 μm.
$Cr_2O_3(80)-MgO(8)-Fe_2O_3(6)-Al_2O_3(5)-SiO_2(1)$, fused-cast brick (Monofrax E, Carborundum)	Little reaction occurred. Very thin layer (∿5 μm) of spinel formed, Fe,Al enriched and Cr,Mg depleted compared with original refractory. Original spinel was Fe-enriched and Mg-depleted to a depth of ∿1000 μm.
$Al_2O_3(84.5)-Cr_2O_3(10.5)-SiO_2(0.2)-P_2O_5(4.6)$, plastic (86B, Lava Crucible)	Loose network of spinel reaction product formed, $(Fe,Mg)Al_2O_4$, 200-300 μm thick. Very extensive intergranular attack occurred.
$MgO(41.6)-Cr_2O_3(27.1)-Al_2O_3(13.3)-Fe_2O_3(15.9)-CaO(0.5)-SiO_2(1.2)$, sintered brick (---/---)	Magnesia matrix was preferentially dissolved leaving Cr-rich spinel inclusions.

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(Table Continued)

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ANALYSIS OF REACTION PRODUCTS[a] OF REFRACTORIES EXPOSED[b] TO AN
ACIDIC HIGH-IRON COAL SLAG[c][32], Continued

Footnotes _____

[a]The information was obtained by metallographic, scanning electron micro-
scopic, and electron microprobe examination of the refractories (see
Section B.1.2.23 for microprobe analysis and Section B.1.2.15 (last test)
for material removed in testing).

[b]Plenum temperature was maintained at 1575 °C for 493 hours with an oxygen
partial pressure of $\sim 10^{-2}$ to 10^{-4} Pa.

[c]Average slag composition (in weight percent) during exposure: SiO_2 44.6,
Al_2O_3 18.7, CaO 10.1, Fe_2O_3 1.2, FeO 18.0, Fe 0.5, MgO 3.0, Na_2O 1.2,
K_2O 1.4, TiO_2 0.9, other 0.3; base to acid ratio 0.55.

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COMPOSITION AS A FUNCTION OF DISTANCE[a] FROM SLAG-REFRACTORY INTERFACE FOR REFRACTORIES EXPOSED TO ACIDIC HIGH-IRON COAL SLAG[b][32]

(Data Continued)

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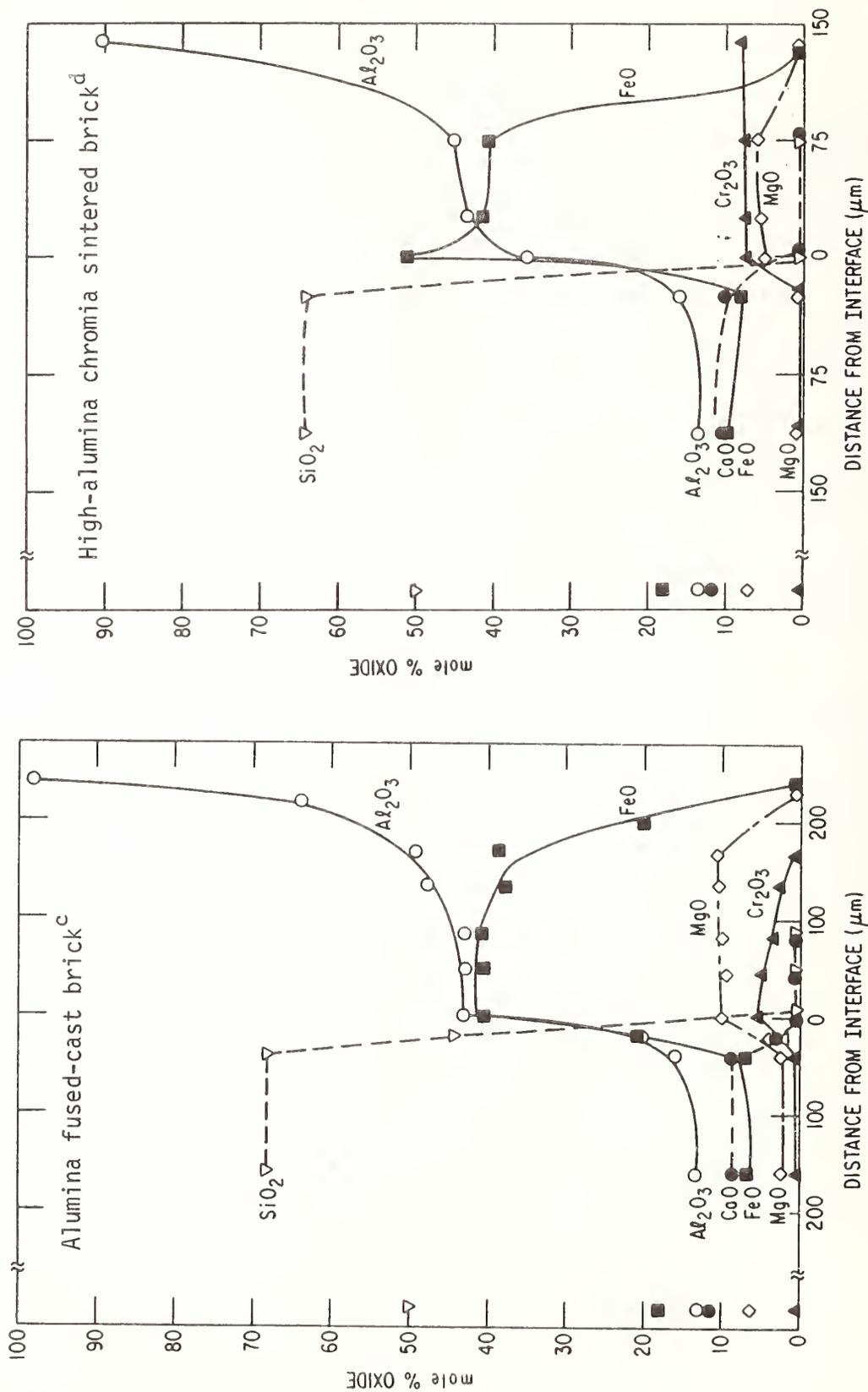
COMPOSITION AS A FUNCTION OF DISTANCE[a] FROM SLAG-REFRACTORY INTERFACE FOR REFRACTORIES EXPOSED

TO ACIDIC HIGH-IRON COAL SLAG[b][32], Continued
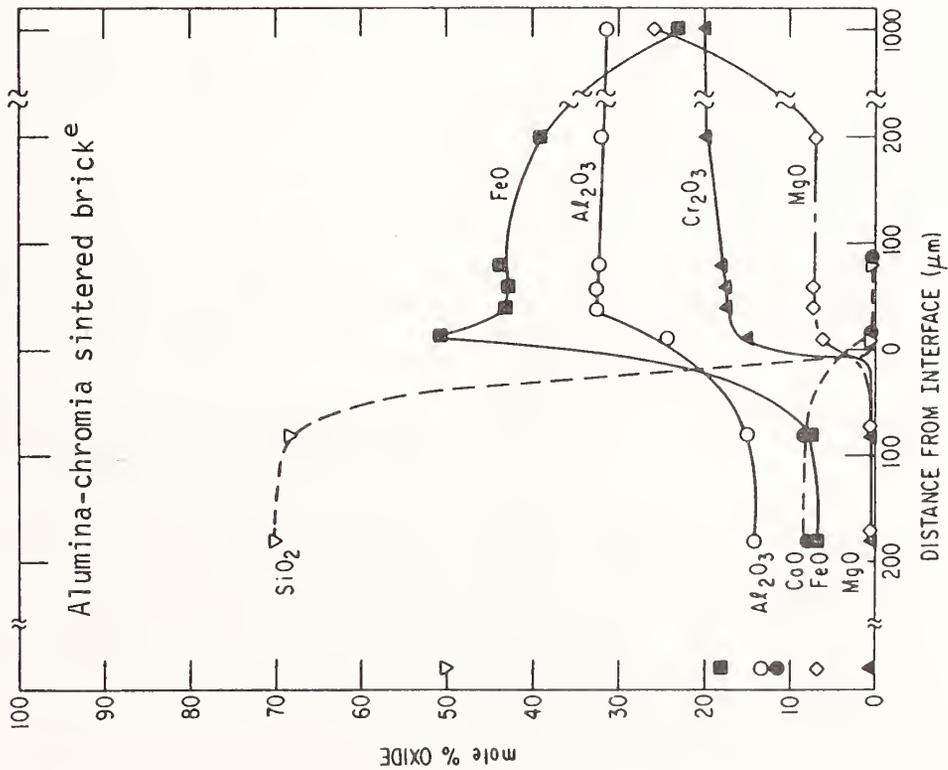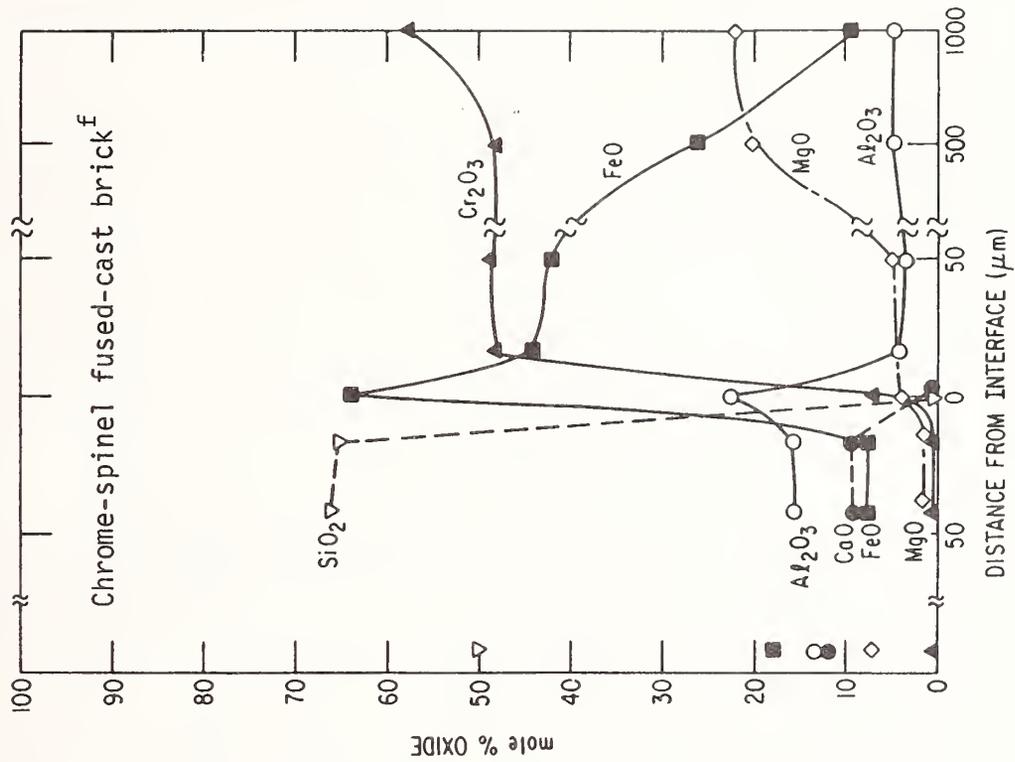
(Data Continued)

COMPOSITION AS A FUNCTION OF DISTANCE[a] FROM SLAG-REFRACTORY INTERFACE FOR REFRACTORIES EXPOSED

TO ACIDIC HIGH-IRON COAL SLAG[b][32], Continued

Footnotes

[a]As determined by electron microprobe analyses, calibrated with standards of MgO, Fe, Cr, Al_2O_3, CaF_2, and SiO_2. Wet chemical analyses indicated that >90% of the iron is Fe^{+2} so all iron is given as FeO. Symbols at the extreme left of each graph indicate the as-formulated composition of the slag (see Section B.1.2.22 for descriptive information and Section B.1.2.15 (last test) for material removed in testing). Note that composition on graphs is in mole percent not weight percent.

[b]Average slag composition (in weight percent) during testing: SiO_2 44.6, Al_2O_3 18.7, CaO 10.1, Fe_2O_3 1.2, FeO 18.0, Fe 0.5, MgO 3.0, Na_2O 1.2, K_2O 1.2, TiO_2 0.9, other 0.3; base to acid ration 0.55.

[c]Al_2O_3(99)-Na_2O(0.5), Monofrax Al (Carborundum).

[d]Al_2O_3(89.7)-Cr_2O_3(10), CS 612 (Kaiser Refractories).

[e]Al_2O_3(60.4)-Cr_2O_3(27.3)-MgO(6.0)-Fe_2O_3(4.2)-SiO_2(1.8), Monofrax K-3 (Carborundum).

[f]Cr_2O_3(80)-MgO(8)-Fe_2O_3(6)-Al_2O_3(5)-SiO_2(1), Monofrax E (Carborundum).

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ANALYSIS OF REACTION PRODUCTS[a] OF REFRACTORIES EXPOSED[b] TO A
BASIC COAL SLAG[c][32]

Refractory	Results of Exposure
$Al_2O_3(99)$–$Na_2O(0.5)$, fused-cast brick (Monofrax Al, Carborundum)	Slag (gehlenite, $Ca_2Al_2SiO_7$) plus refractory produced calcium hexaluminate ($CaO.6Al_2O_3$) with some Fe-rich spinel. Some intergranular corrosion but no swelling of the refractory was observed.
$Al_2O_3(89.7)$–$Cr_2O_3(10)$, sintered brick (CS 612, Kaiser Refractories)	Reaction produced calcium hexaluminate with some Cr ($Ca\{Al,Cr\}_{12}O_{19}$) and some spinel ($Fe\{Fe,Al,Cr\}_2O_4$). Extensive intergranular reaction produced substantial swelling of the outer 20 mm of the brick.
$Al_2O_3(81.1)$–$Cr_2O_3(16.6)$–$P_2O_5(0.8)$–$SiO_2(0.5)$–$Fe_2O_3(0.5)$–$Na_2O(0.5)$, sintered brick (852Z {Serv-M}, Taylor Refractories	More resistant to reaction than the brick above. Besides calcium hexaluminate, significant amounts of spinel were produced. No intergranular attack was observed, depth of penetration was small, no swelling of the brick occurred.
$Al_2O_3(65)$–$Cr_2O_3(32)$–$FeO(1)$–$CaO(0.6)$–$MgO(0.6)$, fused-cast brick (Corhart AC-28, Corhart Refractories)	Better resistance exhibited than first two bricks above. No calcium hexaluminate found. Dense $(Mg,Fe)(Al,Cr,Fe)_2O_4$ spinel layer formed.
$MgO(65)$–$Al_2O_3(35)$, fused-cast brick (X-317, Corhart Refractories)	Periclase (MgO) grains and spinel ($MgAl_2O_4$) matrix of as-received material underwent Fe enrichment. Grains were more readily dissolved by slag than was the matrix.
$MgO(60)$–$Cr_2O_3(15.5)$–$Al_2O_3(15)$–$Fe_2O_3(7)$–$SiO_2(1.5)$–$CaO(1)$, sintered brick (Krilex 911, Kaiser Refractories)	MgO grains were attacked preferentially. Chrome-spinel phase was highly resistant to attack by slag. Fe enrichment of both phases near the slag-refractory interface was again observed.

[a]The information was obtained by metallographic and scanning electron microscope examination of the refractories.

[b]Exposure was for ∼500 hours at 1500 °C as described in footnote a of Section B.1.2.15 (see B.1.2.15 for corrosion data, next-to-last test).

[c]Average slag composition (in weight percent) during the exposure: SiO_2 24.4, Al_2O_3 15.5, CaO 38.9, Fe_2O_3 4.2, FeO 9.0, Fe 1.0, MgO 5.7, Na_2O <0.1, K_2O <0.1, TiO_2 1.0; base to acid ratio 1.4.

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ANALYSIS OF REACTION PRODUCTS[a] OF SOME REFRACTORIES EXPOSED[b] TO
SOME ACIDIC COAL SLAGS[32]

Refractory   Results of Exposure

  Average Slag Composition During Test (wt %), Exposure Time 500 hours[c]

SiO_2	Al_2O_3	CaO	Fe_2O_3	FeO	MgO	Na_2O	K_2O	TiO_2	Cr_2O_3	B/A[d]
39.5	20.0	24.0	0.4	6.6	6.4	1.4	0.8	0.9	0.3	0.64

$MgO(63)-Cr_2O_3(18)-Al_2O_3(5)-$ Microstructure of as-received refrac-
 $Fe_2O_3(12)-SiO_2(1)-CaO(1)$,   tory is Mg-rich grain with some Cr,Fe,Al
 brick   in the form of spheroidal precipitates
(Guidon, Harbison-Walker)   of spinel. Reaction produced a lace-
                                         work structure of $(Mg,Fe)(Cr,Al,Fe)_2O_4$
                                         spinel which recrystallized withing the
                                         slag (mellilite, $Ca_2Al_2SiO_7-Ca_2MgSi_2O_7$)
                                         after the MgO grain dissolved to form
                                         diopside ($CaMgSi_2O_6$).

  Average Slag Composition During Test (wt %), Exposure Time 500 hours[e]

SiO_2	Al_2O_3	CaO	Fe_2O_3	FeO	MgO	Na_2O	K_2O	TiO_2	Other	B/A[d]
39.4	17.1	25.8	2.8	4.9	6.4	1.3	1.0	1.1	0.2	0.75

$Al_2O_3(81.1)-Cr_2O_3(16.6)-P_2O_5(0.8)-$ As-received refractory is mostly large
 $SiO_2(0.5)-Fe_2O_3(0.5)-Na_2O(0.5)$, Al_2O_3 grains in Cr_2O_3-rich matrix.
 sintered brick   Reaction formed a solid layer of complex
(852Z {Serv-M}, Taylor Refrac-   spinel $(Mg,Fe)(Cr,Al,Fe)_2O_4$.
  tories)

  Average Slag Composition During Test (wt %), Exposure Time 1000 hours[f]

SiO_2	Al_2O_3	CaO	Fe_2O_3	FeO	Fe	MgO	Na_2O	K_2O	TiO_2	B/A[d]
36.9	17.4	29.2	2.8	3.2	0.4	7.3	1.4	0.8	0.6	0.9

$Cr_2O_3(80)-MgO(8)-Fe_2O_3(6)-$ Refractory recrystallized at slag inter-
 $Al_2O_3(5)-SiO_2(1)$, fused-cast   face forming a sharp line of demarcation,
 brick   a solid band of spinel $\sim$0.5 mm deep.
(Monofrax E, Carborundum)

$Al_2O_3(60)-Cr_2O_3(27)-MgO(6)-$ Refractory-slag interface not as sharp
 $Fe_2O_3(4)-SiO_2(2)$, fused-cast   as in above brick; spinel layer was up
 brick   to 2 mm thick. $(Mg,Fe)(Cr,Al,Fe)_2O_4$
(Monofrax K-3, Carborundum)   spinel zone graded rapidly to a mixture
                                         of chrome-spinel and solid solution of
                                         $(Al,Cr,Fe)_2O_3$ typical of as-received
                                         refractory.

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(Table Continued)

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ANALYSIS OF REACTION PRODUCTS[a] OF SOME REFRACTORIES EXPOSED[b] TO
SOME ACIDIC COAL SLAGS[32], Continued

Refractory	Results of Exposure
$Al_2O_3(91.6)$–$SiO_2(8.0)$–$Fe_2O_3(0.15)$–Alkali(0.15), sintered brick (Kricor, Kaiser Refractories)	Refractory-slag interface not as sharply defined as in first brick above. Slag penetrated refractory enveloping the largest sintered Al_2O_3 grains; spinel, $MgAl_2O_4$, formed shells around the larger grains. Smaller Al_2O_3 grains entered into spinel formation.
$SiC(75)$–$Si_3N_4(23)$, sintered brick (Refrax 20, Carborundum)	Sharp, uneven line formed the refractory slag interface. Slag attached both SiC and Si_3N_4 bonding matrices. Some SiC was reduced. Small Fe- and Si-rich metallic inclusions were dispersed in slag at the interface. Iron oxides in the slag reacted with SiC grains to produce ferrosilicon alloy and carbon oxide gases.
$SiC(73)$–$Al_2O_3(14)$–$SiO_2(11)$, sintered brick (Harbide, Harbison-Walker)	Reaction of slag and refractory similar to that of preceding brick.

[a] The information was obtained by metallographic and scanning electron microscope examination of the refractories.

[b] Exposure was at ∿1500 °C as described in footnote a of Section B.1.2.15.

[c] See Section B.1.2.15, second test for corrosion data.

[d] B/A = base to acid ratio.

[e] See Section B.1.2.15, fourth test, for corrosion data.

[f] See Section B.1.2.15, sixth test, for corrosion data.

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SLAG CORROSION TESTING[a] OF MAGNESIA-CHROMIA SPINEL REFRACTORY BRICKS[32,89]

Refractory	Composition(wt %)	Open Porosity[b] %	Slag[c]	Test Temperature °C	Oxygen Pressure Pa (atm)	Test Time h	Radial Decrease[d] mm (mils)
Commercial rebonded, fused-grain picromite (magnesia-chromia spinel)	Cr_2O_3 72.8, MgO 25.5, Al_2O_3 0.4, Fe_2O_3 0.6, SiO_2 0.6, TiO_2 0.1	16.8	1	1480	$\sim10^{-3}$ $(\sim10^{-8})$[e]	4	(0)
		15.7	1	1480	$\sim10^{-3}$ $(\sim10^{-8})$[e]	16	0.08(3)
		15.5	1	1580	$\sim10^{-2.4}$ $(\sim10^{-7.4})$[e]	4	(10)
		22.9	2	1600	(3.5×10^{-9})[f]	24	1.12(2)
Developmental high-chromia brick	Cr_2O_3 77.8, MgO 18.8, Al_2O_3 0.01, Fe_2O_3 0.01, SiO_2 3.0, CaO 0.04	26.3	1	1480	$\sim10^{-3}$ $(\sim10^{-8})$[e]	4	(0)
Developmental high-chromia brick	Cr_2O_3 71.3, MgO 17.0, Al_2O_3 3.0, Fe_2O_3 5.7, SiO_2 3.1, CaO 0.03	~25.0	1	1500	$\sim10^{-3}$ $(\sim10^{-8})$[e]	8	(0)
Commercial magnesia spinel, direct bond	Cr_2O_3 43.0, Al_2O_3 9.0, Fe_2O_3 12.5, MgO 33.5, SiO_2 1.3, CaO 0.5	22.6	1	1480	$\sim7\times10^{-4}$ $(\sim7\times10^{-9})$[e]	8	0 (0)
Commercial chromia spinel, direct bond	Cr_2O_3 78.0, Al_2O_3 1.0, Fe_2O_3 1.5, MgO 18.0, SiO_2 0.5, CaO 0.5	24.4	1	1480	$\sim7\times10^{-4}$ $(\sim7\times10^{-9})$[e]	8	0.28(11)[g]
		19.3	2	1600	(3.5×10^{-9})[f]	24	0.23(9)
		10.9	2[h]	1600	(3.5×10^{-9})[f]	24	0.89(36)
Commercial chromia spinel, direct bond	Cr_2O_3 79.7, Al_2O_3 4.7, Fe_2O_3 6.1, MgO 8.1, SiO_2 1.3	12.3	1	1480	$\sim7\times10^{-4}$ $(\sim7\times10^{-9})$[e]	8	0 (0)
Commercial chromia-alumina solid solution, Si-rich glass bond	Cr_2O_3 60.0, Al_2O_3 20.0, Fe_2O_3 0.1, MgO 0.1, ZrO_2 12.0, SiO_2 6.5, CaO 0.1, TiO_2 0.7	16.2	2	1500	$\sim2\times10^{-3}$ $(\sim2\times10^{-8})$[f]	16	0 (0)
		15.7	2	1500	(0.6×10^{-9})[f]	24	0 (0)
		18.4	2	1600	(3.5×10^{-9})[f]	24	0.58(23)
		17.5	2[i]	1600	(3.5×10^{-9})[f]	24	0.05(2)
Developmental chromia spinel, direct bond	Cr_2O_3 82.0, MgO 18.0	18.0	2	1600	(3.5×10^{-9})[f]	24	0.10(4)
		14.1	3	1500	(6×10^{-9})[e]	24	0.13(5)
		13.8	2[h]	1600	(3.5×10^{-9})[f]	24	0.36(14)
		14.3	3[h]	1500	(6.0×10^{-9})[e]	24	0.09(3.6)
		14.4	3[j]	1500	(6.0×10^{-9})[e]	24	0.10(4.0)
		14.2	3	1600	(25.0×10^{-9})[e]	24	0.45(18.0)
Commercial alumina-chromia solid solution	Cr_2O_3 10.0, Al_2O_3 89.7, Fe_2O_3 0.1, SiO_2 ~0.1	17.1	2	1500	(0.6×10^{-9})[f]	24	Sample completely dissolved
Developmental alumina-chromia solid solution	Cr_2O_3 54.4, Al_2O_3 45.0, SiO_2 0.1	15.6	2	1600	(3.5×10^{-9})[f]	24	0.69(27.0)
Developmental magnesia spinel, direct bond	Cr_2O_3 62.4, Al_2O_3 8.4, Fe_2O_3 10.8, MgO 17.5, SiO_2 0.4, CaO 0.4	24.9	2	1600	(3.5×10^{-9})[f]	24	Sample disintegrated

[a] Tests conducted in a high-temperature controlled-atmosphere rotating-spindle apparatus. A cylindrical specimen (25.4 mm diameter x 25.4 mm high) was mounted on a Mo rod which rotated the specimen in a slag bath. Rotational speed was 100 rpm except as noted under the slag heading.

[b] As determined by ASTM method C20-74.

[c] Slag compositions (wt %)--#1: 50 SiO_2, 5 CaO, 20 Al_2O_3, 25 FeO; #2: 53 SiO_2, 16 CaO, 17 Al_2O_3, 6 FeO, 2 Na_2O, 1 K_2O, 1 TiO_2, 4 MgO; #3: 50 SiO_2, 5 CaO, 20 Al_2O_3, 15 FeO, 10 MgO.

[d] As determined with any adhering slag film.

[e] Produced by gas mixture of 3% H_2, 3% CO_2, balance N_2.

[f] Produced by gas mixture of 4% H_2, 1.7% CO_2, balance Ar.

[g] Operating problems probably caused high corrosion loss.

[h] Rotational speed was 150 rpm.

[i] Rotational speed was 50 rpm.

[j] Rotational speed was 200 rpm.

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VISIBLE DEGRADATION EFFECTS OF CARBON MONOXIDE GAS MIXTURES[a] ON IRON-DOPED[b] REFRACTORIES[27]

Feed Gas Composition	90% Alumina Castable[c]	50% Alumina Castable[d]	90% Alumina Ramming Mix[e]
99.9% CO	Fe dopant-- Considerable spalling in one of 5 samples with 0.5% Fe. Spalling increased with increased Fe content. Fe$_2$O$_3$ dopant-- No spalling occurred. Color changed from red or yellow to dark gray or black.	Fe dopant-- Samples with 0.5% Fe showed some corner spalling. Complete disintegration occurred for higher Fe levels. Fe$_2$O$_3$ dopant-- No spalling occurred. Color changed to dark gray or black.	Fe dopant-- No advanced spalling occurred. Hairline cracks seen almost full length of sample face on 2 of 4 samples of 1.5 and 2.0% Fe. Matrix turned gray. Fe$_2$O$_3$ dopant-- Hairline cracks seen almost full length of sample face on 2 of 4 samples of 1.5 and 2.0% Fe. Color turned blue or black.
95% CO- 5% CO$_2$	Fe dopant-- Minor spalling occurred in 1 of 4 samples with 1.5% Fe, in 2 of 4 with 2.0% Fe. Fe$_2$O$_3$ dopant-- No spalling occurred.	Fe dopant-- Large cracks appeared in 2 of 4 samples with 0.5% Fe. More severe spalling for higher Fe level; at least 2 of 4 samples at each level completely disintegrated. Fe$_2$O$_3$ dopant-- No spalling occurred.	Fe dopant-- Corner and edge spalling seen on 2 of 4 samples with 1.0% Fe. Fe$_2$O$_3$ dopant-- Carbon balls and large cracks seen on 2 of 4 samples with 1.5% Fe. Significant spalling occurred in 3 of 4 samples with 2.0% Fe.
85% CO- 15% CO$_2$	Fe dopant-- Minor edge or corner spalling in 1 of 4 samples with 1.0% Fe and in 3 of 4 with 2.0% Fe. Fe$_2$O$_3$ dopant-- Color appeared white to light yellow rather than gray.	Fe dopant-- Large cracks seen in 1 of 4 samples with 2.0% Fe. Fe$_2$O$_3$ dopant-- Color ranged from off-white for 0.5% Fe samples to very light gray for 1.0 and 1.5% Fe samples to light gray for 2.0% Fe samples.	Fe dopant-- Corner and edge spalling occurred in 3 of 4 samples with 1.0% Fe. More Fe produced significant spalling in at least 2 of 4 samples at each Fe level and minor spalling in the rest of the samples. Fe$_2$O$_3$ dopant-- Carbon balls appeared in 2 of 4 samples with 1.0% Fe; significant spalling in 1 of 4 samples at 1.5% Fe with cracks appearing in 2 of the 3 remaining. 1 of 4 samples with 2.0% Fe had a deep crack with spalling.
99.8% CO- 0.2% NH$_3$	Fe dopant-- Corner spalling occurred on 1 of 4 samples with 1.0% Fe; corner and edge spalling on 2 of 4 samples with 1.5% Fe, and on 3 of 4 samples with 2.0% Fe. Fe$_2$O$_3$ dopant-- Samples similar in appearance to those in the plain CO exposure but the color was lighter.	Fe dopant-- Cracking or heavy spalling in 3 of 4 samples with 1.5% Fe and in all 4 samples with 2.0% Fe. Fe$_2$O$_3$ dopant-- Samples looked the same as those in the plain CO exposure.	Fe dopant-- Moderate to heavy spalling occurred in 3 of 4 samples with 1.0% Fe. Heavy spalling occurred in 1 of 4 samples with 1.5% Fe; 2 others showed minor edge and corner spalling. Corner and edge spalling occurred in 2 of 4 samples with 2.0% Fe. Fe$_2$O$_3$ dopant-- No spalling occurred.

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(Table Continued)

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VISIBLE DEGRADATION EFFECTS OF CARBON MONOXIDE GAS MIXTURES[a] ON IRON-DOPED[b] REFRACTORIES[27], Continued

Feed Gas Composition	90% Alumina Castable[c]	50% Alumina Castable[d]	90% Alumina Ramming Mix[e]
99.2% CO-0.8% NH_3	Fe dopant-- Moderate spalling occurred on 1 of 4 samples with 1.0 and 2.0% Fe. 2 of 4 samples with 1.5% Fe spalled moderately. Remaining samples showed minor edge or corner spalling. Fe_2O_3 dopant-- Samples similar in appearance to those in the plain CO exposure but slightly darker in color.	Fe dopant-- Heavy spalling occurred in all 4 samples at 1.0 and 2.0% Fe levels. 2 of 4 samples with 1.5% Fe were completely disintegrated. Fe_2O_3 dopant-- Samples similar in appearance to those in the plain CO exposure but lighter in color.	Fe dopant-- Moderate to heavy spalling occurred in all 4 samples with 1.0% Fe. Moderate spalling occurred in 2 of 4 samples at 1.5 and 2.0% Fe. Fe_2O_3 dopant-- No spalling occurred.
99.8% CO-0.2% H_2S	Fe dopant-- Large crack appeared in 1 of 4 samples with 1.0% Fe. Moderate spalling occurred on 1 of 4 samples with 1.5% Fe. Heavy spalling occurred on 2 of 4 samples with 2.0% Fe. Fe_2O_3 dopant No spalling occurred. Color ranged from off-white for 0.5% Fe levels to medium gray for 2.0% Fe levels.	Fe dopant-- 1 of 4 samples with 0.5% Fe spalled heavily. All 4 samples with 1.0 and 1.5% Fe levels completely disintegrated. Of the 4 samples with 2.0% Fe one spalled heavily and another disintegrated. Fe_2O_3 dopant-- Color ranged from off-white to light gray at 0.5% Fe to dark gray at 2.0% Fe.	Fe dopant-- Moderate spalling occurred on 1 of 4 samples for the 0.5, 1.5, and 2.0% Fe levels For the 1.0% Fe level, one spalled heavily and another of the 4 samples spalled moderately. Fe_2O_3 dopant-- Heavy spalling occurred on 3 of 4 samples with 1.5% Fe and the 4th completely disintegrated. At 2.0% Fe level 2 of 4 samples spalled heavily and the remaining showed minor corner and edge spalling.
99.2% CO-0.8% H_2S	Fe dopant-- Moderate spalling on 1 and heavy spalling on another occurred for 4 samples with 2.0% Fe. No spalling occurred at lower levels of Fe. Fe_2O_3 dopant-- Color of samples ranged from off-white at 0.5% Fe to dark gray at 2.0% Fe.	Fe dopant-- Large cracks appeared in 1 of 4 samples with 0.5 and 1.0% Fe. 2 of 3 samples with 1.0% Fe cracked into 2 pieces. All 4 samples with 1.5% Fe cracked into 2 or more pieces as did 1 of 4 with 2.0% Fe. Fe_2O_3 dopant-- Color of samples ranged from off-white to light gray at 0.5% Fe to dark gray at 2.0% Fe.	Fe dopant-- Light spalling occurred in 1 of 4 samples with 1.0% Fe. All 4 samples with 1.5% Fe spalled moderately. Moderate spalling occurred in 2 of 4 samples with 2.0% Fe. Fe_2O_3 dopant-- Heavy spalling occurred in 2 of 4 samples with 1.0, 1.5, and 2.0% Fe levels. Remaining 2 at 1.5 and 2.0% Fe levels completely disintegrated.
80% CO-20% H_2O	Fe dopant-- No spalling occurred. Fe_2O_3 dopant-- No spalling occurred. Color of samples ranged from off-white at 0.5% Fe to medium gray at 2.0% Fe.	Fe dopant-- No spalling occurred. Fe_2O_3 dopant-- No spalling occurred. Color ranged from light gray at 0.5% Fe to medium gray at 2.0% Fe with a yellowish tint at 1.0, 1.5, and 2.0% Fe.	Fe dopant-- No spalling occurred. Matrix of all samples was black. Fe_2O_3 dopant-- No spalling occurred. Matrix of all samples was black.

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(Table Continued)

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VISIBLE DEGRADATION EFFECTS OF CARBON MONOXIDE GAS MIXTURES[a] ON IRON-DOPED[b] REFRACTORIES[27], Continued

Feed Gas Composition	90% Alumina Castable[c]	50% Alumina Castable[d]	90% Alumina Ramming Mix[e]
60% CO- 40% H_2O	Fe dopant-- No spalling occurred. Fe_2O_3 dopant-- No spalling occurred. Color for 0.5% Fe samples was white, for 1.0 and 1.5% Fe light gray, and for 2.0% Fe yellow-tinted medium gray.	Fe dopant-- No spalling occurred. Fe_2O_3 dopant-- No spalling occurred. Color for 0.5% Fe samples was light gray, for 1.0 and 1.5% Fe yellow-tinted gray, and for 2.0% Fe dark gray.	Fe dopant-- No spalling occurred. Matrix of all samples was black. Fe_2O_3 dopant-- No spalling occurred. Matrix of all samples was black.
80% CO- 20% H_2	Fe dopant-- Heavy spalling occurred in 3 of 4 samples with 2.0% Fe. Other levels of Fe doping showed no damage. Fe_2O_3 dopant-- Color of samples changed to off-white.	Fe dopant-- No spalling occurred. Color of samples darkened more than in plain CO exposure. Fe_2O_3 dopant-- No spalling occurred. Color of samples darkened more than in plain CO exposure.	Fe dopant-- Spalling occurred on 2 faces of 1 of 4 samples with 2.0% Fe. Fe_2O_3 dopant-- No spalling occurred. Color of samples remained light gray.
60% CO- 40% H_2	Fe dopant-- No spalling occurred. Fe_2O_3 dopant-- Color of samples turned yellowish-white.	Fe dopant-- No spalling occurred. Fe_2O_3 dopant-- Color of samples ranged from yellowish-white at the 0.5% Fe level to light gray at 2.0% Fe.	Fe dopant-- No spalling occurred. All samples turned dark gray to black. Fe_2O_3 dopant-- All samples turned dark gray to black.

[a]Samples were exposed to the gas mixtures for 100 hours at 500 °C and 1 atmosphere pressure of the gases.

[b]The refractories were doped with 0.5, 1.0, 1.5, and 2.0 wt% Fe added either as metallic iron (Fe) or as hematite (Fe_2O_3). Five samples of each dopant concentration were exposed to the CO atmosphere, four samples of each dopant concentration were exposed in each of the other mixed gases.

[c]A DOE-specified generic composition; prepared from 65 wt% tabular alumina (25 wt% 6 to 10 mesh, 20 wt% 10 to 20 mesh, 20 wt% less than 20 mesh), 10 wt% calcined alumina (less than 325 mesh), 25 wt% calcium aluminate cement (Casting Grade CA-25 cement), all Alcoa materials. Materials were dry mixed for 2 min, then water added with continued mixing to ball-in-hand consistency (∿605 ml); refractory was then poured into 2 in. x 2 in. x 2 in. aluminum molds, vibrated for 5 min, sealed in plastic for 24 h, then fired in air for 5 h at 1100 °C.

[d]A DOE-specified generic composition; prepared from 75 wt% calcined kaolin (25 wt% 6 to 10 mesh, 20 wt% 10 to 20 mesh, 15 wt% less than 20 mesh, 20 wt% less than 200 mesh), 25 wt% calcium aluminate cement (Casting Grade CA-25, Alcoa). Samples were prepared as described in footnote c except that the water used was ∿700 ml.

[e]A DOE generic ramming mix; prepared from 82 wt% tabular alumina (30 wt% 6 to 10 mesh, 20 wt% 10 to 20 mesh, 15 wt% less than 20 mesh, 17 wt% less than 48 mesh), 15 wt% calcined alumina (less than 325 mesh), 3 wt% bentonite; to this was added 1 wt% hydrated alumina, 6 wt% phosphoric acid (85%), and 0.25 wt% water. Dry materials were mixed and then hand mixed in plastic bags with the liquids; mixture was aged for 18 h; samples were formed by die pressing at 500 psi, cured at 250 °C in air for 12 h, and then fired at 1100 °C for 5 h.

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EFFECT OF ALKALI-IMPREGNATION[a] ON THE CO DISINTEGRATION[b] OF IRON-
DOPED ALUMINA CASTABLES[c][27]

Castable	Dopant	Visual Appearance
90% Alumina[d]	0	No damage[f]
90% Alumina	1.0 wt% Fe	Samples disintegrated
50% Alumina[e]	0	No damage[f]
50% Alumina	1.0 wt% Fe	Samples disintegrated

[a] Cured castable samples were soaked in 50% Na_2CO_3-50% K_2CO_3 saturated solution for 24 hours. They were then air dried and prefired at 1100 °C for 12 hours.

[b] Samples were exposed to 99.9% carbon monoxide at 500 °C at atmospheric pressure for 100 hours.

[c] Three samples were prepared of each castable with no iron added and three each with iron dopant to provide 1.0 wt% Fe.

[d] A DOE generic composition: 65 wt% tabular alumina (25 wt% 6 to 10 mesh, 20 wt% 10 to 20 mesh, 20 wt% less than 20 mesh), 10 wt% calcined alumina (less than 325 mesh), 25 wt% calcium aluminate cement (Casting Grade CA-25 cement), all Alcoa materials. Materials were dry mixed for 2 min, then water added with continued mixing to ball-in-hand consistency (∿605 ml); refractory was then poured into 2 in. x 2 in. x 2 in. aluminum molds, vibrated for 5 min, sealed in plastic for 24 h, then fired for 5 h in air at 1100 °C.

[e] A DOE generic composition: 75 wt% calcined kaolin (25 wt% 6 to 10 mesh, 20 wt% 10 to 20 mesh, 15 wt% less than 20 mesh, 20 wt% less than 200 mesh), 25 wt% calcium aluminate cement (Casting Grade CA-25, Alcoa). Samples were prepared in the same way as the 90% alumina castable except that the water used was ∿700 ml.

[f] Compressive strength was unusually high (8000+ psi), apparently due to the effect of the alkali soaking.

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EFFECT OF CO ATMOSPHERE[a] ON STAINLESS STEEL REINFORCED[b] 50% ALUMINA
CASTABLE[c][27]

Type of Fiber	No. of Samples	Pressure	Time	Visual Appearance
310 SS	4	atmospheric	100 h	All samples contained pop-outs and surface cracks. Samples very weak.
446 SS	4	atmospheric	100 h	All samples contained pop-outs and surface cracks. Samples very weak.
310 SS	2	1000 psi	50 h	Samples completely destroyed.

[a] Samples were exposed to 99.9% carbon monoxide at the specified pressure at 500 °C.

[b] Specimens were prepared with the addition of 4 wt% stainless steel fibers.

[c] Castable was a high-strength 50% alumina prototype castable (Mix 36C, Babcock & Wilcox).

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EFFECT OF PREFIRING[a] ON 90% ALUMINA CASTABLES[b] EXPOSED TO CARBON
MONOXIDE[c][91]

Castable Composition[b]	Prefired		Condition
90% Alumina	no		Heavy Carbon Deposition
90% Alumina		yes	Light Carbon Deposition
90% Alumina, 0.1% Fe doped	no		Sample Disintegrated
90% Alumina, 0.1% Fe doped		yes	Sample Disintegrated
90% Alumina, 310 SS fibers[d]		yes	Sample Disintegrated

[a] Firing in air at 1100 °C for 5 hours.

[b] A DOE generic castable; see footnote d of Section B.1.2.28 for the preparation.

[c] Samples were exposed to 99.9% CO at 500 °C and 1000 psi for 50 hours.

[d] Sample from the U.S. Bureau of Mines, stainless steel fibers added, 4 weight percent.

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EFFECT OF WATER-IMMERSION IN CARBON MONOXIDE/STEAM/H_2S ATMOSPHERE[a] ON CaO CONTENT

OF ALUMINA REFRACTORIES[b][39]

Atmosphere[a]	Temperature °C / °F[h]	Time days	Calcium Oxide Content,[c] %			
			93% Alumina[d]	91% Alumina[e]	57% Alumina[f]	54% Alumina[g]
– – – – –Control Value– – – –	– – –	– – –	5.4	7.8	5.5	2.9
CO/H_2O = 0.1 (CO 90, H_2O 900, H_2S 10, all psia)	278/532	20	3.60	4.67	3.73	2.40
CO/H_2O = 1.0 (CO 495, H_2O 495, H_2S 10, all psia)	241/466	20	1.60	2.00	2.43	2.53
CO/H_2O = 3.0 (CO 742.5, H_2O 247.5, H_2S 10, all psia)	204/400	20	0.80	0.72	1.28	0.67
CO/H_2O = 0.44 (CO 18, CO_2 12, H_2 24, CH_4 5, H_2O 40, H_2S 1, all vol %)	231/447	60	not analyzed	4.40	3.06	6.45

[a] Samples were exposed in the liquid in the bottom of a steam generator pressure vessel. All tests were at 1000 psia (6.9 MPa). See Sections B.3.2.57, B.3.2.58, B.4.2.12-14, and B.4.2.32 for other data for these tests and for the test procedures.

[b] Refractories were cast in molds, cured 24 hours in air at 100% humidity at ambient temperature, and dried at 230 °F for 24 hours. All specimens were stored at 230 °F until tested.

[c] Percent CaO determined by x-ray fluorescence analysis.

[d] 93% Alumina dense castable, UMR-1 generic preparation (70% tabular alumina + 30% calcium aluminate cement) (T-61 alumina, Alcoa; CA-25 Calcium Aluminate Cement, Alcoa).

[e] 91% Alumina dense castable, UMR-2 generic preparation (70% tabular alumina + 30% calcium aluminate cement) (T-61 alumina, Alcoa; Secar 71(250) cement, Lone Star Lafarge).

[f] 57% Alumina, 34% silica castable, calcium aluminate bonded (RC-3, General Refractories; CA-25, Alcoa).

[g] 54% Alumina, 40% silica insulating castable, calcium aluminate bonded (Cer-Lite #75, C-E Refractories; CA-25, Alcoa).

[h] Control value is based on manufacturer's chemical analysis.

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EFFECT OF EXPOSURE[a] TO CARBON MONOXIDE/STEAM/HYDROGEN SULFIDE IN SATURATED VAPOR OR LIQUID ON THE PHASES PRESENT IN VARIOUS ALUMINA CASTABLE REFRACTORIES[b][39]

93% ALUMINA DENSE CASTABLE[d]

Atmosphere[a]	Location	Temperature	Alumina	Boehmite	Calcite				% Weight Loss	% Boehmite	% Calcite	% CaO, Dry Basis
$CO/H_2O = 0.1 + 1\% H_2S$	Unsaturated vapor	1000 °F	M		M				4.1	0	7.8	4.5
	Saturated vapor	532 °F	M	M	M				8.4	30.0	7.0	4.3
	Liquid	532 °F							9.5	25.3	6.0	3.7

91% ALUMINA DENSE CASTABLE[e]

Atmosphere[a]	Location	Temperature	Alumina	Boehmite	Calcite				% Weight Loss	% Boehmite	% Calcite	% CaO, Dry Basis
$CO/H_2O = 0.1 + 1\% H_2S$	Unsaturated vapor	1000 °F	M		M				5.3	0	10.9	6.5
	Saturated vapor	532 °F	M	M	M				10.2	28.8	8.4	5.3
	Liquid	532 °F							9.2	28.9	6.0	3.7

87% ALUMINA DENSE CASTABLE[f]

Atmosphere[a]	Location	Temperature	Alumina	Boehmite	Calcite	C2AS			% Weight Loss	% Boehmite	% Calcite	% CaO, Dry Basis
$CO/H_2O = 0.1 + 1\% H_2S$	Unsaturated vapor	1000 °F	M		m	m			4.1	0	7.1	4.2
	Saturated vapor	532 °F							8.6	23.9	7.9	4.8
$CO/H_2O = 1.0 + 1\% H_2S$	Unsaturated vapor	1000 °F							7.7	23.5	7.2	4.4
	Saturated vapor	466 °F	M	m	M	m			8.1	16.6	9.9	6.1
	Liquid	466 °F	M	m	M	m						

88% ALUMINA DENSE CASTABLE[g]

Atmosphere[a]	Location	Temperature	Alumina	Boehmite	Calcite	α-Quartz	C3AH6		% Weight Loss	% Boehmite	% Calcite	% CaO, Dry Basis
$CO/H_2O = 1.0 + 1\% H_2S$	Saturated vapor	466 °F	M	m	M	m	tr		5.4	10.8	5.5	3.2

84% ALUMINA DENSE CASTABLE[h]

Atmosphere[a]	Location	Temperature	Alumina	Boehmite	Calcite	α-Quartz	CA2		% Weight Loss	% Boehmite	% Calcite	% CaO, Dry Basis
$CO/H_2O = 1.0 + 1\% H_2S$	Saturated vapor	466 °F	M	m	tr	M	tr		5.3	9.7	4.0	3.4

57% ALUMINA–34% SILICA DENSE CASTABLE[i]

Atmosphere[a]	Location	Temperature	Alumina	Boehmite	Calcite	Mullite	α-Cristobalite		% Weight Loss	% Boehmite	% Calcite	% CaO, Dry Basis
$CO/H_2O = 0.1 + 1\% H_2S$	Unsaturated vapor	1000 °F	M		tr	M	M		1.9	0	3.0	1.7
	Saturated vapor	532 °F	tr	M	m	M			5.1	13.6	2.1	1.2
	Liquid	532 °F							6.0	18.2	4.2	2.5
$CO/H_2O = 1.0 + 1\% H_2S$	Saturated vapor	466 °F	m	m	tr	M	M					

59% ALUMINA–30% SILICA DENSE CASTABLE[j]

Atmosphere[a]	Location	Temperature	Alumina	Boehmite	Calcite	Mullite			% Weight Loss	% Boehmite	% Calcite	% CaO, Dry Basis
$CO/H_2O = 0.1 + 1\% H_2S$	Unsaturated vapor	1000 °F							0.8	0	1.1	0.6
	Saturated vapor	532 °F							5.3	14.1	3.0	1.8
	Liquid	532 °F							5.1	15.7	3.7	2.2
$CO/H_2O = 1.0 + 1\% H_2S$	Saturated vapor	466 °F	m	m	M	M			6.6	10.0	7.5	4.5

54% ALUMINA–40% SILICA INSULATING CASTABLE[k]

Atmosphere[a]	Location	Temperature	Alumina	Boehmite	Calcite	Kyanite	α-Quartz		% Weight Loss	% Boehmite	% Calcite	% CaO, Dry Basis
$CO/H_2O = 0.1 + 1\% H_2S$	Unsaturated vapor	1000 °F	m		tr	m	M		1.2	0	1.8	1.0
	Saturated vapor	532 °F	m			m	M		4.4	8.3	1.6	0.9
	Liquid	532 °F							4.5	8.7	3.5	2.1
$CO/H_2O = 1.0 + 1\% H_2S$	Unsaturated vapor	1000 °F	m		tr	m	M					
	Saturated vapor	466 °F	m	tr	tr	m	M					
	Liquid	466 °F	m	tr	tr	m	M					

46% ALUMINA–40% SILICA INSULATING CASTABLE[l]

Atmosphere[a]	Location	Temperature	Alumina	Boehmite	Kyanite	Mullite	α-Quartz	C12A7	C2AS	% Weight Loss	% Boehmite	% Calcite	% CaO, Dry Basis
$CO/H_2O = 0.1 + 1\% H_2S$	Unsaturated vapor	1000 °F			m	M	m		m	2.2	0	3.4	1.9
	Saturated vapor	532 °F								11.0	21.8	4.1	2.6
$CO/H_2O = 1.0 + 1\% H_2S$	Saturated vapor	466 °F		tr	m		M	m					
	Liquid	466 °F	m	m	m		M	tr					

35% ALUMINA–53% SILICA INSULATING CASTABLE[m]

Atmosphere[a]	Location	Temperature	Alumina	Boehmite	α-Quartz	CA2	C2AS		% Weight Loss	% Boehmite	% Calcite	% CaO, Dry Basis
$CO/H_2O = 0.1 + 1\% H_2S$	Unsaturated vapor	1000 °F	m		M		m		1.8	0	2.3	1.3
	Saturated vapor	532 °F							4.0	10.8	0.9	0.5
$CO/H_2O = 1.0 + 1\% H_2S$	Saturated vapor	466 °F	M	tr	M	m						
	Liquid	466 °F	m		M	m						

(Table Continued)

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EFFECT OF EXPOSURE[a] TO CARBON MONOXIDE/STEAM/HYDROGEN SULFIDE IN SATURATED VAPOR OR LIQUID ON THE PHASES PRESENT IN VARIOUS
ALUMINA CASTABLE REFRACTORIES[b][39], Continued

Atmosphere[a]	Location	Temperature	XRD Analysis Results[c]			DTA-TGA Analyses % Weight Loss

- 96% ALUMINA PHOSPHATE-BONDED RAMMING MIX[n] -

Atmosphere[a]	Location	Temperature	Alumina	Berlinite	α-CaSiO3	% Weight Loss
$CO/H_2O = 0.1 + 1\%$ H_2S	Unsaturated vapor	1000 °F	M	m		0
	Saturated vapor	532 °F	M	m		0.2
	Liquid	532 °F				0.4
$CO/H_2O = 1.0 + 1\%$ H_2S	Saturated vapor	466 °F	M	m		0.6
	Liquid	466 °F	M	m	tr	0.8

[a]Samples were exposed in two pressure vessels. One vessel was a steam generator and tests which specify saturated conditions occurred in this vessel. After placing samples, the vessel was heated to the desired steam pressure and the other gases were then added so as to obtain the concentrations specified in the table. The second vessel was connected to the steam generator and after samples were placed and the vessel heated to the test temperature, the vessel was pressurized with the steam/gas mixture from the steam generator. Tests specified as unsaturated occurred in this vessel. A gaseous flow rate for both vessels was maintained so as to provide one complete change in atmosphere every 24 hours. The specimens were exposed at the temperatures specified in the table at 1000 psia for 20 days. For $CO/H_2O = 0.1$: CO 90 psia, H_2O 900 psia, H_2S 10 psia. For $CO/H_2O = 1.0$: CO 495 psia, H_2O 495 psia, H_2S 10 psia.

[b]All cement-bonded castables were cast in molds, cured 24 hours in air at 100% humidity at ambient temperature, and dried at 230 °F for 24 hours. Phosphate-bonded specimens were prepared by hand ramming in molds, immediately drying for 24 hours at 230 °F and then firing at 1000 °F for 18 hours. All specimens were stored at 230 °F until tested.

[c]Phases were identified by x-ray diffraction analysis; M = major phase, m = minor phase, tr = trace. Cement notation used to specify some compounds (C = CaO, A = Al_2O_3, S = SiO_2, H = H_2O).

[d]93% Alumina dense castable, UMR-1 generic preparation (70% tabular alumina + 30% calcium aluminate cement) (T-61 alumina, Alcoa; CA-25 Calcium Aluminate Cement, 79% alumina-18% calcia, Alcoa).

[e]91% Alumina dense castable, UMR-2 generic preparation (70% tabular alumina + 30% calcium aluminate cement) (T-61 alumina, Alcoa; Secar 71(250), 72% alumina-26% calcia cement, Lone Star Lafarge).

[f]87.4% Alumina dense castable, UMR-4 generic preparation (70% tabular alumina + 30% calcium aluminate cement) (T-61 alumina, Alcoa; Refcon, 58% alumina-33% calcia cement, Universal Atlas).

[g]88% Alumina dense castable, UMR-6 generic preparation (UMR-1 generic preparation with 5% SiO_2 added as 99.9% pure bone dry Wedron silica flour).

[h]84% Alumina dense castable, UMR-7 generic preparation (UMR-1 generic preparation with 10% SiO_2 added as 99.9% pure bone dry Wedron silica flour).

[i]57% Alumina, 34% silica castable, calcium aluminate (79% alumina, 18% calcia) bonded (RC-3, General Refractories; CA-25 Calcium Aluminate Cement, Alcoa).

[j]58.6% Alumina, 29.6% silica castable, UMR-5 generic preparation (75% Mulcoa + Mulgrain M-60, C-E Minerals; 25% Refcon, 58% alumina-33% calcia cement, Universal Atlas).

[k]54% Alumina, 40% silica insulating castable, calcium aluminate (70% alumina-18% calcia) bonded (Cer-Lite #75, C-E Refractories; CA-25 Calcium Aluminate Cement, Alcoa).

[l]46.7% Alumina, 40.2% silica insulating castable (Litecast 60-25, General Refractories).

[m]34.5% Alumina, 52.5% silica insulating castable (VSL-50, A.P. Green).

[n]96% Alumina dense phosphate-bonded ramming mix (Greenpak-90P, A.P. Green).

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EFFECT OF ALKALI-CONTENT[a] OF SIMULATED CATALYTIC COAL GASIFICATION ATMOSPHERE[b] ON
SEVERAL ALUMINA-CONTAINING REFRACTORIES[c][9]

Refractories included[c]	Gas used[b]	Alkali addition[a]	Exposure Time, h	Observations
95% Al_2O_3 dense castable (Castolast G) 45% Al_2O_3 dense castable (H.S. Brikcast BF) 45% Al_2O_3 fired super duty brick (KX-99)	CCG gas[b]	Molten KOH[d] KOH/coal mix[e] Aqueous KOH injection[f]	100	Refractories submerged in molten alkali were severely attacked. Considerable absorption of alkali by refractory occurred due to vapor phase transport.
90% Al_2O_3 dense castable (DOE 90 generic preparation) 55% Al_2O_3 dense castable (Lo-Abrade) 50% Al_2O_3 dense castable (Mix 36-C generic preparation) 45% Al_2O_3 dense castable (H.S. Brikcast BF)	CCG gas	Aqueous KOH injection Molten KOH (as alkali vapor source)	250	Refractories doubled in alkali content due to exposure. 90% Al_2O_3 castable showed significantly lower hot flexural strength than specimens fired in air at the same temperature for the same time. 55% Al_2O_3 castable showed about the same hot strength as the air-fired specimens. 50 and 45% Al_2O_3 castables had a higher hot strength than air-fired specimens but less than half the increase in strength following exposure to dry ash gasifier environments.
90% Al_2O_3 dense castable (DOE 90 generic) 55% Al_2O_3 dense castable (Lo-Abrade) 50% Al_2O_3 dense castable (Mix 36-C generic) 45% Al_2O_3 dense castable (H.S. Brikcast BF)	Steam-argon	Molten KOH (as alkali vapor source)	250	K_2O content increased. Wt % K_2O

Sample	before	after
90%	0.02	0.05
55%	0.58	3.40
50%	0.25	0.29
45% (1st)	0.06	0.22
45% (2nd)	--	0.33

(Table Continued)

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EFFECT OF ALKALI-CONTENT[a] OF SIMULATED CATALYTIC COAL GASIFICATION ATMOSPHERE[b] ON SEVERAL ALUMINA-CONTAINING REFRACTORIES[c][9], Continued

Refractories included[c]	Gas used	Alkali addition[a]	Exposure Time, h	Observations
45% Al_2O_3 dense castable (B&W Kaocrete D)				Absorbed K_2O indicated above was water soluble.
95% Al_2O_3 dense castable (Castolast G) 55% Al_2O_3 dense castable (Lo-Abrade) 45% Al_2O_3 dense castable (H.S. Brikcast BF)	CCG gas with no H_2S	Molten KOH (as alkali vapor source)	50	Refractories showed no absorption of K_2O from vapor phase transport.
95% Al_2O_3 dense castable (Castolast G) 55% Al_2O_3 dense castable (Lo-Abrade) 45% Al_2O_3 dense castable (H.S. Brikcast BF)	Steam-argon + H_2S with no CO_2	Molten KOH (as alkali vapor source)	50	Refractories showed no absorption of K_2O from vapor phase transport.
95% Al_2O_3 dense castable (Castolast G) 55% Al_2O_3 dense castable (Lo-Abrade) 45% Al_2O_3 dense castable (H.S. Brikcast BF) 45% Al_2O_3 fired super duty brick (KX-99)	CCG gas	KOH/coal mix Molten KOH (as alkali vapor source)	100	Refractories showed no corrosion either from contact with the KOH/coal mix or the alkali vapor environment. Cold compressive strengths were unaffected by exposure.
95% Al_2O_3 dense castable (Castolast G) 45% Al_2O_3 dense castable[g] (H.S. Brikcast BF) 45% Al_2O_3 fired super duty brick (KX-99)	CCG gas	KOH/coal mix Molten KOH (as alkali vapor source)	2000	In vapor phase exposure, the lower Al_2O_3 castable showed an increase in compressive strength, the high Al_2O_3 castable showed a decrease (see B.3.2.108 for data). The same

(Table Continued)

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EFFECT OF ALKALI-CONTENT[a] OF SIMULATED CATALYTIC COAL GASIFICATION ATMOSPHERE[b] ON
SEVERAL ALUMINA-CONTAINING REFRACTORIES[c][9], Continued

Refractories included[c]	Gas used	Alkali addition[a]	Exposure Time, h	Observations
90% Al_2O_3 ramming mix, phosphate bonded (Brikram 90R)				trends were observed for abrasion resistance. The K_2O content increased by a factor of 4 in the refractories (chemical analysis by atomic absorption). In the KOH/coal exposure, no deterioration was noted during 500-h inspection intervals, but after 2000 h obvious degradation was observed. SEM analysis of sections of all refractories showed large amounts of K had migrated throughout the samples.

[a] Alkali was introduced to the test environment by several methods (see footnotes d, e, and f), in order to simulate the catalytic coal gasification conditions where alkali is the catalytic agent.

[b] The atmosphere was a reducing one: 21.6 H_2, 32.2 H_2O, 6.1 CO, 14.2 CO_2, 22 CH_4, 2.1 N_2, 1.2 NH_3, and 1.0 H_2S (mole %). All tests were at 730 °C (~1350 °F) and 500 psig.

[c] Samples were prepared as follows: fired brick was taken as received; castables (all calcium aluminate bonded) were thoroughly mixed, oven dried for 24 h at 230 °F, cooled in a moisture-proof container, stored at 75 °F for 24 h, mixed at 144 rpm with 25 °C water to give ball-in-hand consistency, troweled into molds and vibrated for 3 minutes, cured covered with plastic for 24 h at 90 °F and 100% humidity, and dried 24 h at 230 °F; the ramming mix, as-received, was pressed at 1000 psi into mold, dried at 250 °F for 28 h, heated at 50 °F/h to 500 °F, held 24 h, heated at 50 °F/h to 1000 °F, held 24 h, and cooled slowly to ambient temperature.

[d] KOH was placed inside the test chamber in crucibles. Some specimens were placed in the molten KOH so as to be exposed both to the vapor phase above the molten alkali and to the effects of the molten KOH on the submerged portion. In some tests samples were not placed in the crucibles but the molten KOH was included to provide alkali in the vapor.

(Table Continued)

EFFECT OF ALKALI-CONTENT[a] OF SIMULATED CATALYTIC COAL GASIFICATION ATMOSPHERE[b] ON

SEVERAL ALUMINA-CONTAINING REFRACTORIES[c][9], Continued

Footnotes continued

[e]Some refractory specimens were subjected to contact with KOH-impregnated coal. A mix of 80% Illinois No. 6 coal and 20% KOH was prepared and packed in holes in the specimens.

[f]During some tests a KOH-water solution (44.34 g KOH/liter) was pumped into the test chamber at a rate such that if all the alkali vaporized, a concentration of 50 ppm (mole basis) of KOH vapor would result. This concentration corresponds to a KOH saturation of the atmosphere at 730 °C.

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EFFECT OF HEAT TREATMENT[a] ON THE PHASE ASSEMBLAGE[b] OF A HIGH-ALUMINA

CASTABLE REFRACTORY[c][9]

Maximum Cure Temperature, °C	Phases Present[b]
120	α-Al_2O_3, CA, C_3AH_6, Al(OH)$_3$
600	α-Al_2O_3, CA, β-$NaAl_{11}O_{17}$, CA_2
800	α-Al_2O_3, $C_{12}A_7$, CA, CA_2
1,000	α-Al_2O_3, CA, CA_2, $C_{12}A_7$
1,200	α-Al_2O_3, CA_2, CA
1,400	α-Al_2O_3, CA_2, β-$NaAl_{11}O_{17}$, CA

[a] Heat treatment consisted of raising the temperature at the rate of 30 °C/h from ambient to 540 °C, holding for 6 hours, then raising at 60 °C/h to the "maximum cure temperature". Samples were soaked at the cure temperature for 8 hours and then the furnace and contents were cooled naturally to ambient conditions.

[b] Phases were determined by x-ray diffraction and are listed in order of decreasing abundance. Cement notation used to abbreviate phase designations: A = Al_2O_3, C = CaO, H = H_2O.

[c] Castable used is a high-purity tabular alumina, calcium aluminate bonded (Castolast G, Harbison-Walker). Samples were prepared by mixing 10 ml H_2O per 100 g dry refractory in a Hobart mixer for 1 minute, then casting, curing under 100% relative humidity at 29 °C for 24 hours, and drying at 120 °C for 24 hours.

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SUMMARY OF VISUAL INSPECTION[a] OF ALUMINA CASTABLES[b] AFTER EXPOSURE[c] TO SIMULATED COAL GASIFICATION ATMOSPHERES[d][95]

Castable tested[b]	Post-Exposure Castable Condition[a]			
	Gas A--58 h	Gas A--1000 h	Gas B--1000 h	Gas C--1000 h
DENSE CASTABLES:				
90% Al$_2$O$_3$	Unaffected, no carbon spots evident	Unaffected	Affected: small surface popouts	Unaffected
90% Al$_2$O$_3$ + 0.1% Fe	Unaffected, no carbon spots evident	Unaffected	Destructive conditions: can be broken by hand pressure	Unaffected
90% Al$_2$O$_3$ + 310 SS[e]	Carbon deposits evident; extensive surface cracking; corners and edges showed deterioration	Not tested	Not tested	Not tested
90% Al$_2$O$_3$ + alkali[f]	Unaffected, no carbon spots evident	Not tested	Not tested	Not tested
50% Al$_2$O$_3$	Unaffected, no carbon spots evident	Unaffected	Affected: large surface popouts (1/4 in.), many carbon spots	Unaffected
50% Al$_2$O$_3$ + 0.1% Fe	Unaffected, no carbon spots evident	Not tested	Not tested	Not tested
50% Al$_2$O$_3$ + 310 SS[e]	Carbon deposits evident; extensive surface cracking; corners and edges showed deterioration	Not tested	Not tested	Not tested
50% Al$_2$O$_3$ + alkali[f]	Unaffected, no carbon spots evident	Not tested	Not tested	Not tested
INSULATING CASTABLES:				
95% Al$_2$O$_3$	Not tested	Unaffected	Unaffected	Unaffected

(Table Continued)

SUMMARY OF VISUAL INSPECTION[a] OF ALUMINA CASTABLES[b] AFTER EXPOSURE[c] TO SIMULATED COAL GASIFICATION ATMOSPHERES[d][95], Continued

Castable tested[b]	Post-Exposure Castable Condition[a]			
	Gas A--58 h	Gas A--1000 h	Gas B--1000 h	Gas C--1000 h
95% Al_2O_3 + rust	Not tested	Unaffected	Unaffected	Unaffected
55% Al_2O_3	Not tested	Unaffected	Affected: small surface popouts	Unaffected
55% Al_2O_3 + rust	Not tested	Unaffected	Affected: small surface popouts	Unaffected
40% Al_2O_3	Not tested	Unaffected	Affected: small surface popouts and cracks	Unaffected
40% Al_2O_3 + rust	Not tested	Unaffected	Affected: small surface popouts	Unaffected

[a]Reports state that the specimens subjected to the Gas B exposure were inspected visually following ANSI/ASTM-C288 guidelines. No such statement is made explicitly for the other exposures.

[b]No preparative information or other characterization was given in the reports and no commercial brand names were included. All castables are calcium aluminate bonded. No information was given about the manner of the Fe addition or the rust addition. See sections for reference [9] for earlier reports from the same organization.

[c]All exposures to the gases were at 500 °C and 1000 psi for the stated exposure times.

[d]Gas compositions in mole percent:
A = 38 H_2O, 24 H_2, 18 CO, 12 CO_2, 5 CH_4, 2 NH_3, 1 H_2S
B = 40 H_2O, 25 H_2, 18 CO, 12 CO_2, 5 CH_4
C = 40 H_2O, 25 H_2,　　 12 CO_2, 5 CH_4, 18 N_2.

[e]310 stainless steel fibers (4 wt%) were added to the mix before casting.

[f]Alkali-containing specimens were prepared by soaking for 6 hours in a 50% Na_2CO_3-50% K_2CO_3 saturated solution prior to firing.

EFFECT OF ALKALI ATTACK[a] IN A HIGH-BTU GASIFIER ATMOSPHERE[b] ON VARIOUS REFRACTORIES[95]

Refractory Type	Composition	Brand Name/Manufacturer	Observations after Exposure 50 hours	100 hours
Fused-cast spinel brick	72 Al_2O_3-28 MgO	X1233/?	Failed catastrophically, removed.	----
Fused-cast spinel brick	80 Cr_2O_3-8 MgO-6 Fe_2O_3	Monofrax E/Carborundum		No visual or mineralogical changes.
Phosphate-bonded ramming mix	67 Al_2O_3-32 Cr_2O_3-1 P_2O_5	Lavalox B-135/Lava Crucible		↑ All suffered some volume expansion, cracking or spalling. X-ray diffraction showed that in Al_2O_3-bearing refractories either carnegieite, β-Al_2O_3, or gibbsite, or a combination of these had formed causing volume expansion.
Fused-cast brick	60 Al_2O_3-27 Cr_2O_3-6 MgO-4 Fe_2O_3	Monofrax K-3/Carborundum		
Fireclay dense castable	58 Al_2O_3-28 SiO_2-5 CaO	?/?	← No changes apparent.	
Thermochemically-bonded ramming mix	80 Al_2O_3-12 SiO_2-6 ZrO_2 SiO_2	Valle-623/North American		
Resintered fused-cast mullite grain	78 Al_2O_3-20 SiO_2	Mulfrax W/?		
Fused-cast spinel brick	65 MgO-35 Al_2O_3	Corhart 317/Corhart		
High-alumina dense castable	92 Al_2O_3-3 CaO-1 SiO_2	?/?		
Resintered fused-cast grain	49 Al_2O_3-24 ZrO_2-15 Cr_2O_3-11 SiO_2	Unichrome/?		
Sintered brick	99 Al_2O_3	B&W SR 99/Babcock & Wilcox	→	↓
Sintered brick	90 Al_2O_3-10 Cr_2O_3	Ruby/Harbison-Walker		
Fused-cast spinel brick	80 Cr_2O_3-8 MgO-6 Fe_2O_3	Monofrax E/Carborundum	No changes apparent.	No visual or mineralogical changes.
Resintered fused-cast mullite grain	78 Al_2O_3-20 SiO_2	Mulfrax W/?	Began to crack.	----

(Table Continued)

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EFFECT OF ALKALI ATTACK[a] IN A HIGH-BTU GASIFIER ATMOSPHERE[b] ON VARIOUS REFRACTORIES[95], Continued

Refractory Type	Composition	Brand Name/Manufacturer	Observations after Exposure 50 hours	100 hours
Chromate-bonded magnesia ramming mix	Not given	F 264/?	Severely damaged, removed.	---
Spinel-bonded periclase burned brick	Not given	Magnel/ Harbison-Walker	Severely damaged, removed.	---
Chromate-bonded magnesia ramming mix	Not given	GUNTAPITE-382/ Quigley	Severely damaged, removed.	---
Phosphate-bonded fireclay ramming mix	Not given	HW 23-75/?	Began to crack.	Some conversion of mullite to car-negieite observed.
Phosphate-bonded high-alumina burned brick	Not given	Coral BP/ Harbison-Walker		[No comments given in reports; apparently unaffected.]
Fireclay super-duty brick	Not given	Bison/ Harbison-Walker	[No changes described in the reports.]	No volume change or cracking.[c]
Fireclay super-duty brick	Not given	HW 1-77/?		No volume or mineral change or cracking.
Fireclay super-duty brick	Not given	KX-99/ A.P. Green		No volume or mineral change or cracking.
Fireclay castable	Not given	B&W Kaocrete D/ Babcock & Wilcox		Some cracking; some mullite→carnegieite.
Fireclay super-duty brick	Not given	Varnon BF/ Harbison-Walker		No volume or mineral change or cracking.

a Crystals of NaOH were packed into holes drilled in refractory test specimens and the specimens were then subjected to high-Btu gas.

b The atmosphere (40 H_2O, 25 H_2, 18 CO, 12 CO_2, 5 CH_4) simulates that in the devolatilizer section of a

(Table Continued)

EFFECT OF ALKALI ATTACK[a] IN A HIGH-BTU GASIFIER ATMOSPHERE[b] ON VARIOUS REFRACTORIES[95], Continued

Footnotes continued

of a fixed-bed slagging gasifier (Grand Forks Energy Technology Center plant using lignite with an ash of approximately 5% Na_2O. Two tests were run, both at 1000 °C and 360 psig. Specimens were examined after 50 hours and 100 hours of testing.

[c]X-ray diffraction analysis showed a trace of carnegieite.

VISUAL APPEARANCE AND CRYSTALLINE PHASES[a] OF CERAMIC TUBES AND NODULES[b] FORMED ON THEM AFTER FUEL OIL COMBUSTION[c][106]

Material	As-Received	Post-Exposure Results[a]			
		Inside Tube	Upstream Side	Downstream Side	Nodule[b]
Al$_2$O$_3$, high-purity (AD 998, Coors Porcelain)	(Corundum)	Black, no cracks.	No nodules, no cracks, black. (Corundum, hercynite, low cristobalite)	No nodules, no cracks, black. (Corundum, hercynite)	---
Aluminosilicate, high-purity (Mullite, Coors Porcelain)	---	No change in color.	Longitudinal & transverse cracks; black, crusty appearance.	Longitudinal & transverse cracks; black.	---
Magnesium aluminosilicate (MAS 8200, GTE Sylvania)	(Cordierite, corundum)	Darkened in cracked areas.	Longitudinal & transverse cracks; black, dark green, tan; crusty appearance. (Hercynite, low cristobalite, cordierite)	Appearance the same as the upstream side of tube. (Tridymite, hercynite, cordierite, corundum)	---
Magnesium aluminosilicate (MAS 8400, GTE Sylvania)	(Cordierite, corundum)	Darkened in cracked areas.	Appearance the same as MAS 8200. (Cordierite, corundum, hercynite, tridymite)	Appearance the same as MAS 8200.	---
Zirconia plus mullite (Zirmul, GTE Sylvania)	(Zirconia, corundum, mullite)	Darkened in cracked areas.	Extensive cracks; black. (Hercynite, low cristobalite, tridymite)	Extensive cracks; black; some deposit at air exit end, black.	---

(Table Continued)

VISUAL APPEARANCE AND CRYSTALLINE PHASES[a] OF CERAMIC TUBES AND NODULES[b] FORMED ON THEM AFTER
FUEL OIL COMBUSTION[c][106], Continued

Post-Exposure Results[a]

Material	As-Received	Inside Tube	Upstream Side	Downstream Side	Nodule[b]
SiC, sintered-α (Carborundum)	(6H SiC, 2H SiC, graphite)	No change.	Nodules formed; no cracks, dark greenish. (6H SiC, 2H SiC, tridymite, cristobalite)	No nodules; no cracks; dark greenish. (6H SiC, 2H SiC, graphite)	(6H SiC, 2H SiC, low cristobalite, hercynite, corundum)
SiC, siliconized (KT-SiC, Carborundum)	(β-SiC, Si)	No change.	Nodules formed; no cracks; green-gray; tube slightly deformed and bent upward from the upstream side. (β-SiC, 6H SiC, Si, low cristobalite)	No nodules; no cracks; green-gray. (β-SiC, 6H SiC, Si, low cristobalite)	(β-SiC, Si, hercynite, low cristobalite)
SiC, siliconized (NC 430, Norton)	(6H SiC, β-SiC, Si)	No change.	Nodules formed; no cracks; dark greenish. (6H SiC, β-SiC, Si, high and low cristobalite)	No nodules; no cracks; dark greenish.	---
SiC, siliconized (Refel SiC, Pure Carbon Co.)	(β-SiC, Si)	No change.	Nodules formed; no cracks; dark brown. (β-SiC, Si, low cristobalite, tridymite)	No nodules; no cracks; dark brown.	(β-SiC, low cristobalite)
CVD SiC[d] (Deposits and Composites Inc.)	---	No change.	Same as α-SiC. (21R SiC, 6H SiC, low cristobalite)	Same as α-SiC. (21R SiC, 6H SiC)	---

(Table Continued)

VISUAL APPEARANCE AND CRYSTALLINE PHASES[a] OF CERAMIC TUBES AND NODULES[b] FORMED ON THEM AFTER FUEL OIL COMBUSTION[c][106], Continued

Material	As-Received	Inside Tube	Post-Exposure Results[a]		
			Upstream Side	Downstream Side	Nodule[b]
CVD SiC on SiC[e] (Deposits and Composites Inc.)	---	No change.	Same as α-SiC. (21R SiC, high cristobalite)	Same as α-SiC.	(21R SiC, low and high cristobalite)
SiC, clay-bonded (Carbofrax A, Carborundum)	(β-SiC, SiC, mullite)	No change.	Nodules formed; no cracks; green-gray. (Low cristobalite, β-SiC, hercynite, mullite)	No nodules; no cracks; green-gray.	(Low cristobalite, hercynite, tridymite)
SiC, clay-bonded (Carbofrax M, Carborundum)	(β-SiC, Si, BaSO₄)	No change.	Same as Carbofrax A.	Same as Carbofrax A.	---
SiC, Si₃N₄-bonded (Refrax 20, Carborundum)	(21R SiC, β-Si₃N₄)	No change.	Nodules formed; no cracks, dark brown.	No nodules; no cracks; dark brown.	(Low cristobalite, tridymite)

[a]Phases determined by x-ray diffraction. The phases are reported for the various locations after the visual observations and are in parentheses. Not all of the information was supplied for every portion or condition of the tubes in the column headings.

[b]Nodules formed on the upstream side of all SiC-based ceramic tubes and dark deposits developed on the outer surface of all oxide-based ceramic tubes.

[c]Ceramic tubes were exposed in the flue gas of a combustor. The combustor was fueled with No. 6 fuel oil with a high vanadium content (~83 weight ppm); 7 heating-holding-cooling cycles occurred during the test. The test is the Test Exposure 1 named in Section B.3.2.150. The test period was 500 hours, tube temperatures were 1100-1250 °C; flue gas temperatures-inlet 1410 °C, outlet 1385 °C; air temperatures-inlet ambient, outlet 340 °C; air velocity 8.1 m/s; heat transfer rate 23.7 kW; heat flux 54 kW/m².

[d]CVD = chemically vapor deposited.

[e]Chemically vapor deposited SiC on reaction-sintered SiC substrate.

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GLASS TRANSITION POINTS[a] FOR SiC CERAMICS AND NODULES FORMED AFTER
FUEL OIL COMBUSTION TESTING[b][106]
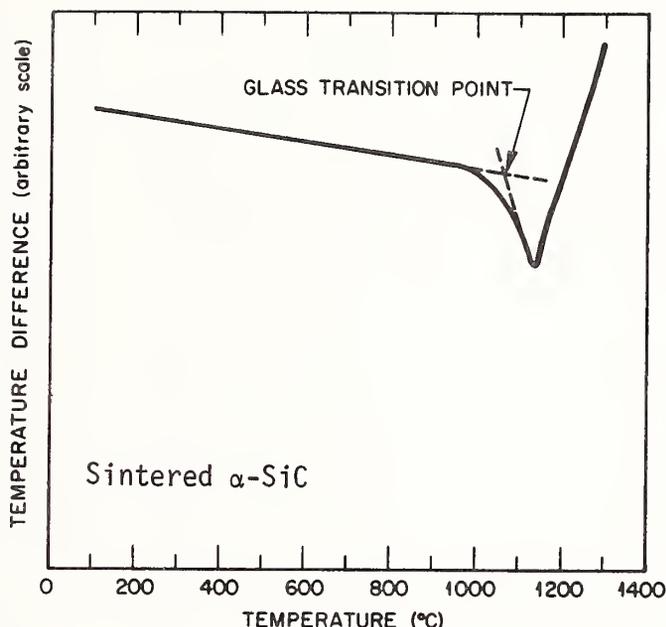
[a]Determined by differential thermal analysis on the nodules and parent SiC
after combustion exposure. The above is a typical DTA curve for these ma-
terials.

SiC Ceramic	Measured Glass Transition Points
Sintered-α (Carborundum)	1063 °C
Siliconized (KT-SiC, Carborundum)	1088
Siliconized (NC 430, Norton)	1070
Siliconized (Refel SiC, Pure Carbon Co.)	1117
CVD* (Deposits and Composites Inc.)	1076
CVD* on reaction-sintered SiC (Deposits and Composites Inc.)	1126
Clay-bonded (Carbofrax M, Carborundum)	1112
Si_3N_4-bonded (Refrax 20, Carborundum)	1126

*CVD = chemically vapor deposited

[b]SiC ceramic tubes were exposed in the flue gas of a combustor. Nodules
formed on the upstream side of the tubes. See Section B.1.2.37, footnote c,
for combustion conditions.

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CRYSTALLINE PHASES[a] IN SILICON-BASED CERAMICS AFTER COMBUSTION TESTING WITH A COAL-OIL MIXTURE FUEL[b][106]

Material	As-Received	Phases Present[a] after Testing		
		Interior[c]	Interface Region[d]	Surface Layer[e]
SiC, sintered-α (Hexoloy SA, Carborundum) densification aids B, C	12H SiC 15R SiC FeWSi[f] Unknown phase	15R SiC 12H SiC	15R SiC 12H SiC α-cristobalite α-quartz	15R SiC 12H SiC α-cristobalite (increase) α-quartz (increase) mullite amorphous peak (~4.2 Å)
SiC, siliconized (KT-SiC, Carborundum)	12H SiC 15R SiC Si FeWSi[f]	15R SiC 12H SiC	15R SiC 12H SiC α-cristobalite	15R SiC 12H SiC 4H SiC α-cristobalite (increase)
SiC, hot-pressed (NC 203, Norton) densification aids alumina, WC	12H SiC 15R SiC α-alumina WC Unknown phase	15R SiC 12H SiC WC α-alumina	15R SiC 12H SiC WC α-cristobalite	15R SiC 12H SiC WC α-cristobalite (increase) amorphous peak
Si$_3$N$_4$, hot-pressed (NC 132, Norton) densification aids MgO, WC	β-Si$_3$N$_4$ Si$_2$ON$_2$ α-Si$_3$N$_4$ α-cristobalite tridymite WC WCoSi[f] MgSiN$_2$[f]	β-Si$_3$N$_4$ WC WCoSi Si$_2$ON$_2$	β-Si$_3$N$_4$ WC WCoSi Si$_2$ON$_2$ (increase) α-cristobalite (increase)	β-Si$_3$N$_4$ WC Si$_2$ON$_2$ (decrease) α-cristobalite (increase) amorphous peak (~3.9 Å)
Si$_3$N$_4$, hot-pressed (NCX 34, Norton) densification aids Y$_2$O$_3$, WC	β-Si$_3$N$_4$ Si$_2$ON$_2$ WC WCoSi MgY$_4$Si$_3$O$_{13}$ Y$_2$Si$_2$O$_7$ α-cristobalite tridymite Y$_2$SiWO$_8$[f] Si[f]	β-Si$_3$N$_4$ WC β-Y$_2$Si$_2$O$_7$ RY$_5$Si$_6$O$_{21}$ α-cristobalite	β-Si$_3$N$_4$ WC β-Y$_2$Si$_2$O$_7$ Y$_5$Si$_3$O$_{12}$N Si$_2$ON$_2$ (increase) α-cristobalite (increase) amorphous peak (~7.1 Å) X$_2$-Y$_2$SiO$_5$	β-Si$_3$N$_4$ WC β-Y$_2$Si$_2$O$_7$ RY$_5$Si$_6$O$_{21}$ (increase) α-cristobalite (increase) amorphous peak (~7.1 Å) X$_2$-Y$_2$SiO$_5$

(Table Continued)

CRYSTALLINE PHASES[a] IN SILICON-BASED CERAMICS AFTER COMBUSTION TESTING WITH A COAL-OIL

MIXTURE FUEL[b][106], Continued

Footnotes

[a]As determined by x-ray diffraction.

[b]Ceramic bars suitable for flexure testing were exposed to the flue gases and hot particulates of a combustor burning a coal-oil mixture. The fuel was a Venezuelan crude-derived No. 6 fuel oil containing 10 wt% powdered bituminous coal, with over 98.9% of particle size <44 μm; ash content was acidic with base to acid ratio = 0.29; the slag was viscous. There were three thermal cycles during the test with the temperature going from 1200 °C to 800 °C back to 1200 °C. The duration of the test was 500 hours, flue gas inlet temperature 1390 °C and outlet temperature 1349 °C.

[c]Slag layers and an oxidized layer had formed on the surface of the test bars. The "interior" is defined as being about 1.2 mm below the interface between the original test bar and the oxidized layer.

[d]Interface between the the oxidized layer and the ceramic.

[e]Oxidized ceramic layer on the surface of the specimen.

[f]Uncertain about the composition of the phase.

[g]R = Na, Fe, Mg, Al. This phase is apparently deleterious to the ceramic.

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PHASES FORMED[a] IN SURFACE LAYERS OF OXIDIZED[b] SILICON-BASED CERAMICS[106]

Material Treatment	Surface Layer Structure	Phases in Surface Layer
SINTERED α-SiC[c]		
As-received SiC oxidized at 1325 °C for 250 hours	Thin, glass-like oxidation layer	α-cristobalite
Oxidized layer removed and sample reoxidized for another 340 hours	Glass-like oxidation layer reduced in thickness	α-cristobalite
Oxidized layer again removed and same sample reoxidized for another 250 hours	Glass-like oxidation layer now localized around surface pits	α-cristobalite
HOT-PRESSED SiC[d]		
As-received SiC oxidized at 1325 °C for 250 hours	Heavy glassy oxide coating containing gas bubbles	α-cristobalite α-alumina
Oxidized layer removed and sample reoxidized for another 340 hours	Glassy layer reduced in thickness	α-cristobalite
Oxidized layer again removed and same sample reoxidized for another 250 hours	Glass-like oxidation layer now localized around surface pits	α-cristobalite
HOT-PRESSED Si_3N_4[e]		
As-received Si_3N_4 oxidized at 1325 °C for 250 hours	Thick porous polycrystalline oxide layer	$MgSiO_3$ α-cristobalite some Si_2ON_2
Oxidized layer removed and sample reoxidized for another 340 hours	Polycrystalline oxide layer reduced in thickness	$MgSiO_3$ α-cristobalite Si_2ON_2 increased
Oxidized layer again removed and same sample reoxidized for another 250 hours	Scattered oxide grains on Si_3N_4; layer is not continuous	α-cristobalite Si_2ON_2 some $MgSiO_3$

[a]As determined by x-ray analysis.

[b]Specimens were subjected to oxidation cycles by heating in air at 1325 °C and cooling to ambient temperature according to the pattern given in the column headed Material Treatment. See Section B.3.2.154 for strength values.

[c]Hexoloy SA, Carborundum.

[d]NC 203, Norton.

[e]NC 132, Norton.

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EFFECTS OF TEMPERATURE[a] ON THE PHASES PRESENT[b] IN SOME SILICON NITRIDES[c][107]

| Material[c] β-Si$_3$N$_4$+ | Preparation[d] & Treatment | As-Received Phases β-Si$_3$N$_4$ plus | | | Film Thickness[e] | Phases Observed After High-Temperature Test[b] | | | | |
		Y$_3$Al$_5$O$_{12}$	Glass	Other		β-Si$_3$N$_4$	β-cristo-balite	β-Y$_2$Si$_2$O$_7$	mullite	unknown[f]
10% YAG	HP 30 min. 1850 °C; HT 24 h at 1300 °C				1	D	A	B	B	A
10% YAG	HP 30 min. 1750 °C			x	5	B	D	0	B	D
10% YAG	HP 30 min. 1750 °C				3	C	C	B	A	B
10% YAG + Pt	S 30 min. at 1700 °C in BN; HP 30 min. at 1750 °C		x	x	1	D	C	B	B	B
10% YAG	HP 30 min. 1750 °C; HT 24 h at 1300 °C	x			5	B	C	C	B	A
10% YAG + Pt	S 30 min. at 1600 °C; HP 30 min. at 1750 °C			x	4	B	B	A	A	B
10% YAG + Pt	S 30 min. at 1750 °C; HP 1 h 1750 °C; HT 24 h at 1300 °C		x	x	3	C	C	B	C	B
10% YAG + Pt	HP 1 h at 1750 °C; HT 24 h at 1300 °C				3	C	C	A	B	B

(Table Continued)

EFFECTS OF TEMPERATURE[a] ON THE PHASES PRESENT[b] IN SOME SILICON NITRIDES[c][107], Continued

Material[c] β-Si$_3$N$_4$+	Preparation & Treatment[d]	As-Received Phases β-Si$_3$N$_4$ plus Y$_3$Al$_5$O$_{12}$	Glass	Other	Film Thickness[e]	Phases Observed After High-Temperature Test[b] β-Si$_3$N$_4$	β-cristo-balite	β-Y$_2$Si$_2$O$_7$	mullite	unknown[f]
10% YAG + Pt	HP 1 h at 1800 °C; HT 24 h at 1300 °C				1	D	B	A	B	A
10% YAG + Pt	HP 1 h at 1750 °C; HT 24 h at 1300 °C				5	B	B	A	A	B
10% YAG + Pt	HP 1 h at 1800 °C; HT 24 h at 1300 °C				6	A	B	0	B	C
10% YAG as crystal	HP 1 h at 1800 °C; HT 24 h at 1300 °C				4	C	B	0	B	A
10% YAG as crystal	HP 1 h at 1800 °C			x	2	D	D	A	C	C
10% YAG + Pt	HP 1 h at 1800 °C; HT 24 h at 1300 °C			x	4	B	A	0	B	A
20% YAG + Pt	HP 1 h at 1800 °C; HT 24 h at 1300 °C			x	4	B	C	C	D	B

(Table Continued)

EFFECTS OF TEMPERATURE[a] ON THE PHASES PRESENT[b] IN SOME SILICON NITRIDES[c][107], Continued

Material[c] β-Si3N4+	Preparation[d] & Treatment	As-Received Phases β-Si3N4 plus Y3Al5O12	Glass	Other	Film Thickness[e]	β-Si3N4	β-cristobalite	β-Y2Si2O7	mullite	unknown[f]
								Phases Observed After High-Temperature Test[b]		
20% YAG	HP 1 h at 1800 °C; HT 24 h at 1300 °C	x			5	B	C	D	D	D
20% YAG	HP 1 h at 1800 °C				5	B	D	C	D	D

[a] Specimens were fracture tested (see Section B.3.2.159) at 1300 °C in air for approximately 1 hour and examined for phase changes.

[b] Phases determined by x-ray diffraction. The amounts of the phases present are given relative to each other and to the other samples: $D > C > B > A > 0$.

[c] The materials are silicon nitrides hot-pressed with yttrium aluminum garnet (YAG) plus Pt or $PtCl_2$. They were prepared at the University of Michigan. The amount of YAG is in weight percent.

[d] S = pre-sintered, HP = hot-pressed, HT = heat-treated.

[e] The examination was limited to the surface material to preserve the samples for other analyses. A film formed on the surface due to the air oxidation and the thickness is given in the table on an arbitrary scale from 1 to 6, with 1 being the thinnest and 6 being the thickest.

[f] The unknown phase was well developed but remained unidentified.

EFFECT OF TIME AT HIGH TEMPERATURE[a] ON THE RELATIVE AMOUNTS[b] OF REACTION PRODUCTS FORMED ON SEVERAL SILICON CARBIDES[c] [107]

Figure A

Figure B

ALL SILICONIZED MATERIALS
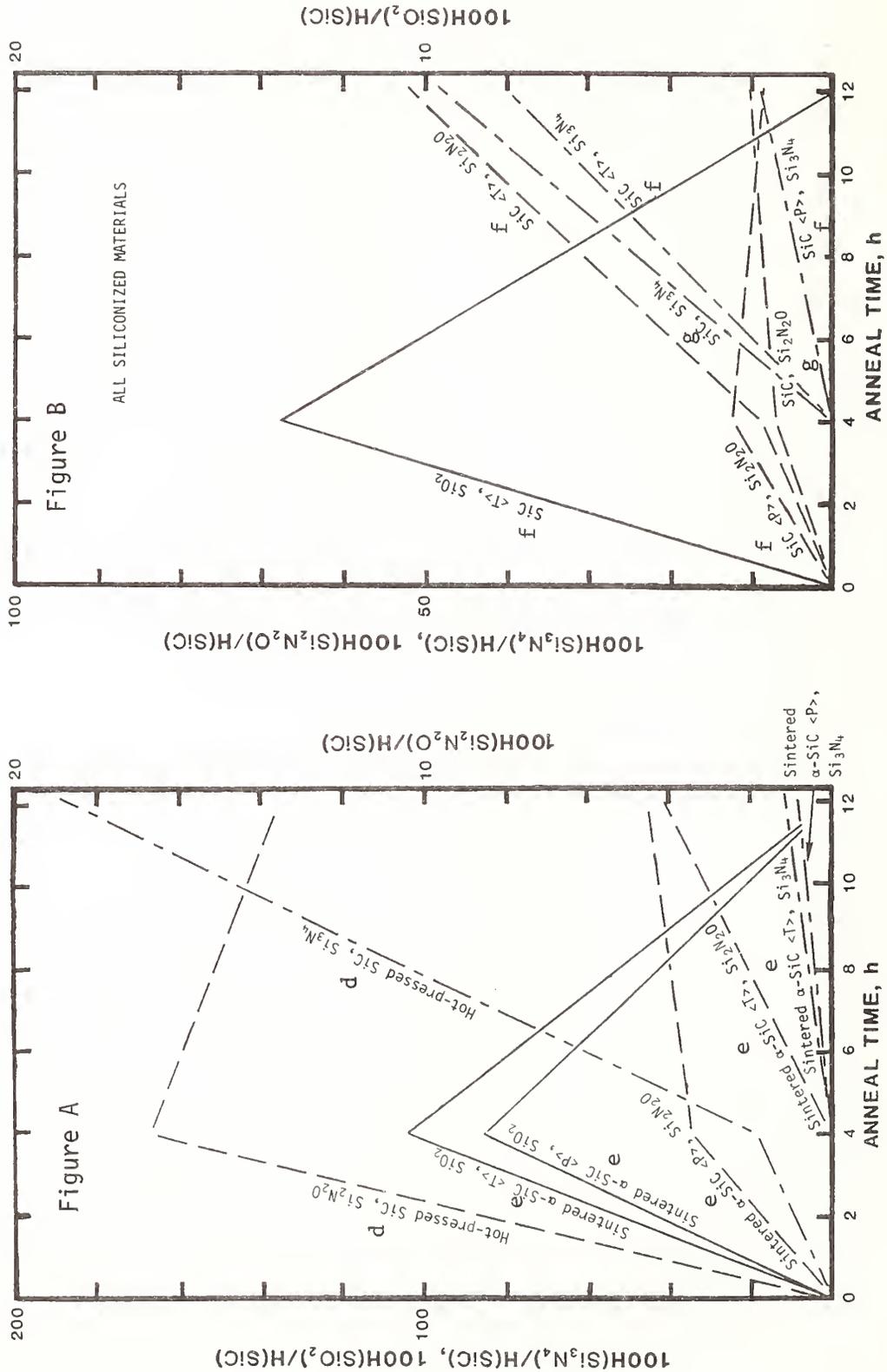
(Data Continued)

EFFECT OF TIME AT HIGH TEMPERATURE[a] ON THE RELATIVE AMOUNTS[b] OF REACTION PRODUCTS FORMED ON SEVERAL SILICON CARBIDES[c][107], Continued
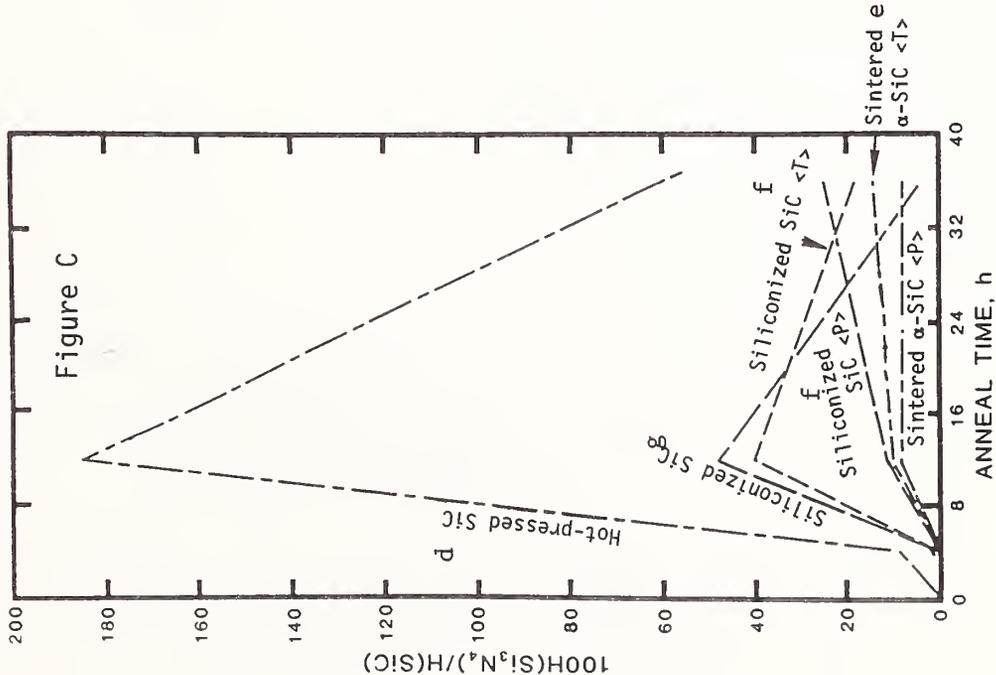
[a] Figure A shows the effect of annealing in an atmosphere of nitrogen with 1000 ppm SO_2 on two silicon-free SiC materials, one sintered and one hot-pressed. The annealing times were 4, 8, and 24 hours at 1400 °C. The data include the 4 and 8 hour periods. Three products are considered, SiO_2, Si_2N_2O and Si_3N_4.
Figure B shows the effect of the same annealing procedure on two siliconized SiC materials.
Figure C shows the effect of cumulative annealing times of 4, 12, and 40 hours on the formation of Si_3N_4 on SiC, presumably for the same conditions as Figures A and B.

[b] Phases were determined by x-ray diffraction. The ratio of peak heights (H) of specific diffraction peaks for each compound are given as a measure of the amount of material formed relative to the original material.

[c] The labelling <T> and <P> on the figures refers to the orientation of the specimen surface with respect to the large surface of the slab from which specimens were cut. <P> means the specimen surface was parallel to the surface of the slab, <T> refers to a specimen surface transverse to the slab surface.

[d] NC 203 (Norton).

[e] Hexoloy SA-80 (Carborundum).

[f] Hexoloy KX-01 (Carborundum).

[g] NC 430 (Norton).

Figure C

100H(Si₃N₄)/H(SiC) vs ANNEAL TIME, h

Curves: Hot-pressed SiC[d]; Siliconized SiC[g]; Siliconized SiC <P>[f]; Siliconized SiC <T>[f]; Sintered α-SiC <P>[e]; Sintered α-SiC <T>[e]

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WEIGHT CHANGES[a] VERSUS TIME CURVES FOR OXIDATION OF SILICON CARBIDE IN DRY

OXYGEN AT ONE ATMOSPHERE PESSURE[15]
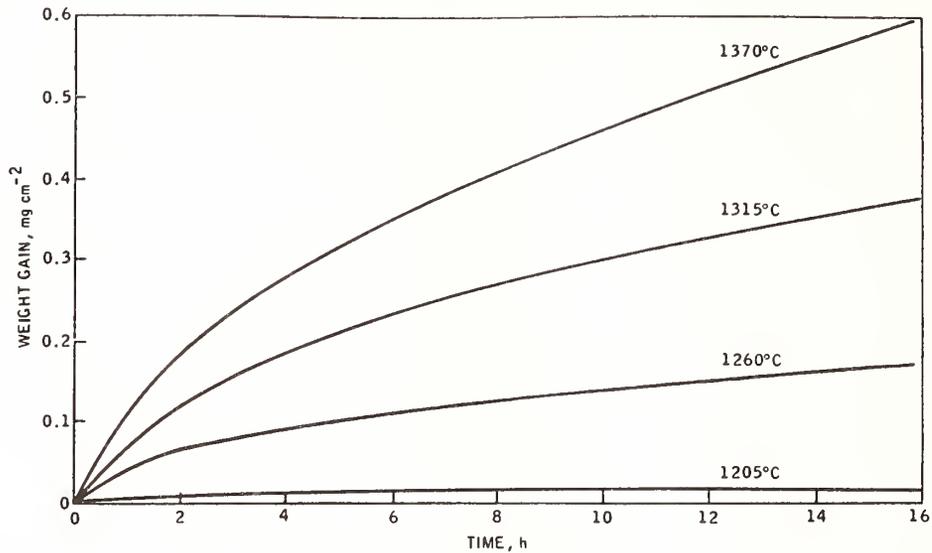
a Weight change indicates the formation of silicon dioxide in pure oxygen.
A thick coating on the SiC is desirable in order to prevent the SiC from
reacting with glass adhesive during firing of the glass to form a relaxing
joint between SiC tubes. A sufficient oxide coat is formed at 1360 °C for
periods over 15 hours with air circulating around the ceramic.

b A hot-pressed SiC (probably NC 203, Norton).

EFFECT[a] OF GAS TURBINE ENVIRONMENT[b] ON CERAMIC CANDIDATE HEAT EXCHANGER TUBES[c][15]

Ceramic	As-Received	After 100 h, V_2O_5 Contaminant[b]	After 1000 h, Fly Ash & Sea Salt Contaminant[b]
All three materials			General results--amount of ash deposit found on tubes is small because deposits spalled off tubes during periodic shut downs. Ash deposit composition found to be consistent with the original fly ash composition. Deposit on the front bank of tubes is glassy and translucent indicating melting of the ash on the surface. Ash on the rear bank tubes is more inhomogeneous and contains spherical metallic dendrites (Fe).
SiC, reaction sintered, siliconized (NC-430, Norton)	SiC grains (~100 μm) surrounded by fine-grained SiC and Si. Some large Si grains with some regions probably SiO_2. No evidence of porosity or internal cracking.	No changes observed.	Front and back tube banks--No preferential attack of Si occurred. No apparent oxidation layer or reaction with contaminants; fly ash fused to the surface. Front bank tubes--No significant change in tube cross section; ash merely deposited on surface.
SiC, siliconized (Super KT, Carborundum)	Fine-grained SiC and Si grains. Fully dense.	No changes observed.	Front and back tube banks--No changes in microstructure occurred due to exposure. Front bank tubes--Isolated areas on the outside diameter exhibited reduction in area.
SiC, sintered α (Carborundum)	SiC grains only, no free Si. Significant macroporosity and internal flaws.	No changes observed.	Front and back tube banks--No evidence of reaction products or preferential attack. Some thinning of tube walls observed in rear bank. Significant uniform thinning of front bank tubes observed in the leading edge region (thickness decreased by 0.5 mm, or ~20%).

(Data Continued)

EFFECT[a] OF GAS TURBINE ENVIRONMENT[b] ON CERAMIC CANDIDATE HEAT EXCHANGER TUBES[c][15], Continued

Footnotes

[a]As determined by metallographic examination and scanning electron microscopy with dispersive x-ray capability. Specimens were machined from the center of the tubes using a diamond wafering blade. Sections were mounted metallographically using low pressure. To avoid loss of the ash deposit there was no rough grinding but only polishing with diamond either impregnated in wheels or in paste form.

[b]The environmental chamber used in testing was a gas turbine simulator consisting of a small burner connected to a heat exchanger section which was sized to produce velocities (27 m/s) typical of those in low pressure drop heat exchangers. The combustor was run on No. 2 diesel fuel with corrosive additions which were introduced upstream of the test area. The additives were fly ash from the Pennsylvania Electric Homer City power plant, sea salt as specified by Federal Test Method Standard No. 151, and vanadium oxide which is a common contaminant of liquid fuels. For one series of tests the fly ash contaminant was added in the amount of 0.688 g/ft³ of gas, and the sea salt 0.00025 g/ft³ of gas. For other tests the V_2O_5 added was 0.00019 g/ft³ of gas.

[c]Two specimen tube bundles consisting of 5 tubes each in a staggered arrangement were placed in the heat exchanger test section so that the inside tube wall temperature was controlled by flowing air. Tubes from both the front bank (air temperature 1400–1315 °C, tube temperature 1200–1040 °C) and the rear bank (air temperature 1260–1095 °C), tube temperature 1090–980 °C) were examined after testing.

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COAL SLURRY EROSION DATA[a] FOR A NICKEL-TITANIUM ALLOY[b][103]

| Composition[b] (wt %) | Erosion Crater[c] | |
	Depth (μm)	Length (μm)
WC-10% Co-4% Cr (K701, reference standard)	3.4-5.6	$2.31-3.15 \times 10^3$
Ni 53.1, Ti 45.7, B 1.1	163.	1.12×10^3
Ni 49.1, Ti 47.5, B 3.4	130.	1.36×10^3

[a]The coal slurry consisted of 8% by weight of solids from the SRC-1 coal
liquefaction process (Wilsonville pilot plant, Illinois #6 coal) suspended
in anthracene oil. The jet of slurry impinged on the specimens at an angle
of 20° for 1 hour at 343 °C (650 °F) at a 100 m/s velocity. The slurry
was recycled for use with regular substitution of fresh slurry to main-
tain a consistent erosive quality. The surface of the materials was fin-
ished to 3.3×10^{-2} microns by grinding on a diamond wheel in the direction
of slurry flow to eliminate irregularities on the initial surface. A ce-
mented tungsten carbide was frequently retested to check test conditions
and is reported as the reference standard.

[b]Alloys are designated as Nitinol + B, an alloy of 50-50 atomic percent
Ni-Ti, plus TiB_2. Supplied by Oak Ridge National Laboratory.

[c]The depth of the erosion craters was measured using a Talysurf Model 4
Profilometer.

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EROSION TEST[a] DATA FOR SILICON NITRIDE REFRACTORIES[1]

Material	Brand Name or Other Designation	Source	Relative Erosion Factor[b]		
			20° C, 20° imp[c]	20° C, 90° imp[c]	700° C, 90° imp[c]
Si_3N_4, hot-pressed	Lot 1321	Ceradyne	0.03	0.18	0.57
Si_3N_4, reaction-bonded			6.17	5.50	6.51
Si_3N_4, hot-pressed		Norton	0.04	0.40	0.12
Si_3N_4-SiC, hot-pressed	Noroc-33	Norton	0.05	0.20	0.42
87% Si_3N_4-13% Al_2O_3	GE129 Sialon	Gen. Electric	8.24	4.61	2.20
50% Si_3N_4-50% Al_2O_3	GE128 Sialon	Gen. Electric	0.39	1.55	2.27
40% Si_3N_4-60% Al_2O_3	GE102 Sialon	Gen. Electric	0.66	2.03	2.05
Si_3N_4-Al_2O_3, 90% dense, 10% porosity	Sialon		1.00+	1.00+	1.00+
19% Si_3N_4-80%SiC-SiO_2	Refrax 20C	Carborundum	0.27	0.91	1.15

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[a] Erosion test conditions: 1/2-inch square specimens subjected to erosion by 27μ Al_2O_3 abrasive, 170 m/sec particle velocity, 3 minutes test duration, N_2 atmosphere, 5 g/min abrasive flow.

[b] Relative Erosion Factor = $\dfrac{\text{Volume loss of sample}}{\text{Volume loss of Stellite 6B}}$, volume loss calculated from weight loss and density data; Stellite 6B = 30Cr-4.5W-1.2C-bal Co.

[c] imp = angle of impingement.

===

EROSION TEST[a] DATA FOR MISCELLANEOUS REFRACTORIES[1]

Material	Brand Name or Other Designation	Source	Relative Erosion Factor[b]		
			20 °C, 20° imp[c]	20 °C, 90° imp[c]	700 °C, 90° imp[c]
B_4C, HP[d]	Norbide	Norton	0.16	0.38	0.21
B_4C, HP	Lot 1076	Ceradyne	0	0	0.38
TiB_2, P&S[e]	KB TiB_2		0.05	0.13	weight gain
TiB_2	Refractory boride		0.19	0.60	1.30
TiB_2, Ni binder HP		ORNL[f]	0.059	0.370	
TiB_2, 20% Ni binder HP	NT-80B	ORNL	0.008	0.036	
TiB_2, 15% Ni binder HP	NT-85B	ORNL	0.017	0.046	
TiB_2, 10% Ni binder HP	NT-90B	ORNL	0.013	0.108	
ZrB_2-SiC, HP	ZRBSC-D	Norton	0.14	0.32	0.69
ZrB_2-SiC-graphite,[g] HP	ZRBSC-M	Norton		6.36	
$MgAl_2O_4$, HP		Trans Tech		1.84	2.56
$45MgAl_2O_4$-55MgO, HP	5527	Trans Tech		3.39	2.84
$91MgAl_2O_4$-9MgO, HP	4879	Trans Tech		2.44	2.77
$97MgAl_2O_4$-3MgO, HP	4310	Trans Tech		2.56	2.76
TiC-Al_2O_3, P&S		Babcock & Wilcox		0.19	0.30
TiC, 25% Ni-6% Mo binder, P&S	K162B	Kennametal		1.35	1.67
TiC, 19% Ni binder P&S	K151A	Kennametal		1.37	1.62
CbC [NbC], cast				3.56	
HfC, cast				3.49	
TaC, cast				2.48	
Chromite, P&S		Union Carbide		2.44	3.43
CBN		General Electric		0	0
C	diamond	General Electric		0	0

[a] Erosion test conditions: 1/2-inch square specimens subjected to erosion by 27μm Al_2O_3 abrasive, 170 m/s particle velocity, 3 minutes test duration, N_2 atmosphere, 5 g/min abrasive flow.

[b] Relative Erosion Factor = $\dfrac{\text{Volume loss of sample}}{\text{Volume loss of Stellite 6B}}$, volume loss calculated from weight loss and density data; Stellite 6B = 30Cr-4.5W-1.2C-balance Co.

[c] imp = angle of impingement.

[d] HP = hot-pressed.

[e] P&S = pressed and sintered.

[f] Oak Ridge National Laboratory.

[g] Oxidation was observed.

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RELATIONSHIP BETWEEN EROSIVE WEAR[a] AND FLEXURAL STRENGTH[b]

FOR HYDROTHERMALLY-TREATED[c] ALUMINA REFRACTORIES[23]

Material	Treatment Temperature[c] °C	Flexural Strength[b] MPa	Erosion[a] mg sample lost/ g abrasive used
Calcined Flint Clay[d] Castable	untreated	10.1	20.3
	110	13.9	13.3
	310	10.9	13.3
	410	15.2	16.0
	610	15.5	14.0
	710	15.2	16.0
	910	14.8	13.6
High-Purity Alumina[e] Castable	untreated	17.4	18.2
	110	23.4	9.6
	310	24.1	14.6
	410	27.1	15.2
	610	24.8	9.2
	710	25.1	10.2
	910	28.0	11.6

[a]Specimens were subjected to erosion by 100 mesh (150 μm) SiC at 25 °C; impingement angle 90°; particle velocity 72 m/s.

[b]Flexural strength as determined in four-point bend tests, loading rate 5 x 10^{-3} cm/min (2 x 10^{-3} in/min); rectangular specimens 5 x 10 x 50 mm; upper loading span 10 mm, lower loading span 40 mm; tests at ambient temperatures.

[c]Treatment consisted of sealing samples in a pressure chamber and heating to test temperature, then injecting steam to raise the pressure to 1000 psig.

[d]An NBS-prepared medium-alumina castable refractory bonded with high-purity calcium aluminate cement: 55.6 wt% Al_2O_3, 4.5 wt% CaO, 37.0 wt% SiO_2, 0.8 wt% Fe_2O_3, 0.2 wt% Na_2O, 1.9 wt% others and loss on ignition.

[e]An NBS-prepared high-alumina castable bonded with high-purity calcium aluminate cement: 94.4 wt% Al_2O_3, 4.5 wt% CaO, 0.1 wt% SiO_2, 0.1 wt% Fe_2O_3, 0.2 wt% Na_2O.

EROSION/CORROSION MATERIAL LOSS[a] OF SOME REFRACTORIES SUBJECTED TO EROSION BY COARSE FMC CHAR[b]
IN COAL GASIFICATION ATMOSPHERE[c][11]

Refractory	Average[d] Corrosion mils	Maximum[e] Erosion/ Corrosion mils	Comments	Average[d] Corrosion mils	Maximum[e] Erosion/ Corrosion mils	Comments
Pressure		atmospheric			1000 psi	
Velocity		100 ft/s			100 ft/s	
Time		100 hours			100 hours	
90% Al_2O_3 castable, CA bond[f] (Castolast G)	0	7.8	Localized binder removal E/C[g] surface	+1.4	2.5	Limited visible attack
60% Al_2O_3 castable, CA bond (Super Brik- cast A)	0.4	34.4	Localized loss of binder E/C surface	0.4	23.6	Localized binder and particle loss E/C surface
54% Al_2O_3 castable, insulating, CA bond (Litecast 75-28)	6.0	243.8	Specimen completely penetrated	2.3	246.7	Specimen completely destroyed
85% Al_2O_3 brick, phosphate bond (Chemal 85 B)	+2.0	17.6	Some binder removal E/C surface	+1.8	55.6	Major binder and particle loss E/C surface
90% Al_2O_3 brick, self-bond (Kricor)	+1.5	5.4	Possible binder removal E/C surface	+1.9	26.6	Moderate binder loss E/C surface
77% Al_2O_3 insulating brick, vitreous bond (B&W Insalcor)	0	7.0	No apparent attack	+4.7	49.2	Erosion area on E/C surface; corner of specimen broken after test
70% Al_2O_3 brick, mullite (Alumex 70-HD)	+2.4	13.9	Some binder removal E/C surface	+0.7	45.7	Major binder loss E/C surface; three broken corners after test
60% Al_2O_3 brick, pitch impregnated (Ufala TI)	1.0	3.4	Possible binder removal E/C surface	+0.1	15.3	Light binder loss E/C surface
45% Al_2O_3 superduty fire brick (KX-99)	+0.7	5.2	Possible binder removal E/C surface	1.6	27.8	Moderate binder loss E/C surface
90% Al_2O_3-10% Cr_2O_3 brick (Ruby)	+1.7	5.0	Possible binder removal E/C surface	+1.2	14.0	Light binder loss E/C surface
Pressure		atmospheric			1000 psi	
Velocity		50 ft/s			50 ft/s	
Time		5 hours			5 hours	
54% Al_2O_3 castable,[h] insulating, CA bond (Litecast 75-28)	0.4	149.1	Oval erosion pit	2.8	72.9	Large erosion area
	1.8	93.8	Small oval erosion pit	2.5	89.9	Large erosion area
	1.1	71.0	Small oval erosion pit	3.5	·81.0	Large erosion area
	2.1	81.4	Small oval erosion pit	1.7	86.3	Large erosion area
	1.5	82.4	Small erosion pit	3.5	101.6	Large erosion area
	1.6	133.6	Oval erosion pit	3.7	120.5	Large erosion area
Pressure		atmospheric			1000 psi	
Velocity		100 ft/s			100 ft/s	
Time		5 hours			5 hours	
54% Al_2O_3 castable,[h] insulating, CA bond (Litecast 75-28)	1.5	15.9	Surface discoloration only	2.3	252.6	Large very deep E/C area
	2.4	252.5	Sample penetrated	3.0	250.6	Completely penetrated
	0.7	124.8	Deep oval erosion pit	3.2	156.8	Large deep E/C area
	0.7	13.3	Sample discoloration only	2.3	174.9	Two deep E/C areas
	1.3	245.7	Sample penetrated	2.1	158.6	Two deep E/C areas
	1.3	254.2	Sample penetrated	3.7	213.4	Two deep E/C areas
	1.3	251.6	Sample penetrated			
	1.0	35.3	Small shallow erosion pit			
	0.6	158.1	Small deep erosion pit			
	0.8	90.5	Small erosion pit			
	1.2	256.9	Sample penetrated			
	1.1	252.9	Sample penetrated			

(Table Continued)

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EROSION/CORROSION MATERIAL LOSS[a] OF SOME REFRACTORIES SUBJECTED TO EROSION BY COARSE FMC CHAR[b]

IN COAL GASIFICATION ATMOSPHERE[c][11], Continued

Refractory	Average[d] Corrosion mils	Maximum[e] Erosion/ Corrosion mils	Comments
Test run in atmosphere of N_2 only			
Temperature- - - - - - - - - - -1800 °F- - - - - - - -			
Pressure		atmospheric	
Velocity		100 ft/s	
Time - - - - - - - - - - - - - - 5 hours - - - - - -			
54% Al_2O_3 castable,[h]	3.5	49.6	E/C pit
insulating, CA bond	2.1	102.9	Large E/C pit
(Litecast 75-28)	1.3	96.7	Large E/C pit
	2.3	93.6	E/C pit
	1.8	47.5	E/C pit
	1.2	62.9	E/C pit

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[a] Refractory samples 2 x 2 x 3/4 in. were subjected to erosion in coal gasification atmosphere under the indicated conditions. Impingement angle 90°. Values are for one specimen per test.

[b] Char from Western Kentucky coal prepared by COED process, −20 +40 mesh (840 to 420 μm) used in the 100 hour tests, −30 +50 mesh (600 to 300 μm) used in the 5 hour tests.

[c] Coal gasification atmosphere input gas: 12% CO_2, 18% CO, 24% H_2, 5% CH_4, 1% NH_3, 1.0% H_2S, and the balance H_2O. All tests were run at 1800 °F.

[d] Average corrosion of the one side of the sample exposed to both erosion and corrosion; calculated from thickness measurements (by micrometer) of samples before exposure and after exposure and cleaning; measurements after exposure made in uneroded areas. A plus (+) sign indicates increased thickness apparently due to swelling of the refractory and to adhering erodent residue. Refractories which swelled had also gained weight.

[e] Maximum effect on the one side of the sample exposed to both erosion and corrosion; calculated from thickness measurements (by micrometer) of samples before exposure and after exposure and cleaning; measurements after exposure were made in eroded areas and pits.

[f] CA bond = calcium aluminate bonded.

[g] E/C = erosion/corrosion.

[h] Refractory, which would show appreciable erosion and no corrosion in the short test time, was used with alloy specimens (see Section B.2.1.23 for alloy results) to compare results in atmospheric and high-pressure apparatus. Multiple specimens were used to indicate the variability of the data.

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EROSION/CORROSION TESTING[a] OF REFRACTORIES[b][33]

Weight Change, g (±0.0005 g)

Refractory	Initial Weight, g	During First 50 h	During Second 50 h	Total Change for 100 h	Physical Description after 100 h
98% Al_2O_3 fused cast brick (Monofrax A2; reference)	86.7751	-0.003	+0.002	-0.001	No visible erosion. Gray/black deposit on impinged surface.
99+% Al_2O_3 fused cast brick (Monofrax A1)	92.8273	+0.023	+0.006	+0.029	No visible erosion. Gray/black deposit on impinged surface.
90+% Al_2O_3 dense castable (Castolast G)	91.5132	-0.414	-0.062	-0.476	No visible erosion. Gray/black deposit on impinged surface.
90% Al_2O_3/10% Cr_2O_3 solid solution fired brick (CS 612)	102.0899	-0.062	-0.003	-0.065	No visible erosion. Gray/black deposit on impinged surface.
∿70% Si_2ON_2, ∿20% Si_3N_4, ∿10% SiC brick (SION)	68.6982	+2.721	+0.180	+2.901	No visible erosion. Light gray discoloration. Slight weight loss during first 50 h, which resulted from damage in handling.
90% Al_2O_3 phosphate bond ramming mix (Brickram 90)	99.4256	-0.027	-0.012	-0.039	No visible erosion. Gray/black deposit on impinged surface.
90+% Al_2O_3 phosphate bonded castable (Resco Cast AA-22)	88.9209	-0.285	-0.085	-0.370	No visible erosion. Gray deposit with pink/yellow tint.
72% Al_2O_3, 23% SiO_2 brick (Alumex 70)	73.6872	-0.406	-0.094	-0.500	Some binder erosion. Black deposit with yellowish tint.
62% Al_2O_3 castable (AR 400)	78.9711	-1.319	-0.160	-1.479	Corners, edges, and binder show some erosion. Slight damage during first 50 h. Partial black deposit.
55% Al_2O_3 fire clay aggregate gunning mix (Lo-Abrade GR)	63.8424	-0.955	-0.120	-1.075	Some binder erosion. Partial black deposit. Slight damage during first 50 h.
50% Al_2O_3 fused cast brick (Monofrax R)	125.0410	+0.042	+0.006	+0.048	No visible erosion. Black deposit with yellowish tint.
∿57% Al_2O_3 castable (Fraxcast ES)	73.7900	-4.590[c]	-0.016	-4.606	No visible erosion. Black deposit with yellowish tint. Some weight loss during first 50 h, which resulted from damage in handling.
46% Al_2O_3, 40% SiO_2 castable (B&W Kaocrete HS)	57.9730	-1.963	-0.364	-2.327	Obvious erosion of binder over entire impinged surface. Partial black deposit.
79% SiC, ∿18% Si_3N_4 bonded, dense fired brick (Refrax 20)	90.6470	+1.755	+0.097	+1.852	No visible erosion. Black deposit with yellowish tint.
73% SiC, 14% Al_2O_3, ∿10% SiO_2 brick (Siltec)	78.6791	+0.060	+0.018	+0.078	No visible erosion. Black deposit with yellowish tint.

[a] Test temperature was controlled at 980 °C (1800 °F), pressure was maintained at 240 KPa (35 psi), and char particle velocity averaged 39 m/s (125 ft/s). Char used was FMC char from Western Kentucky coal. Gas composition was close to the following with variations due to problems with ammonia and steam control: (in vol %), 39 H_2O, 24 H_2, 12 CO_2, 18 CO, 5 CH_4, 1 NH_3, 1 H_2S. Total exposure time was 100 hr.

[b] After exposure, specimens were lightly brushed, air blown, and weighed after testing.

[c] Visual examination did not indicate any erosion or other damage to account for the high measured weight loss. Experimental error is suspected.

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ABRASION LOSS[a] OF IRON-DOPED ALUMINA CASTABLES EXPOSED[b] TO CARBON-MONOXIDE ATMOSPHERES AT HIGH PRESSURE[c][91]

Gas Composition (vol %)	Wt% Dopant	Exposure Time, h	Abrasion Volume Loss[a], cm^3		Abrasion Ratio[d]
			Prefired	Exposed	
— 90% Alumina Castable[e] —					
99.9 CO	0	50	2.2	5.5	2.5
	0.1 Fe[f]	3	completely disintegrated		
	0.1 Fe$_2$O$_3$[f]	3	—	0.181	0.58[g]
99.2 CO-0.8 H$_2$S	0	50	2.2	2.6	1.2
	0.1 Fe	50	completely disintegrated	0.96	1.16
	0	25	—	0.23	0.371
	0.1 Fe	25	completely disintegrated	0.67	1.093
	0	10	—	0.544	1.209
	0.1 Fe	10	completely disintegrated		
	0.1 Fe	3	—		
79.36 CO-20 H$_2$O-0.64 H$_2$S	0	50	2.2	0.5	0.2
78.37 CO-20 H$_2$O-1 NH$_3$-0.63 H$_2$S	0	50	2.2	—	
— 50% Alumina Castable[h] —					
99.2 CO-0.8 H$_2$S	0	50		2.98	—
	0.1 Fe	50	completely disintegrated		
60 CO-40 H$_2$O	0	50	—	2.64	—
	0.1 Fe$_2$O$_3$	50	—	3.44	—
	0	50	—	3.67	—
	0.1 Fe	50	—	14.45	—
60 CO-40 H$_2$	0	50	3.292	2.778[i]	0.84
	0.1 Fe$_2$O$_3$	50	1.667	2.315[i]	1.39
	0	50	3.183	4.093	1.29
	0	18 h 40 min	3.183	2.274	0.71
	0.1 Fe	50	3.640	24.568	6.75
	0.1 Fe	18 h 40 min	3.640	15.200	4.18
59.5 CO-40 H$_2$O-0.5 H$_2$S	0	50	—	3.44	—
	0.1 Fe	50	—	3.75	—

(Table Continued)

ABRASION LOSS[a] OF IRON-DOPED ALUMINA CASTABLES EXPOSED[b] TO CARBON-MONOXIDE ATMOSPHERES
AT HIGH PRESSURE[c][91], Continued

Footnotes

[a]Abrasion volume loss was determined using a modified version of ASTM C-704 test procedure. The distance from the nozzle to the sample was reduced to 3.75 in. and the amount of SiC abrasive used was 500 g.

[b]Castables were exposed to the gas mixtures indicated at 500 °C for the indicated times.

[c]Gas pressure was 1000 psi.

[d]Abrasion Ratio = $\dfrac{\text{Abrasion Volume Loss of Exposed Samples}}{\text{Abrasion Volume Loss of Prefired Samples}}$. Abrasion loss of prefired samples was not always measured.

[e]A DOE generic castable: 65 wt% tabular alumina (25 wt% 6 to 10 mesh, 20 wt% 10 to 20 mesh, 20 wt% less than 20 mesh), 10 wt% calcined alumina (less than 325 mesh), 25 wt% calcium aluminate cement (Casting Grade CA-25 cement), all Alcoa materials. Materials were dry mixed for 2 min, then water was added with continued mixing to ball-in-hand consistency (~605 ml); refractory was then poured into 2 in. x 2 in. x 2 in. aluminum molds, vibrated for 5 min, sealed in plastic for 24 h, then fired in air for 5 h at 1100 °C.

[f]These samples were prefired at 500 °C instead of 1100 °C.

[g]This ratio was calculated using the value for samples prefired at 500 °C.

[h]A DOE generic castable: 75 wt% calcined kaolin (25 wt% 6 to 10 mesh, 20 wt% 10 to 20 mesh, 15 wt% less than 20 mesh, 20 wt% less than 200 mesh), 25 wt% calcium aluminate cement (Casting Grade CA-25, Alcoa). Samples were prepared as described in footnote e except that the water used was ~700 ml.

[i]Values are somewhat low due to test apparatus malfunction.

===

EFFECT OF CARBON MONOXIDE ATMOSPHERES[a] ON ABRASION LOSS[b] OF STAINLESS STEEL
FIBER-REINFORCED[c] ALUMINA CASTABLES AT HIGH PRESSURE[d][91]

Gas Composition (vol %)	Fiber Type[c]	Exposure Time, h	Abrasion Volume Loss[b] cm^3
- - - - - - - - - - - - - -90% Alumina Castable[e]- - - - - - - - - - - - - - -
N_2	446	50	1.06
	30-20	50	0.50
	310	50	1.86
CO	446	50	1.91
	30-20	50	5.00
	310	50	2.29
40 CO-40 N_2-20 H_2O	446	50	1.83
	446	100	2.04
	30-20	50	1.38
	30-20	100	0.81
	310	50	3.26
80 CO-20 H_2O	310	50	5.78
- - - - - - - - - - - - - -50% Alumina Castable[e]- - - - - - - - - - - - - - -
N_2	446	50	0.40
	30-20	50	0.85
	310	50	2.53
CO	446	50	1.35
	30-20	50	0.85
	310	50	2.26
40 CO-40 N_2-20 H_2O	446	50	1.03
	446	100	0.96
	30-20	50	0.90
	30-20	100	0.36
	310	50	1.04
80 CO-20 H_2O	310	50	0.82

[a] Castables were exposed to the indicated gases at 500 °C.

[b] Abrasion volume loss was determined using a modified version of ASTM C-704 test procedure. The distance from the nozzle to the sample was reduced to 3.75 in. and the amount of SiC abrasive used was 500 g.

[c] Four weight percent of the stainless steel fibers was added to the mix before samples were casted. The 30-20 steel is the same alloy as the 310 but with an additional 5% chromium.

[d] Gas pressure was 1000 psi.

[e] These are the same castables as are described in footnotes e and h of Section B.2.2.20 except that they were not fired at 1100 °C. These specimens were held at 500 °C for 12 hours before the test exposure. Prefiring of these reinforced materials has a deleterious effect on them.

==

EFFECT OF PREFIRING[a] ON CO DISINTEGRATION[b] OF STAINLESS STEEL FIBER-REINFORCED[c]
CASTABLES AS MEASURED BY ABRASION LOSS[d][91]

Castable Composition[e]	Fiber Type[c]	Prefired[a]		Abrasion Volume Loss[d]
90% Alumina	446	no		2.04 cm^3
	446		yes	0.91
	30-20	no		0.81
	30-20		yes	0.81
50% Alumina	446	no		0.76
	446		yes	3.00
	30-20	no		0.36
	30-20		yes	1.74

[a] Samples were fired in air at 1100 °C for 12 hours.

[b] Samples were exposed to an atmosphere of 40% CO-40% N_2-20% H_2O for 100 hours at 500 °C and 1000 psi.

[c] Four weight percent of the stainless steel fiber was added to the mix before samples were prepared. The 30-20 alloy is the same as 310 SS but with an additional 5% chromium.

[d] Abrasion volume loss was determined using a modified version of ASTM C-704 test procedure. The distance from the nozzle to the sample was reduced to 3.75 in. and the amount of SiC abrasive used was 500 g.

[e] The castables are DOE generic compositions. See footnotes e and h of Section B.2.2.20 for the preparation and see also Section B.2.2.21.

==

EROSION DATA[a] FOR A TUNGSTEN CARBIDE[b] AT VARIOUS PARTICLE VELOCITIES AND
IMPINGEMENT ANGLES[1]
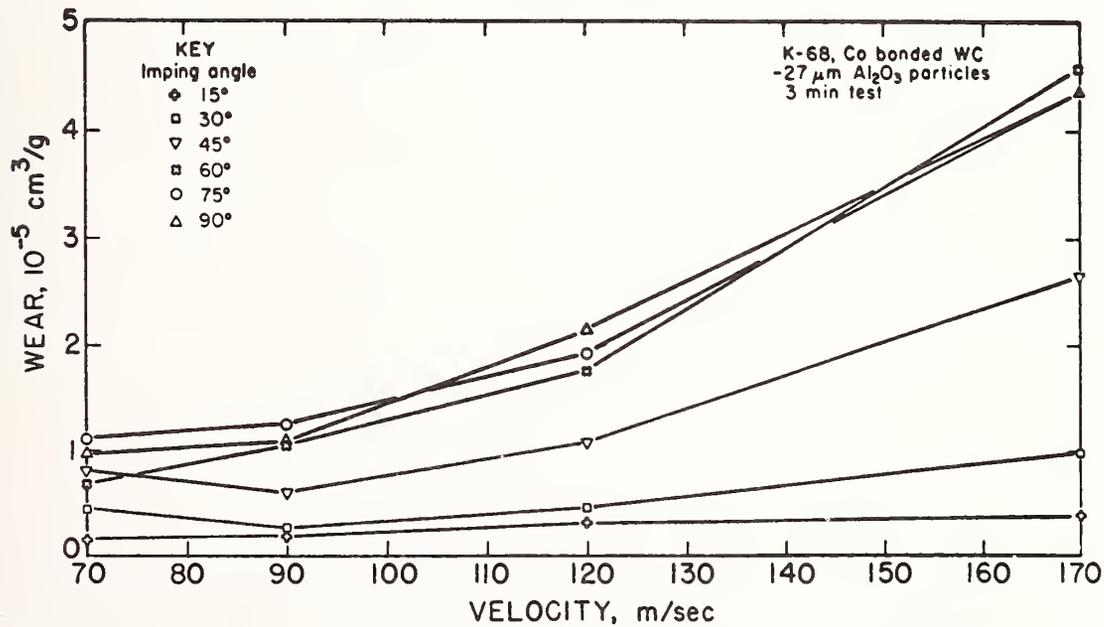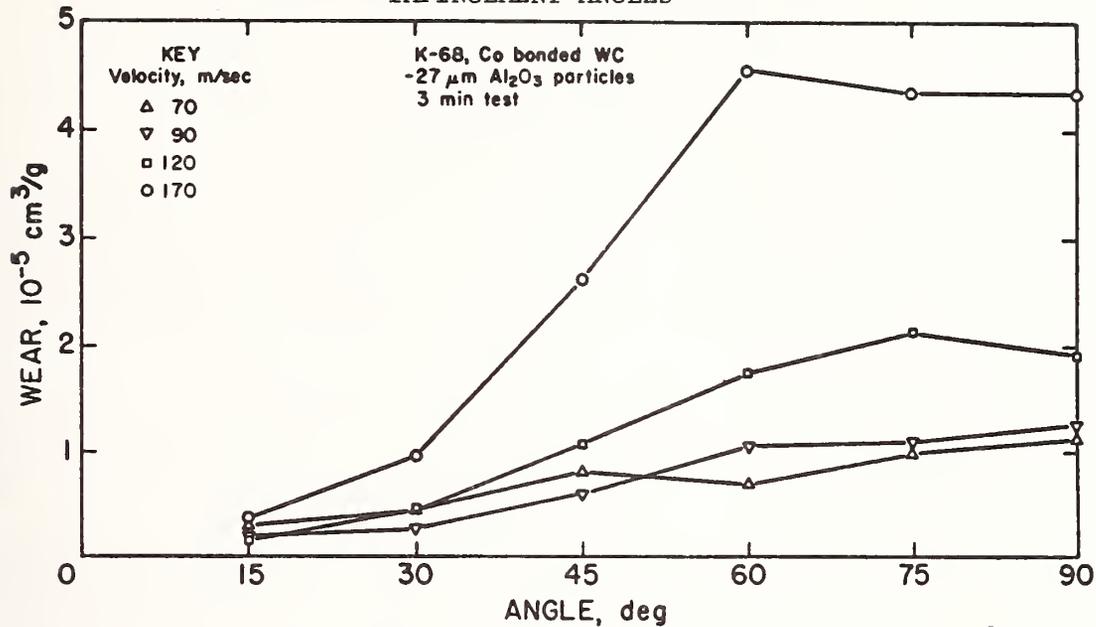
[a]Erosion testing was performed on 1/2-inch square specimens subjected to
erosion by 27 μm Al_2O_3 in three minute tests in N_2 atmosphere. The ab-
rasive flow was 5 g/min. There were 4 different particle velocities and
6 impingement angles. The wear value plotted is the volume of material
removed divided by the weight of Al_2O_3 expended. See Section B.2.1.16
for tabulated data for this and other tungsten carbides.

[b]A cobalt-bonded tungsten carbide with 5.8% binder, pressed and sintered
(K68, Kennametal).

==

EFFECT OF CERAMIC FIBER[a] ADDITION ON THE ABRASION RESISTANCE[b] OF A
HIGH-ALUMINA CASTABLE REFRACTORY[c] [9]

$\triangle$ No Fibers Added
$\bigcirc$ 1% AB-312
$\bigtriangledown$ 2% AB-312
⬡ 5% AB-312
■ 10% AB-312
★ 5% Saffil
• Hycol Added
▵ GP 2500 Added

ABRASION WEIGHT LOSS, g

WATER, percent

---

[a]Fibers used were AB-312 from 3M Company, high-alumina Al_2O_3-B_2O_3-SiO_2,
11 μm diameter, and Saffil from Imperial Chemical Industries, high-temper-
ature 98% Al_2O_3-2% SiO_2, 3 μm diameter. Fibers were chopped in a high-
speed Waring blender; length after chopping varied 20-150 μm. Castable,
fibers, and water were mixed in blender before casting. Cast samples were
moist cured for 24 h, oven dried for 24 h. Wetting agents were used to re-
duce the amount of water necessary, 6 ounces added per 100 pounds of cement:
Hycol from W.R. Grace Company and GP 2500 from Cresset Chemical Company.

[b]Sample bars 1 in. x 1 in. x 3 1/2 in. were abraded with 1000 g of 36 grit
SiC according to ASTM C704-72; above values are the average of 3 separate
samples of each preparation.

[c]Castable used is a high-purity tabular alumina, calcium aluminate bonded
(Castolast G, Harbison-Walker).

==

EFFECT OF VARIOUS HIGH-BTU GAS COMPOSITIONS[a] ON THE ABRASION RESISTANCE[b] OF VARIOUS ALUMINA CASTABLES[c][95]

Castable tested[c]	Abrasion Resistance, Volume of Refractory Lost, cm^3					
	Air 1 1100 °C 1 atm 6 h	Gas A 500 °C 1000 psi 58 h	Air 2 500 °C 1 atm 1000 h	Gas A 500 °C 1000 psi 1000 h	Gas B 500 °C 1000 psi 1000 h	Gas C 500 °C 1000 psi 1000 h
90% Al_2O_3	2.98	4.45	1.30	9.59[de]	4.42[df]	10.28[df]
90% Al_2O_3 + 0.1% Fe	4.94	4.41	1.26	6.56[de]	16.39[defg]	7.70[df]
90% Al_2O_3 + 310 SS[h]	3.30	4.01				
90% Al_2O_3 + alkali[i]	1.63	1.65				
50% Al_2O_3	4.57	6.38	1.09	1.29	1.34	1.51
50% Al_2O_3 + 0.1% Fe	5.58	2.74				
50% Al_2O_3 + 310 SS[h]	6.13	12.50				
50% Al_2O_3 + alkali[i]	1.74	3.44				

[a] Gas compositions in mole percent:
  A = 38 H_2O, 24 H_2, 18 CO, 12 CO_2, 5 CH_4, 2 NH_3, 1 H_2S
  B = 40 H_2O, 25 H_2, 18 CO, 12 CO_2, 5 CH_4
  C = 40 H_2O, 25 H_2,   12 CO_2, 5 CH_4, 18 N_2.
  Temperature, pressure, and exposure time are given in the table for each exposure. Specimens were prefired before exposure according to the conditions given for Air 1 or Air 2.

[b] Abrasion resistance determined according to Modified ANSI/ASTM C704-76a Test Method.

[c] No preparative information or other characterization was given in the reports and no commercial brand names were included. Both castables are dense, calcium aluminate bonded. No information was given about the manner of the Fe addition. See sections for reference [9] for earlier reports from the same organization.

[d] The reports indicated a difference significant at the 99% level of confidence between the results for this exposure and for the Air 2 firing.

[e] Values for Gas A and Gas B reported to show a difference significant at the 99% confidence level.

[f] Values for Gas B and Gas C reported to show a difference significant at the 99% confidence level.

[g] In other reports of the series giving these same data, this value is also reported as 6.74 and 16.36.

[h] 310 stainless steel fibers (4 wt%) were added to the mix before casting.

[i] Alkali-containing specimens were prepared by soaking for 6 hours in a 50% Na_2CO_3-50% K_2CO_3 saturated solution prior to firing.

EFFECT OF UNSATURATED STEAM AND CO/STEAM ATMOSPHERES[a] ON THE ABRASION RESISTANCE[b] OF ALUMINA REFRACTORIES[c][95]

Refractory[c]	Brand Name/Manufacturer	Volume of Refractory Lost by Abrasion,[b] cm³				
		Oven-Dried 110 °C 24 h	Air-Fired 1000 °C 250 h	Steam 1000 °C 250 h 105 psig	CO/H_2O=3.5 1000 °C 250 h 105 psig	CO/H_2O=7.0 1000 °C 250 h 105 psig
High-Al_2O_3 dense castable	Plicast L-22-84/Plibrico	1.64[d]	1.37[d]	1.75[e]	1.55[e]	1.14[e]
High-Al_2O_3 dense castable	Greenkon-33/A.P. Green	1.26	2.65	3.14	NA[f]	3.12
High-Al_2O_3 dense castable	H-W 9/79/[Harbison-Walker]	1.70	1.73	1.98	1.34	1.09[g]
90+% Al_2O_3 brick	Kricor/Kaiser	2.26	2.14	2.35	1.98	1.82
High-Al_2O_3 chrome phosphate-bonded ramming mix	Lavalox X-8/Lava Crucible Refractories	0.99	0.86	0.83	0.92	0.82
Intermediate-Al_2O_3 dense castable	Resco Cast RS-17E/Resco Products	2.54	5.89	2.41[g]	3.88[g]	3.90[g]
Fireclay dense castable	Plicast HyMOR 3000/Plibrico	5.22	4.09	3.62[g]	4.35	2.81[g]
Fireclay castable	H-W 8/80/[Harbison-Walker]	2.68	4.07	3.16[g]	3.14	2.31[g]

[a]Refractories were exposed under the indicated conditions to unsaturated atmospheres of steam and carbon monoxide/steam. The two CO/steam atmospheres had two different ratios of CO to steam, 3.5 and 7.0.

[b]Refractories were tested using the Modified ANSI/ASTM C704-76a Test Method for Abrasion Resistance.

[c]Specimens were 2-inch cubes prepared and supplied by the manufacturers. Before testing the specimens were oven-dried at 110 °C for 24 hours.

[d]Values in this column are the average of five samples.

[e]Values in this column are the average of three samples.

(Table Continued)

EFFECT OF UNSATURATED STEAM AND CO/STEAM ATMOSPHERES[a] ON THE ABRASION RESISTANCE[b] OF

ALUMINA REFRACTORIES[c][95], Continued

Footnotes continued

[f] Samples not available for abrasion testing due to damage in the exposure test vessel.

[g] Value is significantly different at the 99% level of confidence from the value for samples fired in air at 1000 °C.

===

COAL SLURRY EROSION TESTING[a] OF ALUMINA-CONTAINING CERAMICS[103]

Material	Erosion Crater[b]			
	Depth (µm)		Length (µm)	
	As-received	Ground	As-received	Ground
Test Set 1[c]				
Cemented WC (WC-10% Co-4% Cr, Kennametal K701, reference standard)	---	1.9-3.5[d]	---	1070-3430[d]
Al$_2$O$_3$, hot-pressed (SNG452T, K-060, Kennametal)	17.8	22.3[d]	1440	3870[d]
Al$_2$O$_3$-TiC, hot-pressed (K-090, Kennametal)	21.3	19.1[d]	1220	3660[d]
SiAlON (Kyon 2000, Kennametal)	1.4	2.3[d]	1020	2350[d]
Test Set 2[e]				
Cemented WC (WC-8.5 Co-4.5 Cr, Kennametal K701, reference standard)	---	3.1[f]	---	---
Al$_2$O$_3$-TiC (Babcock & Wilcox)	---	33.8[f]	---	not given

[a] The slurry consisted of 8% by weight of solids from the SRC-1 coal liquefaction process (Wilsonville pilot plant) suspended in anthracene oil. A jet of slurry impinged on the specimens at a 20° angle for 1 hour, under the conditions specified for the test sets.

[b] The depth of the erosion crater was measured using a Talysurf Model 4 Profilometer.

[c] The coal was Illinois #6, temperature 343 °C (650 °F), slurry velocity 100 m/s.

[d] The surface of these specimens was finished to 3.3×10^{-2} microns by grinding on a diamond wheel in the direction of slurry flow.

[e] The coal was Lafayette #9, temperature 589 K (600 °F), slurry velocity 140 m/s.

[f] [Pretest surface finish not given, but for most tests the specimens were finished to eliminate irregularities on the initial surface.]

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COMPARISON OF EROSION RESULTS FOR CERAMICS TESTED WITH COAL SLURRY[a] AND
SILICA SLURRY[b][103]

| Material[c] | Source | Erosion Crater Depth[d] (μm) | |
		Coal slurry[a]	Silica slurry[b]
WC-10% Co-4% Cr[e]	Kennametal (K701)	5.5	2.5
SiC, sintered α	Carborundum	0.6	2.0
SiC, hot-pressed	Norton (NC-203)	28	35.6
B_4C, sintered I	Norton	3.8	2.0
B_4C, sintered II	Norton	2.8	2.3
Al_2O_3, hot-pressed	Avco	not tested	5.0
Soda-lime glass	PPG Industries	not tested	1270

[a] Coal-slurry consisted of 8% by weight of solids from the SRC-I coal lique-
faction process (Wilsonville pilot plant) suspended in anthracene oil. A
jet of slurry impinged on the specimens at an angle of 20° for 1 hour,
145 m/s velocity, temperature not given; slurry was recycled during these
tests.

[b] Silica-slurry consisted of 8% by weight of 99.5% pure fused silica from
Harbison-Walker Refractories suspended in an oxidation-resistant, petro-
leum-derived machine oil (Energol HL-32 Sohio grade). A jet of slurry
impinged on the specimens at an angle of 90° for 5 minutes, 145 m/s veloc-
ity, temperature not given; slurry was used in a once-through mode for
these tests.

[c] The surface of the specimens was finished to 3.3×10^{-2} microns by grind-
ing on a diamond wheel in the direction of slurry flow.

[d] The depth of the erosion crater was measured using a Talysurf Model 4 Pro-
filometer.

[e] This material is the reference standard for the tests.

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EFFECT OF VARIOUS PARAMETERS ON SLURRY[a] EROSION TESTING[b][103]

(Data Continued)

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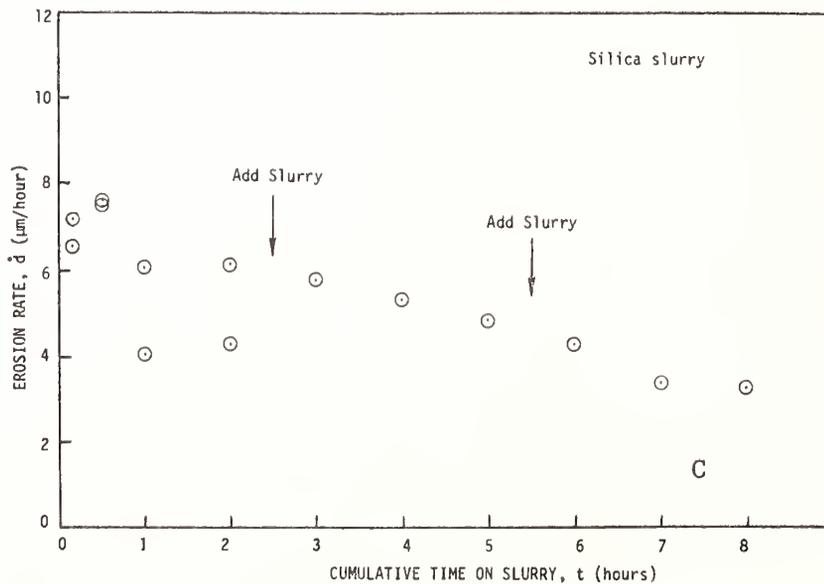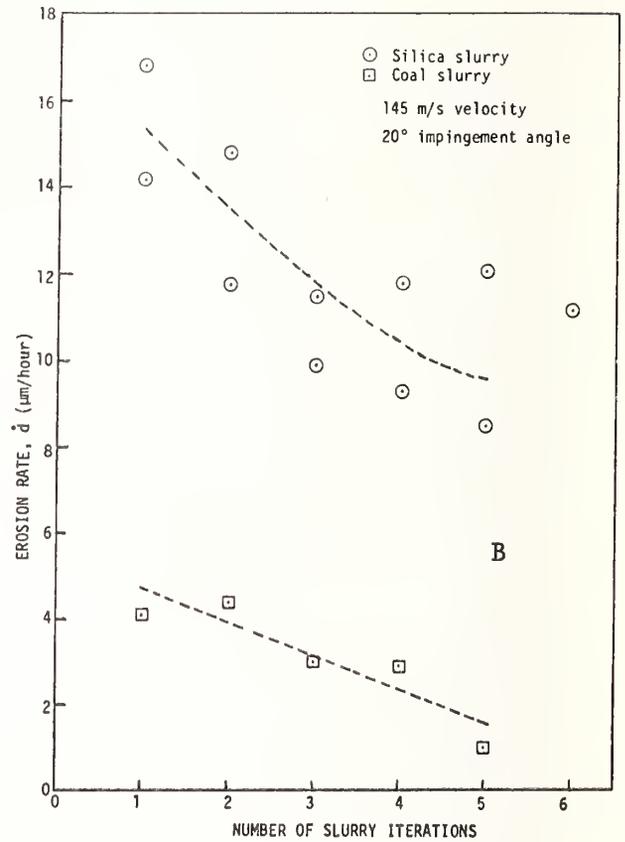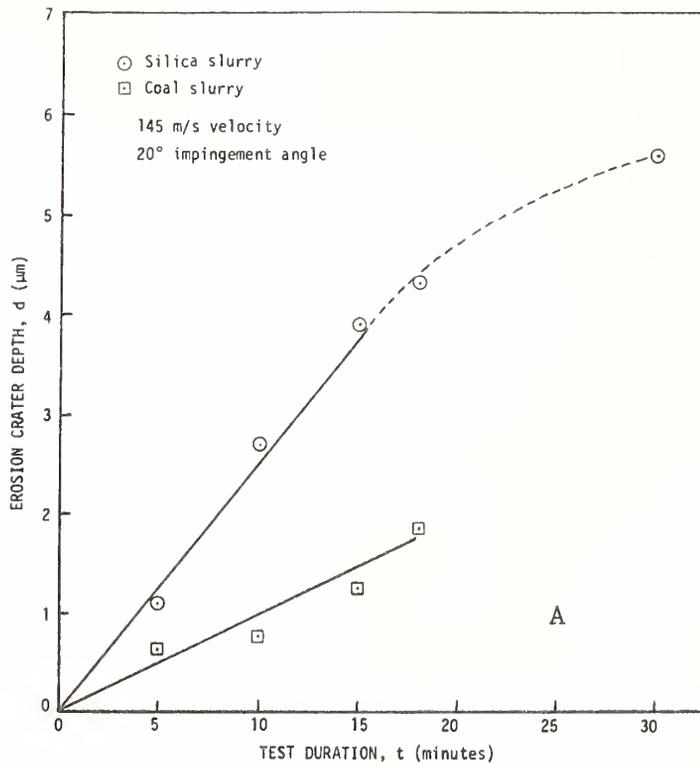
EFFECT OF VARIOUS PARAMETERS ON SLURRY[a] EROSION TESTING[b][103], Continued

Footnotes _____

[a]The coal slurry consisted of 8% by weight of solids from the SRC-I coal lique-
faction process (Wilsonville pilot plant) suspended in anthracene oil. The
silica slurry consisted of 8% by weight of 99.5% pure fused silica from Har-
bison-Walker Refractories suspended in an oxidation-resistant, petroleum-
derived machine oil (Energol HL-32 Sohio grade).

[b]A jet of slurry was impinged on specimens of the target material, a cemented
tungsten carbide, WC-10% Co-4% Cr, Kennametal K701, which was used as the
reference material in all the slurry erosion tests given in the series of re-
ports for reference [103]. Erosion was determined by the depth of the erosion
crater which was measured using a Talysurf Model 4 profilometer.
  Figure A compares the erosion crater depth for the two slurries as a function
of the duration of the test. The tests were conducted in such a way that a
constant supply of fresh slurry was used to avoid complications from slurry
degradation from particle breakage or blunting.
  Figure B compares the effect of aging of the slurry. For these tests the
20-gallon reservoir was emptied in testing after 45 minutes. After each test
the slurry was returned to the reservoir and reused in a new test. The pro-
cedure was repeated a number of times. In Figure B are plotted erosion rates
(crater depth normalized to 1 hour) versus the repetitive uses of the same
batch of slurry. The data for the silica slurry include two separate series
of tests.
  Figure C shows the effect of periodic additions of fresh slurry to continu-
ously re-cycled slurry on the erosion rates of specimens. At the times in-
dicated by "Add Slurry", a substitution of 25% volume of fresh slurry occurred.
For coal slurry (not shown) the same substitution every three hours resulted
in nearly constant erosion rates after two cycles of slurry addition.

COAL SLURRY EROSION DATA[a] FOR CEMENTED TUNGSTEN CARBIDES [103]

Material	Composition	Source	Erosion Crater[b] Depth (μm)	Length (μm) all x10³
Test Set 1[c]				
WC-Co-Cr (reference)	WC-10% Co-4% Cr	Kennametal (K701)	3.4-5.6	2.31-3.15
WC-Co	WC-9.0% Co	GTE (WA-41)	9.6	2.72
WC-Co	WC-6.0% Co	GTE (WA-1)	7.8	2.44
WC-Co	WC-6.0% Co	GTE (WA-2)	6.7	2.29
WC-Co	WC-6.0% Co	GTE (WA-35)	5.6	2.34
WC-Co	6% Co, total comp. unknown	GTE (X-614)	6.6	2.84
WC-Co	WC-4.3% Co	GTE (WA-3)	6.6	2.59
WC-Co	WC-3.0% Co	GTE (WA-4)	4.7	3.00
WC-Co	composition unknown	GTE (WA-107)	5.1	2.49
WC-Co	submicron carbide, composition unknown	GTE (WA-110)	6.6	3.00
WC-Co	composition unknown	GTE (WA-114)	12.7	2.49
WC-Co	composition unknown	GTE (WA-119)	15.2	2.39
WC-Co	non-magnetic carbide, composition unknown	GTE (WA-510)	86.4	2.64
Test Set 2				
WC-Co-Cr (reference)			1.9-3.5	1.07-3.43
WC	binderless, comp. not given	Kennametal (SP-278)	8.5	3.00
WC-TaC-Co	<1.5% Co	Kennametal (K-602)	3.3	3.34
WC-Co	low Co, micrograin WC	Kennametal (SPZ313)	1.5/none	0.70
WC-Co	2.8% Co	Kennametal (K-11)	2.5	2.51
WC-Co	5.8% Co	Kennametal (K-68)	2.0	3.23
WC-Co	7.8% Co	Kennametal (K-3406)	3.3	0.84
WC-Co	12.2% Co	Kennametal (K-3109)	4.3	1.84
WC-Ni	6% Ni	Kennametal (K-801)	11.4	2.59
WC-Ni	composition not given	Kennametal (K-803)	7.1	3.35
WC-Ni-Mo	composition not given	Kennametal (K-165)	53.3	2.44
WC-Ni-Cu-Fe	94% W, 2.6% (Ni,Cu,Fe)	Kennametal (W-2)	too deep to measure	too deep to measure
WC-Ni-Cu-Fe	composition not given	Kennametal (W-11)	too deep to measure	too deep to measure
WC-Co-Cr	8.5% Co, 4.5% Cr	Kennametal (K701)	1.8	3.56
WC-Co-Cr	6.0% Co, 1.0% Cr	Kennametal (K-703)	1.2	2.08
WC-Co-Cr	14% Co + Cr	Kennametal (K-714)	2.0	2.84

(Table Continued)

COAL SLURRY EROSION DATA[a] FOR CEMENTED TUNGSTEN CARBIDES[103], Continued

Material	Composition	Source	Erosion Crater[b] Depth (µm)	Length (µm) all x10³
Test Set 3				
WC-Co-Cr (reference)			1.2-1.5	0.79-0.91
WC	WC (binderless)	Kennametal (WCX)	15.2	2.08
WC-Co	6% Co, 0.5% (Ta,Ti,NbC)	Kennametal (K-6T)	2.7	0.75
WC-Co	9.5% Co	Kennametal (K-3560)	~2.5	---
WC-Co-multicarbides	composition not given	Kennametal (CRZ 316-25)	4.3	1.52
WC-Ni-multicarbides	composition not given	Kennametal (SP 269)	14.8	1.12
WC-proprietary binder	composition not given	Kennametal (SP 317)	6.8	0.51
WC-TiCN	high Ti, fine grained	Kennametal (SP 293A)	10.4	1.47
WC-TiCN	hot-isostatic-pressed version of SP 293A	Kennametal (SP 293B)	10.8	1.57
Test Set 4--temperature given for test as 288 °C (550 °F)				
WC-Co-Cr (reference)			1.6-2.3	2.133-2.879
WC-Co-Cr	standard grade	Kennametal (K701)	0.9	1.402
WC-Co-Cr	modified K701	Kennametal (E-839)	3.9	2.113
WC-Co-Cr	standard grade	Kennametal (K-703)	0.8	1.666
WC-Co-Cr	modified K-703	Kennametal (E-817A)	1.4	1.646
WC	infiltrated macro-crystalline WC	Kennametal (FAP90)	68.1	2.306
Test Set 5--temperature given for test as 288 °C (550 °F)				
WC-Co-Cr (reference)			2.30	
WC-Co	WC-12.2 Co	Kennametal (K-3109)	3.81	
WC-Co	WC-20 Co	Kennametal (K-3520)	5.40	
WC-Co	WC-11 Co	Kennametal (K-3833)	1.78	

[a]The coal slurry consisted of 8% by weight of solids from the SRC-1 coal liquefaction process (Wilsonville pilot plant, Illinois #6 coal) suspended in anthracene oil. Unless otherwise noted, the jet of slurry impinged on the specimens at an angle of 20° for 1 hour at 343 °C (650 °F) at a 100 m/s velocity. The slurry was recycled for use with regular substitution of fresh slurry to maintain a consistent erosive quality. The surface of the materials was finished to 3.3 x 10⁻² microns by grinding on a diamond wheel in the direction of slurry flow to eliminate irregularities on the initial surface. One of the tungsten carbides was frequently retested to check test conditions and is reported as the reference standard.

(Table Continued)

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COAL SLURRY EROSION DATA[a] FOR CEMENTED TUNGSTEN CARBIDES[103], Continued

Footnotes continued

[b]The depth of the erosion craters was measured using a Talysurf Model 4 Profilometer.

[c]The individual "test sets" simply refer to groups of data which all have the same reference standard sample. Any deviation from the test conditions given in footnote a is noted.

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COAL SLURRY EROSION DATA[a] FOR CERMET MATERIALS[103]

Material	Composition	Source	Erosion Crater[b] Depth (µm)	Length (µm) all x 10³
Test Set 1[c]				
WC-Co-Cr (reference)	WC-10% Co-4% Cr	Kennametal (K701)	3.1-4.7	1.04-1.73
B₄C	hot-isostatically pressed (27 ksi, 200 °C, 1 hour)	Norton (Norbide)	2.8	1.16
Test Set 2				
WC-Co-Cr (reference)		Oak Ridge Natl. Lab.	1.9-3.5	1.07-3.43
B₄C-Ni	94% B₄C, 6% Ni		7.1	1.15
B₄C-Co	98% B₄C, 2% Co	ORNL	6.6	0.90
B₄C-Co	96% B₄C, 4% Co	ORNL	6.0	1.32
B₄C-Co	92% B₄C, 8% Co	ORNL	1.9	0.76
B₄C-SiB₄	0.9/0.1 molar B₄C/SiB₄	ORNL	too deep to measure	
B₄C-SiB₄-Co	0.9/0.1 molar B₄C+4%Co/SiB₄	ORNL	111.7	1.19
B₄C-SiB₄-Ni	0.9/0.1 molar B₄C+6%Ni/SiB₄	ORNL	68.6	1.34
B₄C-SiB₄-Ni	0.9/0.1 molar B₄C+10%Ni/SiB₄	ORNL	25.0	0.98
Test Set 3—temperature given for test as 288 °C (550 °F)				
WC-Co-Cr (reference)			2.03	not given
SiC-β	sintered β-SiC	not given	0.73	not given
Test Set 4				
WC-Co-Cr (reference)			3.4-5.6	2.31-3.15
TiB₂-Ni	0.4% Ni, 1% O₂, balance TiB₂	ORNL	17.0	not given
TiB₂-Ni	0.4% Ni, 1% O₂, balance TiB₂	ORNL	19.1	not given
TiB₂-Ni	1.5% Ni, 2.2% O₂, bal. TiB₂	ORNL	22.5	not given
TiB₂-Ni	1.0% Ni, 2.0% O₂, bal. TiB₂	ORNL	14.9	not given
TiB₂-Ni	8.6% (Ni, 90%-Mo, 10%), balance TiB₂	ORNL	16.8	not given
Diamond	commercial sintered diamond	GE (Stratapax 2538)	0.0[d]	0.0[d]
Diamond/Si-SiC	low diamond, size 1[e]	GE	135	2.95
Diamond/Si-SiC	high diamond, size 1	GE	~22.9	not given
Diamond/Si-SiC	low diamond, size 2	GE (developmental	135	2.57
Diamond/Si-SiC	high diamond, size 2	GE materials)	25-27	~0.150
Diamond/Si-SiC	low diamond, size 3	GE	187	2.94

(Table Continued)

COAL SLURRY EROSION DATA[a] FOR CERMET MATERIALS[103], Continued

Material	Composition	Source	Erosion Crater[b] Depth (μm)	Length (μm) all x 10^3
Test Set 4, continued				
Diamond/Si-SiC	high diamond, size 3	GE	7.7-10.2	not given
Diamond/Si-SiC	high diamond, size 6	GE (developmental	~4.8	not given
Diamond/Si-SiC	high diamond, size 5	GE	~6.9	not given
Diamond/Si-SiC	high diamond, size 4	GE materials)	~4.6	not given
Diamond/Si-SiC	high diamond, size 3	GE	~7.9	not given
Cr-cermet	56.7% Al_2O_3, 38.8% B_4C, 4.5% Cr	ORNL	19.1	2.93
Cr-cermet	38.9% Al_2O_3, 56.6% CrB, 4.5% Cr	ORNL	30.0	3.00
Cr-cermet	47.3% Al_2O_3, 48.2% B_4C, 4.5% Cr	ORNL	9.7	2.59
Cr-cermet	50.5% CrB, 49.5% Colmonoy No. 6	ORNL	35.1	2.27
CrB-cermet	CrB-2% Co	ORNL (100/CBC)	excessive erosion in 1 h.	
CrB-cermet	CrB-2% Fe	ORNL (101/CBF)	excessive erosion in 35 min.	
Test Set 5,[f] --reduced severity test: 600 °F, 20° impingement angle, 185 ft/sec, 10 minutes duration				
CrB-cermet	CrB-2% Co	ORNL (100/CBC)	23	0.50
CrB-cermet	CrB-12% Co	ORNL	0.5	1.30
CrB-cermet	CrB-16% Co	ORNL	1.5	1.93
CrB-cermet	CrB-20% Co	ORNL	~2.5	0.97
CrB-cermet	CrB-2% Fe	ORNL (101/CBF)	340	not given
CrB-cermet	CrB-12% Fe	ORNL	13.7	1.14
CrB-cermet	CrB-20% Fe	ORNL	1.2	1.23
CrB-cermet	CrB-2% Ni	ORNL	204	1.02
CrB-cermet	CrB-12% Ni	ORNL	1.8	1.33
CrB-cermet	CrB-16% Ni	ORNL	1.5	1.17

[a]The coal slurry consisted of 8% by weight of solids from the SRC-1 coal liquefaction process (Wilsonville pilot plant, Illinois #6 coal) suspended in anthracene oil. Unless otherwise noted, the jet of slurry impinged on the specimens at an angle of 20° for 1 hour at 343 °C (650 °F) at a 100 m/s velocity. The slurry was recycled for use with regular substitution of fresh slurry to maintain a consis-

(Table Continued)

COAL SLURRY EROSION DATA[a] FOR CERMET MATERIALS[103], Continued

Footnotes continued

tent erosive quality. The surface of the materials was finished to 3.3×10^{-2} microns by grinding on a diamond wheel in the direction of slurry flow to eliminate irregularities on the initial surface. A cemented tungsten carbide was frequently retested to check test conditions and is reported as the reference standard.

[b] The depth of the erosion craters was measured using a Talysurf Model 4 Profilometer.

[c] The individual "test sets" simply refer to groups of data which all have the same reference standard sample. Any deviation from the test conditions given in footnote a is noted.

[d] No measurable erosion.

[e] Diamond particulate size designations are 1 = coarse, 2 = fine. Low and high refer to the volume fraction of diamond.

[f] No value for a reference standard is given for this test set. The severity of the erosive conditions was greatly reduced for these materials because of the excessive erosion results under the standard test conditions. See the immediately preceding data above for CrB-cermets.

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COAL SLURRY EROSION DATA[a] FOR PARTIALLY STABILIZED ZIRCONIA[103]

Material	Erosion Crater Depth (μm)[b]
Test Set 1[c]	
Cemented WC (WC-10% Co-4% Cr, Kennametal K701, reference standard)	2.03
ZrO_2(MgO), partially stabilized, fabricated[e]	30.5
ZrO_2(MgO), partially stabilized, as-cut[e]	7.42
ZrO_2(MgO), partially stabilized, fabricated[f]	8.50
ZrO_2(MgO), partially stabilized, as-cut[f]	24.6
Test Set 2[d]	
Cemented WC (WC-8.5 Co-4.5 Cr, Kennametal K701, reference standard)	3.1
ZrO_2(MgO), partially stabilized (American Feldmuehle)	12.4
ZrO_2(3 mol% MgO), partially stabilized (Coors Porcelain)	26.7
ZrO_2(16.9 wt% Y_2O_3), partially stabilized (American Feldmuehle)	177.8

[a] The coal slurry consisted of 8% by weight of solids from the SRC-1 coal liquefaction process (Wilsonville pilot plant) suspended in anthracene oil. The jet of slurry impinged on the specimens at an angle of 20° for 1 hour under the conditions specified for the test sets. The slurry was recycled for use with regular substitution of fresh slurry to maintain a consistent erosive quality. The surface of specimens was finished by grinding on a diamond wheel in the direction of slurry flow to eliminate irregularities on the initial surface. A cemented tungsten carbide was frequently retested to check test conditions and is reported as the reference standard.

[b] The depth of the erosion craters was measured using a Talysurf Model 4 Profilometer.

[c] The coal was Illinois #6, temperature 288 °C (550 °F), slurry velocity 100 m/s.

[d] The coal was Lafayette #9, temperature 589 K (600 °F), slurry velocity 140 m/s.

[e] This material is optimized for maximum mechanical strength.

[f] This material is optimized for thermal shock resistance.

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INFLUENCE OF HARDNESS[a] ON COAL SLURRY EROSION BEHAVIOR[b] OF CEMENTED
TUNGSTEN CARBIDES[c] [103]

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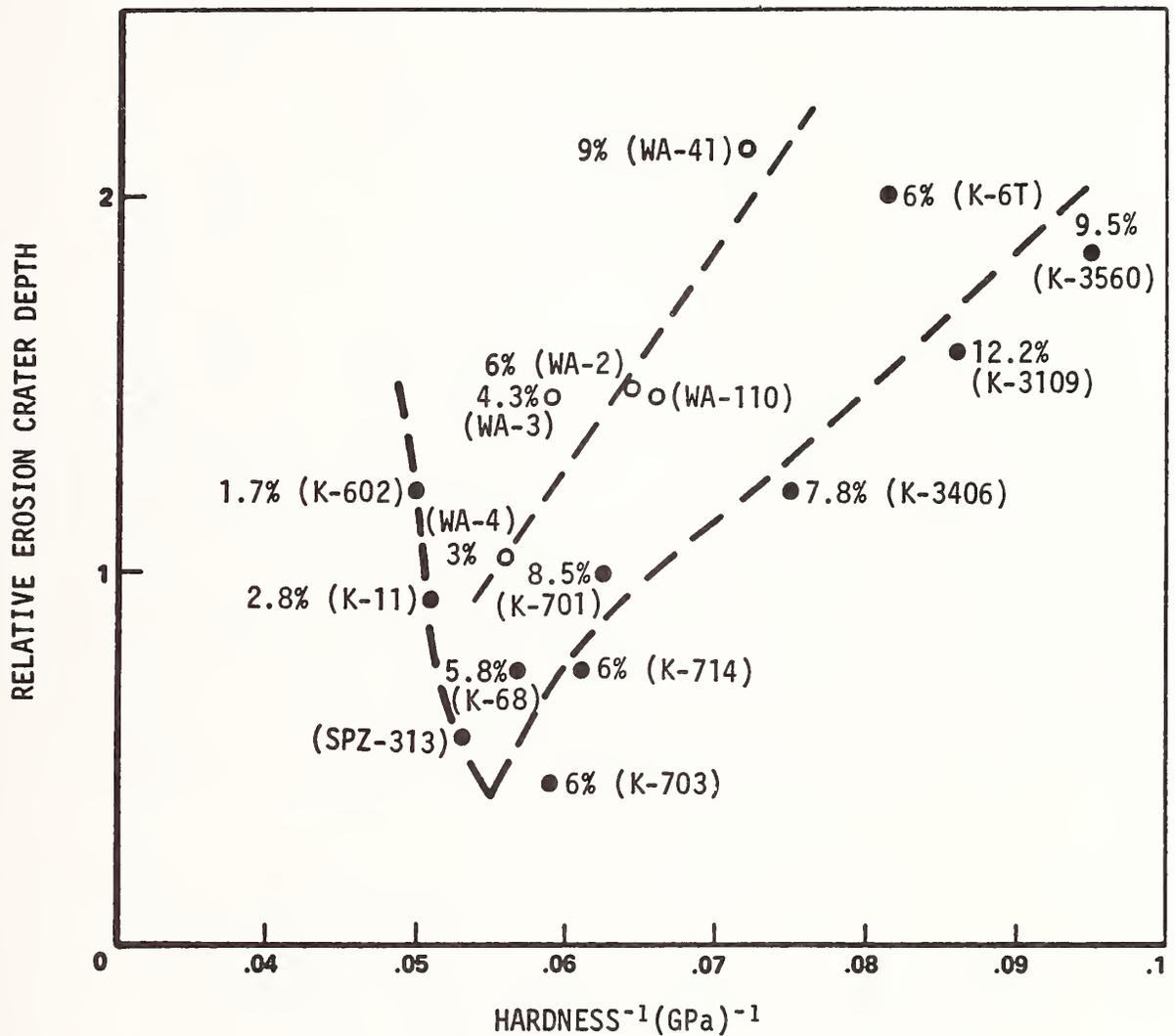
[a] See Section B.3.2.141 for the hardness data plotted here.

[b] See Section B.2.2.30 for the erosion testing data. The erosion crater depth
plotted here is relative to the crater depth of the reference standard
values given in B.2.2.30.

[c] The percentage values labelling the data points correspond to the cobalt
binder content, where known.

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SILICA SLURRY EROSION DATA[a] FOR SEVERAL CERAMIC MATERIALS[103]

| Material[c] | Source | Erosion Crater Depth[a] (μm) | |
		Maximum[b]	Average[b]
WC-10% Co-4% Cr (reference standard)	Kennametal (K701)	3.0	1.9
		4.8	4.0
SiC, sintered	Carborundum (α-SiC-I)	7.6	1.1
SiC, sintered	Carborundum (α-SiC-I)	4.0	1.8
SiC, hot-pressed	Norton (NC-203)	39.4	25.8
SiC, hot-pressed	Norton (NC-203)	61.0	46.7
B_4C, sintered	Norton (Norbide-I)	3.0	1.1
B_4C, sintered	Norton (Norbide-II)	3.6	1.0
Al_2O_3, hot-pressed	Avco	7.4	2.8
Si_3N_4, hot-pressed	Norton (NC-132)	26.7	12.1
73% SiO_2-14% Na_2O-9% CaO	PPG, Ind. (Soda-lime glass)	1320.	1364.

[a]The silica slurry consisted of 8% by weight of 99.5% pure silica (Harbison-Walker fused silica, grade GP7I) suspended in Energol HL-32 oil. A jet of slurry impinged on the specimens at an angle of 90° for 5 minutes, 145 m/s velocity, at 250 °C. The slurry was not recycled but was used once. The depth of the erosion crater was measured using a Talysurf Model 4 Profilometer.

[b]The average crater depth is included because some materials do not erode uniformly and may show localized depth variations due to the microstructure. The average crater profile was determined by averaging over a fixed crater width (∿800 μm).

[c]Specimen surfaces were finish ground with a 600-grit diamond wheel.

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SILICA SLURRY EROSION DATA[a] FOR CEMENTED TUNGSTEN CARBIDES[b][103]

Material[b]	Erosion Crater[c] Average Volume (mm^3)	Erosion Crater[c] Maximum Depth (μm)
WC-10 Co-4Cr (refer-	---	4.3
ence standard)	---	4.0
WC-8 Co	6.41 x 10^{-4}	4.2
	6.98 x 10^{-4}	5.5
WC-8 Co	---	3.3
	---	3.5
WC-8 Co (hot isostat-	---	3.7
ically pressed)	1.15 x 10^{-3}	6.7
WC-8 Co (hot isostat-	---	3.0
ically pressed)	---	4.5
WC-10 Co (hot isostat-	---	7.7
ically pressed)	---	10.8
WC-10 Co (hot isostat-	1.31 x 10^{-3}	5.9
ically pressed)	2.32 x 10^{-3}	10.6
WC-10 Co (hot isostat-	---	8.9
ically pressed)	---	8.8
WC-11 Co (hot isostat-	---	73.7
ically pressed)	---	77.5
WC-18 Co (hot isostat-	1.86 x 10^{-2}	5.7
ically pressed)	1.95 x 10^{-2}	7.9

[a]The silica slurry consisted of 8% by weight of 99.5% pure silica (Harbison-Walker fused silica, grade GP7I) suspended in Energol HL-32 oil. A jet of slurry impinged on the specimens at an angle of 90° for 5 minutes, 140 m/s velocity, at 177 °C (350 °F). The slurry was not recycled but was used once.

[b]Except for the reference material which was Kennametal K701, all the tungsten carbides were supplied by Terra Tek, Salt Lake City, Utah. The specimen surfaces were finished to 1 μm diamond.

[c]The depth of the erosion crater was measured using a Talysurf Model 4 Profilometer. To obtain the crater volume, the craters were measured by the surface profilometer traces across the centerline; two traces were made at 90° to each other, yielding two cross-sectional views or four half-cross-sectional planes from the crater centerline to the outer edge. The crater volumes were calculated by integrating over 360° the measured area of each half-cross-sectional plane to give four values of volume for each crater which were then averaged. Duplicate specimens were tested.

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SILICA SLURRY[a] EROSION RATES[b] FOR SEVERAL CERAMIC MATERIALS[103]

Material	Source	Erosion Rate[b] $(\Delta V/V) \times 10^{10}$	Erosion Characteristics
WC-10% Co-4% Cr[c]	Kennametal (K701)	1.8 ± 0.7	---
B_4C, hot-pressed	Norton (Norbide)	0.3 ± 0.15	} { Transgranular delamination or microflaking.
SiC, sintered	Carborundum (α-SiC)	0.7 ± 0.2	
SiC, hot-pressed	Norton (NC-203)	$19. \pm 2.$	Intergranular failure.
SiC, hot-pressed	Ceradyne	$10. \pm 1.$	Intergranular failure.
Al_2O_3, sintered	3M Co. (Alsimag 614)	6.5	Intergranular failure.
Al_2O_3, hot-pressed	Avco	1.4	} { Transgranular delamination or microflaking.
AlN, hot-pressed	Battelle	1.6	
Si_3N_4, hot-pressed	Norton (NC-132	$5. \pm 1.$	Intergranular failure.
Glass-ceramic	Corning (Pyroceram 9606)	$210. \pm 10.$	Transgranular delamination or microflaking.
Soda-lime glass	PPG Ind. (Float glass)	$2200. \pm 100.$	Microcutting indentation fracture.

[a] The silica slurry consisted of 8% by weight of 99.5% pure silica (Harbison-Walker fused silica, grade GP7I) suspended in Energol HL-32 oil. A jet of slurry impinged on the specimens at an angle of 90° for 10 minutes, 135 m/s velocity, temperature not given. The slurry was not recycled but was used once.

[b] The erosion crater depth was measured using a Talysurf Model 4 Profilometer. To obtain the crater volume, the craters were measured by the surface profilometer traces across the centerline; two traces were made at 90° to each other, yielding two cross-sectional views or four half-cross-sectional planes from the crater centerline to the outer edge. The crater volumes were calculated by integrating over 360° the measured area of each half-cross-sectional plane to give four values of volume for each crater which were then averaged. The erosion rate reported is equal to the specimen erosion crater volume (ΔV) divided by the volume (V) of slurry used in the test.

[c] The cemented tungsten carbide is the reference standard.

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SILICA SLURRY EROSION DATA[a] FOR CEMENTED TUNGSTEN CARBIDES[b] WITH
VARIED COBALT BINDER CONTENT[103]

| Cobalt Binder Volume Fraction | Erosion Crater[c] | | |
| | 20° Impingement Angle[d] | 90° Impingement Angle[e] | |
	Maximum Depth (μm)	Maximum Depth (μm)	Volume (mm³)
0.1 (reference)[f]	1.50	9.61	3.32×10^{-3}
		9.55	3.30×10^{-3}
0.051	0.26	3.91	1.35×10^{-3}
		4.01	1.38×10^{-3}
0.076	0.84	5.70	1.97×10^{-3}
	0.86		
0.101	0.89	11.10	3.86×10^{-3}
		12.40	4.30×10^{-3}
0.148	1.70	15.20	5.27×10^{-3}
	1.78		
0.171	1.64	45.2	1.56×10^{-2}
		42.4	1.46×10^{-2}
0.193	2.37	79.2	2.74×10^{-2}
0.208	1.39	88.4	3.05×10^{-2}
	1.01		
0.236	8.13	111.7	3.69×10^{-2}
		123.4	4.21×10^{-2}
0.306	4.72	231.3	7.99×10^{-2}
0.369	20.8	312.0	1.08×10^{-1}

[a]The silica slurry consisted of 8% by weight of 99.5% pure silica (Harbison-Walker fused silica, grade GP7I) suspended in Energol HL-32 oil. A jet of slurry impinged on the specimens at a velocity of 133 m/s for 10 minutes at 20 and 90° impingement angles (see footnotes d and e). The slurry was not recycled but was used once.

[b]Specimen surfaces were prepared by grinding on diamond-grit wheels, 15 minutes on 180 mesh grit, 15 minutes on 400 mesh, 5 minutes on 600 mesh, and by polishing 10 minutes with 3 μm diamond, 5 minutes with 1 μm diamond, and a final polishing with 0.25 μm alumina (Linde B) for 10 minutes.

[c]The erosion crater depth was measured with a Talysurf Model 4 Profilometer. The crater volume was calculated from the profilometer traces assuming axial symmetry about the jet axis. See Section B.2.2.36, footnote b.

[d]Temperature for slurry, 165 °C (438 K). The surfaces of these specimens were not optically flat but had pronounced curvature in the direction of the axis of the erosion craters. It was therefore difficult to obtain proper reference planes for surface profilometer measurements so these crater depths are considered less accurate than others measured in the original reports.

[e]Temperature for slurry, 436 K (163 °C).

[f]The reference standard was Kennametal K701.

==

EFFECT OF SURFACE STRESS STATE[a] ON SLURRY EROSION[b] OF PARTIALLY STABILIZED ZIRCONIA[103]

| Material | Surface Finish[a] | Erosion Crater[c] | |
		Depth (μm)	Volume (mm³)
Partially stabilized zirconia	As-fabricated	51.8	0.0180
(optimized for maximum	As-cut	49.0	0.0169
mechanical strength)	Ground	48.2	0.0166
Partially stabilized zirconia	As-fabricated	66.0	0.0232
(optimized for thermal	As-cut	68.0	0.0237
shock resistance)	Ground	66.0	0.0232

[a]As-fabricated material is presumed to have the stabilized cubic matrix with tetragonal precipitates. The specimens designated as "ground" were ground on a 320 grit diamond wheel to transform the precipitates (the tetragonal to monoclinic transformation is believed to be martensitic). The as-cut specimens were included for comparison with coal slurry erosion data also given, see Section B.2.2.32.

[b]The silica slurry consisted of 8% by weight of 99.5% pure silica (Harbison-Walker fused silica, grade GP7I) suspended in Energol HL-32 oil. A jet of slurry impinged on the specimens at an angle of 90° for 10 minutes, 130 m/s velocity, at 177 °C. The slurry was not recycled but was used once.

[c]The erosion crater depth was measured with a Talysurf Model 4 Profilometer. The crater volume was calculated from the profilometer traces assuming axial symmetry about the jet axis. See Section B.2.2.36, footnote b.

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EFFECT OF SURFACE STRESS STATE[a] ON SLURRY EROSION[b] OF CEMENTED
TUNGSTEN CARBIDES[c][103]

| Material[c] | Surface Condition[a] | Erosion Crater[d] | |
		Depth (μm)	Volume (mm³)
WC-5.1 Co	Polished (unstressed)	2.5	0.00089
	Ground (stressed)	3.8	0.00134
WC-36.9 Co	Polished (unstressed)	161.	0.056
	Ground (stressed)	137.	0.047

[a] Specimens designated polished (unstressed) were polished for prescribed
periods of time on 600 grit diamond and then on 6, 3, and 0.3 μm diamond
wheels to remove at least 40 μm of material. Microhardness indentation
tests compared with other tests for the series of materials (see Section
B.3.2.144) indicated that the desired surface conditions had been achieved.
Specimens designated as ground were ground for two minutes each on 320
and 600 diamond grit paper to provide a stressed surface.

[b] The silica slurry consisted of 8% by weight of 99.5% pure silica (Harbison-
Walker fused silica, grade GP7I) suspended in Energol HL-32 oil. A jet
of slurry impinged on the specimens at an angle of 90° for 10 minutes,
80 m/s velocity, at 177 °C. The slurry was not recycled but was used once.

[c] See Section B.2.2.37 for erosion data for the series of cermets to which
these materials belong. The proportions of cobalt refer to the minimum
and maximum Co binder volume fraction values of the cermets in the series.

[d] The depth of the craters was measured with a Talysurf Model 4 Profilometer.
The crater volume was calculated from the profilometer traces assuming
axial symmetry about the jet axis. See Section B.2.2.36, footnote b.
The values given are the averages of data from three craters.

B.2 Erosion, Erosion/Corrosion, and Abrasion Effects

B.2.2 Refractories

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SILICA SLURRY EROSION DATA[a] FOR VARIOUS TUNGSTEN CARBIDE-COBALT-BASED CERMETS[b][103]

Nominal Composition					Erosion Crater[c]	
WC	Co	TaC	TiC	Other	Depth (μm)	Volume (mm^3)
99+	<1.0	--	--	--	14.0	0.00483
96.7	3.0	0.3	--	--	5.8	0.00202
96.0	3.0	1.0	--	--	4.2	0.0014
93.2	6.0	0.5	0.3	--	14.0	0.00483
86.0	10.0	--	--	4.0 Cr	10.9	0.00378
83.2	6.5	4.8	4.5	1.0 Cr_2C_3	6.6	0.00228
87.0	8.4	--	--	4.5 Cr (reference)[d]	6.4	0.00216

[a]The silica slurry consisted of 8% by weight of 99.5% pure silica (Harbison-Walker fused silica, grade GP7I) suspended in Energol HL-32 oil. A jet of slurry impinged on the specimens at an angle of 90° for 10 minutes, 130 m/s velocity, at 177 °C. The slurry was not recycled but was used once.

[b]Materials from Valenite Die and Wear Parts (Division of Valeron Corporation).

[c]The depth of the craters was measured with a Talysurf Model 4 Profilometer. The crater volume was calculated from the profilometer traces assuming axial symmetry about the jet axis. See Section B.2.2.36, footnote b.

[d]One material, Kennametal K701, was tested to check experimental conditions and to be a reference standard for comparison with data from other tests.

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EFFECT OF SURFACE FINISH[a] AND TEST TIME ON THE SILICA SLURRY EROSION[b]
OF CEMENTED TUNGSTEN CARBIDES[c][103]

| Material[c] | Volume of Erosion Crater[d] (mm^3/h) | |
	Standard Finish 5 minute test	Special Finish 10 minute test
Reference Standard[e]	2.12×10^{-2}	1.74×10^{-2}
WC-8 Co	1.62×10^{-2}	2.64×10^{-2}
WC-8 Co	1.14×10^{-2}	1.75×10^{-2}
WC-8 Co (hot isostatically pressed)	1.61×10^{-2}	---
WC-8 Co (hot isostatically pressed)	1.67×10^{-2}	1.64×10^{-2}
WC-10 Co (hot isostatically pressed)	3.4×10^{-2}	6.24×10^{-2}
WC-10 Co (hot isostatically pressed)	2.34×10^{-2}	4.21×10^{-2}
WC-10 Co (hot isostatically pressed)	2.43×10^{-2}	3.17×10^{-2}
WC-11 Co (hot isostatically pressed)	3.50×10^{-2}	3.56×10^{-2}
WC-18 Co (hot isostatically pressed)	3.38×10^{-1}	2.84×10^{-1}

[a]The standard surface finish was prepared by polishing 10 minutes on 6 μm
diamond, 5 minutes on 1.5 μm diamond, and 1 minute on 0.3 μm diamond. The
special surface finish was prepared by polishing 15 minutes on 6 μm dia-
mond, 15 minutes on 3 μm diamond, 10 minutes on 1 μm diamond, and 10 minutes
on 0.3 μm diamond. The intent of the special polishing is to remove a min-
imum of 40 μm of material from the surface at which point the remaining
stresses are those resulting from fabrication.

[b]The silica slurry consisted of 8% by weight of 99.5% pure silica (Harbison-
Walker fused silica, grade GP7I) suspended in Energol HL-32 oil. A jet of
slurry impinged on the specimens at an angle of 90° for the times given
in the tables, 140 m/s velocity, temperature not specified. The slurry was
not recycled but was used once.

[c]Except for the reference material, all the tungsten carbides were supplied
by Terra Tek, Salt Lake City, Utah. See Section B.2.2.35 for other tests
on these materials.

[d]The depth of the craters was measured using a Talysurf Model 4 Profilometer.
The crater volume was calculated from the profilometer traces assuming
axial symmetry about the jet axis. See Section B.2.2.35, footnote c.
Note that the volumes given in the above table have been adjusted to the
volume which would be eroded in 1 hour.

[e]The reference standard used is the WC-Co-Cr material supplied by Kennametal
(K701).

COMPARISON OF SLURRY EROSION DATA[a] VERSUS HARDNESS[b] FOR TUNGSTEN CARBIDES CEMENTED WITH VARIOUS BINDERS[c] [103]
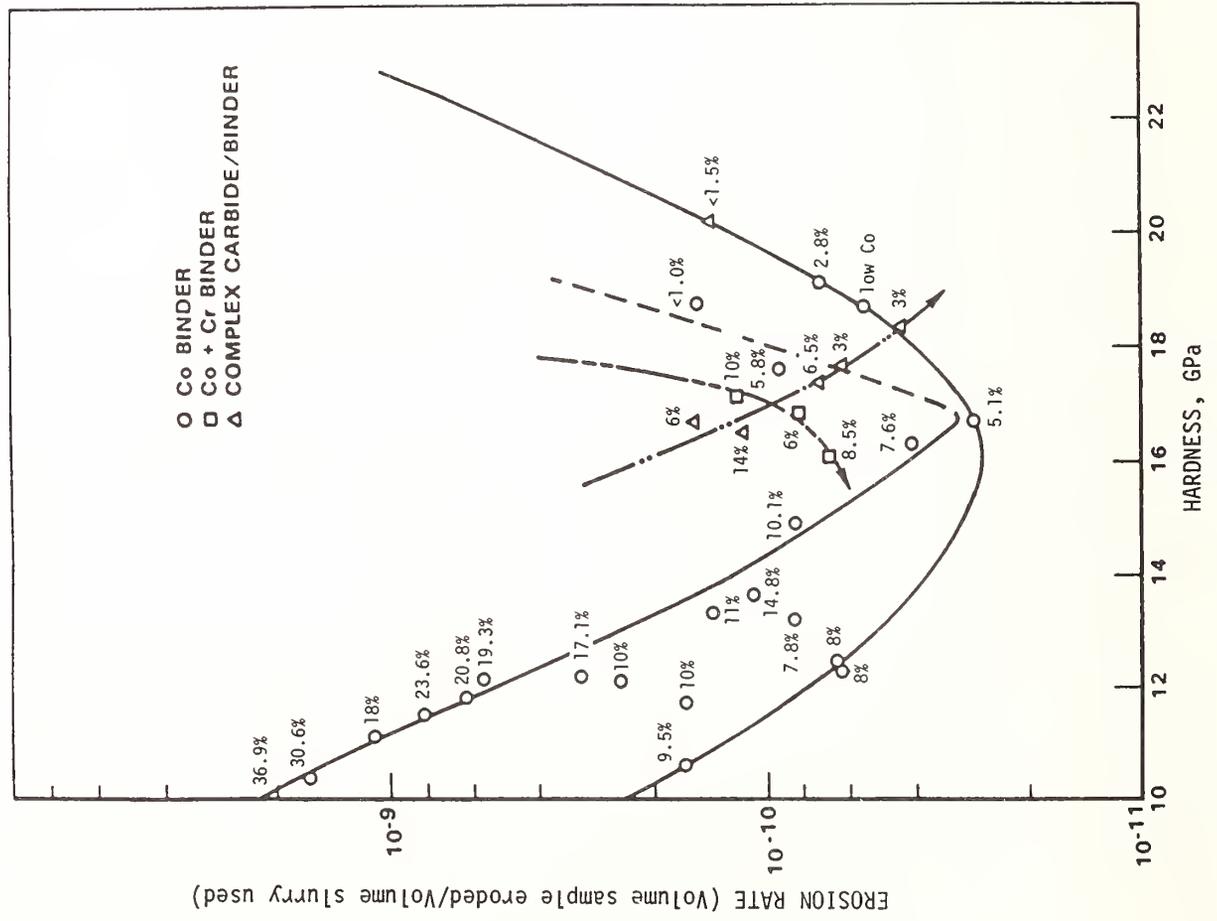
[a]Silica slurry erosion data from various tests were combined for this plot. The crater volume data were converted to volume loss of material per unit volume of slurry used in each test. The data were then normalized with respect to the value for the reference standard (Kennametal K701) for the specific series of tests.

[b]See also Section B.2.2.33.

[c]The data appear in Sections B.2.2.35, B.2.2.37, B.2.2.39, B.2.2.40, and B.2.2.41. Footnotes in these sections give experimental details and the method of measuring the crater volumes.

O Co BINDER
□ Co + Cr BINDER
△ COMPLEX CARBIDE/BINDER

EROSION RATE (Volume sample eroded/Volume slurry used)

HARDNESS, GPa

==

COAL SLURRY EROSION TESTING[a] OF BORIDE CERAMICS[b][105]

Material[b]	Initial Nickel Content[c] (mol %)	Maximum Erosion Crater Depth[d] (μm)
TiB_2-Ni[e]	20	17.8
TiB_2-Ni[e]	15	35.6
TiB_2-Ni[e]	10	30.5
Reference Standard[f]	--	4.6
TiB_2-Ni	[20][g]	33.
TiB_2-Ni	[15][g]	42.
TiB_2-Ni	[10][g]	28.
TiB_2-Ni	[20][g]	31.
TiB_2-Ni	[15][g]	26.
TiB_2-Ni	[10][g]	43.
Reference Standard[f]	--	7.

[a] Testing was performed at Battelle Columbus Laboratories, see Sections B.2.2.7 through B.2.2.42 and B.2.3.5 through B.2.3.9. The coal slurry consisted of 8% by weight of solids from the SRC-I coal liquefaction process (Wilsonville pilot plant, Illinois #6 coal) suspended in anthracene oil. The jet of slurry impinged on the specimens at an angle of 20° for 1 hour, at ∿340 °C at a 100 m/s velocity. The slurry was recycled for use with regular substitution of fresh slurry to maintain a consistent erosive quality.

[b] These materials were prepared by activated sintering to TiB_2 powders during hot pressing by using liquid Ni at temperatures slightly above the Ni melting point. See Section B.2.2.31 for data for other TiB_2-Ni cermets prepared by Oak Ridge National Laboratory and tested by Battelle Columbus Laboratories.

[c] After hot pressing, the secondary products formed in the first three ceramics is stated as being distinctly different for the 10 and 20 mole percent nickel-content materials and these different products vary over the 10-20% starting composition range in an orderly way. The final retained Ni content is often less than 1 wt% of the dense boride product.

[d] The depth of the erosion craters was measured using a Talysurf Model 4 Profilometer.

[e] These are the same materials for which erosion test data are reported in Section B.2.2.6. These three materials were prepared with size-classified TiB_2 powders, mean particle size 5.6 μm and width of particle size distribution 0.5 to 13.5 μm.

[f] The reference standard is the same cemented tungsten carbide used in all of the Battelle Columbus tests, Kennametal K701.

[g] These initial Ni content values are not explicitly stated in the reports but are implied. The comment is made in the original reports that the final Ni content is high for these materials. These were prepared with the as-received powders, mean particle size 7.2 μm and width of particle size distribution 0.5 to 35 μm.

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EROSION TEST[a] DATA FOR VARIOUS COATINGS[1]

Material	Brand Name or Other Designation	Source	Relative Erosion Factor[b]				
			20 °C, 20° imp[c]	20 °C, 30° imp[c]	20 °C, 45° imp[c]	20 °C, 90° imp[c]	700 °C, 90° imp[c]
CVD[d] W pure coating		Rolla[e]				0.53	0.25
CVD W on mild steel		Rolla				0.57	0.34
CVD W on mild steel	550	Rolla				0.48	0.16
TiC coating on WC	Carboloy 523	Carboloy				0.31	0.72
CVD TiC, unknown substrate			pen[f]			pen[f]	pen[f]
71.5WC-12TiC-10TaC-6.5Co	VR 73		pen			pen	
VR 73 + B_4C			pen			pen	
WC-11.5TaC-8TiC-8.5Co	GE 370		pen			pen	
GE 370 + $20NiTiB_2$			pen			pen	
CNTD[g] W-C on Mo	CM-500	Chemetal Corp.		pen[h]	pen[h]	pen[h]	
85WC-15Co	LW-IN40		pen			pen	
WC-10Ni-2.3W	2S5		pen			pen	
CVD SiC pure coating			0.20[i]			0.05[i]	0
CVD SiC on graphite cloth which had been converted to SiC			0.13[i]			0.06[i]	0
CVD SiC on HIP[j] SiC and graphite	Lummus 1-8	MTC & Ceradyne	0			0	0[k]
SiC in Ni over Cu	Nye-Carb		pen			pen	
CNTD SiC on graphite		Chemetal Corp.		0.003-0.006[h]	0.007[h]	0.005-0.009[h]	
Si_3N_4 on graphite			pen			pen	pen
CNTD Si_3N_4 on graphite		Chemetal Corp.		0.011[h]	0.009[h]	0.070[h]	
TiB_2 on nickel[l]		CPMRC[m]	0			0	0
TiB_2 on 310 SS[l]	18B-11	United Tech.	0			0	0[n]
TiB_2 on 310 SS[l]	19A-13	United Tech.	0			0	0[n]
TiB_2 on 410 SS[o]	BM5	Battelle NW	0			0	0[k,p]
TiB_2 on 410 SS[q]	BM6	Battelle NW	0			0	0[k,p]
TiB_2 CNTD	168-179	Chemetal Corp.	0			0	0[k,p]
TiB_2 on Kovar[l]	115-115	Sandia Labs.	0			0	0[k,p]
CVD Al_2O_3, unknown substrate			pen			pen	pen
Cr_2O_3	LC-4		pen			pen	
Cr_2O_3-$5SiO_2$-$3TiO_2$		CWS Corp.	0.61			1.23	
0.5C-4Si-16Cr-4B-4Fe-2.4Cu-2.4Mo-2.4W-balance Ni[r]	Ni-Cr-B-Cu	CWS Corp.	0.98			1.32	2.79
35(WC+8Ni)-11Cr-2.5B-2.5Fe-2.5Si-0.5C-balance Ni[r]	NiWC	CWS Corp.	0.72			1.11	2.06
HfN sputtered on steel			pen			pen	

- -

(Table Continued)

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EROSION TEST[a] DATA FOR VARIOUS COATINGS[1], Continued

| Material | Brand Name or Other Designation | Source | Relative Erosion Factor[b] | | |
			20 °C, 20° imp[c]	20 °C, 90° imp[c]	700 °C, 90° imp[c]
88WC + 12Co on 440C SS[r]	Plasmalloy 702-E	Plasmadyne	pen	pen	pen
75Cr-C + 25Ni-Cr on 440C SS[r]	Plasmalloy 915-E	Plasmadyne	pen	pen	pen
Fe-base TiC on 440C SS[r]	FerroTiC	Plasmadyne	pen	pen	pen
CVD TiC, unknown substrate			pen	pen	pen
CVD TiN, unknown substrate			pen	pen	pen
CVD TiCN on Ti-6Al-4V			0	pen[s]	
CVD TiCN on Inconel 718			0	pen[s]	
CVD TiCN on WC			pen[s]	pen[s]	

[a] Erosion Test conditions: 1/2-inch square specimens subjected to erosion by 27 μm Al_2O_3 abrasive, 170 m/s particle velocity, 3 minutes duration, N_2 atmosphere, 5 g/min abrasive flow.

[b] Relative Erosion Factor = $\dfrac{\text{Volume loss of sample}}{\text{Volume loss of Stellite 6B}}$, volume loss calculated from weight loss and density data; Stellite 6B = 30Cr-4.5W-1.2C-balance Co.

[c] imp = angle of impingement.

[d] CVD = chemical vapor deposition.

[e] Rolla = Rolla Metallurgy Research Center, Bureau of Mines.

[f] pen = coating penetrated.

[g] CNTD = controlled nucleation thermochemical deposition.

[h] Test was 15 minutes duration.

[i] Weight loss noted, but no visible crater.

[j] HIP = hot isostatic pressed.

[k] Also tested at 700 °C, 20° impingement angle, with same results.

[l] Coating electrodeposited.

[m] CPMRC = College Park Metallurgy Research Center, Bureau of Mines.

[n] Electrodeposited TiB_2 showed excellent erosion resistance, even after testing for 8 minutes. There was, however, spalling of these samples at 700 °C due to thermal expansion mismatching.

[o] 1.5-2.2 mils sputtered on at 700 °C.

[p] Retesting at 20 °C after tests at 700 °C revealed some spalling.

[q] 1.5-2.2 mils sputtered on at 360 °C.

[r] Coating applied by plasma-gun process. The three Plasmadyne coatings ranked 702-E > FerroTiC > 915-E in terms of their relative erosion resistance as to which lasted longer during the test time period.

[s] Retarded penetration, coating 0.002 in. thick.

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EROSION TESTS[a] ON NICKEL BASE ALLOYS[b] FOR COATINGS AND CLADDINGS[53],

Continued

Footnotes continued

[d] AMS-4775 flame spray coating too thin.

[e] Tungsten carbide addition consists of WC and W_2C mixture.

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COAL SLURRY EROSION DATA[a] FOR TITANIUM DIBORIDE COATINGS[103]

Coating Information	Base Material	Source	Erosion Crater[b] Depth (μm)	Length (μm) all x 10³
Test Set 1[c]				
Reference material, WC-Co-Cr (WC-10% Co-4% Cr)		Kennametal (K701)	5.3-6.3	0.76
TiB$_2$, 35 μm, CVD[e]	Cemented WC (K701)	SNLA	3.5	1.35
TiB$_2$, 35 μm, CVD	Cemented WC (K701)	SNLA	3.3	2.59
TiB$_2$, 50 μm, CVD	TiC-Ni (K151A)	SNLA	7.4	0.97
TiB$_2$, 50 μm, CVD	TiC-Ni (K151A)	SNLA	3.5	1.12
TiB$_2$, 50 μm, CVD	TiC-Ni (K151A)	SNLA	6.6	1.24
TiB$_2$, 50-100 μm, CVD	Cemented TiB$_2$ (0.8% Ni-[<1% Ni,1% O$_2$,balance TiB$_2$])	ORNL/SNLA	3.4	1.54
TiB$_2$, 50-100 μm, CVD	Cemented TiB$_2$ (<1% Ni-1% O$_2$-balance TiB$_2$)	ORNL/SNLA	no measurable wear	
TiB$_2$, 50-100 μm, CVD	Cemented TiB$_2$ (9.6% Ni-0.7% O$_2$-balance TiB$_2$)	ORNL/SNLA	5.0	2.47
TiB$_2$, 50-100 μm, CVD	Cemented TiB$_2$ (0.9% Ni-8.1% O$_2$-balance TiB$_2$)	ORNL/SNLA	1.5	1.01
Test Set 2--temperature given for test as 288 °C (550 °F)				
Reference material, WC-Co-Cr			2.03	not given
TiB$_2$, ~4 μm	WC-Co (GE 370)	not given	5.23	not given
TiB$_2$, ~7 μm	WC-Co (GE 370)	not given	11.4	not given
TiB$_2$, ~4 μm	WC-Co (GE 370)	not given	8.74	not given
TiB$_2$, ~20 μm	WC-Co (GE 370)	not given	no measurable wear	
Test Set 3--temperature given for test as 288 °C (550 °F)				
Reference material, WC-Co-Cr			1.65	not given
TiB$_2$, ~29 μm	WC-Co (GE 370)	not given	no measurable wear	
TiB$_2$, ~24 μm	WC-Co (GE 370)	not given	no measurable wear	
TiB$_2$, ~25 μm	WC-Co (GE 370)	not given	33.0	not given
Test Set 4[f]--temperature given for test as 589 K (600 °F); coal is Lafayette #9				
Reference material, WC-Co-Cr (WC-8.5% Co-4.5% Cr)		Kennametal (K701)	1.4[g]	not given
TiB$_2$, 50-100 μm, CVD	Cemented TiB$_2$ (<1% Ni-1% O$_2$-balance TiB$_2$)	ORNL/SNLA	3.6[g]	not given
TiB$_2$, 50-100 μm, CVD	Same as above	ORNL/SNLA	3.3[g]	not given
TiB$_2$, 50-100 μm, CVD	Same as above	ORNL/SNLA	4.1[g,h]	not given
TiB$_2$, 50-100 μm, CVD	Same as above	ORNL/SNLA	5.1[g]	not given

(Table Continued)

COAL SLURRY EROSION DATA[a] FOR TITANIUM DIBORIDE COATINGS[103], Continued

Coating Information	Base Material	Source	Erosion Crater[b] Depth (μm)	Length (μm) all x 10³
Test Set 5[f] --temperature given for test as 589 K (600 °F); coal is Lafayette #9				
Reference material, WC-Co-Cr (WC-8.5% Co-4.5% Cr)		Kennametal (K701)	1.6[g,h]	not given
TiB$_2$, 50-100 μm, CVD	Cemented TiB$_2$ (0.9% Ni-8.1% O$_2$-balance TiB$_2$)	ORNL/SNLA	2.9[g,h]	not given
TiB$_2$, 50-100 μm, CVD	Same as above	ORNL/SNLA	3.3[g]	not given
TiB$_2$, 50-100 μm, CVD	Same as above	ORNL/SNLA	4.6[g]	not given
TiB$_2$, 50-100 μm, CVD	Same as above	ORNL/SNLA	4.6[g,h]	not given
TiB$_2$, 50-100 μm, CVD	Cemented TiB$_2$ (9.6% Ni-0.7% O$_2$-balance TiB$_2$)	ORNL/SNLA	3.2[g]	not given
TiB$_2$, 50-100 μm, CVD	Same as above	ORNL/SNLA	2.5[g,h]	not given
TiB$_2$, 50-100 μm, CVD	Same as above	ORNL/SNLA	5.8[g]	not given
TiB$_2$, 50-100 μm, CVD	Same as above	ORNL/SNLA	5.2[g]	not given
Test Set 6[f] --temperature given for test as 589 K (600 °F); coal is Lafayette #9				
Reference material, WC-Co-Cr (WC-8.5% Co-4.5% Cr)		Kennametal (K701)	1.5[h]	not given
TiB$_2$, 50-100 μm, CVD	Cemented TiB$_2$ (0.8% Ni- [<1% Ni,1% O$_2$,balance TiB$_2$])	ORNL/SNLA	3.1[g]	not given
TiB$_2$, 50-100 μm, CVD	Same as above	ORNL/SNLA	4.1[g]	not given
TiB$_2$, 50-100 μm, CVD	Same as above	ORNL/SNLA	5.1[g,h]	not given
TiB$_2$, 50-100 μm, CVD	Same as above	ORNL/SNLA	4.6[g]	not given
Test Set 7 --temperature given for test as 288 °C (550 °F)				
Reference material, WC-Co-Cr (WC-10% Co-4% Cr)		Kennametal	1.4	2.420
TiB$_2$, 10.8 μm[i]	TiC-Ni (K151A)	Kennametal	7.6	3.073
TiB$_2$, 8.5 μm[i]	TiC-Ni (K151A)	Kennametal	6.9	3.353
TiB$_2$, 8.8 μm[i]	TiC-Ni,Mo (K165)	Kennametal	3.1	1.625
TiB$_2$, 7.9 μm[i]	W-10 (9 W, 1 Ni, Cu, Fe)	Kennametal	50.5	2.763
Test Set 8 --temperature given for test as 288 °C (550 °F)				
Reference material, WC-Co-Cr (WC-10% Co-4% Cr)		Kennametal	2.3-2.4	2.169-2.768
TiB$_2$, 29 μm[j]	TiC-Ni (K151A)	Kennametal	24.3	3.454
TiB$_2$, 30 μm[j]	TiC-Ni (K151A)	Kennametal	47.7	2.900
TiB$_2$, 29 μm[j]	TiC-Ni,Mo (K165)	Kennametal	45.2	not given
TiB$_2$, 30 μm[j]	TiC-Ni,Mo (K165)	Kennametal	19.4	2.885

(Table Continued)

COAL SLURRY EROSION DATA[a] FOR TITANIUM DIBORIDE COATINGS[103], Continued

Footnotes

[a] The coal slurry consisted of 8% by weight of solids from the SRC-1 coal liquefaction process (Wilsonville pilot plant, Illinois #6 coal) suspended in anthracene oil. Unless otherwise noted, the jet of slurry impinged on the specimens at an angle of 20° for 1 hour at 343 °C (650 °F) at a 100 m/s velocity. The slurry was recycled for use with regular substitution of fresh slurry to maintain a consistent erosive quality. The surface of the materials was finished to 3.3×10^{-2} microns by grinding on a diamond wheel in the direction of slurry flow to eliminate irregularities on the initial surface. A tungsten carbide material was used as a standard reference to check test conditions.

[b] The depth of the erosion craters was measured using a Talysurf Model 4 Profilometer.

[c] The individual "test sets" simply refer to groups of data which all have the same reference standard sample. Any deviation from the test conditions given in footnote a is noted.

[d] Composition not given.

[e] CVD = chemically vapor deposited.

[f] Pressure drop/velocity relationship is different from that in Test Set 1 and adjustments were made to maintain the jet velocity at 100 m/s. Hence these results vary from those of Test Set 1 for the same material.

[g] CVD-cooled surfaces were curved in direction of jet impact; data represent best estimates.

[h] This test was noted as being the one in the series of test sets 4, 5, and 6 which contained the fresh slurry added for rejuvenation (one quarter volume of fresh slurry replaced every 3 hours of slurry use).

[i] High-temperature process.

[j] Low-temperature process.

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COAL SLURRY EROSION DATA[a] FOR MISCELLANEOUS COATINGS[103]

Coating Information	Base Material	Source	Erosion Crater[b] Depth (μm)	Length (μm) all x 10³
Test Set 1				
Reference material, WC-Co-Cr (WC-10% Co-4% Cr)		Kennametal (K701)	5.3-6.3	0.76
TiC, 20 μm	WC-Co-Cr (K701)	R. P. & C. Valve	5.8	2.03
TiC, 20 μm; WC, 75-100 μm	420 stainless steel (buffed)	R. P. & C. Valve	84.8	2.13
TiC, 20 μm; WC, 75-100 μm	420 stainless steel (as deposited)	R. P. & C. Valve	96.5	2.39
TiC, 25 μm	WC-6% Co-1% Cr (K703)	Kennametal	7.0	2.0
TiN, 37 μm	WC-6% Co-1% Cr (K703)	Kennametal	9.8	1.85
η-K703, 5 μm	WC-6% Co-1% Cr (K703)	Kennametal	4.3	2.79
η-K703, 7 μm	WC-6% Co-1% Cr (K703)	Kennametal	6.3	3.00
TMT-745	WC-10% Co-4% Cr (K701)	Turbine Metals Technology	7.1	1.06
TMT-745	WC-6% Co-1% Cr (K703)	Turbine Metals Technology	7.1	2.02
Test Set 2--temperature given for test as 288 °C (550 °F)				
Reference material, WC-Co-Cr (WC-10% Co-4% Cr)			1.6-2.3	2.133-2.879
TiC[d]	not given	R. P. & C. Valve	10.4[e]	not given
TiC[d]	not given	R. P. & C. Valve	800.[f]	not given
Test Set 3--temperature given for test as 288 °C (550 °F)				
Reference material, WC-Co-Cr (WC-10% Co-4% Cr)			1.4-1.6	2.133-2.420
TMT-745, 13 μm	WC-5% Cr-Co	Turbine Metals Technology	8.2	4.506
TMT-745, 51 μm	WC-6% Cr-Co	Turbine Metals Technology	21.1	2.692
TMT-745, 152-203 μm	WC-6% Cr-Co	Turbine Metals Technology	none measureable	
Test Set 4				
Reference material, WC-Co-Cr (WC-10% Co-Cr)			3.1-4.7	1.04-1.73
SiC, CVD[g,h]	graphite	Not given	1.1	0.97
SiC, CVD[h]	graphite	Not given	0.6	0.29
SiC, CVD[h]	graphite	Not given	2.5(10.1 max.)	0.93
TMT-745[d]	cemented WC (?)	Turbine Metals Technology	too deep to measure	

[a]The coal slurry consisted of 8% by weight of solids from the SRC-1 coal liquefaction process (Wilsonville pilot plant, Illinois #6 coal) suspended in anthracene oil. Unless otherwise noted, the jet of slurry impinged on the specimens at an angle of 20° for 1 hour at 343 °C (650 °F) at a 100 m/s veloc-

(Table Continued)

COAL SLURRY EROSION DATA[a] FOR MISCELLANEOUS COATINGS[103], Continued

Footnotes continued

ity. The slurry was recycled for use with regular substitution of fresh slurry to maintain a consistent erosive quality. The surface of the materials was finished to 3.3×10^{-2} microns by grinding on a diamond wheel in the direction of slurry flow to eliminate irregularities on the initial surface. A tungsten carbide material was used as a standard reference to check test conditions.

[b] The depth of the erosion craters was measured using a Talysurf Model 4 Profilometer.

[c] The individual "test sets" simply refer to groups of data which all have the same reference standard sample. Any deviation from the test conditions given in footnote a is noted.

[d] TiC is assumed. Neither coating material nor base is specified in the table in the original reports.

[e] Duration of test 7.5 minutes; results are normalized to 1 hour.

[f] Duration of test 15 minutes; results are normalized to 1 hour.

[g] CVD = chemically vapor deposited.

[h] These three coatings have widely different morphologies; the most erosion-resistant morphology closely resembles an alternating amorphous/crystalline structure.

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COAL SLURRY EROSION DATA[a] FOR LASER-PROCESSED MATERIALS[b][103]

Material[b]	Erosion Crater Depth[a]	Remarks
Stellite 6B (reference standard)	80 μm	Co-30 Cr-4 W-1 C, wrought plate
Stellite 6B (single laser pass)	147	
Stellite 6B (double laser pass)	101 106	
Stellite 12 (low speed pass)	322 366	Co-30 Cr-8 W-1.5 C, weld deposit; surface ground after laser processing
Stellite 12 (high speed pass)	271	Weld deposit; surface ground after laser processing
Stellite 1 (as laser melted)	34-84	Co-30 Cr-12 W-2.5 C, weld deposit
Stellite 1 (surface ground)	189	Weld deposit
Stellite 6 + TiC	74	Fairly low volume fraction of hard particles; ended by same mode as wrought Stellite 6B

[a] Coal slurry consisted of 8% by weight of solids from the SRC-I coal liquefaction process (Wilsonville pilot plant) suspended in anthracene oil. A jet of slurry impinged on the specimens at an angle of 20° for 10 minutes, 185 ft/s (56.4 m/s) velocity, at 600 °F, [slurry was probably recycled for these tests but that is not explicitly stated]. The depth of the erosion crater was measured using a Talysurf Model 4 Profilometer.

[b] The materials were fused to mild steel substrates with high-power laser radiation. Hardfacing alloys in powder form were placed on the substrate while overlapping linear passes with a continuous wave CO_2 laser melted the powder fusing it to the base material.

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COAL SLURRY EROSION DATA[a] FOR LASER-PROCESSED TUNGSTEN CARBIDE COATINGS WITH
VARYING CARBIDE CONTENT[b][103]

Carbide Volume Fraction[c]	Matrix	Carbide	Erosion Crater Depth (μm)[a]	
			10 minutes	30 minutes
0.63	Co	WC	6.0[d]	20.0[d]
0.62	Co	WC	17.0[d]	39.3[d]
0.50	Co	WC	16.0[d]	45.0[d]
0.50	Co	WC	22.0[d]	50.8
0.25	Haynes 25	WC	7.6	66.0
0.00	Stellite 6	WC	24.7	111.7
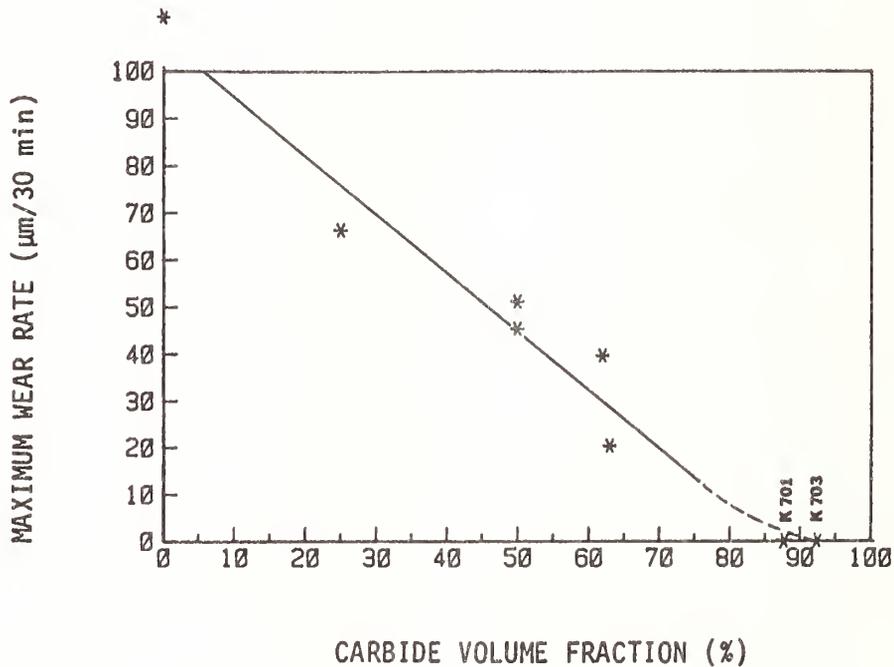
CARBIDE VOLUME FRACTION (%)

_____

[a]Coal slurry consisted of 8% by weight of solids from the SRC-I coal lique-
faction process (Wilsonville pilot plant, Lafayette #9 coal) suspended in
anthracene oil. A jet of slurry impinged on the specimens at an angle of
20° for the times stated in the table, 55-58 m/s velocity, at 315 °C. The
slurry was recycled for use with regular substitution of fresh slurry to
maintain a consistent erosive quality. The depth of the erosion craters
was measured using a Talysurf Model 4 Profilometer unless otherwise noted.

[b]The materials were fused to stainless steel substrates with high-power laser
radiation. Powder mixtures were placed on the substrate while overlapping
linear passes with a continuous wave CO_2 laser melted the powder fusing it
to the base material.

[c]In starting powder. WC powder (-200 +325 mesh) was mixed with Co powder
mixtures of two different particle sizes (one -400 mesh and one -200+325
mesh). The mixed samples varied in carbide volume fraction.

[d]These data obtained from optical microscope measurements, required because
of surface porosity.

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EFFECT OF DEPOSITION TEMPERATURE AND VARYING SUBSTRATES ON COAL SLURRY
EROSION[a] OF TITANIUM DIBORIDE COATINGS[b][103]

Substrate	Deposition Temperature °C	Coating Thickness μm	Maximum Erosion Crater Depth[c] μm
WC-Co-TaC-TiC (GE-370)	900	15	5.46/8.51
WC-Co-TaC-TiC (GE-370)	800	8	6.98 (P)[d]
WC-Co-TaC-TiC (GW-370) [f]	950	29	8.25[e]
Reference Standard[f]	---	--	0.76
WC-Co-TaC-TiC (GE-370)	1000	24	5.10[e]
WC-Co-TaC-TiC (GE-370)	850	9	9.65[e] (P)[d]
WC-Co-TaC-TiC (GE-370)	750	4	6.60[e] (PS)[g]
Reference Standard	---	--	0.90
WC-Co-TaC-TiC (GE-370)	850	14	17.40[e] (PS)[g]
WC-Co-TaC-TiC (GE-370)	1050	15	5.10
WC-Co-TaC-TiC (GE-370)[h]	900	16	1.-3.
WC-Co-Cr (Kennametal K701)	900	15-20	2.80[e]
Reference Standard	---	--	0.60
WC-Co (GE-883)	900	15-20	2.94[e]
WC-Co-TaC-TiC (GE-370)	900	15-20	0-1.24[e]
WC-Co-Cr (Kennametal K703)	900	15-20	7.62[e] (C)[i]
Reference Standard	---	--	0.61
TiB$_2$-Ni, hot-pressed	900	15-20	2.03[e]
WC-Co-TaC-TiC (GE-370)	900	15-20	0-2.33[e]
TiC-Ni (Kennametal K-151A)	900	15-20	20.93[e] (P)[d]
Reference Standard	---	--	1.10
TiB$_2$-Ni, hot-pressed	900	15-20	0[e]
WC-Co-TaC-TiC (GE-370)	900	15-20	17.78 (P,C)[di]

[a] The coal slurry consisted of 8% by weight of solids from the SRC-I coal
liquefaction process (Wilsonville pilot plant, Lafayette #9 coal) suspended
in anthracene oil. A jet of slurry impinged on the specimens at an angle
of 20° for 1 hour, 140 m/s velocity, at 316 °C. The slurry was recycled
for use with regular substitution of fresh slurry to maintain a consistent
erosive quality. The test runs immediately following this slurry rejuvena-
tion are noted.

[b] Coatings are all chemically vapor deposited (CVD) titanium diboride on the
stated substrates. Source of the samples was Oak Ridge National Laboratory.

[c] Crater depths were measured with a Talysurf Model 4 Profilometer. Because
of the surface roughness of the coatings, accurate depth values for shallow
craters were difficult to obtain. These values were generated using a ref-
erence overlay technique involving making a surface profile in an unaffected
area adjacent to a crater parallel to the crater axis. After averaging,
the reference trace was cut and lined up with the trace from the crater cen-
terline and the maximum crater depth measured.

- -

(Table Continued)

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EFFECT OF DEPOSITION TEMPERATURE AND VARYING SUBSTRATES ON COAL SLURRY
EROSION[a] OF TITANIUM DIBORIDE COATINGS[b][103], Continued

Footnotes continued

[d] P denotes complete penetration of the coating at designated thickness,
with minimal wear to the substrate surface.

[e] Slurry rejuvenation took place prior to this test run. See footnote a.

[f] Periodically erosion tests were made using WC-Co-Cr (Kennametal K701) to
check the test procedures and to be a reference standard for comparison
of data.

[g] PS denotes penetration of the coating at designated thickness, with ob-
vious wear into the substrate.

[h] This specimen was processed to have a smooth coating surface prior to
testing.

[i] C denotes that the coating cracked in and around the entire crater, with
no apparent influence on the isolated wear site.

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B.3.2.148 Thermal Shock Estimates Based on Physical and Mechanical Properties of Alumina Castables

B.3.2.149 Strength Loss Agreement with Estimated Thermal Shock Damage Resistance for Alumina Castables

B.3.2.150 Effect of Several Fuel Combustion Environments on the Tensile Strength of Various Ceramic Tube Materials

B.3.2.151 Effect of a Basic Slag in a High-Temperature Environment on Half-Ring Fracture Strength of Sintered Silicon Carbide Tubes

B.3.2.152 Effect of Various Coal-Oil Combustion Products on the Flexure Strength of Various Ceramic Materials

B.3.2.153 Comparison of Fracture Strengths of Silicon-Based Ceramics After Coal-Oil Fuel Combustion Tests

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B.3.2.155 Flexure Strength of SiC Ceramics After Long-Term Oxidation

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B.3.2.157 Crack Growth Data for a Silicon Carbide and a Silicon Nitride Tested in Air

B.3.2.158 Fracture Toughness of Various Silicon-Based Ceramics

B.3.2.159 Fracture Toughness Data for Some Silicon Nitrides

B.3.2.160 Effect of Creep on the Fracture of Silicon-Based Ceramics

B.3.2.161 Creep Data for Two Silicon-Based Ceramics

B.3.2.162 Creep Crack Growth Data for a SiAlON Material

B.3.2.163 Fracture Toughness Data for Silicon Carbides and Silicon Nitride

B.3.2.164 Preliminary Fracture Toughness Data for a Silicon Carbide

B.3.2.165 Fracture Toughness Data for Silicon Carbides in a Simulated Coal Combustion Gas

B.3.2.166 Effect of a Simulated Coal Combustion Gas on the Fracture Toughness of a Silicon Carbide

B.3.2.167 Flexure Strength of Silicon Carbide under Static Load as a Function of Time

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EFFECT OF FLOWING HIGH-BTU GAS[a] ON THE COMPRESSIVE STRENGTH[b]
OF AN ALUMINA-SILICA REFRACTORY[c][9]

| Exposure Time | Compressive Strength[b] | |
hr	Air Fired, 980° C	High-Btu Gas[a], 980° C, 1000 psi
0[d]	3,180±300	3,180±300
50	2,925±240	4,950±660
100	2,450±180	4,100±420
150	2,510±280	5,750±560
200	2,320±160	3,750±360
250	2,080±160	5,970±440

[a]Steam-gas mixture composition: 45% H_2O, 55% high-Btu gas composed of 39.5% H_2, 30.9% CO, 21.0% CO_2, 8.6% CH_4. An average of 6 scf/h of the mixture passed into the reaction vessel; superficial velocity was calculated to be 0.04 ft/min in the vessel.

[b]ASTM C-133-72, specimens were 1 in x 1 in x 2 in; values given are 95% confidence intervals; units are psi.

[c]Refractory is a dense 55% Al_2O_3-fireclay calcine aggregate (A. P. Green's Lo-Abrade) with intermediate-purity calcium aluminate cement (Universal Atlas Cement's Refcon).

[d]All samples were prefired in air at 980° C for 24 hr, then exposed.

EFFECT OF THERMAL SHOCK[a] ON THE MODULUS OF RUPTURE[b] OF SOME ALUMINA REFRACTORIES[2]

Composition	Brand Name	Manufacturer	MOR (ambient) psi	MOR (2000 °F) psi	Ambient MOR after Thermal Shock psi
96% Al_2O_3	Plicast 40	Plibrico	797(42)[c]	516	99(±55)[d]
94.6% Al_2O_3-0.5% SiO_2	Alfrax B 57	Carborundum	111(16)[e]	no test	no test
94.5% Al_2O_3-0.15% SiO_2	Greencast-94	A.P. Green	1054(280)[c]	538(204)[c]	no test
above, steel-fiber reinforced[f]			1517	808	no test
93.7% Al_2O_3-0.1% SiO_2	Castolast G	Harbison-Walker	849(61)[c]	no test	242(±154)[d]
93.7% Al_2O_3	Taycor 414-FH	Taylor	1875(193)[c]	966(601)[e]	133(±82)[d]
93% Al_2O_3	B&W Kao Phos 30	Babcock & Wilcox	2063(125)[c]	no test	150(±108)[d]
92.6% Al_2O_3-6.8% CaO- 0.4% SiO_2	H-W Lightweight Castable 33	Harbison-Walker	78(46)[g]	no test	no test
89.6% Al_2O_3-0.7% SiO_2	Resco Cast AA-22	Resco Products	1789(120)[c]	499(82)[e]	345(±278)[d]
over 87% Al_2O_3	Alfrax 66	Carborundum	722(100)[c]	no test	181(±120)[d]

[a]Thermal shock test: samples heated rapidly to temperatures ranging from 1700 °F to 2000 °F, soaked for 2 to 5 hours, and rapidly cooled to ambient temperatures.

[b]Modulus of rupture (MOR) test complied with ASTM C268-70 for measurements at ambient temperature and ASTM C583-67(72) for measurements at 2000 °F.

[c]Standard deviation based on 5 samples.

[d]Variation of two samples tested.

[e]Standard deviation based on 3 samples.

[f]1.7% Steel fibers (Ribtec) added to Greencast-94 refractory mix.

[g]Standard deviation based on 4 samples.

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COMPRESSIVE, SHEAR, AND IMPACT STRENGTHS OF SOME ALUMINA REFRACTORIES [2]

Composition	Brand Name[a]	Compressive Strength[b] (ambient) psi	Compressive Strength (2000 °F) psi	Shear Strength[c] (ambient) psi	Impact Strength[d] (ambient) in-lb/in^2
96% Al$_2$O$_3$	Plicast 40	5633(374)[e]	6208(584)[f]	2661(454)[e]	8.4(0)[e]
94.6% Al$_2$O$_3$-0.5% SiO$_2$	Alfrax B 57	465(32)[e]	no test	279(118)[e]	no test
94.5% Al$_2$O$_3$-0.15% SiO$_2$	Greencast-94	7571(118)[e]	4815(464)[e]	3613[g]	56.4(44)[e]
above, steel-fiber reinforced[h]		5511	3783	3850	no test
93.7% Al$_2$O$_3$-0.1% SiO$_2$	Castolast G	5668(380)[e]	4358(1215)[f]	2799(731)[e]	16.8(6.7)[e]
93.7% Al$_2$O$_3$	Taycor 414-FH	13685(469)[e]	9438(1682)[f]	4893(1381)[e]	9.6(1.2)[e]
93% Al$_2$O$_3$	B&W Kao Phos 30	5274(607)[e]	2355(±331)[i]	926(70)[e]	no test
92.6% Al$_2$O$_3$-6.8% CaO-0.4% SiO$_2$	H-W Lightweight Castable 33	745(40)[e]	no test	400(93)[e]	no test
89.6% Al$_2$O$_3$-0.7% SiO$_2$	Resco Cast AA-22	7751(465)[e]	2720(763)[e]	4478(746)[e]	15.2(11)[e]
over 87% Al$_2$O$_3$	Alfrax 66	3927(±83)[i]	3565(306)[f]	2132(179)[e]	10.4(5)[e]

[a]See Section B.3.2.10 for manufacturers.
[b]ASTM C133-72 procedure followed with some modification for high-temperature testing.
[c]ASTM D732-46(75) in modified version followed.
[d]Mechanical shock measured following ASTM C589-68.
[e]Standard deviation based on 3 samples.
[f]Standard deviation based on 4 samples.
[g]Earlier reported value 7227 said to be incorrect due to a computational error.
[h]1.7% Steel fibers (Ribtec) added to Greencast-94 refractory mix.
[i]Variation of 2 samples tested.

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EFFECT OF ALKALI-GAS EXPOSURE[a] ON THE COMPRESSIVE STRENGTH[b]
OF SOME REFRACTORIES [9]

| Refractory | Brand Name Manufacturer | Compressive Strength[b] after Treatment[a] | | | |
| | | A | B | C | D |
		High-Btu Gas	Alkali- Gas	Solution, Air Fired	Solution, Alkali- Gas
Dense 95% Al_2O_3 castable, calcium aluminate bond	Castolast G Harbison- Walker	6,880±1310	7,480± 390	9,030	8,020
Dense 90% Al_2O_3 castable, calcium aluminate bond	DOE 90 Generic Preparation	5,360± 280	5,970± 880	9,950	11,770
Dense 55% Al_2O_3 castable, calcium aluminate bond	Lo-Abrade A. P. Green	4,540± 720	7,650±1200	3.060	3,170
Lightweight 50% Al_2O_3 insulating castable, calcium aluminate bond	Litecast 75-28 General Re- fractories	3,130± 260	2,720	1,740	1,700
Dense 45% Al_2O_3 castable, calcium aluminate bond	B&W Kaocrete D Babcock & Wilcox	7,290± 500	7,280±1170	1,600	2,100
Dense 45% Al_2O_3 castable, calcium aluminate bond	H.S. Brikcast BF General Re- fractories	10,960±1440	11,170±5640	2,940	3,830
90% Al_2O_3 ramming mix, phosphate bond	Brikram 90R General Re- fractories	12,690±2100	11,510±4300	14,300	23,070
Dense 90% Al_2O_3 castable, phosphate bond	Resco Cast AA-22 Resco Products	5,040± 320	3,610	7,100	2,090
60% Al_2O_3 ramming mix, phosphate bond	HW 23-75 Harbison- Walker	11,430± 780	10,100±1530	9,430	12,040
90% Al_2O_3 brick	Arco-90 General Re- fractories	20,410±1900	21,750±6370	26,650	24,740
45% Al_2O_3 fired super duty brick	KX-99 A. P. Green	13,880±1900	13,170	13,930	10,630

[a]All samples were fired in air at 980° C for 24 hr and then given one of the following
treatments:
  A--Samples were exposed to a high-Btu gas for 250 hr at 980° C and 1000 psi. Feed
    gas composition: 26% H_2, 21% CO, 14% CO_2, 5% CH_4, 34% H_2O.
  B--A 2:1 mixture by weight of KOH/NaOH was placed in a Pt crucible and surrounded by
    test specimens in the reaction vessel. Feed gas composition was the same as in
    A. The specimens were subjected to the resulting atmosphere for 200 hr at 980° C
    and 1000 psi. At the end of the test the alkali had been reduced about 50% and the
    KOH/NaOH ratio was 1.58 in the crucible.
  C--Samples were soaked for 24 hr in a solution of 500g KOH and 250g NaOH in 600 ml
    water, oven dried at 125° C for 24 hr, and then air fired at 980° C for 200 hr.
  D--Samples were soaked and dried as in C, and then subjected to the same alkali-gas
    atmosphere described in B.

[b]ASTM C-133-72, specimens were 1 in x 1 in x 2 in; values given are 95% confidence in-
  tervals; units are psi.

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EFFECT OF HEAT TREATMENT[a] ON COMPRESSIVE STRENGTH AND POROSITY

OF A HIGH-ALUMINA CASTABLE REFRACTORY[b][9]

Maximum Cure Temperature, °C	Compressive Strength,[c] psi		Pore Diameter[d] Micrometers
	Lot A	Lot B	
120		8,860±550	0.04
600	7,430± 140	7,990±350	
800	12,720± 830	8,750±640	0.17
1,000	10,140± 720	7,850±610	0.23
1,200	12,780±1390	6,960±760	
1,400		7,870±610	0.80

[a]Heat treatment consisted of raising the temperature at the rate of 30 °C/h from ambient to 540 °C, holding for 6 hours, then raising at 60 °C/h to the "maximum cure temperature". Samples were soaked at the cure temperature for 8 hours and then the furnace and contents were cooled naturally to ambient conditions.

[b]Castable used is a high-purity tabular alumina, calcium aluminate bonded (Castolast G, Harbison-Walker). Samples were prepared from two different commercial lots in the form of 2-inch cubes. 10 ml H_2O per 100 g dry refractory was mixed with a Hobart mixer for 1 minute, cast, cured under 100% relative humidity at 29 °C for 24 hours, and dried at 120 °C for 24 hours.

[c]Compressive strength determined according to ASTM C133-72; 95% confidence intervals given.

[d]The diameter of the smallest surface-connected pores which mercury must enter in order to reach a degree of penetration equal to 0.05 cm^3/g.

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EFFECT OF CERAMIC FIBER[a] ADDITION ON THE MODULUS OF RUPTURE[b] OF A
HIGH-ALUMINA CASTABLE REFRACTORY[c][9]
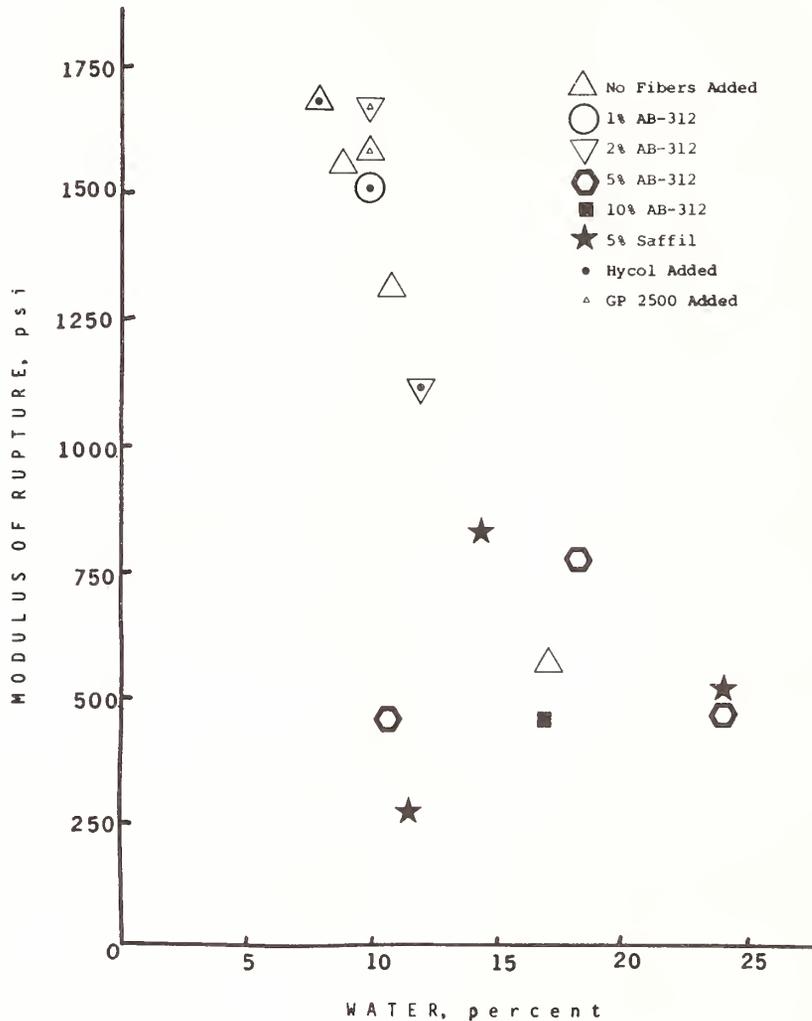
[a]Fibers used were AB-312 from 3M Company, high-alumina $Al_2O_3-B_2O_3-SiO_2$,
11 μm diameter, and Saffil from Imperial Chemical Industries, high-temper-
ature 98% Al_2O_3-2% SiO_2, 3 μm diameter. Fibers were chopped in a high-
speed Waring blender; length after chopping varied 20-150 μm. Castable,
fibers, and water were mixed in blender before casting. Cast samples were
moist cured for 24 h, oven dried for 24 h. Wetting agents were used to re-
duce the amount of water necessary, 6 ounces added per 100 pounds of cement:
Hycol from W.R. Grace Company and GP 2500 from Cresset Chemical Company.

[b]Modulus of rupture measured using bars 1 in. x 1 in. x 7 in., following
ASTM C583-67.   Each point is the average of 2 specimens.

[c]Castable used is a high-purity tabular alumina, calcium aluminate bonded
(Castolast G, Harbison-Walker).

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EFFECT OF CERAMIC FIBER[a] ADDITION ON CORRELATION OF ABRASION

WEIGHT LOSS[b] WITH MODULUS OF RUPTURE[c] OF A HIGH-ALUMINA CASTABLE REFRACTORY[d][9]
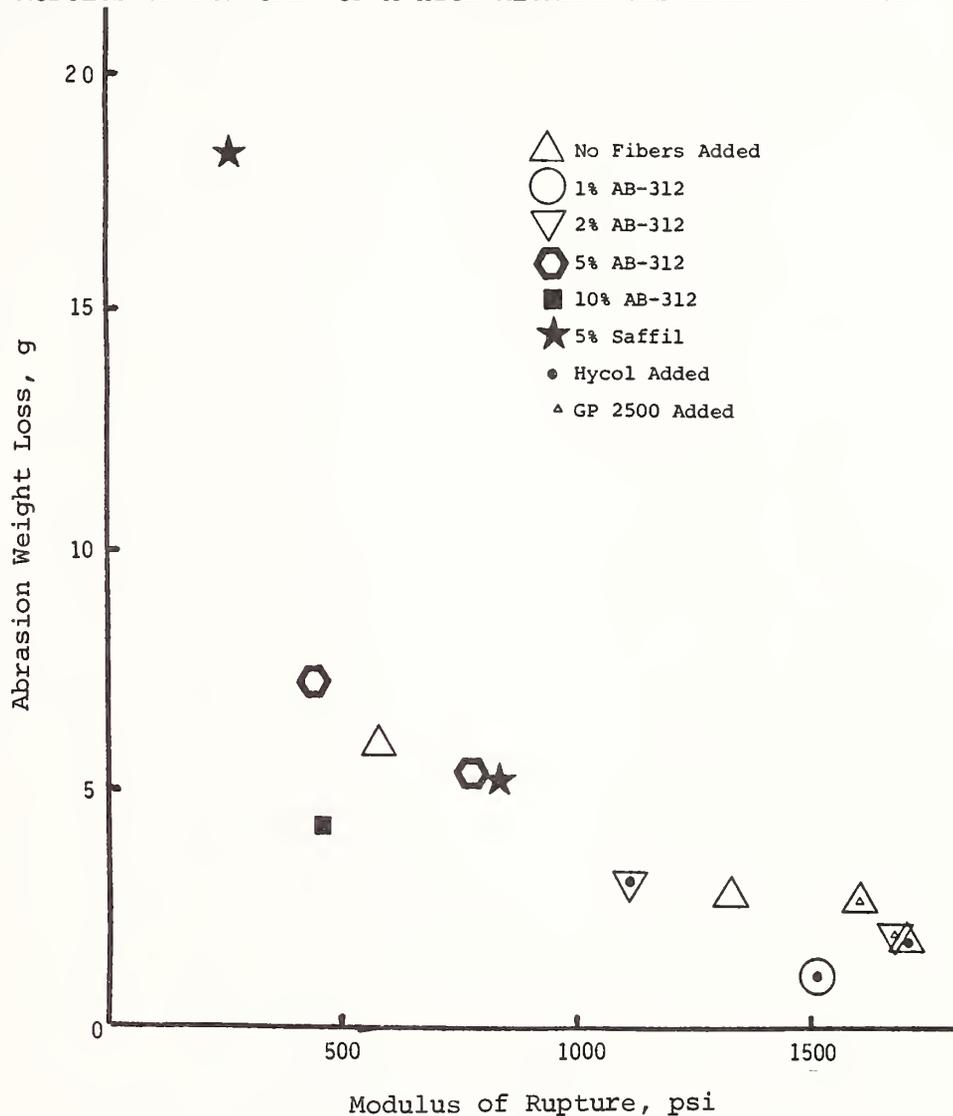
[a]Fibers used were AB-312 from 3M Company, high-alumina Al_2O_3-B_2O_3-SiO_2, 11 μm diameter, and Saffil from Imperial Chemical Industries, high-temperature 98% Al_2O_3-2% SiO_2, 3 μm diameter. Fibers were chopped in high-speed Waring blender, length after chopping varied 20-150 μm; castable, fibers, and water mixed in blender before casting; cast samples were moist cured for 24 h, oven dried for 24 h. Wetting agents were used to reduce the amount of water necessary, 6 ounces added per 100 pounds of cement: Hycol from W.R. Grace Company and GP 2500 from Cresset Chemical Company.

[b]Sample bars 1 in x 1 in x 3 1/2 in were abraded with 1000g of 36 grit SiC according to ASTM C-704; above values are the average of 3 separate samples of each preparation.

[c]Modulus of Rupture measured using bars 1 in x 1 in x 7 in, test method and load not specified; each point is the average of 2 specimens.

[d]Castable used is Harbison-Walker's Castolast G, a high-purity tabular alumina, calcium aluminate bonded.

EFFECT OF TEMPERATURE CYCLING ON LENGTH OF ALUMINA REFRACTORY SAMPLES[2]

Composition	Brand Name[a]	Fired[c]	Average Percent Change in Length[b] at Ambient Temperature after Cycling to 2000 °F				
			Cycle 1	Cycle 2	Cycle 3	Cycle 4	Cycle 5
96% Al_2O_3	Plicast 40	+0.25	-0.01	-0.01	-0.01	-0.01	-0.01
94.6% Al_2O_3-0.5% SiO_2	Alfrax B 57	-0.04					
94.5% Al_2O_3-0.15% SiO_2	Greencast-94	+0.11					+0.07
93.7% Al_2O_3-0.1% SiO_2	Castolast G	+0.57	+0.01	0	+0.02	-0.01	-0.01
93.7% Al_2O_3	Taycor 414-FH	-1.5	0	0	0	0	0
93% Al_2O_3	B&W Kao Phos 30	-0.63					
92.6% Al_2O_3-6.8% CaO-0.4% SiO_2	H-W Lightweight Castable 33	-0.23					
89.6% Al_2O_3-0.7% SiO_2	Resco Cast AA-22	-0.52	-0.02	-0.07	-0.07	-0.07	-0.07
over 87% Al_2O_3	Alfrax 66	-0.02					

a See Section B.3.2.10 for manufacturers.

b ASTM C269-70 procedure followed for determining permanent linear change. Length after each cycle compared to length of fired sample.

c Length of fired sample compared to length of unfired sample.

EFFECT OF THERMAL SHOCK[a] ON CONDITION OF ALUMINA REFRACTORY SAMPLES[2]

Composition	Brand Name[b]	Sample Condition		
		1700-1800 °F	1800-2000 °F	2000 °F
95% Al_2O_3	Plicast 40	No cracking or pitting	No test	Deep cracks after cycle 1, small cracks continued after cycle 3, no further changes
93.7% Al_2O_3-0.1% SiO_2	Castolast G	No cracking or pitting	No test	Small surface cracks after cycle 1, 0.5 in. deep cracks after cycle 2, no further changes
93.7% Al_2O_3	Taycor 414-FH	No cracking or pitting	No test	Slight cracks on surface after cycle 1, few more minor cracks after cycle 2, no further changes
93% Al_2O_3	B&W Kao Phos 30	No test	Cracked after cycle 3, cracked through after cycle 5	No test
89.6% Al_2O_3-0.7% SiO_2	Resco Cast AA-22	No test	Cracked deeply after cycle 5	No test
over 87% Al_2O_3	Alfrax 66	No cracking or pitting	No test	Cracked through in 2 places after cycle 1, small cracks across width after cycle 3, no further changes

[a] Thermal shock test consisted of heating rapidly to the indicated temperature, soaking for 2-5 hours, and rapidly cooling to ambient temperature. Each sample was subjected to 5 such cycles.
[b] See Section B.3.2.10 for manufacturers.

EFFECT OF TEMPERATURE ON THE FLEXURAL STRENGTH[a] OF SEVERAL ALUMINA REFRACTORIES[b][16]
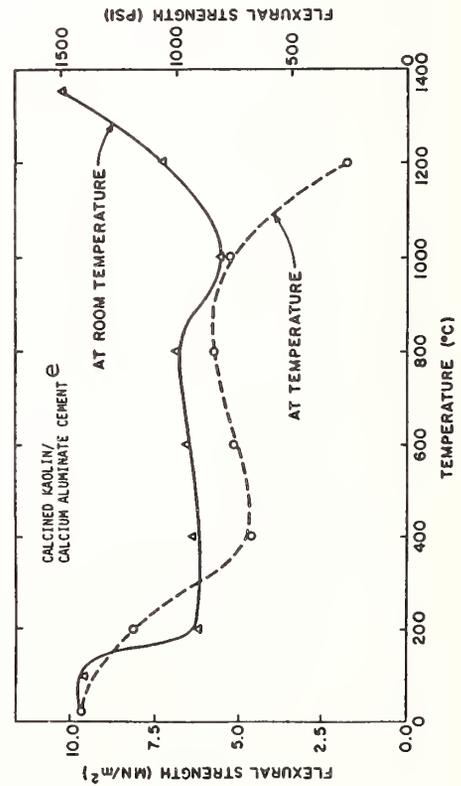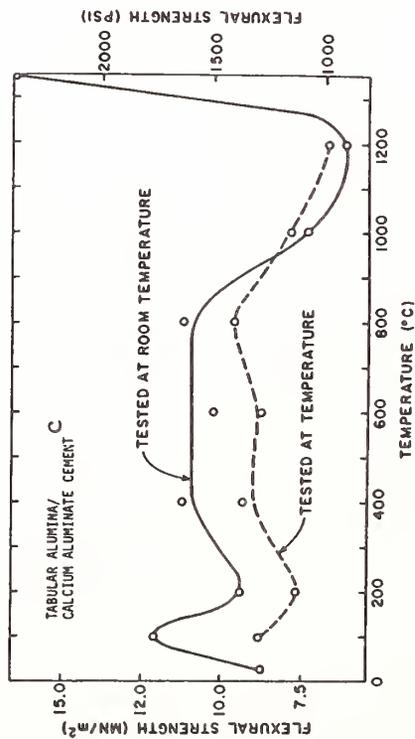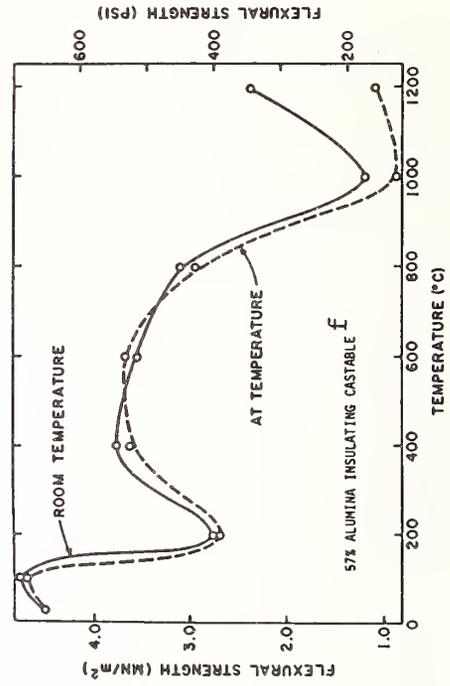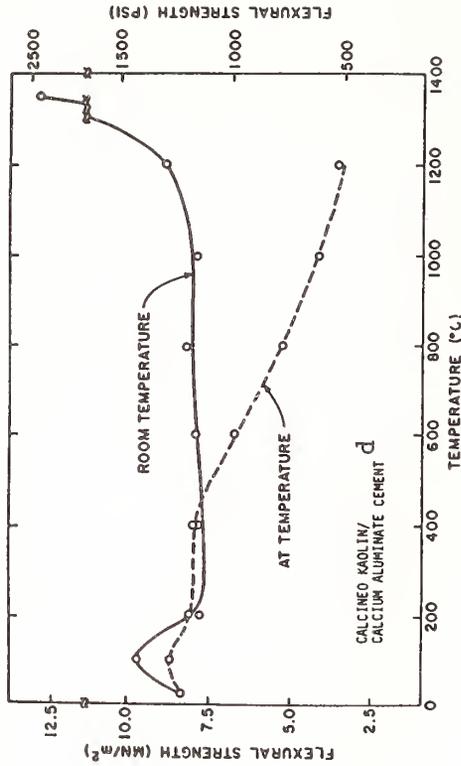
(Data Continued)

EFFECT OF TEMPERATURE ON THE FLEXURAL STRENGTH[a] OF SEVERAL ALUMINA REFRACTORIES[b][16], Continued
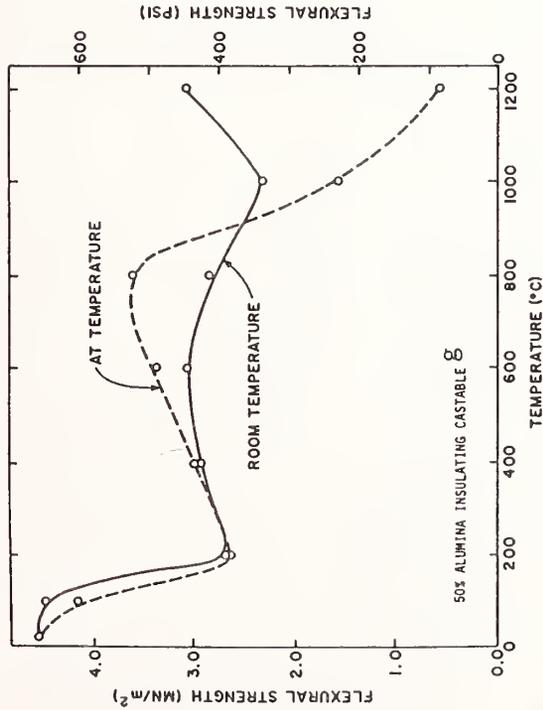
[a]Flexural strength was measured in three-point bending, crosshead speed 0.05 in./min., span 6 in. One set of samples was tested at ambient temperature (solid line), the second set was heated in a globar furnace and tested at firing temperatures when the samples reached thermal equilibrium (dashed line). Samples were 1 in. x 1 in. x 7 in.

[b]Samples were cast, vibrated one minute, cured sealed in plastic 24 hours at ambient temperature, cured in air 24 hours at ambient temperature, dried in air 24 hours at 100 °C, fired at 60 °C/h to 200, 400, 600, 800, 1000, 1200, and 1350 °C, and soaked at temperature 5 hours, and furnace cooled to ambient temperature.

[c]75% T-61 alumina (Alcoa) aggregate, 25% Casting Grade CA-25 cement (Alcoa), water to ball-in-hand consistency 10.4% of aggregate and cement.

[d]75% Mulcoa M-47 (C-E Minerals) aggregate, 25% Casting Grade CA-25 (Alcoa) cement, water to ball-in-hand consistency 11.3% of aggregate and cement.

[e]75% Mulcoa M-47 (C-E Minerals) aggregate, 25% Refcon cement (Universal Atlas), water to ball-in-hand consistency 13.5% of aggregate and cement. [See B.3.2.84 for slightly different tabulated values.]

[f]A commercial lightweight insulating castable (Cer-Lite #75, C-E Refractories), water to ball-in-hand consistency 22% of mix.

[g]A commercial lightweight insulating castable (Litecast 75-28, General Refractories), water to ball-in-hand consistency 24% of mix.

EFFECT OF TEMPERATURE ON THE YOUNG'S MODULUS[a] OF SEVERAL ALUMINA REFRACTORIES[b][16]
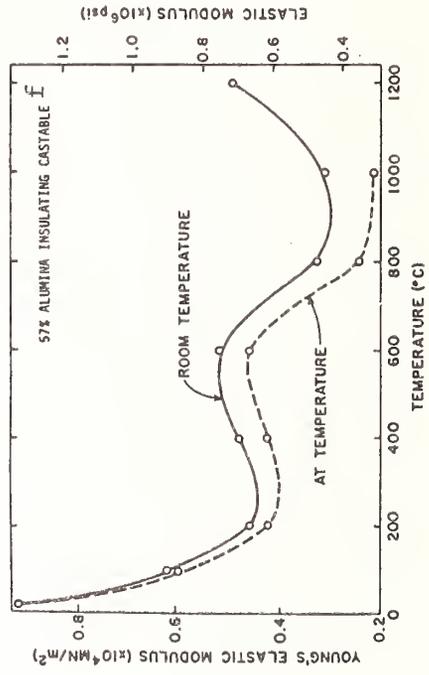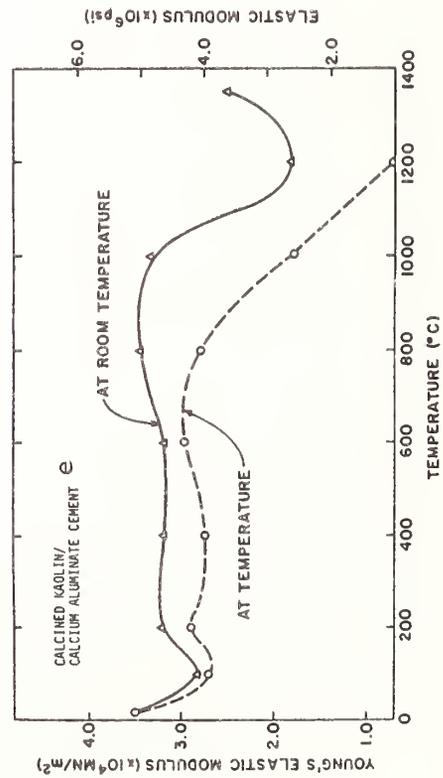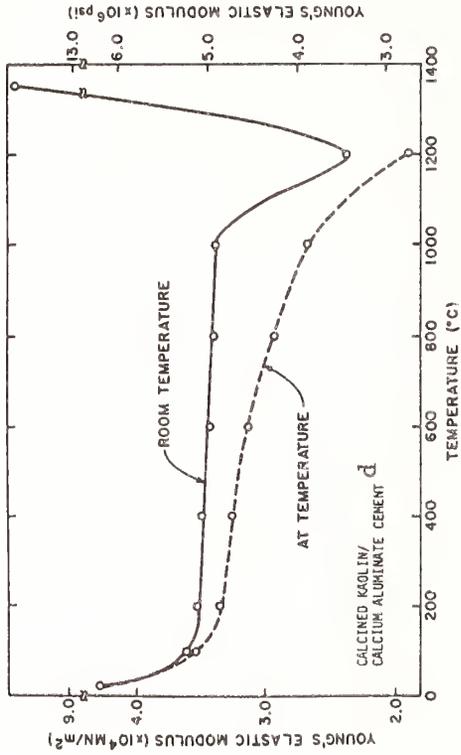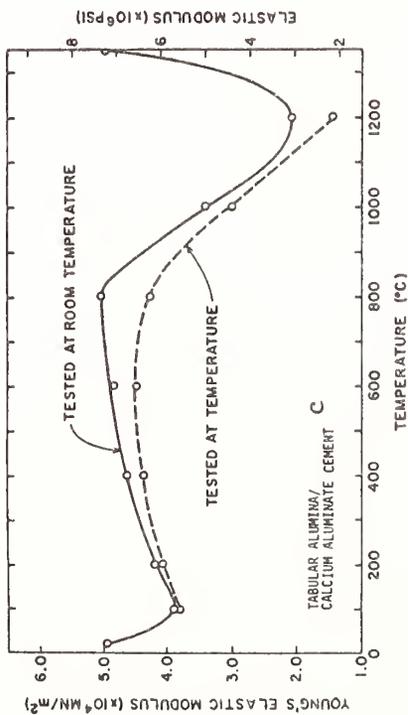
(Data Continued)

EFFECT OF TEMPERATURE ON THE YOUNG'S MODULUS[a] OF SEVERAL ALUMINA REFRACTORIES[b][16], Continued

[a]Young's modulus measured by dynamic mechanical resonance method. Specimens 0.65 cm x 2.54 cm x 17.78 cm were saw cut from larger cast specimens. Two specimens of each refractory at each curing and firing temperature were measured. Ambient temperature and elevated temperature data were obtained on the same specimens. Flexural and torsional resonance frequencies were obtained using an acoustic spectrometer and the modulus then calculated. See Section B.3.2.81 for tabulated data and Section B.3.2.83 for other plotted data.

[b]Samples were cast, vibrated one minute, cured sealed in plastic 24 hours at ambient temperature, cured in air 24 hours at ambient temperature, dried in air 24 hours at 100 °C, fired at 60 °C/h to 200, 400, 600, 800, 1000, 1200, and 1350 °C, soaked at temperature 5 hours, and furnace cooled to ambient temperature.

[c]75% T-61 alumina (Alcoa) aggregate, 25% Casting Grade CA-25 cement (Alcoa), water to ball-in-hand consistency 10.4% of aggregate and cement.

[d]75% Mulcoa M-47 (C-E Minerals) aggregate, 25% Casting Grade CA-25 (Alcoa) cement, water to ball-in-hand consistency 11.3% of aggregate and cement.

[e]75% Mulcoa M-47 (C-E Minerals) aggregate, 25% Refcon cement (Universal Atlas), water to ball-in-hand consistency 13.5% of aggregate and cement.

[f]A commercial lightweight insulating castable (Cer-Lite #75, C-E Refractories), water to ball-in-hand consistency 22% of mix.

[g]A commercial lightweight insulating castable (Litecast 75-28, General Refractories), water to ball-in-hand consistency 24% of mix. [Tabular values for Young's Modulus given in the reports do not agree with these plotted here. See Section B.3.2.81.]

EFFECT OF TEMPERATURE ON THE FRACTURE TOUGHNESS[a] OF SEVERAL ALUMINA REFRACTORIES[b][16]

(Data Continued)

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EFFECT OF TEMPERATURE ON THE FRACTURE TOUGHNESS[a] OF SEVERAL ALUMINA REFRACTORIES[b][16], Continued
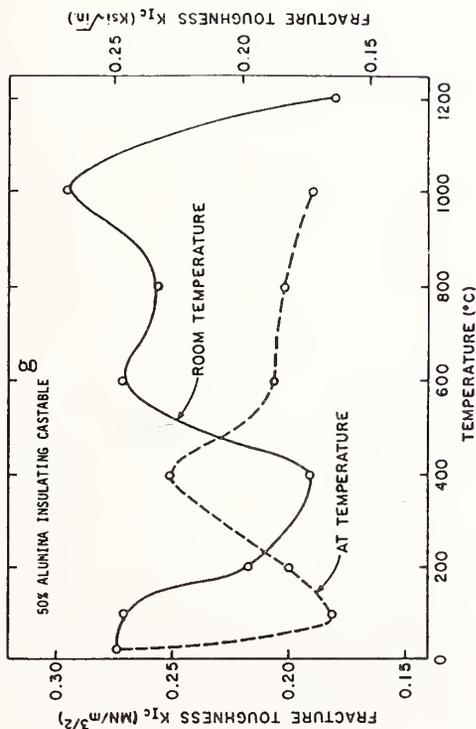
[a] Fracture toughness was measured by single edge notched beam method; notch was saw cut to a depth of 1/2 the specimen thickness, average notch width was about 0.032 in. Specimens were tested in three-point bending, crosshead speed 0.05 in./min. One set of samples was tested at ambient temperature (solid line), a second set was heated in a globar furnace and tested at the firing temperatures when the samples reached thermal equilibrium (dashed line). Samples were 1 in. x 1 in. x 7 in.

[b] Samples were cast, vibrated one minute, cured sealed in plastic 24 hours at ambient temperature, cured in air 24 hours at ambient temperature, dried in air 24 hours at 100 °C, fired at 60 °C/h to 200, 300, 400, 600, 800, 1000, 1200, and 1350 °C, soaked at temperature 5 hours, and furnace cooled to ambient temperature.

[c] 75% T-61 alumina (Alcoa) aggregate, 25% Casting Grade CA-25 cement (Alcoa), water to ball-in-hand consistency 1.4% of aggregate and cement.

[d] 75% Mulcoa M-47 (C-E Minerals) aggregate, 25% Casting Grade CA-25 (Alcoa) cement, water to ball-in-hand consistency 11.3% of aggregate and mix. Room temperature curve agrees with early curves given in the set of reports. Final report contains a room temperature curve corresponding to the dotted line alteration. [See Section B.3.2.82 for tabulated data.]

[e] 75% Mulcoa M-47 (C-E Minerals) aggregate, 25% Refcon cement (Universal Atlas), water to ball-in-hand consistency 13.5% of aggregate and mix. [Values given in tables in reports for the high temperature measurements show the same trend but are slightly higher. See Section B.3.2.82.]

[f] A commercial lightweight insulating castable (Cer-Lite #75, C-E Refractories), water to ball-in-hand consistency 22% of mix. [Values given in tables differ. $MN/m^{3/2}$ values seem to have been plotted on the Ksi√in. scale.]

[g] A commercial lightweight insulating castable (Litecast 75-28, General Refractories), water to ball-in-hand consistency 24% of mix. [Different reports of the series give a second set of curves for this material. This set of curves is more consistent with others in the reports. The high temperature curve agrees with tabulated data. See Section B.3.2.82.]

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EFFECT OF TEMPERATURE ON THE WORK OF FRACTURE[a] OF SEVERAL ALUMINA REFRACTORIES[b][16]
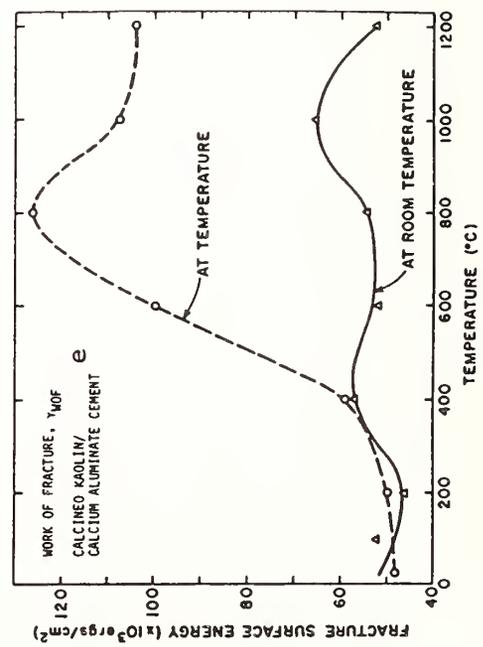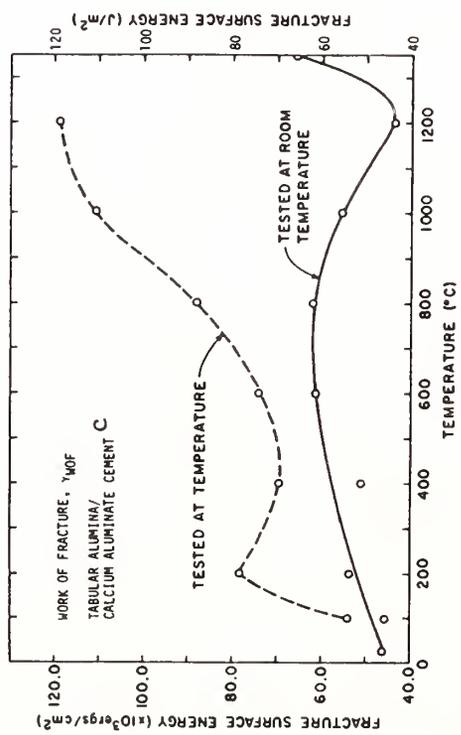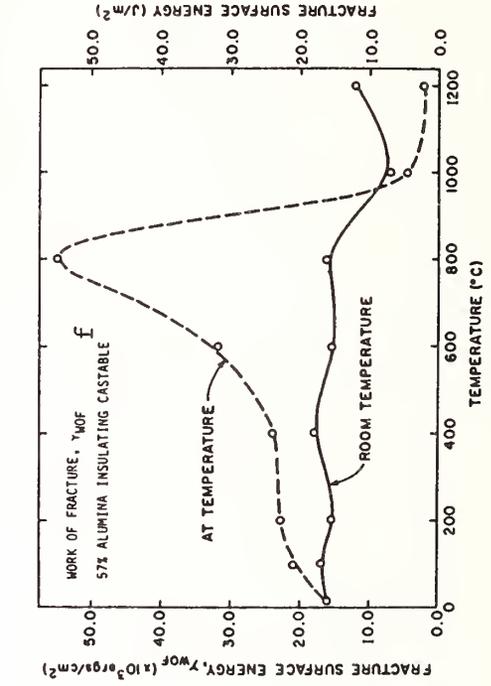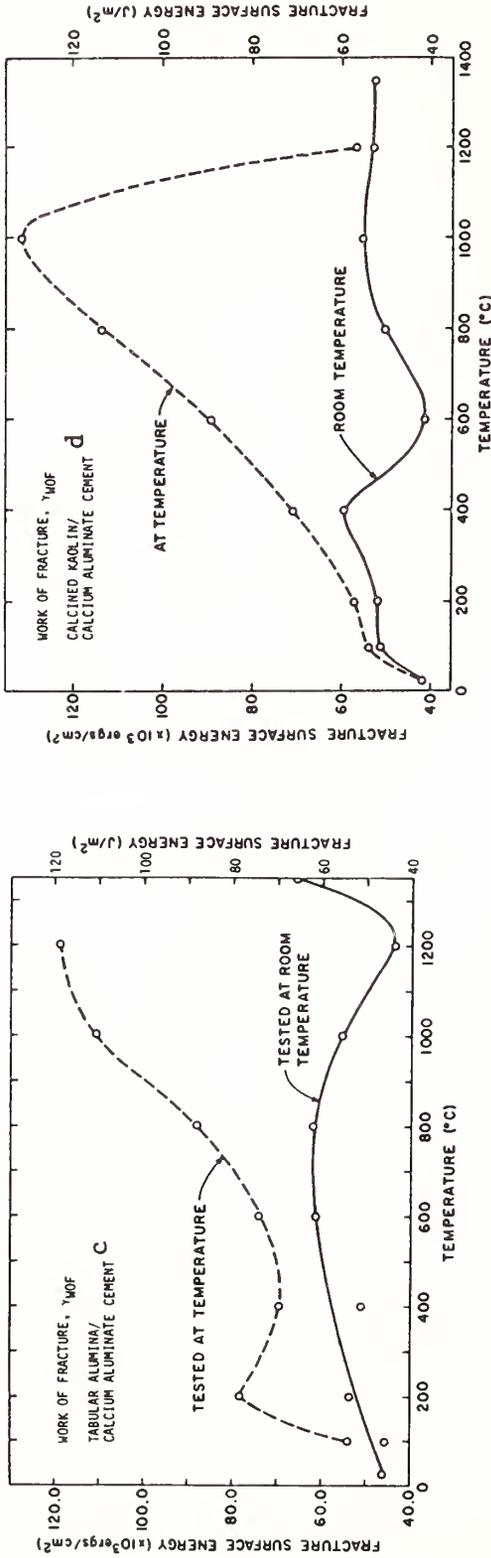
(Data Continued)

EFFECT OF TEMPERATURE ON THE WORK OF FRACTURE[a] OF SEVERAL ALUMINA REFRACTORIES[b][16], Continued

[a]Fracture was measured by the single edge notched beam method; notch was saw cut so that one fourth of the cross sectional area remained, the average notch width was about 0.32 in. Specimens were tested in three-point bending, crosshead speed 0.05 in./min. One set of samples was tested at ambient temperature (solid line), the second set was heated in a globar furnace and tested at the firing temperatures when the samples reached thermal equilibrium (dashed line). Samples were 1 in. x 1 in. x 7 in. γ_{WOF} was measured as the area under the load-displacement curve divided by twice the fracture surface area.

[b]Samples were cast, vibrated one minute, cured sealed in plastic 24 hours at ambient temperature, cured in air 24 hours at ambient temperature, dried in air 24 hours at 100 °C, fired at 60 °C/h to 200, 400, 600, 800, 1000, 1200, and 1350 °C, soaked at temperature 5 hours, and furnace cooled to ambient temperature.

[c]75% T-61 alumina (Alcoa) aggregate, 25% Casting Grade CA-25 cement (Alcoa), water to ball-in-hand consistency 10.4% of aggregate and cement.

[d]75% Mulcoa M-47 (C-E Minerals) aggregate, 25% Casting Grade CA-25 (Alcoa) cement, water to ball-in-hand consistency 11.3% of aggregate and cement.

[e]75% Mulcoa M-47 (C-E Minerals) aggregate, 25% Refcon cement (Universal Atlas), water to ball-in-hand consistency 13.5% of aggregate and cement.

[f]A commercial lightweight insulating castable (Cer-Lite #75, C-E Refractories), water to ball-in-hand consistency 22% of mix.

[g]A commercial lightweight insulating castable (Litecast 75-28, General Refractories), water to ball-in-hand consistency 24% of mix.

EFFECT OF TEMPERATURE ON NOTCH BEAM TEST RESULTS[a] OF SEVERAL ALUMINA REFRACTORIES[b][16]

(Data Continued)

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EFFECT OF TEMPERATURE ON NOTCH BEAM TEST RESULTS[a] OF SEVERAL ALUMINA REFRACTORIES[b][16], Continued

[a] Fracture toughness was measured by single edge notched beam method; notch was saw cut to depth of 1/2 specimen thickness, average notch width about 0.032 in. Specimens were tested in three-point bending, crosshead speed 0.05 in./min. Samples were 1 in. x 1 in. x 7 in. One set of samples was tested at ambient temperature (solid line), second set was heated in a globar furnace and tested at firing temperatures when the samples reached thermal equilibrium (dashed line). γ_{NBT}, a measure of the energy for crack initiation, was then calculated from an equation relating fracture toughness and Young's modulus.

[b] Samples were cast, vibrated one minute, cured sealed in plastic 24 hours at ambient temperature, cured in air 24 hours at ambient temperature, dried in air 24 hours at 100 °C, fired at 60 °C/h to 200, 300, 400, 600, 800, 1000, 1200, and 1350 °C, soaked at temperature 5 hours, and furnace cooled to ambient temperature.

[c] 75% T-61 alumina (Alcoa) aggregate, 25% Casting Grade CA-25 cement (Alcoa), water to ball-in-hand consistency 10.4% of aggregate and cement.

[d] 75% Mulcoa M-47 (C-E Minerals) aggregate, 25% Casting Grade CA-25 (Alcoa) cement, water to ball-in-hand consistency 11.3% of aggregate and cement. [Solid room temperature curve agrees with tabulated strain energy release rate values given and with tabulated strain energy release rate values given. Final report contains a curve corresponding to the dotted line alteration. See Section B.3.2.82 for tabulated data.]

[e] 75% Mulcoa M-47 (C-E Minerals) aggregate, 25% Refcon cement (Universal Atlas), water to ball-in-hand consistency 13.5% of aggregate and cement. [Tabulated high-temperature values, see B.3.2.82, differ.]

[f] A commercial lightweight insulating castable (Cer-Lite #75, C-E Refractories), water to ball-in-hand consistency 22% of mix.

[g] A commercial lightweight insulating castable (Litecast 75-28, General Refractories), water to ball-in-hand consistency 24% of mix. [Two sets of curves are given for this material in the reports. The set of curves labelled I corresponds to data in early reports, the one labelled II corresponds to data tabulated in the final report. See Section B.3.2.82 for tabulated data.]

NOTCH BEAM TEST, γ_{NBT}
50% ALUMINA INSULATING CASTABLE[g]
I
ROOM TEMPERATURE
AT TEMPERATURE
TEMPERATURE (°C)
FRACTURE SURFACE ENERGY, γ_{NBT} ($\times 10^3$ ergs/cm^2)
FRACTURE SURFACE ENERGY (J/m^2)

NOTCH BEAM TEST, γ_{NBT}
50% ALUMINA INSULATING CASTABLE[g]
II
ROOM TEMPERATURE
AT TEMPERATURE
TEMPERATURE (°C)
FRACTURE SURFACE ENERGY, γ_{NBT} ($\times 10^3$ ergs/cm^2)
FRACTURE SURFACE ENERGY (J/m^2)

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EFFECT OF TEMPERATURE ON THE COMPRESSIVE STRENGTH[a] OF PHOSPHATE-BONDED REFRACTORIES[26]

	45% Al_2O_3[b]	90% Al_2O_3[c]	90-95% Al_2O_3[d]
Bulk Density, lb/ft³			
Cured at ambient temp.	150	194	182
Dried 24 h at 450 °F	142.5	185	172
Linear Shrinkage, %			
Cured length relative to dried length	0.3	0.2	0.2

Hot Compressive Strength, Psi (MPa)

°F	°C	45% Al_2O_3[b]	90% Al_2O_3[c]	90-95% Al_2O_3[d]
72	22	2480± 345 (17.0)	2680±385 (18.4)	3490±1050 (24.0)
500	260	3480± 715 (24.0)	4830±545 (33.2)	3740±1825 (25.8)
1000	538	5205± 480 (35.9)	6670±800 (46.0)	6770±2330 (46.6)
1500	816	6080±1100 (41.9)	8000±630 (55.1)	6840±2030 (47.1)
1750	954	8080± 455 (55.6)	6370±710 (43.9)	4160± 980 (28.6)
2000	1098	3640± 640 (25.0)	1530±540 (10.5)	1260± 405 (8.7)

[a]Uniaxial compressive strength was determined using specimens 0.5 in. x 1 in. x 1 in. or 1 in. x 1 in. x 1 in. loaded uniaxially at a 0.020 in./min. strain rate. Values are the average for five samples.

[b]Generic preparation of phosphate-bonded ramming mix: 15% calcined alumina, 65% calcined kaolin, 17% ball mill fines, 3% Bentonite (Wyoming), 1% hydrated alumina; bonded with 6% phosphoric acid (85% strength).

[c]Generic preparation of phosphate-bonded ramming mix: 82% tabular alumina, 15% calcined alumina, 3% Bentonite (Wyoming), 1% hydrated alumina; bonded with 6% phosphoric acid (85% strength).

[d]Commercial phosphate-bonded ramming mix: 90 Ram H.S. from C-E Refractories.

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CREEP AND HOT LOAD DEFORMATION FOR VARIOUS ALUMINA REFRACTORIES[26]

Hot Load Deformation,[a] %

Test Conditions[l] psi	°F	Commercial 50% Al$_2$O$_3$[b]			Generic 50% Al$_2$O$_3$[c]	Modified 50% Al$_2$O$_3$[d]	Commercial 50% Al$_2$O$_3$ +446 SS[e]		Commercial 42% Al$_2$O$_3$[f]	Commercial 40-60% Al$_2$O$_3$[g]	Modified 90+% Al$_2$O$_3$[h]			Generic 45% Al$_2$O$_3$[i]	Generic 90% Al$_2$O$_3$[j]	Commercial 90% Al$_2$O$_3$[k]
% Casting Water		21	24	21	10	10	7.5[e] (2%)[e]	50% SS[e] (4%)[e]	59	11	7.75	8.5	9.0			
100	2000	3.9	--	--	0.3	0.3(0.4)	--	--	7.8	0.5(1.3)	0.0	0.0	0.0	0.6(0.3)	0.4	1.2
200	2000	failed[m]	--	--	--	0.4(0.5)	0.6	0.5	failed	0.8	0.1	0.1	0.1	0.8(0.8)	0.8	--
1000	2000	--	--	--	failed(1 h)[n]	1.7(1.0)	1.6	1.8	--	--	1.4	--	0.6	--	failed(14m)[n]	failed(12m)[n]
1500	2000	--	--	--	--	--	--	--	--	--	--	2.4	3.7	--	--	--
2000	2000	--	--	--	--	--	2.7 failed[o]	failed[p]	--	--	2.3	--	--	--	--	--
2500	2000	--	--	--	--	--	3.4	--	--	--	3.8	5.0	5.6[q]	--	--	--
3300	2000	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
100	1800	1.5	1.9	1.4	0.2	0.2	--	--	--	--	--	--	--	--	--	--
200	1800	3.3(2.8)	5.8	--	0.3	0.3	--	--	--	--	--	--	--	0.1	0.1	--
1000	1800	--	--	--	--	--	--	--	--	--	--	--	--	0.2	0.5	--
1500	1800	4.0	--	--	1.0	1.4	--	--	--	1.6	--	--	--	0.4	--	--
2000	1800	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
2500	1800	--	--	--	1.7	failed(1¼h)[n]	--	--	--	--	--	--	--	--	--	--
100	1500	0.3	0.41	0.42	0.1	--	--	--	--	0.2	--	--	--	--	--	0.08
700	1500	1.7	2.9(8.7)	--	--	--	--	--	--	0.5	--	--	--	--	--	--
1000	1500	3.1	--	--	--	--	--	--	--	--	--	--	--	--	--	--
1500	1500	5.0(3.7)	5.4[r]	--	--	--	--	--	--	1.6	--	--	--	--	--	--
100	1000	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
500	1000	--	0.25	0.22	--	--	--	--	--	--	--	--	--	--	--	--
700	1000	--	--	0.67	--	--	--	--	--	--	--	--	--	--	--	--
1500	1000	0.36	0.40	0.57	--	--	--	--	--	0.13	--	--	--	--	--	--

[a] Tests were performed during creep testing. Specimens were loaded at ambient temperature, heated to the top test temperature and cooled to ambient. The change in length was calculated as the percent deformation. Values in parentheses represent data obtained on different batches of the same material. Not all of the data in a column are necessarily obtained on the same batch of material.

[b] Commercial lightweight insulating castable, calcium aluminate bonded (Litecast 75-28, General Refractories).

[c] Generic preparation: 75% calcined kaolin (60% to -20 mesh, 15% ball milled fines with 50% less than 325 mesh), 25% calcium aluminate cement (Casting Grade CA-25, Alcoa).

[d] Generic preparation: 75% calcined kaolin (70% 6 to -20 mesh, 5% ball milled fines with 50% less than 325 mesh), 25% calcium aluminate cement (Casting Grade CA-25, Alcoa).

[e] Coarse grain size, high-strength 50% alumina prototype castable (B&W Kaocrete XD50, Mix 36C, Babcock & Wilcox); 75% water added to all batches, with and without 1-in. long fibers of 446 SS (Ribtec 446) added. The 7.5% column refers to batches with no fibers added, the 2% and 4% columns refer to the weight percent of 446 SS fibers added to those batches.

[f] Commercial lightweight insulating castable, 42% alumina (B&W Kaolite 2300 LI, Babcock & Wilcox).

[g] Commercial dense castable, calcium aluminate bonded (Lo-Abrade, A. P. Green).

[h] Modified DOE 90 preparation: 70% tabular alumina (-48 mesh), 5% calcined alumina (-325 mesh), 25% calcium aluminate cement (Casting Grade CA-25, Alcoa).

[i] Generic preparation of phosphate-bonded ramming mix: 15% calcined alumina, 65% calcined kaolin, 17% ball mill fines, 3% Bentonite (Wyoming), 1% hydrated alumina; bonded with 6% phosphoric acid (85% strength).

[j] Generic preparation of phosphate-bonded ramming mix: 82% tabular alumina, 15% calcined alumina, 3% Bentonite (Wyoming), 1% hydrated alumina; bonded with 6% phosphoric acid (85% strength).

[k] Commercial phosphate-bonded ramming mix: 90 Ram H.S. from C-E Refractories.

[l] Duration of tests either 10 or 11 hours; in ten hour tests, samples at temperature 11 hours, but tested for only 10 hours.

[m] Samples from three batches failed, one at 1900 °F, a second after 3 hours, a third after 5 hours.

[n] Time at which specimens failed given in hours (h) or minutes (m).

[o] Specimen failed in 55 minutes.

[p] Specimen failed immediately.

[q] Sample began to fail after 3 hours, test was stopped.

[r] Sample began to fail after 7 hours, test was stopped.

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EFFECT OF TEMPERATURE ON THE RESULTS OF RADIAL COMPRESSION TESTS[a]
OF SOME SILICON CARBIDE TUBES[15]

MATERIAL Temperature °C(°F)	Mean Strength ksi	Characteristic[b] Stress (ksi)	Weibull Slope[c]	Number Tested	Failure Rate % 1.0	0.5 Failure Stress	0.1 (ksi)
SINTERED SILICON CARBIDE[d]							
ambient	26.4	29.0	9.30	30	17.68	16.4	13.79
816(1500)	22.7	25.0	6.51	8	12.33	11.08	8.65
926(1700)	31.4	33.0	8.88	4	19.65	18.17	15.16
1037(1900)	26.6	28.0	8.92	4	16.71	15.46	12.91
1260(2300)	26.3	28.0	8.30	4	16.08	14.79	12.18
1371(2500)	16.4	17.8	9.14	4	10.76	9.97	8.36
REACTION-BONDED SILICON CARBIDE[e]							
ambient	35.775	42.0	5.80	30	19.00	16.85	12.76
816(1500)	39.045	41.0	8.65	8	24.10	22.22	18.45
926(1700)	33.043	33.0	8.20	4	18.83	17.29	14.21
1037(1900)	42.099	44.0	11.92	4	29.91	28.21	24.65
1260(2300)	36.301	37.0	11.94	4	25.16	23.74	20.75
1371(2500)	15.445	15.0	7.23	4	7.93	7.21	5.77
SINTERED SILICON CARBIDE[f]							
ambient	29.014	30.5	5.97	32	14.1	12.6	9.6
816(1500)	22.263	23	27.8	4	19.5	19.0	17.9
926(1700)	28.641	31	6.08	5	14.5	13.0	10.0
1037(1900)	28.782	32	5.66	5	14.2	12.6	9.4
1260(2300)	37.285	41	5.67	5	18.2	16.1	12.1

[a] Extruded tubes, 1.905 cm (0.75 in.) outer diameter, 1.27 cm (0.5 in.) inner diameter, and 1.27 cm (0.5 in.) long, were loaded between two flat plates in the radial direction. Tests at ambient temperature were tested using compliant layers (4 sheets of 0.0127 cm thick lead) to prevent point loading. High-temperature tests were made with induction heating via a SiC susceptor; radial compression was applied with the sample between alumina blocks.

[b] Characteristic stress is the stress corresponding to 62.3% cumulative failures in a plot of number of failures versus stress.

[c] Weibull slope or modulus is the slope of a failure distribution curve, i.e., number of failures versus stress.

[d] NC-430 from Norton.

[e] Super-KT SiC from Carborundum.

[f] Sintered alpha SiC from Carborundum; testing stopped because specimens had irregularities, internal circumferential cracks or voids and some microscopic radial cracks.

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FAILURE STRESS[a] AS A FUNCTION OF TEMPERATURE FOR SILICON CARBIDE REFRACTORIES[15]
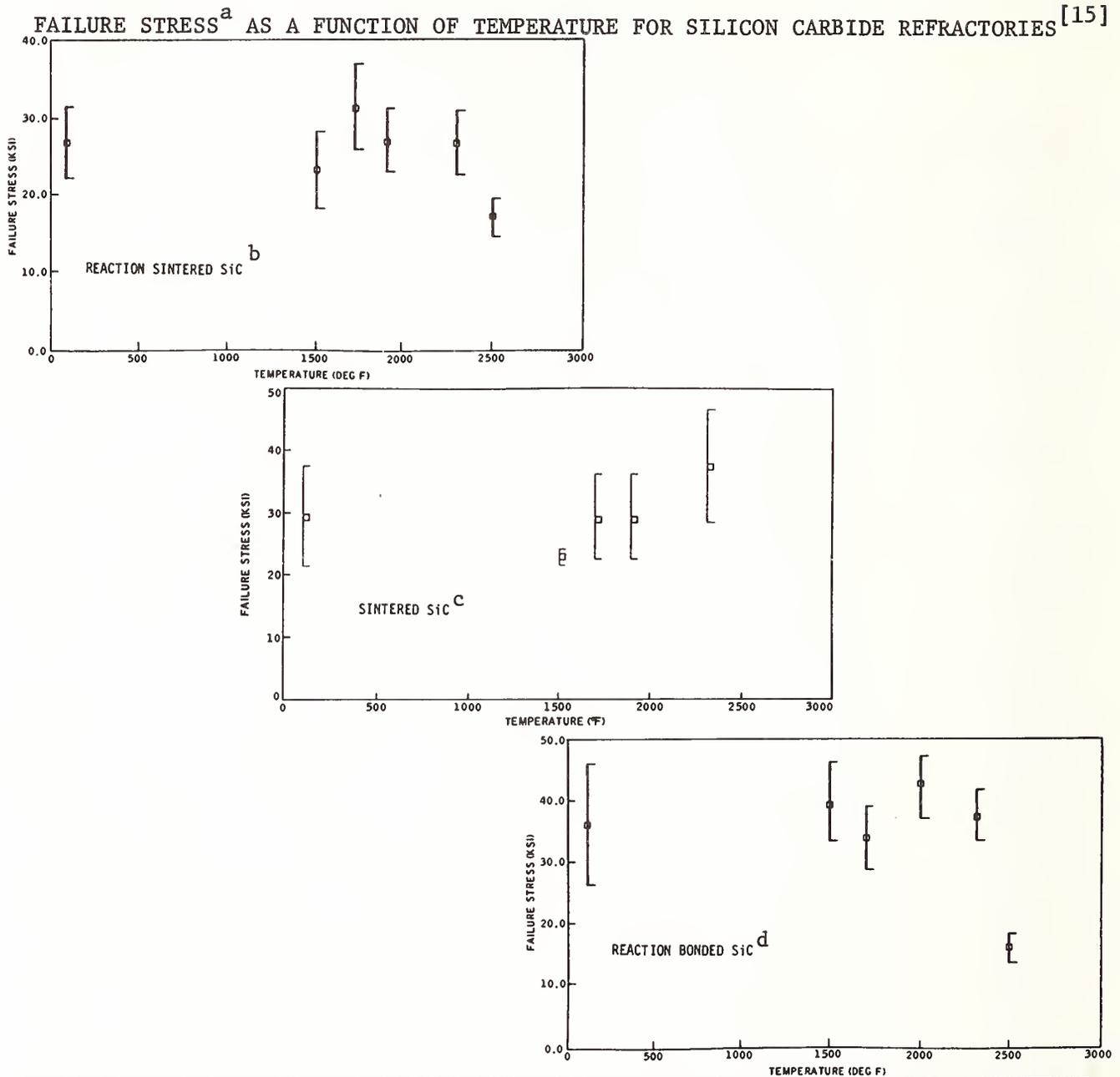
[a]Stress measured by loading extruded tubes between two flat plates in the radial direction. Tube samples were 1.905 cm (0.75 in.) outer diameter, 1.27 cm (0.5 in.) inner diameter, and 1.27 cm (0.5 in.) long. Tests at ambient temperature were tested using compliant layers (4 sheets of 0.0127 cm thick lead) to prevent point loading. High-temperature tests were made with induction heating via a SiC susceptor; radial compression was applied with the sample between alumina blocks.

[b]NC-430 from Norton.

[c]Sintered alpha SiC from Carborundum; testing stopped because specimens had irregularities, internal circumferential cracks or voids and some microscopic cracks.

[d]Super-KT SiC from Carborundum.

EFFECT OF TEMPERATURE ON THE MODULUS OF ELASTICITY[a] OF SOME
CASTABLE REFRACTORIES[26]

Temperature		Hot Modulus of Elasticity,[a] 10^6 psi (MPa)			
°F	°C	Generic 50% Al_2O_3[b]	Commercial 50% Al_2O_3[c]	Commercial 50% Al_2O_3[d]	50% Al_2O_3[d] + 310 SS[e]
72	22	0.9±0.5 (6200)	0.6±0.3 (4130)	0.2	0.13
500	260	0.6±0.3 (4130)	0.3±0.1 (2065)	0.2	0.14
1000	538	1.0±0.4 (6890)	0.4±0.2 (2755)	0.2	0.15
1250	677	--	0.4±0.2 (2755)	--	--
1500	816	0.6±0.2 (4130)	0.2±0.1 (1380)	0.2	0.14
1750	954	0.6±0.3 (4130)	--	0.2	0.12
2000	1098	0.2±0.1 (1380)	--	0.1	0.10

Temperature		Commercial 42% Al_2O_3[f]	Generic 90+% Al_2O_3[g]	Generic 90+% Al_2O_3[h]	Modified 90+% Al_2O_3[i]
°F	°C				
72	22	0.02	1.7±0.8 (11710)	1.19	1.5±0.5
500	260	--	0.7±0.3 (4820)	0.77	0.8±0.3 (5510)
1000	538	0.02	0.5±0.2 (3445)	0.62	0.8±0.3 (5510)
1500	816	0.03	0.4±0.1 (2755)	0.58	0.7±0.3 (4820)
1750	954	--	0.3±0.1 (2065)	0.60	0.4±0.1 (2755)
2000	1098	--	0.2±0.1 (1380)	0.56	0.4±0.2 (2755)

[a] Young's modulus was determined from uniaxial compressive strength test data by calculating the slope of the stress/strain curves in the most linear part of the curves. Specimens 0.5 in. x 1 in. x 1 in. or 1 in. x 1 in. x 1 in. were loaded uniaxially at a 0.020 in./min. strain rate. Values are the average for five samples.

[b] Generic preparation: 75% calcined kaolin (70% to -20 mesh, 5% ball milled fines with 50% less than 325 mesh), 25% calcium aluminate cement (Casting Grade CA-25, Alcoa); 11% water added.

[c] Commercial lightweight insulating castable, calcium aluminate bonded (Litecast 75-28, General Refractories); 21% water added.

[d] Coarse grain size, high-strength 50% alumina prototype castable (B&W Kaocrete XD50, Mix 36C, Babcock & Wilcox); 7.5% water added.

[e] 4 wt% of 310 SS fibers (Ribtec 310), 1 in. long, was added to the Kaocrete XD50.

[f] Commercial lightweight insulating castable, 42% alumina (B&W Kaolite 2300 LI, Babcock & Wilcox); 59% water added.

[g] DOE 90 generic preparation: 65% tabular alumina (6 to -20 mesh), 10% calcined alumina (-325 mesh), 25% calcium aluminate cement (Secar 250, Lone Star Lafarge); 10.5% water added.

[h] DOE 90 generic preparation: as in footnote g but with Casting Grade CA-25 (Alcoa) cement; 9.3% water added.

[i] Modified DOE 90 preparation: 70% tabular alumina (-48 mesh), 5% calcined alumina (-325 mesh), 25% calcium aluminate cement (Casting Grade CA-25); 8.5% water added.

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EFFECT OF TEMPERATURE ON THE COMPRESSIVE STRENGTH[a] OF SOME
CASTABLE REFRACTORIES[26]

Hot Compressive Strength,[a] psi (MPa)

Temperature °F	Generic 50% Al_2O_3[b]	Commercial 50% Al_2O_3[c]	Commercial 42% Al_2O_3[d]
At 72[e]	8020± 880(55.2)	3945±350(27.2)	430± 55
500	6705± 850(46.2)	3490±180(24.0)	505± 25
1000	8570± 910(59.0)	3940±340(27.1)	540± 25
1250	--	4245±740(29.2)	560± 30
1500	10130± 490(69.8)	5330±430(36.7)	820± 20[f]
1750	8690± 390(59.9)	--	--
2000	6300±1020(43.4)	--	--

Temperature °F	Commercial 50% Al_2O_3[g]	50% Al_2O_3[g] + 310 SS[h]	50% Al_2O_3[g] + 446 SS[i]	50% Al_2O_3[g] + 446 SS[j]
At 72[e]	4000	3220±410	2560± 635	3350± 600
500	4195	3480±850	3965±1055	4300± 460
1000	3370	3520±650	3820±1290	4660± 560
1500	5680	4725±370	6960±1250	6090± 670
1750	4550	4380±435[k]	6180± 950[k]	4080±2115[k]
2000	3865	3680±270	5630±1220	4460± 610

Temperature °F	Generic 90+% Al_2O_3[l]	Generic 90+% Al_2O_3[m]	Modified 90+% Al_2O_3[n]	Commercial 90+% Al_2O_3[o]
At 72[e]	11120±1030(76.6)	9815	9100±1750(62.7)	5940
500	6830± 605(47.0)	8750	9220± 580(63.5)	--
1000	7200± 570(49.6)	8650	9690±1040(66.8)	--
1500	8330± 580(57.4)	9740	9130±1480(62.9)	--
1750	5915± 115(40.8)	9680	7300±1160(50.3)	--
2000	5030± 425(34.7)	10440	8455± 420(58.3)	--

[a]Uniaxial compressive strength was determined using specimens 0.5 in. x 1. in x 1 in. or 1 in. x 1 in. x 1 in. loaded uniaxially at a 0.020 in./min. strain rate. Values are the average for five samples.

[b]Generic preparation: 75% calcined kaolin (70% to -20 mesh, 5% ball milled fines with 50% less than 325 mesh), 25% calcium aluminate cement (Casting Grade CA-25, Alcoa); 11% water added.

[c]Commercial light weight insulating castable, calcium aluminate bonded (Litecast 75-28, General Refractories); 21% water added.

[d]Commercial lightweight insulating castable, 42% alumina (B&W Kaolite 2300 LI, Babcock & Wilcox); 59% water added.

[e]Specimens, after molding, were dried for 24 hours at 250 °F.

[f]Another table in the same report does not give a value measured at 1500 °F and does give this value as measured at 1750 °F.

[g]Coarse grain size, high-strength 50% alumina prototype castable (B&W Kaocrete XD50, Mix 36C, Babcock & Wilcox); 7.5% water added.

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EFFECT OF TEMPERATURE ON THE COMPRESSIVE STRENGTH[a] OF SOME
CASTABLE REFRACTORIES[26], Continued

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[h] 4 wt% of 310 SS fibers (Ribtec 310), 1 in. long, was added to the Kaocrete XD50.

[i] 2 wt% of 446 SS fibers (Ribtec 446), 1 in. long, was added to the Kaocrete XD50.

[j] 4 wt% of 446 SS fibers (Ribtec 446), 1 in. long, was added to the Kaocrete XD50.

[k] In another table in the same report the temperature is given as 1800 °F for this measurement.

[l] DOE 90 generic preparation: 65% tabular alumina (6 to -20 mesh), 10% calcined alumina (-325 mesh), 25% calcium aluminate cement (Secar 250, Lone Star Lafarge); 10.5% water added.

[m] DOE 90 generic preparation: as in footnote l but with Casting Grade CA-25 (Alcoa) cement; 9.3% water added.

[n] Modified DOE 90 preparation: 70% tabular alumina (-48 mesh), 5% calcined alumina (-325 mesh), 25% calcium aluminate cement (Casting Grade CA-25, Alcoa); 8.5% water added.

[o] Commercial castable (B&W Kao-Tab 95, Babcock & Wilcox).

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EFFECT OF TEMPERATURE ON THE MODULUS OF RUPTURE[a] OF SOME
CASTABLE REFRACTORIES[26]

Modulus of Rupture,[a] psi (MPa)

Temperature °F	Generic 50% Al_2O_3 [b]	Commercial 50% Al_2O_3 [c]	Commercial 42% Al_2O_3 [d]	
At 72, as-cured[e]	1425	655	--	
72, 250°(24h)[e]	1125±140(7.8)	570± 65	230±30	
500	860± 85(5.9)	320± 30(2.2)	170±20	
1000	890± 50(6.1)	225± 55(1.6)	150±15	
1250	--	220± 25(1.5)	140±10	
1500	1030±150(7.1)	185±115(1.3)	150±25[f]	
1750	660± 85(4.5)	--	--	
2000	435± 90(3.0)	--	--	

Temperature °F	Commercial 50% Al_2O_3[g]	50% Al_2O_3[g] + 310 SS[h]	50% Al_2O_3[g] + 446 SS[i]	50% Al_2O_3[g] + 446 SS[j]
At 72, 250°(24h)[e]	980	1990±430	1150±225	1460±135
500	--	1080±430	585±120	675± 85
1000	950	1070±190	650±140	565±140
1500	1080	990±175	570±170	650± 60
1750	--	730± 90[k]	510± 80[k]	460± 30[k]
2000	850	757±160[l]	470± 75	390± 55

Temperature °F	Generic 90+% Al_2O_3[m]	Generic 90+% Al_2O_3[n]	Modified 90+% Al_2O_3[o]	Commercial 90+% Al_2O_3[p]
At 72, as-cured[e]	2325±170(16.0)	1775	--	1695
72, 250°(24h)[e]	1575	1680	1790±330(12.3)	1620
500	950±150(6.5)	870	980±150(6.8)	--
1000	840±320(5.8)	1090	890±170(6.1)	1320
1500	685± 90(4.7)	815	865± 55(6.0)	--
1750	690±225(4.8)	640	610±210(4.2)	--
2000	615±205(4.2)	1210	760±320(5.2)	1320

[a] Modulus of rupture determined following the ASTM C583-67 (1972) procedure. Bars either 1 in. x 1 in. x 6 in. or 1 in. x 2 in. x 6 in. were subjected to 3-point bending, loaded at a rate of 0.02 in./min. to failure. The above values are averages for five specimens.

[b] Generic preparation: 75% calcined kaolin (70% to -20 mesh, 5% ball milled fines with 50% less than 325 mesh), 25% calcium aluminate cement (Casting Grade CA-25, Alcoa); 11% water added.

[c] Commercial lightweight insulating castable, calcium aluminate bonded (Litecast 75-28, General Refractories); 21% water added.

[d] Commercial lightweight insulating castable, 42% alumina (B&W Kaolite 2300 LI, Babcock & Wilcox); 59% water added.

[e] "As-cured" refers to samples which, after molding, were stored in a water-filled dessicator. "250°(24h)" refers to samples which, after molding, were dried for 24 hours at 250 °F.

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EFFECT OF TEMPERATURE ON THE MODULUS OF RUPTURE[a] OF SOME
CASTABLE REFRACTORIES[26], Continued

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[f] Another table in the same report does not give a value measured at 1500 °F and does give this value as measured at 1750 °F.

[g] Coarse grain size, high-strength 50% alumina prototype castable (B&W Kaocrete XD50, Mix 36C, Babcock & Wilcox); 7.5% water added.

[h] 4 wt% of 310 SS fibers (Ribtec 310), 1 in. long, was added to the Kaocrete XD50.

[i] 2 wt% of 446 SS fibers (Ribtec 446), 1 in. long, was added to the Kaocrete XD50.

[j] 4 wt% of 446 SS fibers (Ribtec 446), 1 in. long, was added to the Kaocrete XD50.

[k] In another table in the same report the temperature is given as 1800 °F for this measurement.

[l] In another table in the same report this value is given as 575±160.

[m] DOE 90 generic preparation: 65% tabular alumina (6 to -20 mesh), 10% calcined alumina (-325 mesh), 25% calcium aluminate cement (Secar 250, Lone Star Lafarge); 10.5% water added.

[n] DOE 90 generic preparation: as in footnote m but with Casting Grade CA-25 (Alcoa) cement; 9.3% water added.

[o] Modified DOE 90 preparation: 70% tabular alumina (-48 mesh), 5% calcined alumina (-325 mesh), 25% calcium aluminate cement (Casting Grade CA-25, Alcoa); 8.5% water added.

[p] Commercial castable (B&W Kao-Tab 95, Babcock & Wilcox).

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COMPRESSIVE STRENGTH[a] AND WEIGHT LOSS DATA FOR REFRACTORIES EXPOSED
TO CARBON MONOXIDE AT 500 °C AND 600 PSI FOR 100 HOURS[27]

Added Dopant	Wt% Fe	Compressive Strength, psi[a]	Ratio wt. After to Wt. Before	No. of Samples	Average Strength[b] After Exposure at One Atmosphere
DOE 90 GENERIC CASTABLE[c]					
none	0.0	7,675±500	0.999	4	7,150±2,050
Fe	0.5	0±0[g]	0.000[g]	3	
Fe_2O_3	2.0	9,725±600	1.007	3	
DOE 50% Al_2O_3 GENERIC CASTABLE[d]					
none	0.0	5,400±1,025	0.999	4[e]	4,825±1,250
Fe	0.5	0±0[g]	0.000[g]	3	
Fe_2O_3	2.0	7,850±325	1.011	3	
DOE PHOSPHATE-BONDED RAMMING MIX[f]					
none	0.0	6,900±950	1.000	4[e]	10,050±3,750
Fe	0.5	0±0[g]	0.000[g]	3	
Fe_2O_3	1.0	6,775±2,200	1.006	3	

[a] Compressive strength was measured on a universal testing machine with a crosshead speed of 0.1 in/min.

[b] The average value for 44 samples of each refractory which had been exposed to a variety of CO-containing atmospheres for 100 hours each.

[c] DOE 90 generic castable: 65 wt% tabular alumina (25 wt% 6 to 10 mesh, 20 wt% 10 to 20 mesh, 20 wt% less than 20 mesh), 10 wt% calcined alumina (less than 325 mesh), 25 wt% calcium aluminate cement (Casting Grade CA-25 cement), all Alcoa materials. Materials were dry mixed for 2 min, then water added with continued mixing to ball-in-hand consistency (∿605 ml); refractory then poured into 2 in x 2 in x 2 in aluminum molds, vibrated for 5 min, sealed in plastic for 24 hr, then fired for 5 hr in air at 1100 °C.

[d] DOE generic castable: 75 wt% calcined kaolin (25 wt% 6 to 10 mesh, 20 wt% 10 to 20 mesh, 15 wt% less than 20 mesh, 20 wt% less than 200 mesh), 25 wt% calcium aluminate cement (Casting Grade CA-25, Alcoa). Samples were prepared as described in footnote c except that the water used was ∿700 ml.

[e] Only three samples were used in strength testing.

[f] DOE generic ramming mix: 82 wt% tabular alumina (30 wt% 6 to 10 mesh, 20 wt% 10 to 20 mesh, 15 wt% less than 20 mesh, 17 wt% less than 48 mesh), 15 wt% calcined alumina (less than 325 mesh), 3 wt% bentonite; to this was added 1 wt% hydrated alumina, 6 wt% phosphoric acid (85%), and 0.25 wt% water. Dry materials were mixed and then hand mixed in plastic bags with the liquids; mixture was aged for 18 hr; samples formed by die pressing at 500 psi, curing at 250 °C in air for 12 hr, then firing at 1100 °C for 5 hr.

[g] Samples disintegrated.

COMPRESSIVE STRENGTH[a] OF IRON-DOPED REFRACTORIES AFTER EXPOSURE[b] TO MIXTURES OF CARBON MONOXIDE AND OTHER GASES [27], Table Continued

Added Dopant	Wt% Fe	DOE 90 Generic Composition[c] Compressive Strength, psi[a]	Ratio Doped to Undoped Strength	Ratio Wt. After to Wt. Before	No. of Samples	DOE 50% Al₂O₃ Generic Composition[d] Compressive Strength, psi[a]	Ratio Doped to Undoped Strength	Ratio Wt. After to Wt. Before	No. of Samples	DOE Generic Ramming Mix[e] Compressive Strength, psi[a]	Ratio Doped to Undoped Strength	Ratio Wt. After to Wt. Before	No. of Samples
FEED GAS COMPOSITION: 99.2% CO-0.8% H₂S													
None	0.0	7,475 ±675		0.998	4	5,450 ±200		.996	4	5,700 ±3,025		.998	4
Fe	0.5	8,075 ±1,350	1.080	0.990	4	3,975 ±1,275	.729	.996	4	6,100 ±2,075	1.070	.997	4
Fe	1.0	9,100 ±475	1.217	0.996	4	2,625 ±1,925	.482	.962	4	9,700 ±3,425	1.702	.987	4
Fe	1.5	7,775 ±2,200	1.040	0.999	4	500 ±625	.092	.986	4	9,625 ±4,175	1.689	.934	4
Fe	2.0	4,125 ±4,775	0.552	0.683	4	4,900 ±3,950	.899	.737	4	8,475 ±2,675	1.489	.931	4
Fe₂O₃	0.5	7,850 ±2,100	1.050	1.000	4	5,825 ±1,100	1.069	1.000	4	6,850 ±2,450	1.202	.999	4
Fe₂O₃	1.0	8,900 ±950	1.191	0.999	4	5,200 ±1,125	.954	1.000	4	2,550 ±3,550	.447	.678	4
Fe₂O₃	1.5	7,150 ±550	0.957	0.999	4	5,975 ±2,775	1.096	1.000	4	0 ±0	0	.706	4
Fe₂O₃	2.0	7,925 ±2,950	1.060	1.000	4	5,225 ±725	.959	1.001	4	675 ±950	.118	.131	4
FEED GAS COMPOSITION: 80% CO-20% H₂O													
None	0.0	6,775 ±2,325		1.005	4	3,475 ±575		.999	4	6,725 ±2,225		1.000	4
Fe	0.5	8,425 ±1,825	1.244	1.004	4	3,225 ±1,825	.928	.999	4	6,375 ±1,675	.948	.998	4
Fe	1.0	9,825 ±4,325	1.450	1.004	4	4,425 ±1,200	1.273	.998	4	4,825 ±1,625	.717	.999	4
Fe	1.5	10,450 ±2,275	1.542	1.002	4	5,325 ±1,300	1.532	.998	4	7,725 ±850	1.149	.999	4
Fe	2.0	9,725 ±2,900	1.435	1.001	4	4,850 ±100	1.396	.997	4	8,075 ±1,775	1.201	.999	4
Fe₂O₃	0.5	9,050 ±1,600	1.336	1.003	4	4,400 ±750	1.266	.999	4	8,100 ±2,750	1.204	.999	4
Fe₂O₃	1.0	11,350 ±4,025	1.675	1.002	4	4,075 ±600	1.173	1.000	4	10,456 ±3,025	1.554	.999	4
Fe₂O₃	1.5	17,700 ±1,550	2.613	1.004	4	6,125 ±2,225	1.763	.999	4	10,500 ±2,900	1.561	.999	4
Fe₂O₃	2.0	16,750 ±3,775	2.472	1.003	4	4,325 ±475	1.245	1.000	4	5,975 ±2,800	.888	.999	4
FEED GAS COMPOSITION: 60% CO-40% H₂O													
None	0.0	8,700 ±2,800		1.005	4	4,775 ±1,250		1.002	4	6,900 ±2,175		.999	4
Fe	0.5	8,150 ±1,500	0.937	1.004	4	4,325 ±975	.906	1.000	4	5,125 ±2,000	0.743	.999	4
Fe	1.0	9,075 ±1,500	1.043	1.003	4	4,275 ±1,350	.895	.999	4	8,525 ±3,175	1.236	.998	4
Fe	1.5	8,550 ±2,025	0.983	1.002	4	4,550 ±550	.953	.999	4	4,900 ±975	.710	.999	4
Fe	2.0	7,775 ±1,300	0.894	1.003	4	3,150 ±875	.660	.998	4	8,625 ±2,225	1.250	.998	4
Fe₂O₃	0.5	8,750 ±725	1.006	1.000	4	3,850 ±1,325	.806	1.000	4	9,375 ±3,025	1.359	.999	4
Fe₂O₃	1.0	9,250 ±475	1.063	1.001	4	3,975 ±875	.832	1.001	4	7,775 ±3,150	1.127	.997	4
Fe₂O₃	1.5	10,625 ±750	1.221	1.001	4	4,175 ±950	.874	1.000	4	10,600 ±2,450	1.536	.998	4
Fe₂O₃	2.0	12,100 ±3,050	1.391	1.001	4	4,250 ±675	.890	1.000	3	11,550 ±3,500	1.674	.999	4
FEED GAS COMPOSITION: 80% CO-20% H₂													
None	0.0	5,325 ±900		0.998	4	5,100 ±600		.998	4	12,000 ±3,250		.999	4
Fe	0.5	6,000 ±850	1.126	0.996	4	5,750 ±1,050	1.127	.996	4	9,900 ±1,400	.825	.997	4
Fe	1.0	11,125 ±1,225	2.089	0.997	4	10,425 ±1,325	2.044	.996	4	10,775 ±2,875	.898	.996	4
Fe	1.5	10,800 ±1,150	2.028	0.996	4	12,150 ±2,125	2.382	.995	4	11,400 ±750	.950	.959	4
Fe	2.0	4,875 ±1,100	0.915	0.765	4	11,525 ±1,475	2.260	.969	4	12,500 ±2,100	1.042	.968	4
Fe₂O₃	0.5	9,950 ±950	1.869	0.998	4	6,000 ±1,850	1.176	.996	4	15,100 ±2,500	1.258	.998	4
Fe₂O₃	1.0	7,625 ±575	1.432	0.998	4	11,050 ±1,300	2.167	.995	4	15,925 ±1,300	1.327	.995	4
Fe₂O₃	1.5	11,050 ±1,825	2.075	0.997	4	13,175 ±1,600	2.583	.995	4	11,925 ±2,375	.994	.994	4
Fe₂O₃	2.0	8,025 ±3,350	1.507	0.997	4	11,625 ±2,650	2.279	.994	4	8,250 ±750	.688	.992	4
FEED GAS COMPOSITION: 60% CO-40% H₂													
None	0.0	6,975 ±875		1.000	4	5,125 ±650		.997	4	10,450 ±3,375		.999	4
Fe	0.5	6,775 ±550	0.971	0.968	4	4,075 ±775	.795	.997	4	8,650 ±2,475	.828	.997	4
Fe	1.0	9,775 ±425	1.401	1.031	4	5,725 ±850	1.117	.996	4	10,300 ±1,900	.986	.996	4
Fe	1.5	8,075 ±925	1.158	0.897	4	5,150 ±400	1.005	.996	4	10,925 ±2,275	1.045	.996	4
Fe	2.0	9,000 ±1,325	1.290	0.997	4	5,100 ±1,125	.995	.996	4	10,925 ±1,225	1.045	.997	4
Fe₂O₃	0.5	7,875 ±1,275	1.129	0.999	4	4,875 ±1,075	.951	.998	4	10,675 ±1,725	1.022	.997	4
Fe₂O₃	1.0	8,050 ±1,750	1.154	0.999	4	5,275 ±375	1.029	.997	4	9,625 ±1,900	.921	.996	4
Fe₂O₃	1.5	8,775 ±2,200	1.258	0.999	3	4,250 ±875	.829	.997	4	11,050 ±950	1.057	.996	4
Fe₂O₃	2.0	8,200 ±2,400	1.176	0.998	4	6,275 ±800	1.224	.997	4	10,400 ±3,375	.995	.995	4

[a] The compressive strength was measured on a universal testing machine with a crosshead speed of 0.1 in/min.

[b] Exposure was for 100 hr at 500 °C and 1 atmosphere pressure of the gases.

[c] DOE generic castable: 65 wt% tabular alumina (25 wt% 6 to 10 mesh, 20 wt% 10 to 20 mesh, 20 wt% less than 20 mesh), 10 wt% calcined alumina (less than 325 mesh), 25 wt% calcium aluminate cement (Casting Grade CA-25 cement), all Alcoa materials. Materials were dry mixed for 2 min, then water added with continued mixing to ball-in-hand consistency (~605 ml); refractory then poured into 2 in x 2 in x 2 in aluminum molds, vibrated for 5 min, sealed in plastic for 24 hr, then fired for 5 hr in air at 1100°C.

[d] DOE generic castable: 75 wt% calcined kaolin (25 wt% 6 to 10 mesh, 20 wt% 10 to 20 mesh, 15 wt% less than 20 mesh, 20 wt% less than 200 mesh), 25 wt% calcium aluminate cement (Casting Grade CA-25, Alcoa). Samples were prepared as described in footnote c except that the water used was ~700 ml.

[e] DOE generic ramming mix: 82 wt% tabular alumina (30 wt% 6 to 10 mesh, 20 wt% 10 to 20 mesh, 15 wt% less than 20 mesh, 17 wt% less than 48 mesh), 15 wt% calcined alumina (less than 325 mesh), 3 wt% bentonite; to this was added 1 wt% hydrated alumina, 6 wt% phosphoric acid (85%), and 0.25 wt% water. Dry materials were mixed and then hand mixed in plastic bags with the liquids; mixture was aged for 18 hr; samples formed by die pressing at 500 psi, curing at 250°C in air for 12 hr, then firing at 1100°C for 5 hr.

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EFFECT OF HEAT TREATMENT AND GASEOUS ENVIRONMENT[a] ON THE MODULUS OF RUPTURE[b] OF VARIOUS ALUMINA REFRACTORIES[c][39]

	Treatment Conditions			Modulus of Rupture,[b] psi				
Atmosphere[a]	Temperature °C / °F	Pressure MPa/psi	Time days					

DENSE HIGH-ALUMINA COMMERCIAL CASTABLES

Atmosphere[a]	Temp °C / °F	Pressure MPa/psi	Time days	94% Al_2O_3[d]	93% Al_2O_3[e]	90-95% Al_2O_3[f]	90-95% Al_2O_3[g]
Air (dried)	110 / 230	ambient	1	2870±290	2500±140	3080±240	3300±370
Air (fired)	260 / 500	ambient	3/4	1940±130	1185±180	2510±190	2410±260
Air (fired)	538 /1000	ambient	3/4	1880± 55	1380±150	2650± 90	2220±350
CGA exposure	260 / 500	3.4/500	10	3030±190	2510±330	2880±320	3120±760
			20	1950±180	1630±210	2520±190	2580±470
			30	2760±420	2610±170	2690±400	2990±380
CGA exposure	538 /1000	6.9/1000	10	1270±230	735± 80	1460±150	1780±300
			20	1240±140	740± 65	1470±160	1500±200
			30	1300±130	750± 60	1570± 55	1720±270
CGA with H_2S	538 /1000	6.9/1000	10	1250±120	970± 80	1800±150	2130±185
			20	1105± 60	925± 75	1620± 45	1940±125
			30	1200±125	945± 30	1700±105	1810±805
CGA, H_2O saturated[h]	231 / 447	6.9/1000	30	4240±130	1984	not tested	4860±380
CGA with H_2S, H_2O satd.[h]	231 / 447	6.9/1000	30	3540±370	1720±140	4150±450	5375±280
CO/H_2O, H_2O saturated	199 / 390	3.2/ 465	10	4470±160	2320±150	4870±320	4780±620
CO/H_2O	260 / 500	3.2/ 465	10	2390±260	2230±150	3270±370	3520±220
	538 /1000	3.2/ 465	10	1175±170	860± 80	1560±210	1580±240

DENSE HIGH-ALUMINA CASTABLES (GENERIC PREPARATIONS)

Atmosphere[a]	Temp °C / °F	Pressure MPa/psi	Time days	93% Al_2O_3[i]	91% Al_2O_3[j]	91% Al_2O_3[k]	88% Al_2O_3[l]	84% Al_2O_3[m]
Air (dried)	110 / 230	ambient	1	2850±190	2930±340	2370±300	--	--
Air (fired)	260 / 500	ambient	3/4	2220±170	970±180	1700±300	1740±100	1440±130
Air (fired)	538 /1000	ambient	3/4	2200±140	1810±290	970±230	1630± 80	1330±160
CGA exposure	260 / 500	3.4/500	10	2680±870	2930±430	2260±560		
			20	2480±420	1800±210	1870±220		
			30	2860±600	3120±160	2740±230		
CGA exposure	538 /1000	6.9/1000	10	1820±210	700± 75	1290±120		
			20	1920±210	640± 65	1260±170		
			30	1800±200	660± 80	1250±140	NOT TESTED	NOT TESTED
CGA with H_2S	538 /1000	6.9/1000	10	1720±100	1125±150	1295±170		
			20	1750± 95	880±185	1290±160		
			30	2020±160	1035±130	1410±100		
CGA, H_2O saturated[h]	231 / 447	6.9/1000	30	6630± 35	not tested	2650±450		
CGA with H_2S, H_2O satd.[h]	231 / 447	6.9/1000	30	4870±210	4050±560	2120±240		
CO/H_2O, H_2O saturated	199 / 390	3.2/465	10	4830±330	3310±270	--		
CO/H_2O	260 / 500	3.2/465	10	2530± 60	3040±280	--		
	538 /1000	3.2/465	10	1620±190	1211±150	--		

INTERMEDIATE-ALUMINA DENSE AND INSULATING CASTABLES

Atmosphere[a]	Temp °C / °F	Pressure MPa/psi	Time days	54-57% Al_2O_3[n]	57% Al_2O_3[o]	59% Al_2O_3[p]	54% Al_2O_3[q]
Air (dried)	110 / 230	ambient	1	2830±560	2200±240	650±100	970±120
Air (fired)	260 / 500	ambient	3/4	1870±340	1570±230	460± 50	790± 80
Air (fired)	538 /1000	ambient	3/4	1760±180	1390±100	480± 35	940± 40
CGA exposure	260 / 500	3.4/500	10	2810±310	2770±900	570±100	890±190
			20	2270±310	2000±310	440± 80	670± 65
			30	2700±450	2130±340	460± 50	710± 60
CGA exposure	538 /1000	6.9/1000	10	2160±450	2390±170	530± 20	1160± 40
			20	2510±370	2730±240	580± 35	1090± 70
			30	2540±330	2450±320	650± 55	1090± 90
CGA with H_2S	538 /1000	6.9/1000	10	1760±370	2245±220	520± 30	865± 90
			20	1760±170	2140±140	555± 45	895± 65
			30	1850±190	2340±190	520± 60	900± 65
CGA, H_2O saturated[h]	231 / 447	6.9/1000	30	3540± 90	3330±830	930± 1	960±270
CGA with H_2S, H_2O satd.[h]	231 / 447	6.9/1000	30	2350±240	3920±385	790± 40	560± 70
CO/H_2O, H_2O saturated	199 / 390	3.2/465	10	2880±210	3280±400	860±140	870± 55
CO/H_2O	260 / 500	3.2/465	10	2280±200	3280±180	610± 80	850± 70
	538 /1000	3.2/465	10	2020±130	1760±280	490± 55	1090±210

(Table Continued)

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EFFECT OF HEAT TREATMENT AND GASEOUS ENVIRONMENT[a] ON THE MODULUS OF RUPTURE[b] OF VARIOUS ALUMINA REFRACTORIES[c][39], Continued

Atmosphere[a]	Treatment Conditions			Modulus of Rupture,[b] psi		
	Temperature $^\circ$C / $^\circ$F	Pressure MPa/psi	Time days	96% Al_2O_3[r]	96% Al_2O_3[s]	90% Al_2O_3[t]
- PHOSPHATE-BONDED RAMMING MIXES -						
Air (fired)	538 /1000	ambient	3/4	3190±320	3410±250	2650±600
CGA exposure	260 / 500	3.4/500	10	3320±270	2420±520	1260±620
			20	3960±450	2970±280	2370±560
			30	4040±380	3120± 38	2520±510
CGA exposure	538 /1000	6.9/1000	10	3340±420	3240±410	1810±390
			20	3190±390	2930±440	2680±450
			30	3610±570	3010±410	1610±640
CGA with H_2S	538 /1000	6.9/1000	10	2350±245	2400±430	2185±330
			20	2175±305	2440±255	2230±400
			30	2240±420	2410±320	2120±200
CGA, H_2O saturated[h]	231 / 447	6.9/1000	30	1620±320	470± 40	440± 60
CGA with H_2S, H_2O satd.[h]	231 / 447	6.9/1000	30	1100±120	610±130	904±325
CO/H_2O, H_2O saturated	199 / 390	3.2/465	10	1290±650	690±280	540±170
CO/H_2O	260 / 500	3.2/465	10	2640±220	2440±195	2800±400
	538 /1000	3.2/465	10	2960±210	2280±160	2090±180

[a]Environments included a simulated coal gasification atmosphere (CGA) with an overall composition (in vol %) of 18 CO, 12 CO_2, 24 H_2, 5 CH_4, and 41 H_2O; another CGA which has 1% H_2S added; and a CO-H_2O atmosphere which is 52.5 CO and 46.5 H_2O (vol %). Samples were exposed in two pressure vessels. One vessel was a steam generator and tests specifying saturated conditions occurred in this vessel. After placing samples, the vessel was heated to the desired steam pressure and the other gases were then added so as to obtain the concentrations specified above. The second vessel was connected to the steam generator and after samples were placed and the vessel heated to the test temperature, the vessel was pressurized with the steam/gas mixture from the steam generator. A gaseous flow rate for both vessels was maintained so as to provide one complete change in atmosphere every 24 hours.

[b]Modulus of rupture from 3-point bending tests, ASTM C268-70, with a span of 2 in. and a crosshead speed of 0.5 cm/min. All values are averages for seven specimens except where noted.

[c]All cement-bonded castables were cast in molds either 12 x 3 x 1/2 in. or 3 x 3/4 x 3/4 in., cured 24 h in air at 100% humidity at ambient temperature, and dried at 230 °F for 24 h. The larger slabs were then cut to 3 x 1/2 x 1/2 in. bars. Phosphate-bonded specimens were prepared by hand ramming in 3 x 3/4 x 3/4 in. molds, drying immediately for 24 h at 230 °F, and then firing at 1000 °F for 18 h. All specimens were stored at 230 °F until tested.

[d]94% alumina dense castable, calcium aluminate (79% alumina, 18% calcia) bonded (Greencast-94, A.P. Green; CA-25 Calcium Aluminate Cement, Alcoa).

[e]93% alumina dense castable, calcium aluminate (72% alumina, 25% calcia) bonded (B&W Kao-Tab 93, Babcock & Wilcox; C-3 cement, Babcock & Wilcox).

[f]90-95% alumina dense castable, calcium aluminate (79% alumina, 18% calcia) bonded (Castolast G, Harbison-Walker; CA-25 Calcium Aluminate Cement, Alcoa).

[g]90-95% alumina dense castable, calcium aluminate (79% alumina, 18% calcia) bonded (Purotab, Kaiser Refractories; CA-25 Calcium Aluminate Cement, Alcoa).

[h]Data are for two specimens only.

[i]93% alumina dense castable, UMR-1 generic preparation (70% tabular alumina + 30% calcium aluminate cement) (T-61 alumina, Alcoa; CA-25 Calcium Aluminate Cement, Alcoa).

[j]91% alumina dense castable, UMR-2 generic preparation (70% tabular alumina + 30% calcium aluminate cement) (T-61 alumina, Alcoa; Secar 71(250), a 72% alumina-26% calcia cement, Lone Star Lafarge).

[k]91% alumina dense castable, UMR-3 generic preparation (70% tabular alumina + 30% calcium aluminate cement) (T-61 alumina, Alcoa; C-3, a 72% alumina-25% calcia cement, Babcock & Wilcox).

[l]88% alumina dense castable, UMR-6 generic preparation (UMR-1 preparation with 5% SiO_2 added as 99.9% pure bone dry Wedron silica flour).

[m]84% alumina dense castable, UMR-7 generic preparation (UMR-1 preparation with 10% SiO_2 added as 99.9% pure bone dry Wedron silica flour).

[n]54-57% alumina, 34-37% silica castable, calcium aluminate (79% alumina, 18% calcia) bonded (Lo-Abrade, A.P. Green; CA-25 Calcium Aluminate Cement, Alcoa).

[o]57% alumina, 34% silica castable, calcium aluminate (79% alumina, 18% calcia) bonded (RC-3, General Refractories; CA-25 Calcium Aluminate Cement, Alcoa).

[p]59% alumina, 33% silica insulating castable, calcium aluminate bonded (Kast-O-Lite 30, A.P. Green; CA-25 cement, Alcoa).

[q]54% alumina, 40% silica insulating castable, calcium aluminate bonded (Cer-Lite #75, C-E Refractories; CA-25 cement, Alcoa).

[r]96% alumina dense phosphate-bonded ramming mix (Greenpak-90P, A.P. Green).

[s]96% alumina dense phosphate-bonded ramming mix (90 Ram H.S., C-E Refractories).

[t]90% alumina dense phosphate-bonded ramming mix (Brikram 90R, General Refractories).

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EFFECT OF SATURATED VAPOR AND LIQUID AND EXPOSURE[a] TO PRESSURE-TEMPERATURE CYCLING CAUSING BOEHMITE FORMATION[b] AND DECOMPOSITION ON THE MODULUS OF RUPTURE[c] OF CASTABLE ALUMINA REFRACTORIES[d][39]

	Treatment Conditions			Modulus of Rupture, [c] psi			
Atmosphere[a]	Temperature °C / °F	Pressure MPa/psi	Time days	- - - - - - - - DENSE CASTABLES - - - - - - - -			
				93% Al_2O_3[e]	87% Al_2O_3[f]	64% Al_2O_3[g]	58% Al_2O_3[h]
Air (dried)	110/ 230	ambient	1	2850±190	2260±170	---	2370±210
Air (fired)	260/ 500	ambient	3/4	2220±170	1620±170	1770±150	1290±120
Air (fired)	538/1000	ambient	3/4	2200±140	1490± 70	1995±180	1290± 70
Saturated steam { vapor	231/ 447	2.8/410	10	4320±330	2460±270	4250±270	2710±210
liquid	231/ 447	2.8/410	10	3900±380	2090±160	4180±460	2620±160
Saturated steam { vapor	285/ 545	6.9/1000	10	3500±215	2145±145	4365±270	2535±290
liquid	285/ 545	6.9/1000	10	4895±240	2515±165	4180±820	3210±345
CGA with H_2S, { vapor H_2O saturated	231/ 447	6.9/1000	10	4270±260	3185±420	4015±240	3560±270
liquid	231/ 447	6.9/1000	10	4110±235	3170±610	4890±355	3365±290
CGA with H_2S, { vapor H_2O saturated	231/ 447	6.9/1000	44	3920±450	2970±325	4670±945	3340±480
liquid	231/ 447	6.9/1000	44	6180±380	not tested	5530±435	not tested
CGA with H_2S, saturated[i] followed by firing in air[j]	240/ 465 538/1000	6.9/1000 ambient	5 3/4	1860±660 1730± 85	3980±370 3900±150	2450±180 2130±210	3040±175 2720±170
Cycling tests--repeat of the preceding exposure followed by the same air firing			1 cycle	1190±130	3980±270	4220±615	3440±365
			2 cycles	1030±245	3830±255	4015±200	3460±300
			3 cycles	1430±180	3570±355	3890±280	3050±300
			4 cycles	1260±160	4070±750	3270±265	2645±275
			5 cycles	1360±720	3510±410	3940±283	3420±350

				- - - - - - - INSULATING CASTABLES - - - - - - -			
				54% Al_2O_3[k]	47% Al_2O_3[l]	35% Al_2O_3[m]	
Air (dried)	110/ 230	ambient	1	970±120	440± 90	145± 15	
Air (fired)	260/ 500	ambient	3/4	795± 80	195± 35	120± 10	
Air (fired)	538/1000	ambient	3/4	940± 40	210± 50	100± 20	
Saturated steam { vapor	231/ 447	2.8/410	10	670± 50	380± 60	60± 40	
liquid	231/ 447	2.8/410	10	640± 70	430± 50	90± 20	
Saturated steam { vapor	285/ 545	6.9/1000	10	510± 50	540± 25	95± 35	
liquid	285/ 545	6.9/1000	10	485±100	660±100	190± 20	
CGA with H_2S, { vapor H_2O saturated	231/ 447	6.9/1000	10	380± 62	845±103	345± 60	
liquid	231/ 447	6.9/1000	10	430± 35	810± 60	210± 95	
CGA with H_2S, { vapor H_2O saturated	231/ 447	6.9/1000	44	790±110	440± 85	220± 65	
liquid	231/ 447	6.9/1000	44	1580± 95	not tested	580± 30	
CGA with H_2S, saturated[i] followed by firing in air[j]	240/ 465 538/1000	6.9/1000 ambient	5 3/4	790± 60 975±120	1065±130 1060±140	190± 40 130± 35	
Cycling tests--repeat of the preceding exposure followed by the same air firing			1 cycle	990± 40	965±140	165± 10	
			2 cycles	965±125	975± 70	150± 25	
			3 cycles	1000± 90	1050± 70	130± 25	
			4 cycles	990± 80	760± 85	130± 20	
			5 cycles	945± 50	990± 75	160± 25	

[a] For testing the effect of water-saturated vapor and of liquid, samples were exposed in a steam generator, both in the vapor and in the liquid in the bottom of the vessel. After placement of samples, the vessel was heated to the desired steam pressure and where tests included other gases, these were then added so as to obtain the desired overall concentrations. The overall composition of the coal gasification atmosphere (CGA) is (in vol %) 18 CO, 12 CO_2, 24 H_2, 40 H_2O, 5 CH_4, and 1 H_2S. A gaseous flow rate was maintained so as to provide one complete change in atmosphere every 24 hours.

[b] Other tests showed that boehmite formation occurred in steam-saturated atmospheres and resulted in very large increases in the flexural strength. These cycling tests were designed to investigate the effect of repetitive

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(Table Continued)

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EFFECT OF SATURATED VAPOR AND LIQUID AND EXPOSURE[a] TO PRESSURE-TEMPERATURE CYCLING CAUSING BOEHMITE FORMATION[b]
AND DECOMPOSITION ON THE MODULUS OF RUPTURE[c] OF CASTABLE ALUMINA REFRACTORIES[d][39], Continued

Footnotes continued

formation and decomposition of boehmite on the properties of the refractories.

[c]Modulus of rupture from 3-point bending tests, ASTM C268-70, span of 2 in. and a crosshead speed of 0.5 cm/min. All values are averages for seven specimens.

[d]After casting, specimens were cured 24 hours in air at 100% humidity at ambient temperature, and dried at 230 °F for 24 hours. Specimens were stored at 230 °F until tested.

[e]93% alumina dense castable, UMR-1 generic preparation (70% tabular alumina + 30% calcium aluminate cement) (T-61 alumina, Alcoa; CA-25 Calcium Aluminate Cement, Alcoa).

[f]87.4% alumina dense castable, UMR-4 generic preparation (70% tabular alumina + 30% calcium aluminate cement) (T-61 alumina, Alcoa; Refcon, a 58% alumina-33% calcia cement, Universal Atlas).

[g]63.8% alumina, 28.3% silica dense castable, UMR-8 generic preparation (75% calcined kaolin aggregate + 25% calcium aluminate cement) (Mulcoa M-60 and Mulcoa M-47, C-E Minerals; CA-25 Calcium Aluminate Cement, a 79% alumina-18% calcia cement, Alcoa).

[h]58.6% alumina, 29.6% silica dense castable, UMR-5 generic preparation (75% calcined kaolin aggregate + 25% calcium aluminate cement) (Mulcoa M-60 and Mulcoa M-47, C-E Minerals; Refcon, a 58% alumina-33% calcia cement, Universal Atlas).

[i]85% steam saturated; this exposure is the one forming the first step in the cycling tests, forming boehmite.

[j]The firing step, following the saturated CGA exposure, completed one cycle, decomposing the boehmite.

[k]54% alumina, 40% silica insulating castable, calcium aluminate (79% alumina, 18% calcia) bonded (Cer-Lite #75, C-E Refractories; CA-25 Calcium Aluminate Cement, Alcoa).

[l]46.7% alumina, 40.2% silica insulating castable (Litecast 60-25, General Refractories).

[m]34.5% alumina, 52.5% silica insulating castable (VSL-50, A.P. Green).

EFFECT OF EXPOSURE[a] TO CARBON MONOXIDE/STEAM/HYDROGEN SULFIDE ON THE MODULUS OF RUPTURE[b] OF VARIOUS ALUMINA REFRACTORIES[c][39]

	Modulus of Rupture, psi					
	CO/H_2O = 0.1 + 1% H_2S		CO/H_2O = 1.0 + 1% H_2S		CO/H_2O = 3.0 + 1% H_2S	
	1000 °F	532 °F	1000 °F	466 °F	1000 °F	400 °F
Refractory[c]	Unsaturated	Saturated[d]	Unsaturated	Saturated[d]	Unsaturated	Saturated[d]
DENSE HIGH-ALUMINA CASTABLES						
93% Alumina[e]	1520±70	4660±320 (4610±630)	1780±90	2910±310 (4720±580)	1680±210	3320±370 (3590±230)
91% Alumina[f]	830±80	2290±560 (4710±100)	1780±90	2000±70 (3770±78)	1190±130	1930±570 (2030±1)
87% Alumina[g]	2180±230	2450±330 (1750±80)	1790±150	3120±600 (3570±210)	2160±170	1410±390 (3100±180)
88% Alumina[h]	not tested	not tested	2560±380	3300±320 (3330±370)	2960±160	3990±440 (3950±180)
84% Alumina[i]	not tested	not tested	2500±340	3540±360 (2190±290)	2310±180	3980±420 (4210±350)
DENSE INTERMEDIATE-ALUMINA CASTABLES						
57% Alumina, 34% silica[j]	3260±250	4470±640 (3960±560)	2480±400	4610±620 (3880±370)	1770±120	4060±560 (3570±250)
59% Alumina, 30% silica[k]	2470±340	3740±430 (1580±100)	1910±220	3280±630 (3700±50)	1550±240	3920±960 (3030±40)
INSULATING CASTABLES						
54% Alumina, 40% silica[l]	990±80	490±70 (250±)	1010±40	1230±160 (1190±90)	800±40	1100±170 (1440±210)
46% Alumina, 40% silica[m]	470±80	520±170 (520±2)	340±90	1000±260 (880±30)	370±50	785±85 (1010±55)
35% Alumina, 53% silica[n]	50±10	120±25 (170±5)	110±10	300±10 (420±20)	110±10	280±50 (390±40)
PHOSPHATE-BONDED RAMMING MIX						
96% Alumina[o]	2460±360	920±220 (1080±190)	2910±190	1920±720 (1910±70)	2430±390	1760±230 (1550±290)

[a] Samples were exposed in two pressure vessels. One vessel was a steam generator and tests which specify saturated conditions occurred in this vessel. After placing samples, the vessel was heated to the desired steam pressure and the other gases were then added so as to obtain the concentrations specified in the table. The second vessel was connected to the steam generator and after samples were placed and the vessel heated to the test temperature, the vessel was pressurized with the steam/gas mixture from the steam generator. Tests specified as unsaturated occurred in this vessel. A gaseous flow rate for both vessels was maintained so as to provide one complete change in atmosphere every 24 hours. The specimens were exposed at the temperatures specified in the table at 1000 psia for 20 days. For CO/H_2O = 0.1: CO 90 psia, H_2O 900 psia, H_2S 10 psia. For CO/H_2O = 1.0: CO 495 psia, H_2O 495 psia, H_2S 10 psia. For CO/H_2O = 3.0: CO 742.5 psia, H_2O 247.5 psia, H_2S 10 psia.

[b] All cement-bonded castables were cast in molds either 12 x 3 x 1/2 in. or 3 x 3/4 x 3/4 in., cured 24 hours in air at 100% humidity at ambient temperature, and dried at 230 °F for 24 hours. The 12 x 3 x 1/2 inch slabs were then cut to 3 x 1/2 x 1/2 in. bars. Phosphate-bonded specimens were prepared by hand ramming in 3 x 3/4 x 3/4 in. molds, immediately drying for 24 hours at 230 °F and then firing at 1000 °F for 18 hours. All specimens were stored at 230 °F until tested.

[c] The modulus of rupture was obtained in 3-point bending tests, ASTM C268-70, span of 2 inches, crosshead speed 0.5 cm/minute. All values are averages for seven specimens except where noted otherwise.

[d] Numbers without parentheses are for samples exposed to saturated vapor. Adjacent numbers in parentheses are for samples immersed in liquid (water) in the bottom of the vessel under the same exposure conditions (values are the average of two samples).

[e] 93% Alumina dense castable, UMR-1 generic preparation (70% tabular alumina + 30% calcium aluminate cement) (T-61 alumina, Alcoa; CA-25 Calcium Aluminate Cement, 79% alumina-18% calcia, Alcoa).

[f] 91% Alumina dense castable, UMR-2 generic preparation (70% tabular alumina + 30% calcium aluminate cement) (T-61 alumina, Alcoa; Secar 71(250), 72% alumina-26% calcia cement, Lone Star Lafarge).

[g] 87.4% Alumina dense castable, UMR-4 generic preparation (70% tabular alumina + 30% calcium aluminate cement) (T-61 alumina, Alcoa; Refcon, 58% alumina-33% calcia cement, Universal Atlas).

(Table Continued)

B.3 Mechanical Properties Testing

B.3.2 Refractories

B.3.2.57
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EFFECT OF EXPOSURE[a] TO CARBON MONOXIDE/STEAM/HYDROGEN SULFIDE ON THE MODULUS OF RUPTURE[b] OF VARIOUS ALUMINA REFRACTORIES[c][39],
Continued

Footnotes continued

[h]88% Alumina dense castable, UMR-6 generic preparation (UMR-1 generic preparation with 5% SiO_2 added as 99.9% pure bone dry Wedron silica flour).

[i]84% Alumina dense castable, UMR-7 generic preparation (UMR-1 generic preparation with 10% SiO_2 added as 99.9% pure bone dry Wedron silica flour).

[j]57% Alumina, 34% silica castable, calcium aluminate (79% alumina, 18% calcia) bonded (RC-3, General Refractories; CA-25 Calcium Aluminate Cement, Alcoa).

[k]58.6% Alumina, 29.6% silica castable, UMR-5 generic preparation (75% Mulcoa + Mulgrain M-60, C-E Minerals; 25% Refcon, 58% alumina-33% calcia cement, Universal Atlas).

[l]54% Alumina, 40% silica insulating castable, calcium aluminate (70% alumina-18% calcia) bonded (Cer-Lite #75, C-E Refractories; CA-25 Calcium Aluminate Cement, Alcoa).

[m]46.7% Alumina, 40.2% silica insulating castable (Litecast 60-25, General Refractories).

[n]34.5% Alumina, 52.5% silica insulating castable (VSL-50, A.P. Green).

[o]96% Alumina dense phosphate-bonded ramming mix (Greenpak-90P, A.P. Green).

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EFFECT OF EXPOSURE TO AN UNSATURATED AND STEAM-SATURATED SIMULATED COAL GASIFICATION ENVIRONMENT[a] ON THE MODULUS OF RUPTURE[b] OF VARIOUS ALUMINA REFRACTORIES[c][39]

| Refractory[c] | Modulus of Rupture,[b] psi | |
	Unsaturated (700 °F)	Saturated (447 °F)[d]
DENSE HIGH-ALUMINA CASTABLES		
94% Alumina[e]	1230	2790 (3210)
93% Alumina[f]	1750±190	4610±880 (5190±770)
91% Alumina[g]	1580±100	3600±550 (2420±310)
91% Alumina[h]	2110	1670 (2010)
88% Alumina[i]	not tested	not tested (4290±360)
84% Alumina[j]	not tested	not tested (4980±480)
DENSE INTERMEDIATE-ALUMINA CASTABLES		
57% Alumina, 34% silica[k]	3560±370	3560±420 (4260± 20)
59% Alumina, 30% silica[l]	not tested	3570±560 (not tested)
INSULATING CASTABLES		
54% Alumina, 40% silica[m]	1050±110	480±160 (1340±480)
46% Alumina, 40% silica[n]	not tested	510± 50 (not tested)
PHOSPHATE-BONDED RAMMING MIXES		
96% Alumina[o]	not tested	1600±260 (not tested)
90% Alumina[p]	not tested	1520 (not tested)

[a] Samples were exposed in two pressure vessels. One vessel was a steam generator and tests which specify saturated conditions occurred in this vessel. Samples were exposed both in the vapor and in the liquid in the bottom of the vessel. After placing samples, the vessel was heated to the desired steam pressure and the other gases were then added to obtain the coal gasification atmosphere. The atmosphere composition is (in vol %): 18 CO, 12 CO_2, 24 H_2, 5 CH_4, 40 H_2O and 1 H_2S. The second vessel was connected to the steam generator and after samples were placed and the vessel heated to the test temperature, the vessel was pressurized with the steam/gas mixture from the steam generator. A gaseous flow rate for both vessels was maintained so as to provide one complete change in atmosphere every 24 hours. The samples were exposed at the temperatures indicated in the table at 1000 psia for 60 days.

[b] Modulus of rupture was obtained in 3-point bending tests, ASTM C268-70, span of 2 in., crosshead speed of 0.5 cm/minute. All values are averages for seven specimens except where noted.

[c] All cement-bonded castables were cast in molds either 12 x 3 x 1/2 in. or 3 x 3/4 x 3/4 in., cured 24 hours in air at 100% humidity at ambient temperature, and dried at 230 °F for 24 hours. The 12 x 3 x 1/2 in. slabs were then cut to 3 x 1/2 x 1/2 in. bars. Phosphate-bonded speci-

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(Table Continued)

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EFFECT OF EXPOSURE TO AN UNSATURATED AND STEAM-SATURATED SIMULATED COAL

GASIFICATION ENVIRONMENT[a] ON THE MODULUS OF RUPTURE[b] OF VARIOUS

ALUMINA REFRACTORIES[c][39], Continued

Footnotes continued

mens were prepared by hand ramming in 3 x 3/4 x 3/4 in. molds, immediately
drying for 24 hours at 230 °F and then firing at 1000 °F for 18 hours. All
specimens were stored at 230 °F until tested.

[d] Numbers without parentheses are for samples exposed to saturated vapor.
Adjacent numbers in parentheses are for samples immersed in liquid (water),
average of two samples.

[e] 94% Alumina dense castable, calcium aluminate (79% alumina-18% calcia)
bonded (Greencast-94, A.P. Green; CA-25 Calcium Aluminate Cement, Alcoa).

[f] 93% Alumina dense castable, UMR-1 generic preparation (70% tabular alumina
+ 30% calcium aluminate cement) (T-61 alumina, Alcoa; CA-25 Calcium Aluminate
Cement, Alcoa).

[g] 91% Alumina dense castable, UMR-2 generic preparation (70% tabular alumina
+ 30% calcium aluminate cement) (T-61 alumina, Alcoa; Secar 71(250), 72%
alumina-26% calcia cement, Lone Star Lafarge).

[h] 91% Alumina dense castable, UMR-3 generic preparation (70% tabular alumina
+ 30% calcium aluminate cement) (T-61 alumina, Alcoa; C-3, 72% alumina-25%
calcia cement, Babcock & Wilcox).

[i] 88% Alumina dense castable, UMR-6 generic preparation (UMR-1 generic prep-
aration with 5% SiO_2 added as 99.9% pure bone dry Wedron silica flour).

[j] 84% Alumina dense castable, UMR-7 generic preparation (UMR-1 generic prep-
aration with 10% SiO_2 added as 99.9% pure bone dry Wedron silica flour).

[k] 57% Alumina, 34% silica castable, calcium aluminate (79% alumina-18% calcia)
bonded (RC-3, General Refractories; CA-25 Calcium Aluminate Cement, Alcoa).

[l] 58.6% Alumina, 29.6% silica castable, UMR-5 generic preparation (75% Mulcoa
+ Mulgrain M-60, C-E Minerals; 25% Refcon, 58% alumina-33% calcia cement,
Universal Atlas).

[m] 54% Alumina, 40% silica insulating castable, calcium aluminate (79% alumina-
18% calcia) bonded (Cer-Lite #75, C-E Refractories; CA-25 Calcium Aluminate
Cement, Alcoa).

[n] 46.7% Alumina, 40.2% silica insulating castable (Litecast 60-25, General
Refractories).

[o] 96% Alumina dense phosphate-bonded ramming mix (Greenpak-90P, A.P. Green).

[p] 90% Alumina dense phosphate-bonded ramming mix (Brikram 90R, General
Refractories).

HOT COMPRESSIVE FRACTURE STRAIN[a] FOR VARIOUS CASTABLE REFRACTORIES[26]

Temperature	Hot Compressive Fracture Strain, mil/in.					
	Generic 50% Al$_2$O$_3$ [b]	Commercial 50% Al$_2$O$_3$ [c]	Commercial 42% Al$_2$O$_3$ [d]	Commercial 50% Al$_2$O$_3$ [e]	50% Al$_2$O$_3$ [e] + 310 SS [f]	Modified 90+% Al$_2$O$_3$ [g]
Ambient	10	11	25.5	30	36	3
At 500 °F	12	11	--	30	45	13
1000	--	11	24.0	30	50	13
1250	--	15	--	--	--	--
1500	21	33	31.5	45	60	12
1750	16	--	--	50	60	27
2000	--	--	--	60	60	24

[a]Uniaxial compressive strength was determined using specimens 0.5 in. x 1 in. x 1 in. or 1 in. x 1 in. x 1 in. loaded uniaxially on an Instron tester at a 0.020 in./min. strain rate. Strain was measured with a linear variable differential transducer. Fracture strain was determined from the stress/strain curves by extrapolating the linear portion of the curve back to the abscissa and subtracting this intercept value from the maximum strain value measured.

[b]Generic preparation: 75% calcined kaolin (70% to -20 mesh, 5% ball milled fines with 50% less than 325 mesh), 25% calcium aluminate cement (Casting Grade CA-25, Alcoa); 11% water added.

[c]Commercial lightweight insulating castable, calcium aluminate bonded (Litecast 75-28, General Refractories); 21% water added.

[d]Commercial lightweight insulating castable, 42% alumina (B&W Kaolite 2300 LI, Babcock & Wilcox); 59% water added.

[e]Coarse grain size, high-strength 50% alumina prototype castable (B&W Kaocrete XD50, Mix 36C, Babcock & Wilcox); 7.5% water added.

[f]4 wt% of 310 SS fibers (Ribtec 310), 1 in. long, was added to the Kaocrete XD50.

[g]Modified DOE 90 preparation: 70% tabular alumina (-48 mesh), 5% calcined alumina (-325 mesh), 25% calcium aluminate cement (Casting Grade CA-25, Alcoa); 8.5% water added.

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EFFECT OF TEMPERATURE ON THE MODULUS OF RUPTURE[a] OF PHOSPHATE-BONDED REFRACTORIES[26]

	45% Al_2O_3 [b]	90% Al_2O_3 [c]	90-95% Al_2O_3 [d]
Modulus of Rupture, psi (ambient temperature)			
As Cured	1315± 80	2135± 50	1790±255
Hot Modulus of Rupture, psi			
At 500 °F	1470±210	2155±320	1800±165
1000	2445±270	3210±890	3290±360
1250	2360±260	3810±320	3205±160
1750	2800±105	3830± 50	1320±460
2000	1770± 60	1260±160	165± 90

[a] Modulus of rupture determined following the ASTM C583-67 (1972) procedure. Bars either 1 in. x 1 in. x 6 in. or 1 in. x 2 in. x 6 in. were subjected to 3-point bending, loaded at a rate of 0.02 in./min. to failure. The above values of averages of the results of testing of five samples of each material at each temperature.

[b] Generic preparation of phosphate-bonded ramming mix: 15% calcined alumina, 65% calcined kaolin, 17% ball mill fines, 3% Bentonite (Wyoming), 1% hydrated alumina; bonded with 6% phosphoric acid (85% strength).

[c] Generic preparation of phosphate-bonded ramming mix: 82% tabular alumina, 15% calcined alumina, 3% Bentonite (Wyoming), 1% hydrated alumina; bonded with 6% phosphoric acid (85% strength).

[d] Commercial phosphate-bonded ramming mix: 90 Ram H.S. from C-E Refractories.

EFFECT OF TEMPERATURE ON THE MODULUS OF ELASTICITY[a] OF PHOSPHATE-BONDED REFRACTORIES [26]

Temperature, °F	Hot Modulus of Elasticity,[a] 10^6 psi		
	45% Al_2O_3[b]	90% Al_2O_3[c]	90-95% Al_2O_3[d]
Ambient	0.3±0.2	0.2±0.1	0.1±0
500	0.2±0.1	0.3±0.2	0.3±0.3
1000	0.5±0.3	0.4±0.2	0.6±0.3
1500	0.4±0.2	0.4±0.2	0.4±0.2
1750	0.4±0.2	0.3±0.2	0.1±0.1
2000	0.2±0.1	0.1±0	0.1±0.1

[a]Young's modulus was determined from uniaxial compressive strength test data by calculating the slope of the stress/strain curves in the most linear part of the curves. Specimens 0.5 in. x 1 in. x 1 in. or 1 in. x 1 in. x 1 in. were loaded uniaxially at a 0.020 in./min. strain rate. Values are the average for five samples.

[b]Generic preparation of phosphate-bonded ramming mix: 15% calcined alumina, 65% calcined kaolin, 17% ball mill fines, 3% Bentonite (Wyoming), 1% hydrated alumina; bonded with 6% phosphoric acid (85% strength).

[c]Generic preparation of phosphate-bonded ramming mix: 82% tabular alumina, 15% calcined alumina, 3% Bentonite (Wyoming), 1% hydrated alumina; bonded with 6% phosphoric acid (85% strength).

[d]Commercial phosphate-bonded ramming mix: 90 Ram H.S. from C-E Refractories.

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CREEP DATA (% DEFORMATION)[a] FOR CASTABLE REFRACTORIES AT DIFFERENT STRESS
LEVELS AND TEMPERATURES[26]

Standard 50% Al_2O_3 generic[b]

Time, h	Temperature, °F	At 1500 psi	At 2500 psi
1	75	--	--
3	500	--	0.29%
3	1000	0.25%	0.16
3	1500	0.31	0.41
10	1800	0.65	0.87
Density, lb/ft³			
Starting		136.0	139.8
Post test		132.6	134.9

Modified 50% Al_2O_3 generic[c]

Time, h	Temperature, °F	At 1500 psi		At 2000 psi	At 2500 psi
1	75	0.14%	0.0%	--	0.09%
3	250	--	--	0.08%	--
3	500	0.19	--	0.30	0.27
3	1000	0.25	0.07	0.11	0.22
3	1500	0.39	0.31	0.41	0.94
3	1800	0.55	--	--	1.85[d]
10	1800	--	0.65	0.87	--
10	2000	0.73	--	--	--
Cumulative total		2.25	1.03	1.76	--
Post test results[a]		--	1.0	1.70	--
Density, lb/ft³					
Starting		137.8	--	--	137.2
Post test		133.5	--	--	--

Commercial 50% Al_2O_3 dense castable[e]

Time, h	Temperature, °F	At 1000 psi			At 2000 psi		steam[f]	At 2500 psi	
1	75	0.07%	0.05%	0.06%	0.08%	0.09%	0.23%	0.05%	0.04%
3	500	0.15	--	0.17	0.22	0.12	0.25	0.08	0.08
3	1000	0.08	0.13	0.09	0.14	0.28	0.24	0.11	0.10
3	1500	0.13	0.20	0.21	0.34	0.56	0.46	0.37	0.37
3	1800	0.16	0.30	0.18	0.49	0.88	0.44	0.46	0.46
10	2000	0.53	1.44	0.76	1.95	3.77	2.18	--	2.69
Cumulative total		1.12	2.12	1.47	3.22	5.70	3.55	--	3.74
Post test results[a]		--	--	1.06	2.73	--	--	--	3.45
Density, lb/ft³									
Starting		--	138.9	143.6	--	146.3	--	142.1	--
Post test		--	137.6	140.6	--	142.0	--	138.7	--

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(Table Continued)

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CREEP DATA (% DEFORMATION)[a] FOR CASTABLE REFRACTORIES AT DIFFERENT STRESS
LEVELS AND TEMPERATURES[26], Continued

Commercial 50% Al_2O_3 castable[d] with 310 SS fibers[g]

Time, h	Temperature, °F	with 2% SS		with 4% SS fibers				
		At 1000	2000	1000	1500	(retest)[h]	2000	psi
1	75	0.06%	0.11%	0.08%	0.14%	0.02%	0.29%	0.10%
3	500	0.16	0.38	0.23	0.27	0.02	0.52	0.29
3	1000	0.11	0.22	0.18	0.20	0.14	0.28	0.28
3	1500	0.24	0.50	0.35	0.37	0.27	0.50	0.54
3	1800	0.24	0.60	0.27	0.38	0.27	0.49	0.71
10	2000	1.38	failed	1.68	2.08	1.32	3.64[i]	failed
Cumulative total		5.74	--	2.79	3.44	2.04	5.72	--
Post test results[a]		--	--	1.78	2.29	--	--	--
Density, lb/ft³								
Start		--	137.7	--	145.4	143.4	141.3	143.0
Post test		--	--	--	143.4	--	--	--

Commercial 50% Al_2O_3 castable[d] with 446 SS fibers[g]

Time, h	Temperature, °F	with 4% 446 SS fibers	
		At 1000 psi	At 2000 psi
1	75	0.08%	0.06%
3	500	0.23	0.12
3	1000	0.27	0.18
3	1500	0.64	0.36
3	1800	0.27	0.36
10	2000	1.58	1.44
Cumulative total		3.07	2.52
Post test results[a]		--	--
Density, lb/ft³			
Start		145.9	141.6
Post test		143.6	138.5

Commercial 50% Al_2O_3 insulating castable[j]

Time, h	Temperature, °F	with 21% water added						
		At 700 psi		At 1000 psi			At 1500 psi		
1	75	--	--	0.21%	--	0.22%	--	0.03%	0.19%
3	250	--	0.08%	0.27	--	--	--	--	0.28
3	500	0.31%	0.30	0.39	0.44%	0.39	0.21%	0.22	0.44
3	1000	0.22	0.20	0.29	0.24	0.29	0.26	0.40	0.33
3	1250	--	--	0.36	--	--	--	--	0.50
10	1500	1.51	1.50	2.93	2.08	2.93	3.10	3.81	5.66
Cumulative total		--	2.08	4.45	--	--	--	--	7.40
Post test results[a]		--	1.73	3.79	--	--	--	--	6.40
Density, lb/ft³									
Start		85.5	--	--	84.9	84.7	84.6	85.4	--
Post test		82.2	--	--	81.6	81.7	81.2	81.3	--

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(Table Continued)

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CREEP DATA (% DEFORMATION)[a] FOR CASTABLE REFRACTORIES AT DIFFERENT STRESS LEVELS AND TEMPERATURES[26], Continued

Time, h	Temperature, °F	with 24% water added At____700____			1000	with 26% water added 500		700 psi
		At						
1	75	0.14%	--	--	0.15%	--	--	--
3	500	0.29	--	--	0.32	--	--	--
3	1000	0.15	--	--	0.35	--	--	--
10	1000	--	0.39%	--	--	--	--	0.57%
30	1000	--	--	--	--	0.45%	0.86%	--
3	1500	--	--	--	4.97	--	--	--
10	1500	2.26	--	8.66%	--	--	--	--
Density, lb/ft³								
Start		79.1	75.9	79.5	80.4	78.0	79.0	77.8
Post test		76.2	73.6	76.4	76.4	75.2	76.9	74.6

Commercial 50% Al$_2$O$_3$ insulating castable[j]

Time, h	Temperature, °F	with 9.3% water	with 8.5% water	with 9.0% water
1	75	--	0.03%	--
3	75	--	--	0.16%
3	500	0.57%	0.52	0.54
3	1000	0.22	0.16	0.24
3	1500	0.35	0.36	0.49
3	1800	--	0.58	1.32
7	2000	--	--	3.65
10	2000	2.22	3.62	--
Density, lb/ft³				
Start		169.8	174.4	174.8
Post test		163.7	166.8	162.1

Standard 90+% Al$_2$O$_3$ generic castable[k] — At 3300 psi

Time, h	Temperature, °F	At 1500 psi		At 2000 psi				2500	At 3300 psi	
1	75	0.02%	0.03%	0.08%	--	0.08%	0.01%	0.10%	0.16%	0.08%
3	500	--	--	0.23	--	0.13	0.13	--	0.52	--
3	1000	0.26	0.08	0.08	0.25%	0.12	0.02	0.25	0.16	0.22
3	1500	0.34	0.14	0.20	0.44	0.29	0.16	0.32	0.36	0.40
3	1800	0.90	0.49	0.32	--	0.51	0.29	0.85	0.58	0.87
10	2000	1.70	1.08	1.80	2.4	2.52	1.65	1.54	3.60	2.77
Cumulative total		3.22	1.82	2.71	3.09	3.65	2.26	3.06	5.38	4.34
Post test results[a]		3.54	1.45	2.36	--	--	--	2.33	5.00	3.75
Density, lb/ft³										
Start		--	--	--	169.8	171.8	175.4	--	--	--
Post test		--	--	--	161.7	163.6	168.7	--	--	--

Modified 90+% Al$_2$O$_3$ generic castable[l]

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(Table Continued)

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CREEP DATA (% DEFORMATION)[a] FOR CASTABLE REFRACTORIES AT DIFFERENT STRESS
LEVELS AND TEMPERATURES[26], Continued

		Phosphate-Bonded Ramming Mixes				
		45% Al_2O_3[m]		90% Al_2O_3[n]		Commercial 90% Al_2O_3[o]
Time, h	Temper- ature, °F	At 1000	2000	1000		1000 psi
1	75	0.03%	0.01%	0.02%	0.04%	0.02%
3	500	--	--	0.01	0.01	0.01
3	1000	0.06	0.24	0.01	0.01	0
3	1500	0.04	0.13	0.04	0.01	0.04
3	1800	--	--	0.76	--	0.76
10	1800	0.22	0.49	--	0.53	--
12 minutes	2000	--	--	1.26[p]	--	1.68[p]
Density, lb/ft^3						
Start		145.0	145.0	187	186.1	181
Post test		143.3	143.3	--	184.9	--

[a]Samples were 6 in. long with 2 in. x 1 in. cross sections for stress levels
less than or equal to 2000 psi and with 1 in. x 1 in. cross sections for
stress levels above 2000 psi. Strain was monitored continuously with a
linear variable differential transducer. Samples were tested by loading
uniaxially to 75% or less of their ultimate strength, monitoring strain, un-
loading and continuing to monitor strain. Three to ten hour tests were found
adequate. One sample was tested at one stress level from ambient to higher
temperatures in a stepwise manner (see Section B.3.2.40). The sample was
loaded at ambient temperature for one hour and unloaded before the furnace
was heated. Then the sample was heated to the first test temperature at
250 °F/h, held one hour, loaded as quickly as possible to the desired
stress, held 3 hours, unloaded as quickly as possible, heated to the next
test temperature and the cycle repeated. At the top temperature a 10-hour
hold was used. Post test deformation is generally lower than the cumula-
tive total, thought to be due mainly to creep recovery during unloading.
Data given for the same stress levels are for different batches of the same
material.

[b]Standard generic preparation: 75% calcined kaolin (60% to -20 mesh, 15% ball
milled fines with 50% less than 325 mesh), 25% calcium aluminate cement
(Casting Grade CA-25, Alcoa).

[c]Modified generic preparation: 75% calcined kaolin (70% 6 to -20 mesh, 5%
ball milled fines with 50% less than 325 mesh), 25% calcium aluminate cement
(Casting Grade CA-25, Alcoa).

[d]Failed at 1.25 hours.

[e]Coarse grain size, high-strength 50% alumina prototype castable (B&W Kaocrete
XD50, Mix 36C, Babcock & Wilcox).

[f]Specimens had been subjected to ∿150 psig steam at 1200 °F.

[g]1-in. long fibers of 310 SS (Ribtec 310) and 446 SS (Ribtec 446) were added

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(Table Continued)

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CREEP DATA (% DEFORMATION)[a] FOR CASTABLE REFRACTORIES AT DIFFERENT STRESS
LEVELS AND TEMPERATURES[26], Continued

Footnotes continued

to the mix before casting in amounts of 2 and 4 weight percent.

[h]Specimen was retested.

[i]Failed at 3.64%.

[j]Commercial lightweight insulating castable, calcium aluminate bonded
(Litecast 75-28, General Refractories); specimen prepared with varying
amounts of water.

[k]DOE 90 generic preparation: 65% tabular alumina (6 to - 20 mesh), 10% cal-
cined alumina (-325 mesh), 25% calcium aluminate cement (Casting Grade
CA-25, Alcoa); specimens prepared with varying amounts of water.

[l]Modified DOE 90 preparation: 70% tabular alumina (-48 mesh), 5% calcined
alumina (-325 mesh), 25% calcium aluminate cement (Casting Grade CA-25,
Alcoa).

[m]Generic preparation of phosphate-bonded ramming mix: 15% calcined alumina,
65% calcined kaolin, 17% ball mill fines, 3% Bentonite (Wyoming), 1%
hydrated alumina; bonded with 6% phosphoric acid (85% strength).

[n]Generic preparation of phosphate-bonded ramming mix: 82% tabular alumina,
15% calcined alumina, 3% Bentonite (Wyoming), 1% hydrated alumina; bonded
with 6% phosphoric acid (85% strength).

[o]Commercial phosphate-bonded ramming mix: 90 Ram H.S. from C-E Refractories.

[p]Specimens failed in 12 minutes.

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UNIT CREEP DATA[a] FOR CASTABLE REFRACTORIES[26]
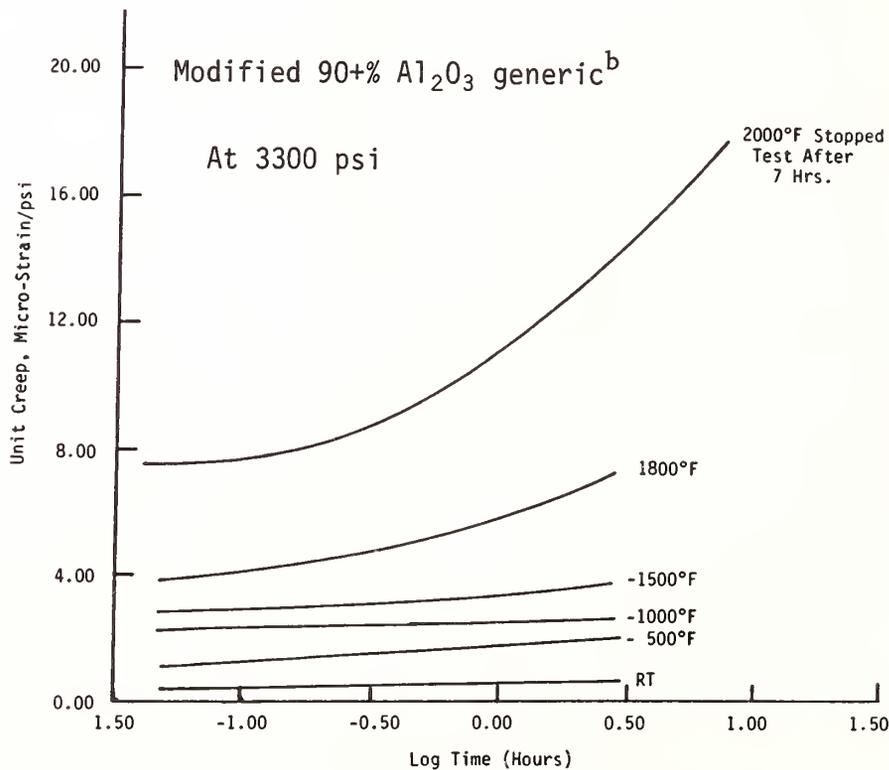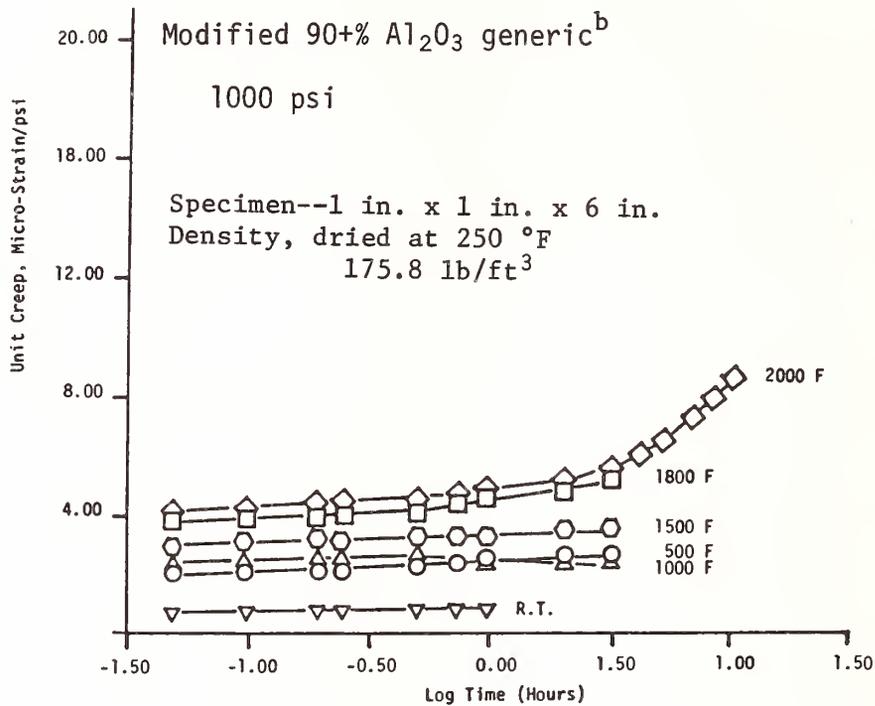
(Data Continued)

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UNIT CREEP DATA[a] FOR CASTABLE REFRACTORIES[26], Continued

Commercial 50% Al_2O_3 dense castable[c]

Commercial 50% Al_2O_3 dense castable[c]

50% Al_2O_3 generic dense castable[e]

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(Data Continued)

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UNIT CREEP DATA[a] FOR CASTABLE REFRACTORIES[26], Continued

50% Al_2O_3 commercial insulating castable[f]

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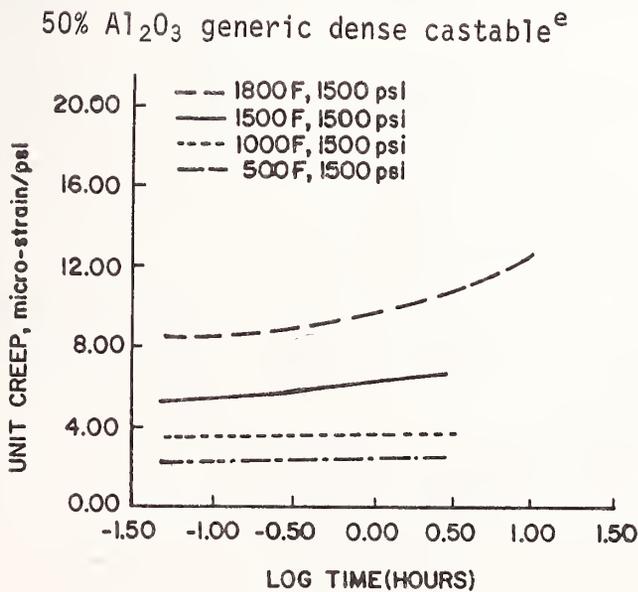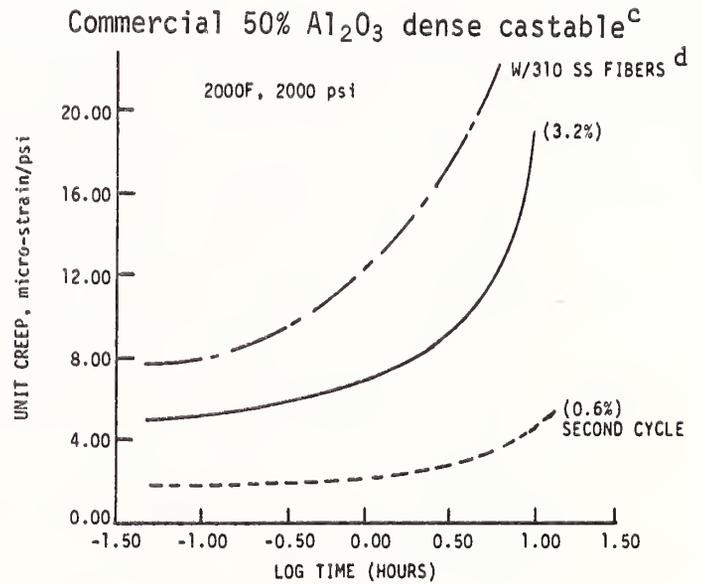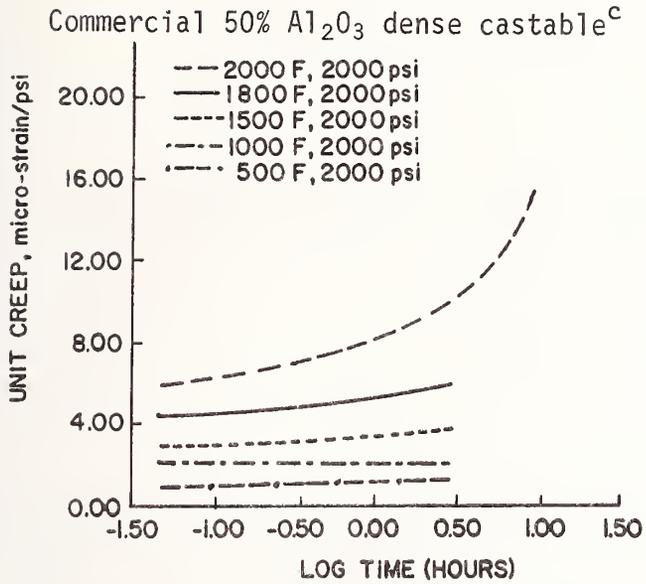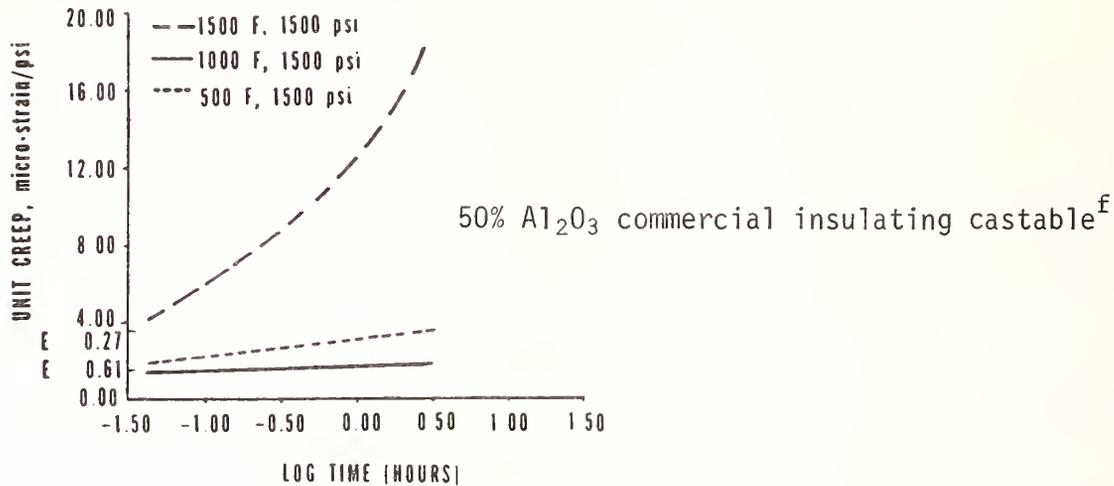
[a] Samples were 6 in. long with 2 in. x 1 in. cross sections for stress levels less than or equal to 2000 psi and 1 in. x 1 in. cross sections for stress levels above 2000 psi. Strain was monitored continuously with a linear variable differential transducer. Samples were loaded uniaxially to 75%, or less, of their ultimate strength, monitoring the strain, unloading and continuing to monitor the strain. Three to ten hour tests were found adequate. One sample was tested at one stress level from ambient to higher temperatures in a stepwise manner (see Section B.3.2.40). Samples were loaded at ambient temperature for one hour and unloaded before the furnace was heated. Then the samples were heated to the first test temperature at 250 °F/h, held one hour, loaded as quickly as possible to the desired stress, held 3 hours, unloaded as rapidly as possible, heated to the next test temperature and the cycle repeated. At the top temperature a 10-hour hold was used. Data were transformed into unit creep (strain/psi stress) versus log time plots.

[b] Modified DOE 90 preparation: 70% tabular alumina (-48 mesh), 5% calcined alumina (-325 mesh), 25% calcium aluminate cement (Casting Grade CA-25, Alcoa); 9.0% water added.

[c] Coarse grain size, high-strength 50% alumina prototype castable (B&W Kaocrete XD50, Mix 36C, Babcock & Wilcox); 7.5% water added.

[d] 4 wt% of 310 SS fibers (Ribtec 310), 1 in. long, was added to the Kaocrete.

[e] Generic preparation: 75% calcined kaolin (70% to -20 mesh, 5% ball milled fines with 50% less than 325 mesh), 25% calcium aluminate cement (Casting Grade CA-25, Alcoa); 11% water added.

[f] Commercial lightweight insulating castable, calcium aluminate bonded (Litecast 75-28, General Refractories); 21% water added.

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STRESS-STRAIN DATA[a] FOR SEVERAL CASTABLE REFRACTORIES[26]
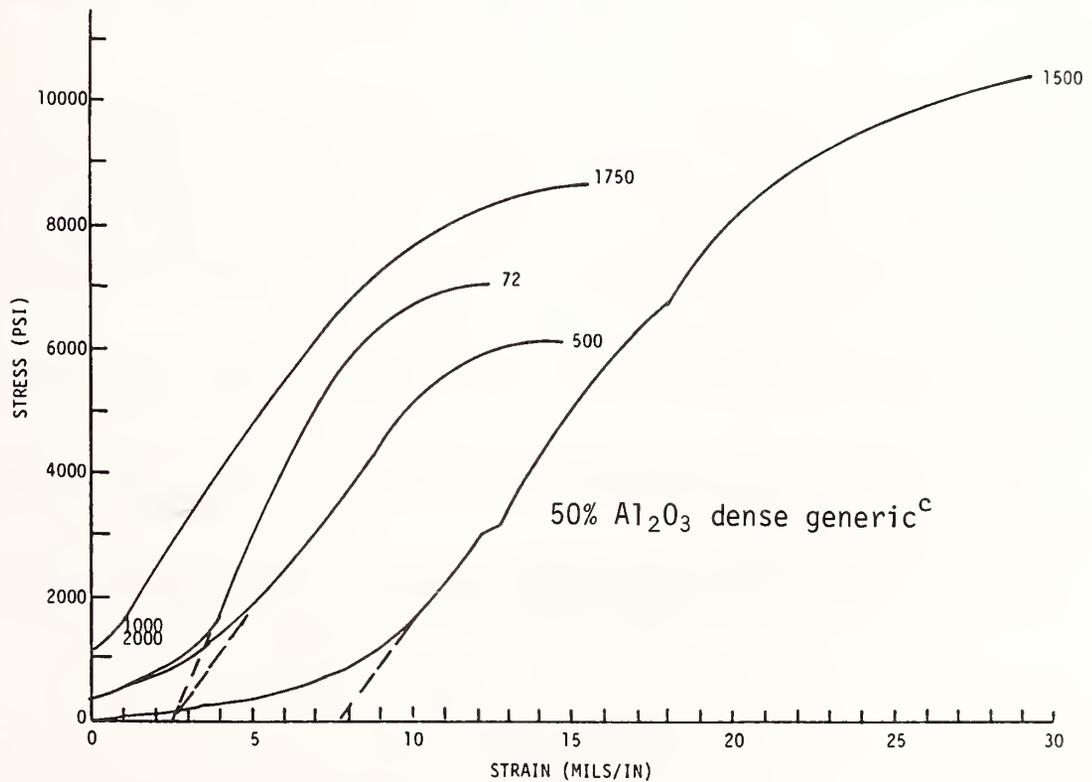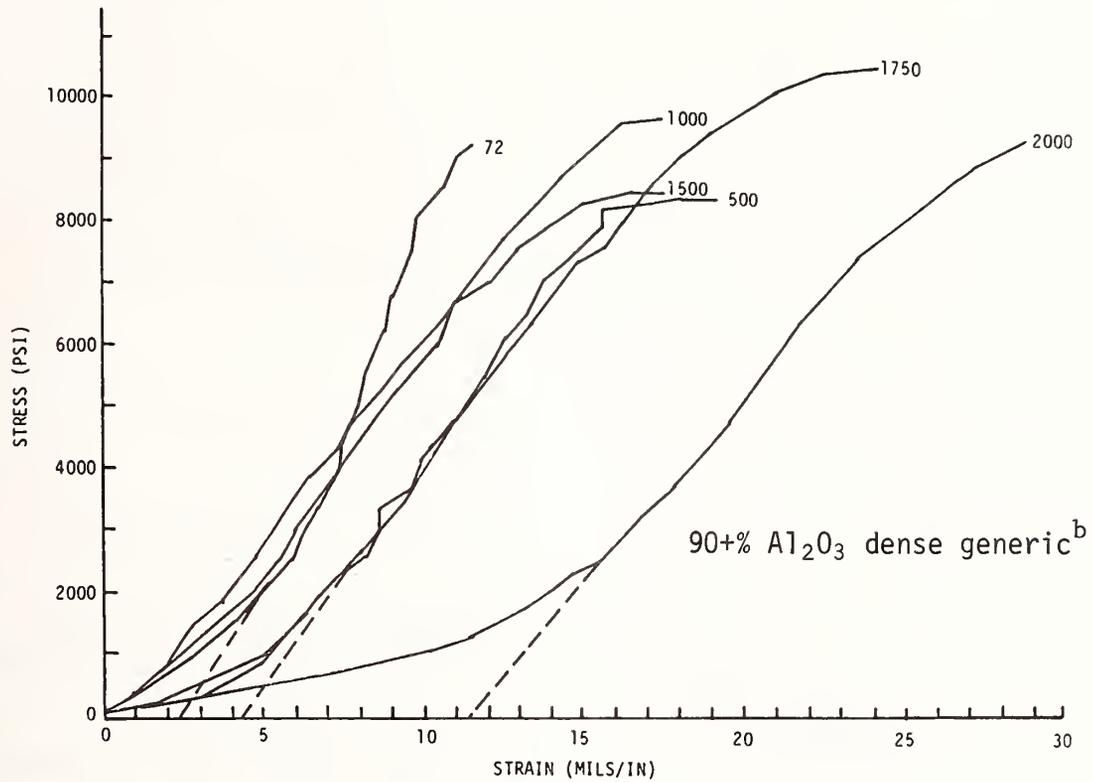
(Data Continued)

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STRESS-STRAIN DATA[a] FOR SEVERAL CASTABLE REFRACTORIES[26], Continued
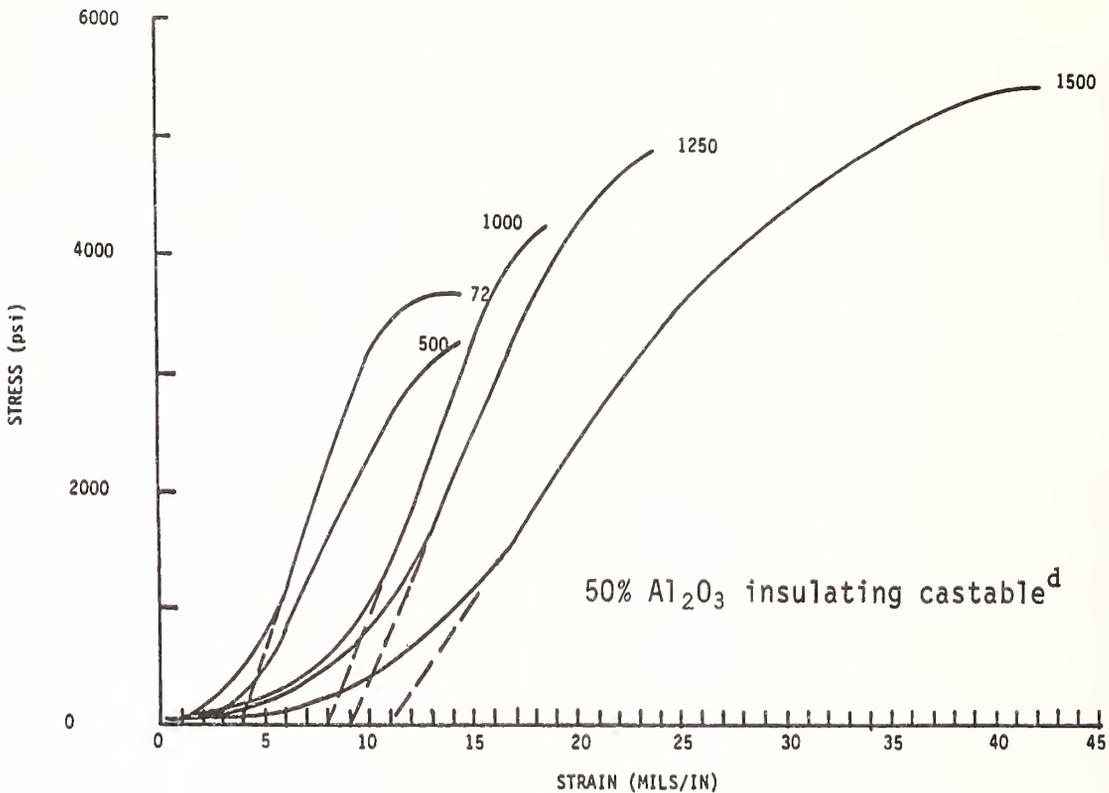
<hr>

[a]Uniaxial compressive strength was determined using specimens 0.5 in. x 1 in.
x 1 in. or 1 in. x 1 in. x 1 in. loaded uniaxially at a 0.020 in./min.
strain rate. After loading the samples in the test furnace they were heated
at 250 °F/h to the test temperature. Strain was monitored with a linear
variable differential transducer. Stress versus strain is plotted for a
variety of temperatures (temperatures are °F).

[b]DOE 90 generic preparation : 65% tabular alumina (6 to -20 mesh), 10% cal-
cined alumina (-325 mesh), 25% calcium aluminate cement (Secar 250, Lone
Star Lafarge).

[c]Generic preparation: 75% calcined kaolin (60% 6 to -20 mesh, 15% ball milled
fines with 50% less than 325 mesh), 25% calcium aluminate cement (Casting
Grade CA-25, Alcoa).

[d]Commercial lightweight insulating castable, calcium aluminate bonded
(Litecast 75-28, General Refractories).

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STRESS RELAXATION DATA[a] FOR ALUMINA CASTABLE REFRACTORIES[26]

90+% Al_2O_3 Generic[b]

50% Al_2O_3 Generic[c]

Commercial 50% Al_2O_3[d]

- -
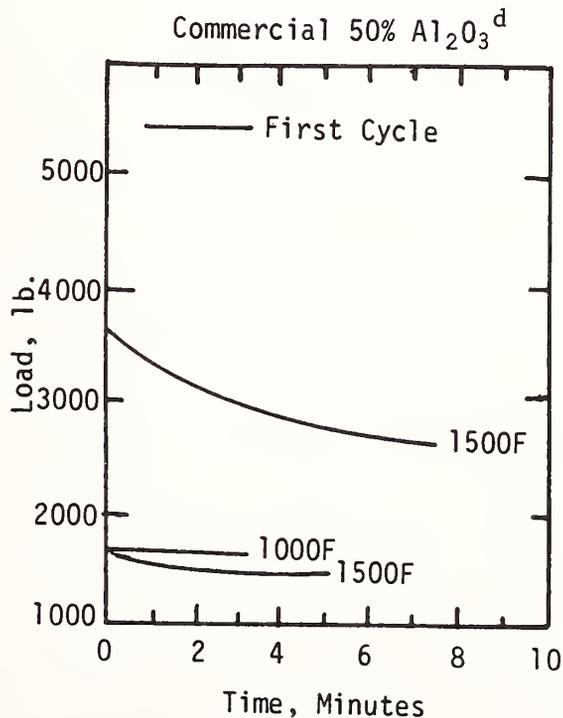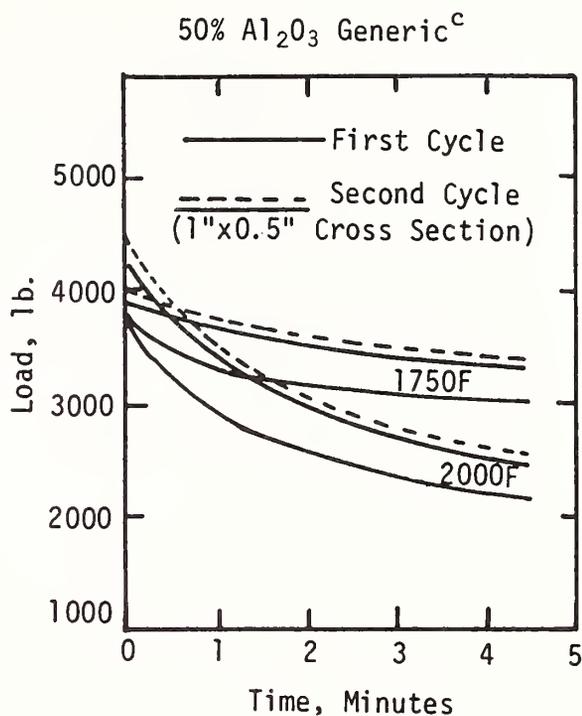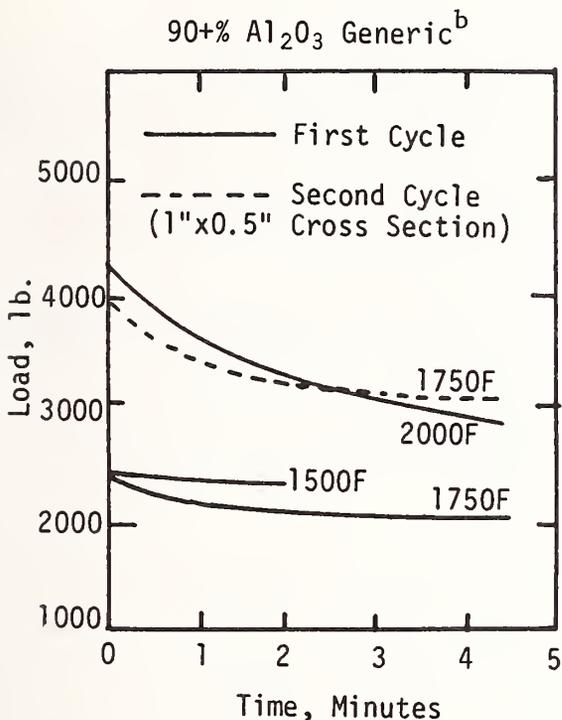
(Data Continued)

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STRESS RELAXATION DATA[a] FOR ALUMINA CASTABLE REFRACTORIES[26], Continued

Footnotes _____

[a]Stress relaxation data are for uniaxially compressed specimens. Specimens
were 0.5 in. x 1 in. x 1 in. or 1 in. x 1 in. x 1 in. loaded uniaxially at
a 0.020 in./min. strain rate. After loading the samples in the test furnace
they were heated at 250 °F/h to the test temperature.

[b]Modified DOE 90 preparation: 70% tabular alumina (-48 mesh), 5% calcined
alumina (-325 mesh), 25% calcium aluminate cement (Casting Grade CA-25,
Alcoa).

[c]Generic preparation: 75% calcined kaolin (60% to -20 mesh, 15% ball milled
fines with 50% less than 325 mesh), 25% calcium aluminate cement (Casting
Grade CA-25, Alcoa).

[d]Commercial lightweight insulating castable, calcium aluminate bonded
(Litecast 75-28, General Refractories).

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EFFECT OF FIRING TEMPERATURE[a] ON THE COLD FLEXURAL STRENGTH OF VARIOUS REFRACTORIES[88]

Refractory[c]	Water Content %	Cold Flexural Strength,[b] psi				
		240 °F	500 °F	1000 °F	1500 °F	2000 °F
90-95% Al_2O_3 dense castable (B&W Kao-Tab 95)	9.25	1339	911.0	967.2	1056	723.3
	9.5	1291	903.7	920.0	1035	769.7
	9.4-10.4[d]	1850	--	1400	1000	1200
54% Al_2O_3 lightweight insulating castable (Litecast 75-28)	25.0	409.9	199.7	235.7	241.0	118.3
	24.0	426.8	230.8	264.2	284.5	146.3
	19.5-23.6[d]	300-400	--	--	--	175-250
50-60% Al_2O_3 low-iron castable (Steelkon)	10.75	1088	540.6	508.8	505.8	386.4
	9.4+[d]	600-900	--	--	400-600	350-550
90% Al_2O_3 phosphate-bonded ramming mix (90 Ram H.S.)	as received[e]	1887	1670	2276	2965	3503
	--[d]	1080	--	--	2250	--
45-60% Al_2O_3 pitch-impregnated brick (Ufala TI)	--	2644[f]	--	--	--	--
	--[d]	1900-2600	--	--	--	--
50 lb/ft³ perlite-containing insulating castable (Kaolite 2300 LI)	74.0	54.4	--	48.9	--	58.4
	62.0[d]	150	--	140	110	190

[a] Specimens were heated and cooled at 100 °F/h with a 5-hour soak at the maximum temperature.

[b] Determined by ASTM test method C-133-72, except that the specimen size was nominal 2 x 2 x 7 in. (bricks sectioned lengthwise); loading rate 0.05 in./min. Values are averages of 5 determinations except for manufacturer's data (see footnote d) and brick specimens (see footnote f).

[c] Castable specimens were fabricated by "spade casting", moist-curing overnight at ambient temperature, drying overnight at ambient temperature, drying 8 hours at 140 °F and overnight at 240 °F. Ramming mix specimens were formed by air-ramming, air-drying overnight at ambient temperature, heating at ∿10 °F/h to 140 °F, holding overnight, heating at 10 °F/h to 200 °F, holding overnight, heating to 240 °F and holding overnight.

[d] Manufacturer's data.

[e] Approximately 6.5% based on weight loss measurements.

[f] Average value for 6 determinations; cut surface of brick was in tension for three specimens, in compression for three other specimens.

COMPRESSIVE STRENGTHS[a] OF IRON-DOPED REFRACTORIES[b] EXPOSED[c] TO VARIOUS CARBON MONOXIDE GAS MIXTURES[27]

Feed Gas Composition: 99.99% CO
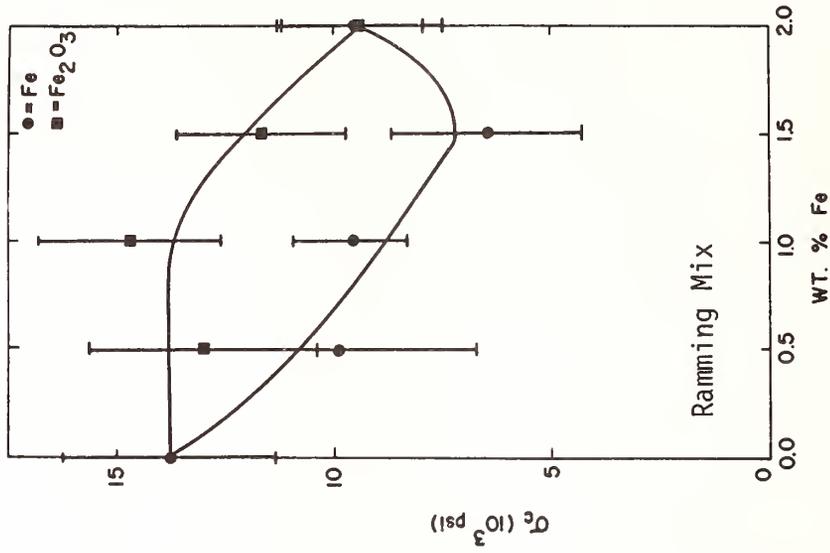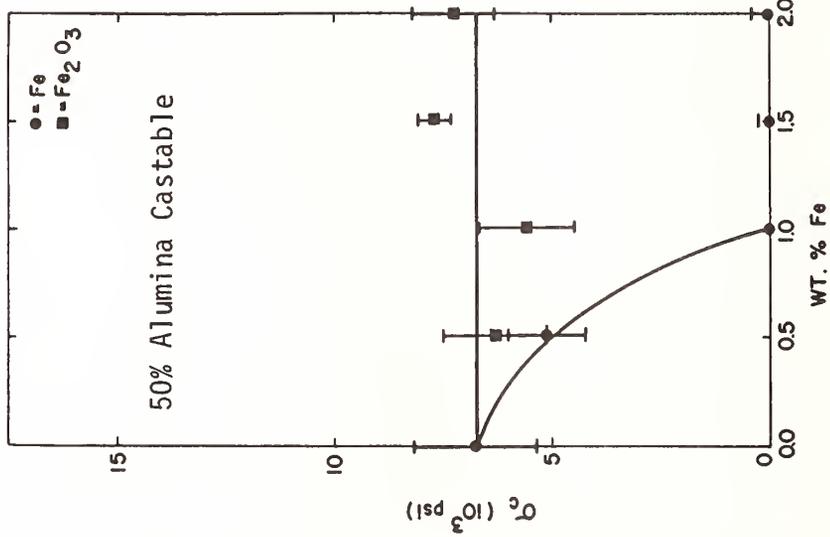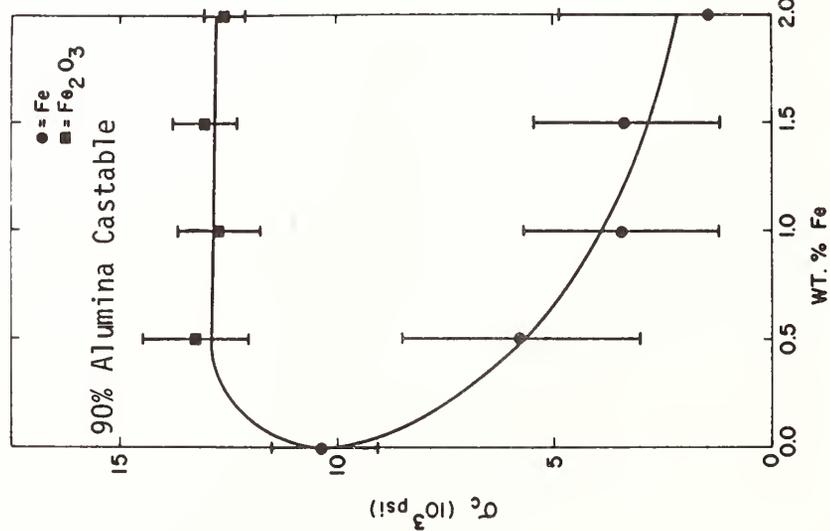
(Data Continued)

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COMPRESSIVE STRENGTHS[a] OF IRON-DOPED REFRACTORIES[b] EXPOSED[c] TO VARIOUS CARBON MONOXIDE GAS MIXTURES[27], Continued

Feed Gas Composition: 95% CO-5% CO_2
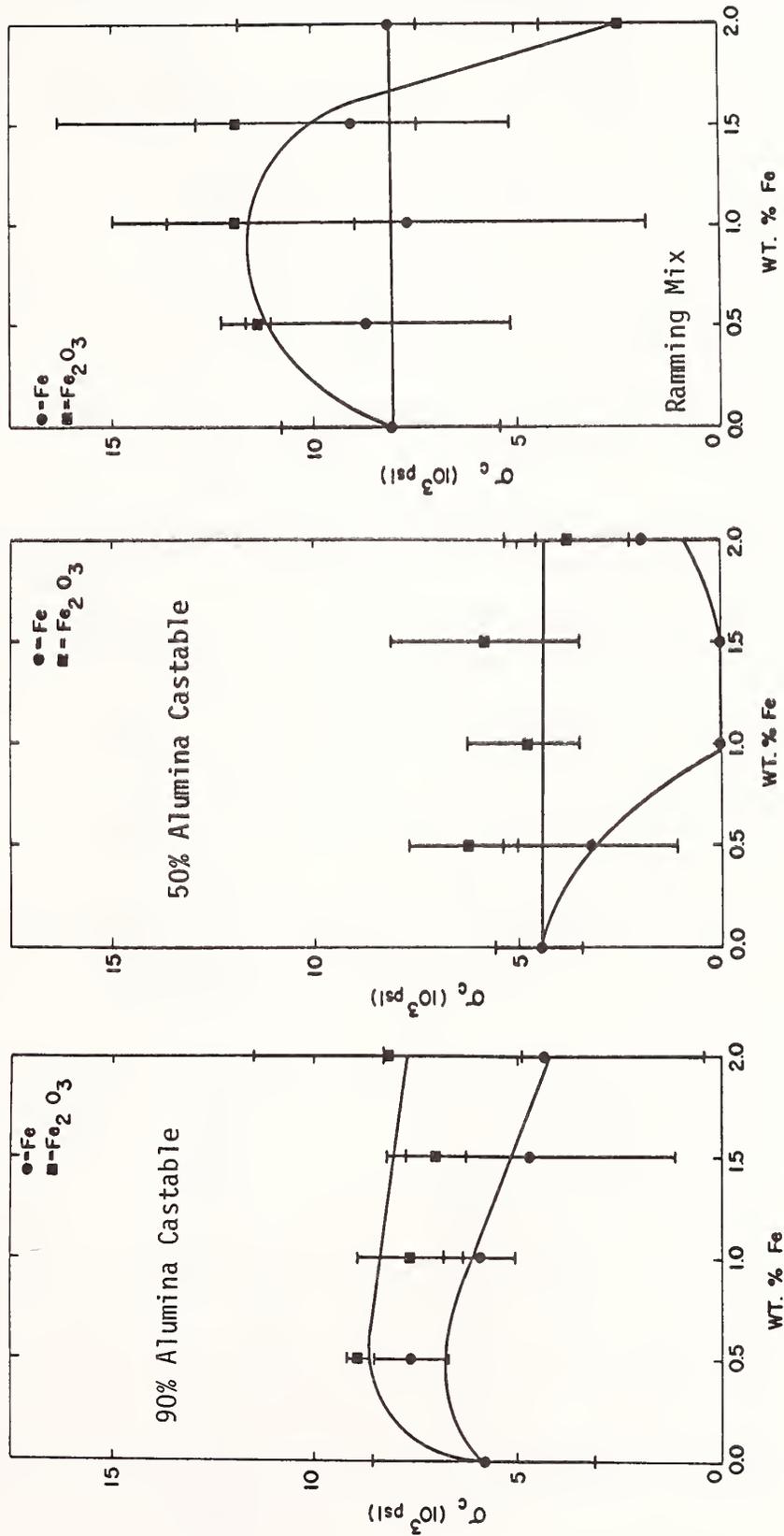
(Data Continued)

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COMPRESSIVE STRENGTHS[a] OF IRON-DOPED REFRACTORIES[b] EXPOSED[c] TO VARIOUS CARBON MONOXIDE
GAS MIXTURES[27], Continued

Feed Gas Composition: 85% CO–15% CO_2

(Data Continued)

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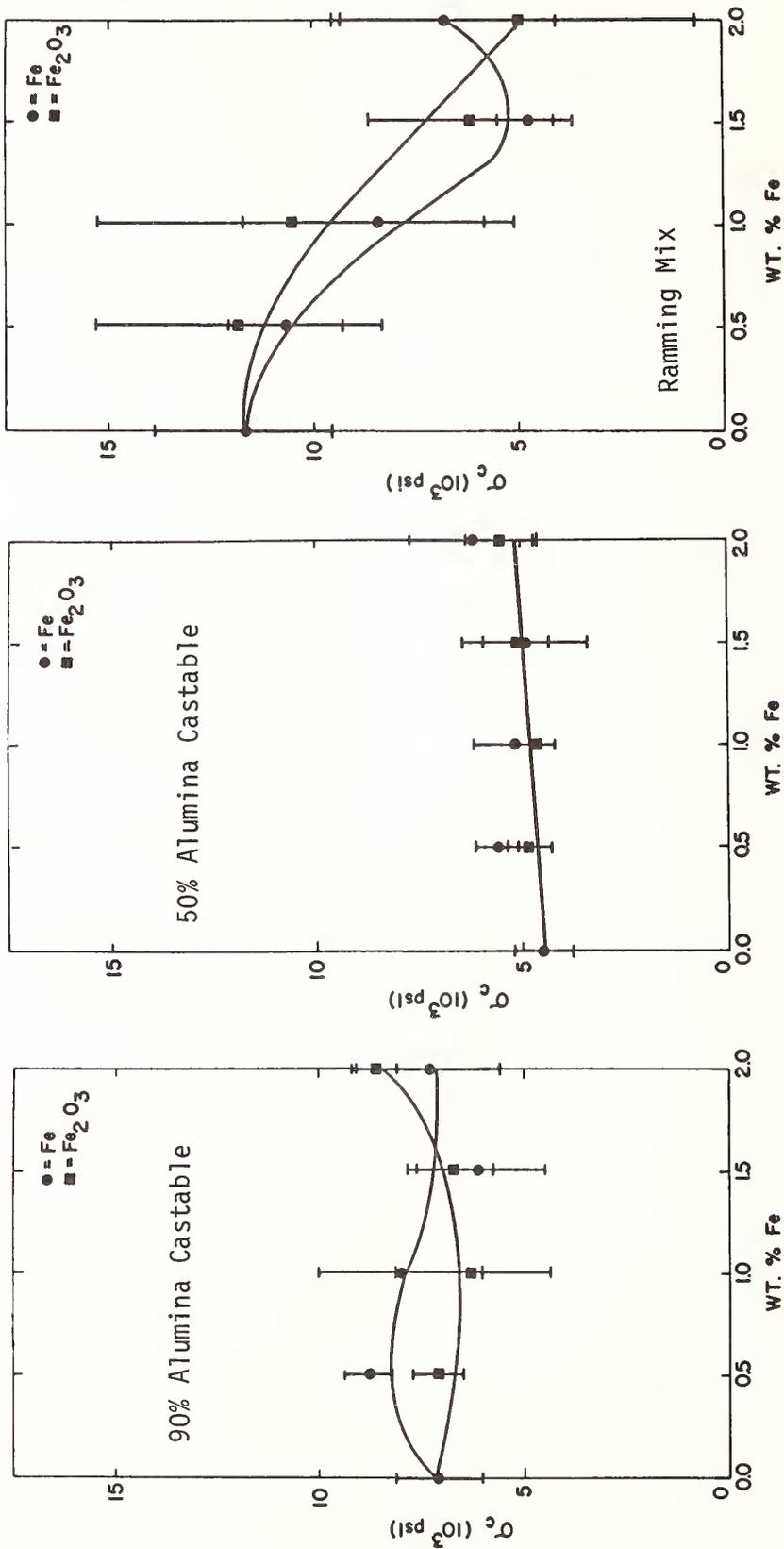
COMPRESSIVE STRENGTHS[s] OF IRON-DOPED REFRACTORIES[b] EXPOSED[c] TO VARIOUS CARBON MONOXIDE GAS MIXTURES[27], Continued

Feed Gas Composition: 99.8% CO—0.2% NH_3
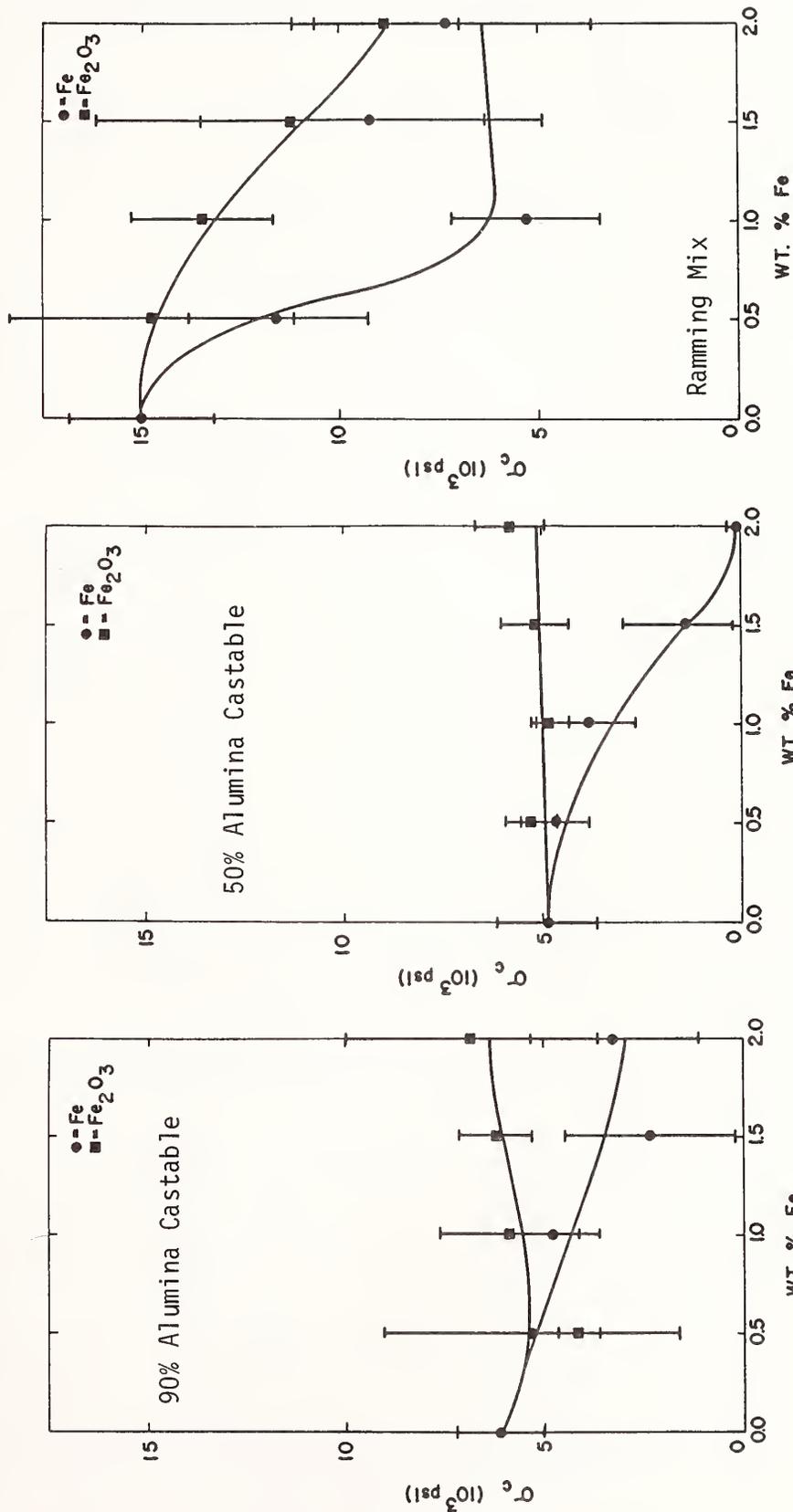
(Data Continued)

COMPRESSIVE STRENGTHS[a] OF IRON-DOPED REFRACTORIES[b] EXPOSED[c] TO VARIOUS CARBON MONOXIDE
GAS MIXTURES[27], Continued

Feed Gas Composition: 99.2% CO-0.8% NH_3
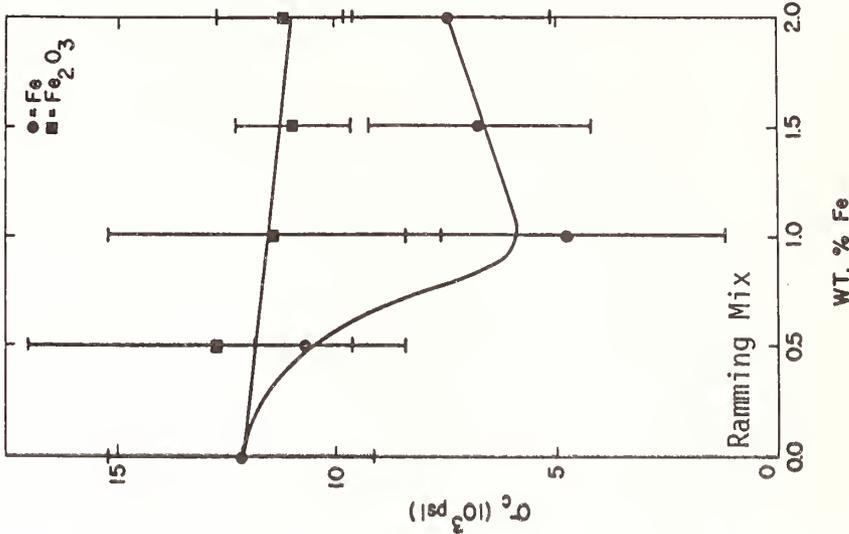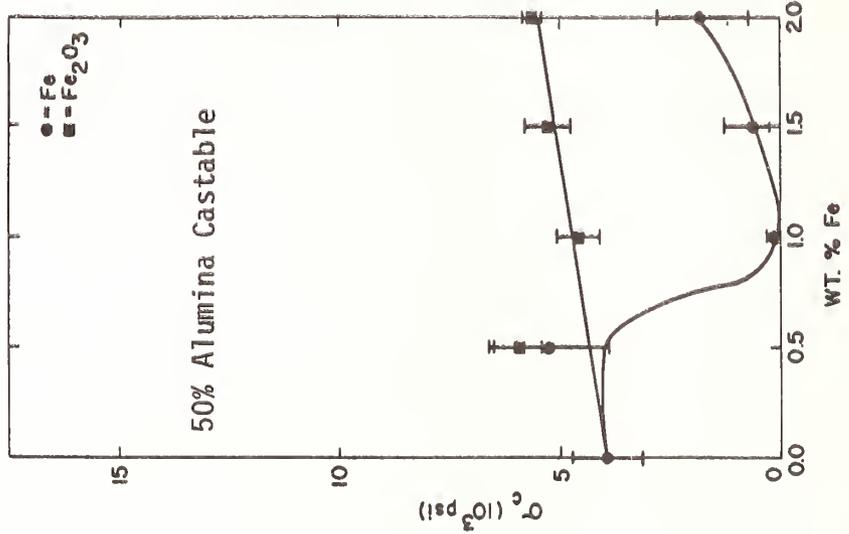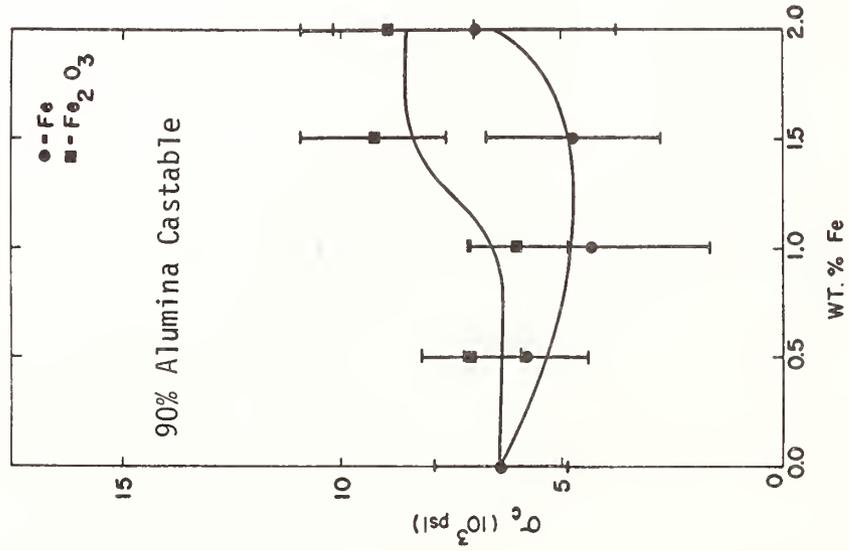
(Data Continued)

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COMPRESSIVE STRENGTHS[a] OF IRON-DOPED REFRACTORIES[b] EXPOSED[c] TO VARIOUS CARBON MONOXIDE

GAS MIXTURES[27], Continued

Feed Gas Composition: 99.8% CO-0.2% H_2S
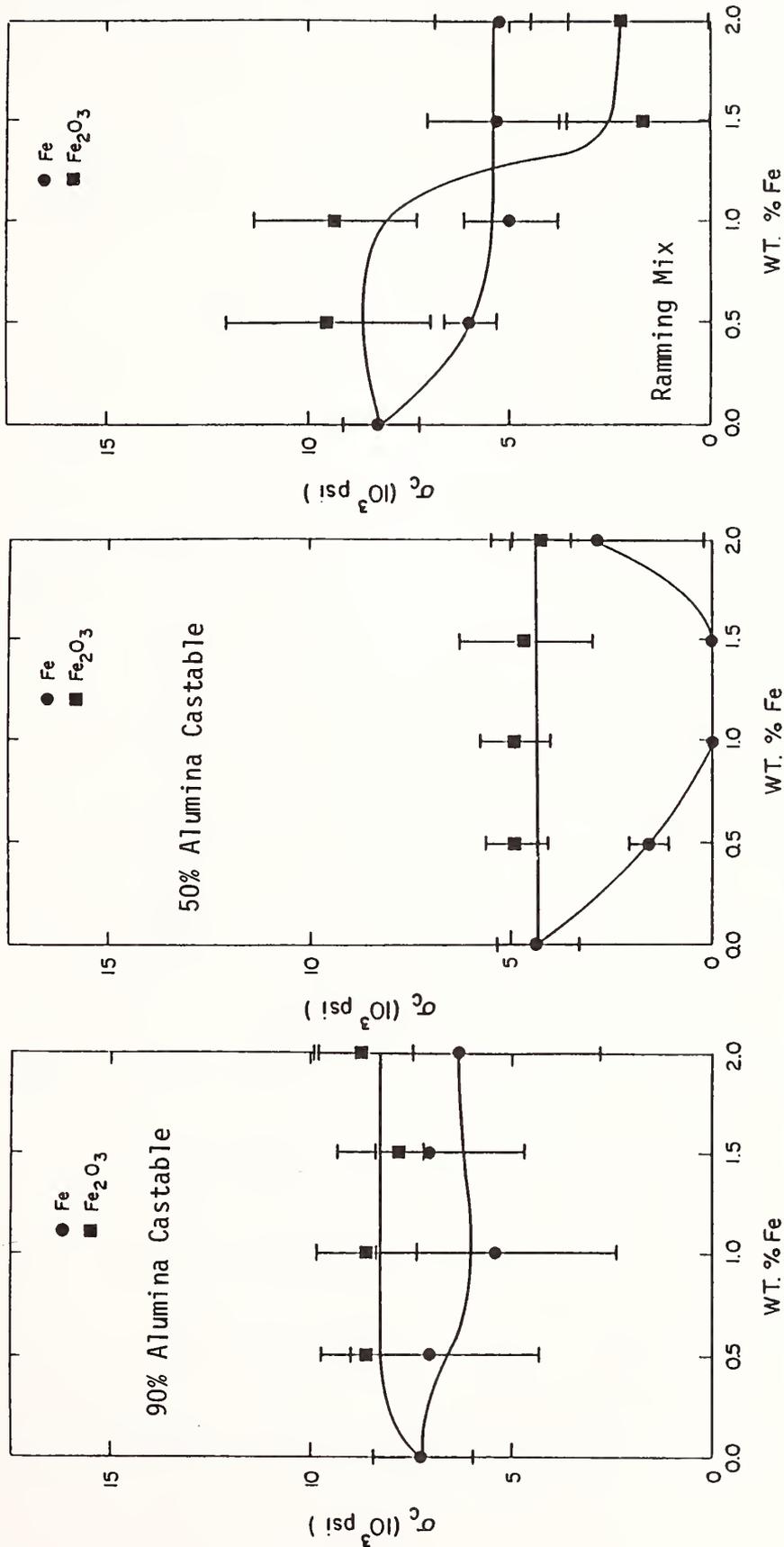
(Data Continued)

COMPRESSIVE STRENGTHS[a] OF IRON-DOPED REFRACTORIES[b] EXPOSED[c] TO VARIOUS CARBON MONOXIDE
GAS MIXTURES[27], Continued

Feed Gas Composition: 99.2% CO-0.8% H_2S

(Data Continued)

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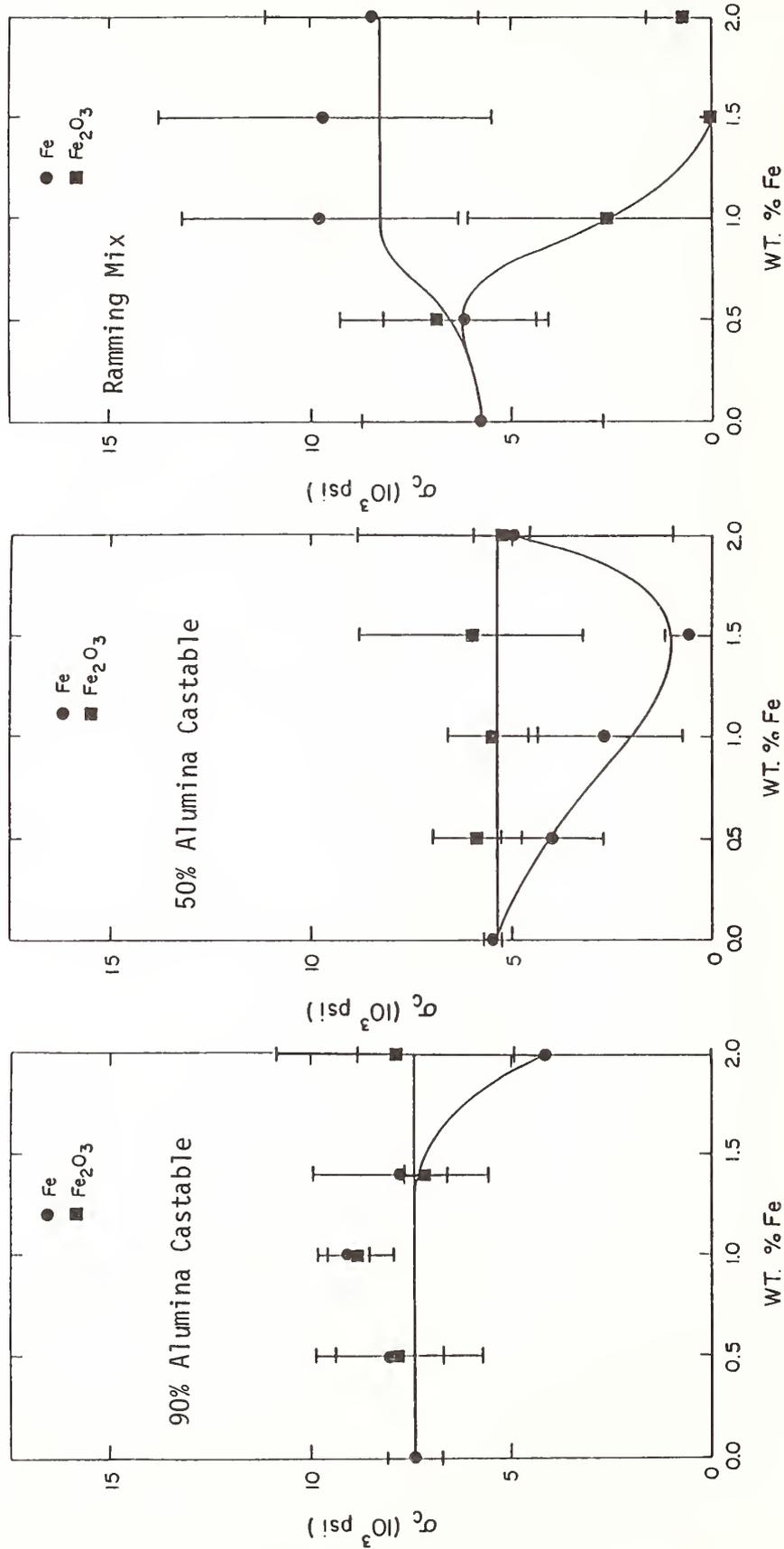
COMPRESSIVE STRENGTHS[a] OF IRON-DOPED REFRACTORIES[b] EXPOSED[c] TO VARIOUS CARBON MONOXIDE GAS MIXTURES[27], Continued
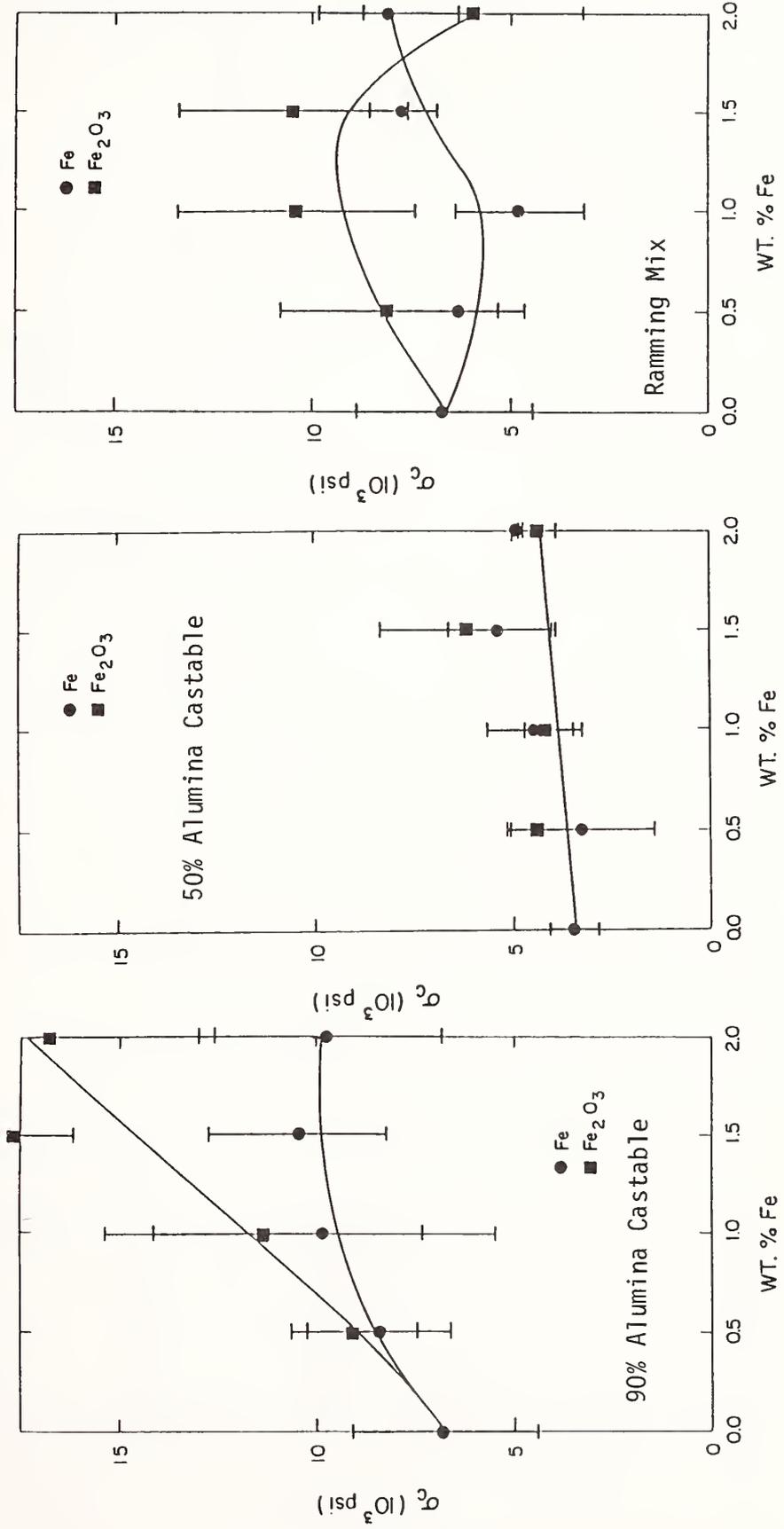
Feed Gas Composition: 80% CO-20% H_2O

(Data Continued)

COMPRESSIVE STRENGTHS[a] OF IRON-DOPED REFRACTORIES[b] EXPOSED[c] TO VARIOUS CARBON MONOXIDE
GAS MIXTURES[27], Continued

Feed Gas Composition: 60% CO–40% H_2O

(Data Continued)

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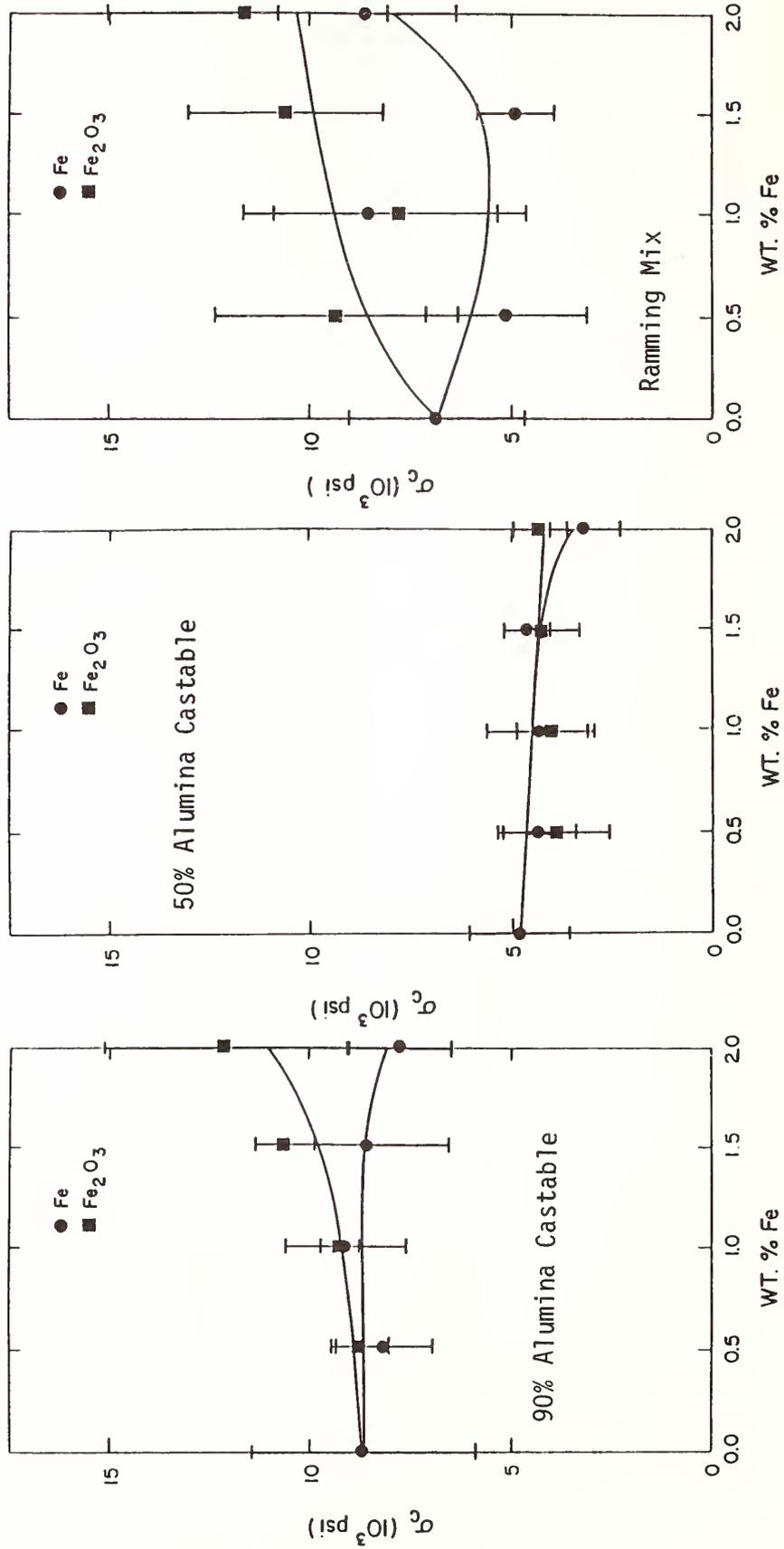
COMPRESSIVE STRENGTHS[a] OF IRON-DOPED REFRACTORIES[b] EXPOSED[c] TO VARIOUS CARBON MONOXIDE

GAS MIXTURES[27], Continued

Feed Gas Compositions: 80% CO-20% H_2
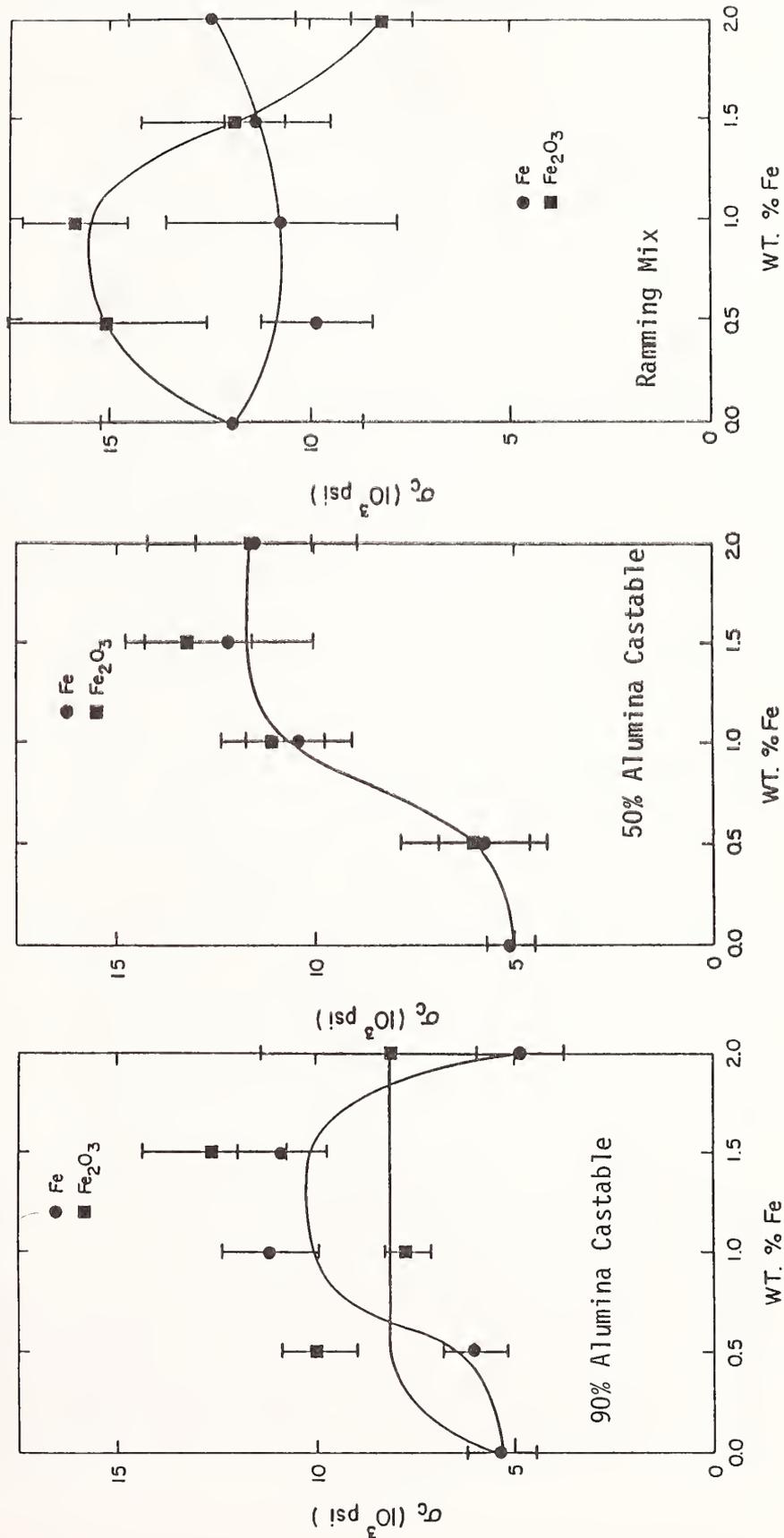
(Data Continued)

COMPRESSIVE STRENGTHS[a] OF IRON-DOPED REFRACTORIES[b] EXPOSED[c] TO VARIOUS CARBON MONOXIDE

GAS MIXTURES[27], Continued

Feed Gas Composition: 60% CO–40% H$_2$

(Data Continued)

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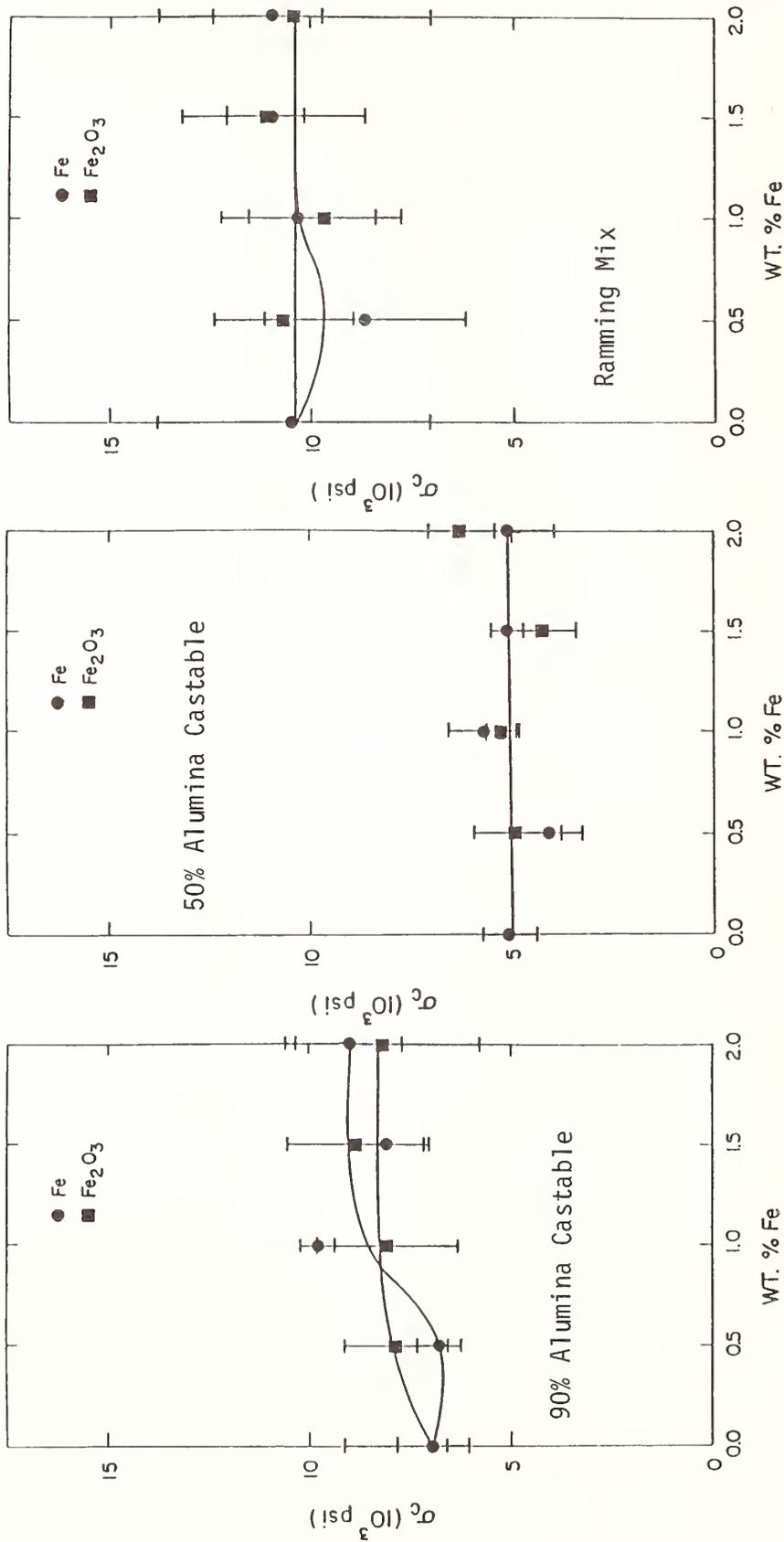
COMPRESSIVE STRENGTHS[a] OF IRON-DOPED REFRACTORIES[b] EXPOSED[c] TO VARIOUS CARBON MONOXIDE
GAS MIXTURES[27], Continued

Footnotes

[a]The compressive strength was measured on a universal testing machine with a crosshead speed of 0.1 in./min. See Section B.3.2.54 for these same data in tabular form.

[b]Refractories were doped with iron added either as metallic iron (Fe) or as hematite (Fe_2O_3). The refractories were DOE-specified generic preparations. The 90% alumina castable was prepared from 65 wt% tabular alumina (25 wt% 6 to 10 mesh, 20 wt% 10 to 20 mesh, 20 wt% less than 20 mesh), 10 wt% calcined alumina (less than 325 mesh), 25 wt% calcium aluminate cement (Casting Grade CA-25 cement), all Alcoa materials. Materials were dry mixed for 2 min, then water added with continued mixing to ball-in-hand consistency (∿605 ml); refractory was then poured into 2 in. x 2 in. x 2 in. aluminum molds, vibrated for 5 min, sealed in plastic for 24 h, then fired for 5 h in air at 1100 °C. The 50% alumina castable was prepared from 75 wt% calcined kaolin (25 wt% 6 to 10 mesh, 20 wt% 10 to 20 mesh, 15 wt% less than 20 mesh, 20 wt% less than 200 mesh), 25 wt% calcium aluminate cement (Casting Grade CA-25, Alcoa). Samples were prepared in the same way as the 90% alumina castable except that the water used was ∿700 ml. The ramming mix was prepared from 82 wt% tabular alumina (30 wt% 6 to 10 mesh, 20 wt% 10 to 20 mesh, 15 wt% less than 20 mesh, 17 wt% less than 48 mesh), 15 wt% calcined alumina (less than 325 mesh), 3 wt% bentonite. To this mix was added 1 wt% hydrated alumina, 6 wt% phosphoric acid (85%) and 0.25 wt% water. Dry materials were mixed and then hand-mixed in plastic bags with the liquids; mixture was aged for 18 h; samples were formed by die pressing at 500 psi, cured at 250 °C in air for 12 h, then fired at 1100 °C for 5 h.

[c]Exposure to the gas mixtures was for 100 hours at 500 °C and 1 atmosphere gas pressure.

COMPARISON OF STRENGTH INDEX[a] AND SPALLING DAMAGE[b] FOR REFRACTORIES[c] DOPED WITH METALLIC IRON AFTER EXPOSURE[d] TO CO GAS MIXTURES[27]

Gas Composition	Strength Index[a]	No. of Samples in Each Damage Category					Total No. Samples	Spalling Index[b]
		No Damage 1.00	Cracking 0.75	Light Spalling 0.50	Heavy Spalling 0.25	Complete Disintegration 0.0		
		—90% ALUMINA CASTABLE[e]—						
99.9% CO	0.336	4	0	0	13	3	20	0.362
99.8%CO-0.2%NH$_3$	0.637	10	0	6	0	0	16	0.812[f]
99.2%CO-0.8%NH$_3$	0.855	11	0	3	2	0	16	0.812[f]
95%CO-5%CO$_2$	0.971	13	0	3	0	0	16	0.906
85%CO-15%CO$_2$	1.074	13	0	3	0	0	16	0.906
		—50% ALUMINA CASTABLE[g]—						
99.9%CO	0.197	4	0	1	1	14	20	0.238
95%CO-5%CO$_2$	0.287	5	1	0	2	8	16	0.391
99.8%CO-0.2%NH$_3$	0.508	8	4	0	2	2	16	0.719
85%CO-15%CO$_2$	1.217	15	1	0	0	0	16	0.984
		—90% ALUMINA RAMMING MIX[h]—						
99.8%CO-0.2%NH$_3$	0.554	8	0	4	4	0	16	0.687[i]
99.2%CO-0.8%NH$_3$	0.610	8	0	4	4	0	16	0.687[i]
99.9%CO	0.650	12	4	0	0	0	16	0.938
85%CO-15%CO$_2$	0.656	5	0	6	5	0	16	0.578
95%CO-5%CO$_2$	1.032	12	0	2	2	0	16	0.844

[a] See Section B.3.2.54 for the compressive strength data on which the strength index is based. The strength index is obtained by averaging strength fractions at all dopant levels for a refractory in a specific gas composition. (The strength fraction is the ratio of the doped strength to the undoped strength.) If the combined fraction, called a strength index, is zero, there is no retained strength. A value of one is the average undoped strength index.

[b] The spalling index is calculated by arbitrarily assigning the following values to the damage condition of the exposed refractory: 1.00 = no damage
0.75 = cracking observed
0.50 = light spalling observed (edge and corner)
0.25 = heavy spalling observed (whole face and deeper)
0.0 = complete disintegration.

COMPARISON OF STRENGTH INDEX[a] AND SPALLING DAMAGE[b] FOR REFRACTORIES[c] DOPED WITH METALLIC
IRON AFTER EXPOSURE[d] TO CO GAS MIXTURES[27], Continued

Footnotes continued

These numbers are multiplied by the number of samples exhibiting that particular condition and then divided by the total number of samples examined.

[c]The refractories were DOE-specified generic preparations. Refractories were doped with 0.5, 1.0, 1.5, and 2.0 wt% metallic Fe.

[d]Exposures to the gas mixtures were for 100 hours at 500 °C and 1 atmosphere gas pressure.

[e]The 90% alumina castable was prepared from 65 wt% tabular alumina (25 wt% 6 to 10 mesh, 20 wt% 10 to 20 mesh, 20 wt% less than 20 mesh), 10 wt% calcined alumina (less than 325 mesh), 25 wt% calcium aluminate cement (Casting Grade CA-25 cement), all Alcoa materials. Materials were dry mixed for 2 min, then water added with continued mixing to ball-in-hand consistency (∿605 ml); refractory was then poured into 2 in. x 2 in. x 2 in. aluminum molds, vibrated for 5 min, sealed in plastic for 24 h, then fired for 5 h in air at 1100 °C.

[f]In another table of this series of reports this value is given as 0.875 which cannot be correct if the information in this table is correct.

[g]The 50% alumina castable was prepared from 75 wt% calcined kaolin (25 wt% 6 to 10 mesh, 20 wt% 10 to 20 mesh, 15 wt% less than 20 mesh, 20 wt% less than 200 mesh), 25 wt% calcium aluminate cement (Casting Grade CA-25, Alcoa). Samples were prepared in the same way as the 90% alumina castable except that the water used was ∿700 ml.

[h]The ramming mix was prepared fro 82 wt% tabular alumina (30 wt% 6 to 10 mesh, 20 wt% 10 to 20 mesh, 15 wt% less than 20 mesh, 17 wt% less than 48 mesh), 15 wt% calcined alumina (less than 325 mesh), 3 wt% bentonite. To this mix was added 1 wt% hydrated alumina, 6 wt% phosphoric acid (85%) and 0.25 wt% water. Dry materials were mixed and then hand-mixed in plastic bags with the liquids; mixture was aged for 18 h; samples were formed by die pressing at 500 psi, cured at 250 °C in air for 12 h, then fired at 1100 °C for 5 h.

[i]In another table of this series of reports this value is given as 0.641 which cannot be correct if the information in this table is correct.

STRENGTH[a] AND SPALLING[b] INDEXES OF IRON-DOPED REFRACTORIES[c] EXPOSED[d] TO VARIOUS CARBON MONOXIDE GAS MIXTURES[27]

| Dopant/ | 90 wt% Alumina Castable | | | 50 wt% Alumina Castable | | | 90 wt% Alumina Ramming Mix | | | |
| | Metallic Fe | | Fe_2O_3 | Metallic Fe | | Fe_2O_3 | Metallic Fe | | Fe_2O_3 | |
Gas Composition	Str. Index	Sp. Index	Str. Index	Str. Index	Sp. Index	Str. Index	Str. Index	Sp. Index	Str. Index	Sp. Index
99.9% CO	0.336	0.362	1.254	0.197	0.238	0.994	0.650	0.938	0.885	1.000
95% CO-5% CO_2	0.971	0.906	1.365	0.287	0.391	1.143	1.032	0.844	1.160	0.766
85% CO-15% CO_2	1.074	0.906	1.015	1.217	0.984	1.136	0.656	0.578	0.751	0.859
99.8% CO-0.2% NH_3	0.637	0.812	0.939	0.508	0.719	1.072	0.554	0.641[f]	0.802	0.953
99.2% CO-0.8% NH_3	0.855	0.875[e]	1.226	0.486	0.469	1.363	0.610	0.641[f]	0.950	1.000
99.8% CO-0.2% H_2S	0.892	0.844	1.168	0.253	---	1.076	0.654	0.891	0.693	0.609
99.2% CO-0.8% H_2S	0.972	0.906	1.064	0.818	0.546	1.020	1.487	0.813	0.442	0.442
80% CO-20% H_2O	1.418	1.000	2.024	1.282	1.000	1.362	1.004	1.000	1.302	1.000
60% CO-40% H_2O	0.953	1.000	1.170	0.854	1.000	0.850	0.985	1.000	1.424	0.984
80% CO-20% H_2	1.540	0.844	1.721	1.953	0.984	2.051	0.929	0.953	1.067	1.000
60% CO-40% H_2	1.205	1.000	1.179	0.978	1.000	1.008	0.976	1.000	0.999	1.000

[a] See Section B.3.2.54 for the compressive strength data on which the strength index is based. The strength index is obtained by averaging strength fractions at all dopant levels for a refractory in a specific gas composition. (The strength fraction is the ratio of the doped strength to the undoped strength.) If the combined fraction, called a strength index, is zero, there is no retained strength. A value of one is the average undoped strength index.

[b] The spalling index is calculated by arbitrarily assigning the following values to the damage condition of the exposed refractory:
1.00 = no damage
0.75 = cracking observed
0.50 = light spalling observed (edge and corner)
0.25 = heavy spalling observed (whole face and deeper)
0.0 = complete disintegration.

[c] Refractories were doped with iron added either as metallic iron (Fe) or as hematite (Fe_2O_3). The refractories were DOE-specified generic preparations. The 90 wt% alumina castable was prepared from 65 wt% tabular alumina (25 wt% 6 to 10 mesh, 20 wt% 10 to 20 mesh, 20 wt% less than 20 mesh), 10 wt% calcined alumina (less than 325 mesh), 25 wt% calcium aluminate cement (Casting Grade CA-25 cement), all Alcoa materials. Materials were dry mixed for 2 min, then water added with continual mixing to ball-in-hand consistency (~605 ml); refractory was then poured into 2 in. x 2 in. x 2 in. aluminum molds, vibrated for 5 min, sealed in plastic for 24 h, then fired for 5 h in air at 1100 °C. The 50% alumina castable was prepared from 75 wt% calcined kaolin (25 wt% 6 to 10 mesh, 20 wt% 10 to 20 mesh, 15 wt% less than 20 mesh, 20 wt% less than 200 mesh), 25 wt% calcium aluminate cement (Casting Grade CA-25, Alcoa). Samples were prepared in the same way as the 90% alumina castable except that the water used was ~700 ml. The ramming mix was prepared from 82 wt% tabular alumina (30 wt% 6 to 10 mesh, 20 wt% 10 to 20 mesh, 15 wt% less than 20 mesh, 17 wt% less than 48 mesh), 15 wt% calcined alumina (less than 325 mesh), 3 wt% bentonite. To this mix was added 1 wt% hydrated alumina, 6 wt% phosphoric acid (85%) and 0.25 wt% water. Dry materials were mixed and then hand-mixed in plastic bags with the liquids; mixture was aged for 18 h; samples were formed by die pressing at 500 psi, cured at 250 °C in air for 12 h, then fired at 1100 °C for 5 h.

[d] Exposure to the gas mixtures was for 100 hours at 500 °C and 1 atmosphere gas pressure.

[e] See footnote f in Section B.3.2.68.

[f] See footnote i in Section B.3.2.68.

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CORRELATION[a] OF STRENGTH DEGRADATION[b] AND SPALLING DAMAGE[c] FOR
REFRACTORIES EXPOSED TO CARBON MONOXIDE GAS MIXTURES[27]

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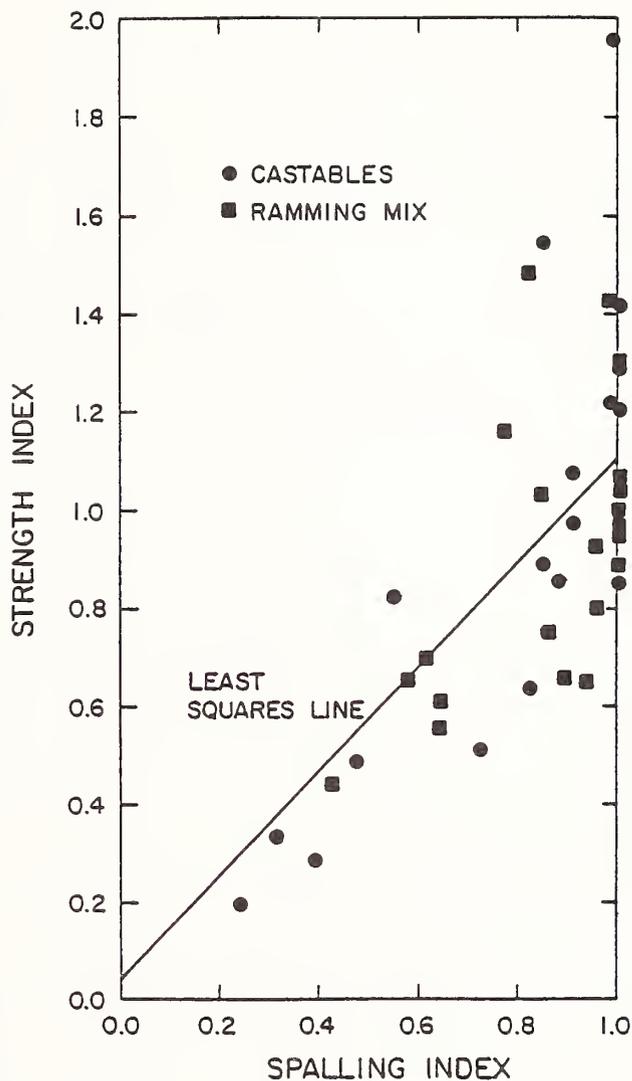
[a]The figure shows that strength degradation and spalling damage follow
similar trends. See Sections B.3.2.68 and B.3.2.69 for the data on which
this figure is based. The data for all exposures of the refractories are
plotted here. The data are for the metallic Fe-doped castables and for
both metallic Fe-doped and Fe_2O_3-doped ramming mix. The least squares
line has a slope of 1.04, indicating extremely good correlation between
the two methods of assessing disintegration damage. The intercept point
near the origin has a value of 0.042.

[b]As measured by the Strength Index, see Sections B.3.2.68 and B.3.2.69.

[c]As measured by the Spalling Index, see Sections B.3.2.68 and B.3.2.69.

EFFECT OF SPECIFIC COMPONENTS OF GAS MIXTURES[a] ON THE COMPRESSIVE STRENGTH[b] OF IRON-DOPED CASTABLE REFRACTORIES[c][27]

(Data Continued)

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EFFECT OF SPECIFIC COMPONENTS OF GAS MIXTURES[a] ON THE COMPRESSIVE STRENGTH[b] OF IRON-DOPED CASTABLE REFRACTORIES[c][27], Continued

(Data Continued)

EFFECT OF SPECIFIC COMPONENTS OF GAS MIXTURES[a] ON THE COMPRESSIVE STRENGTH[b] OF IRON-DOPED CASTABLE REFRACTORIES[c][27], Continued

(Data Continued)

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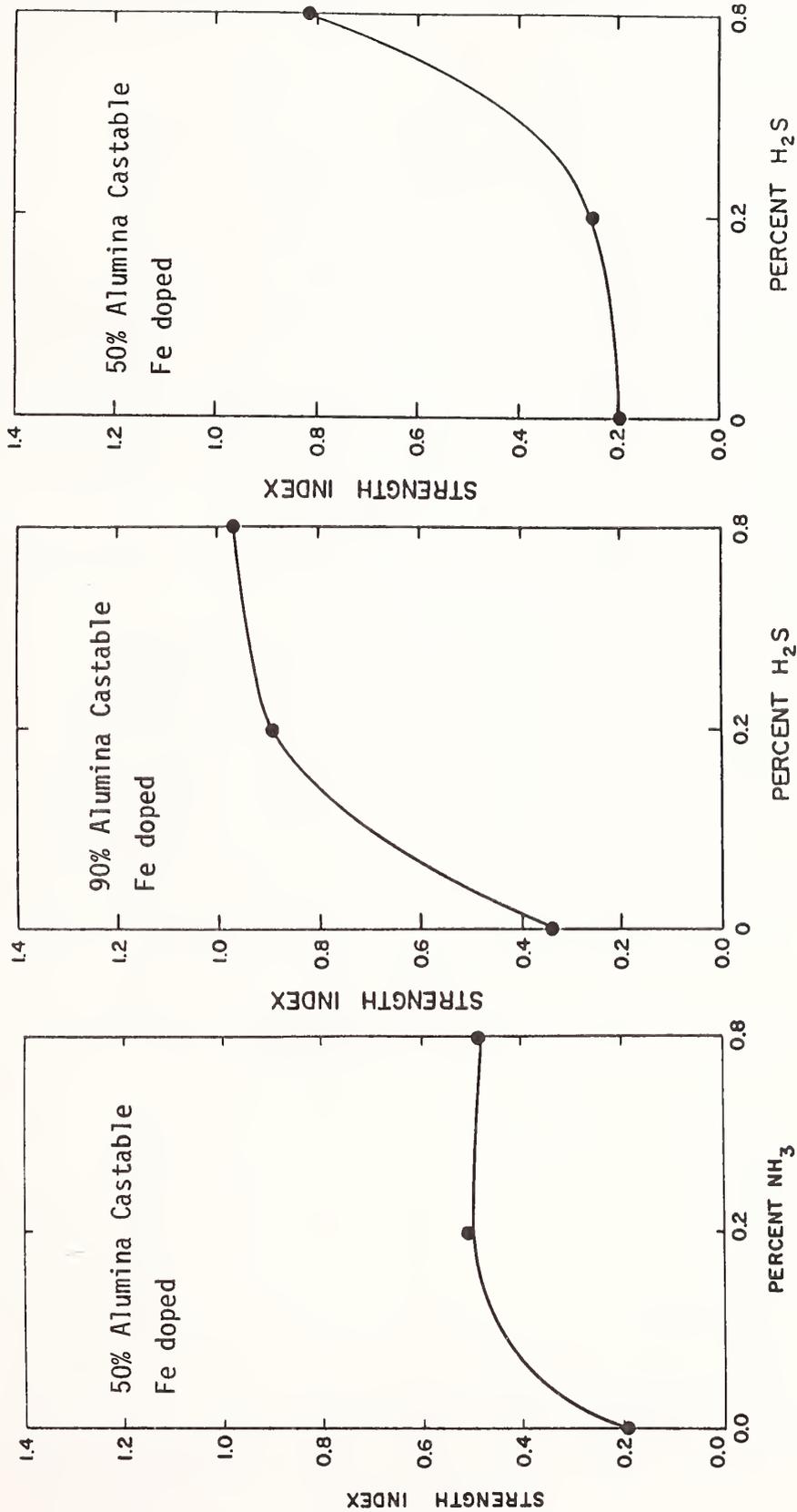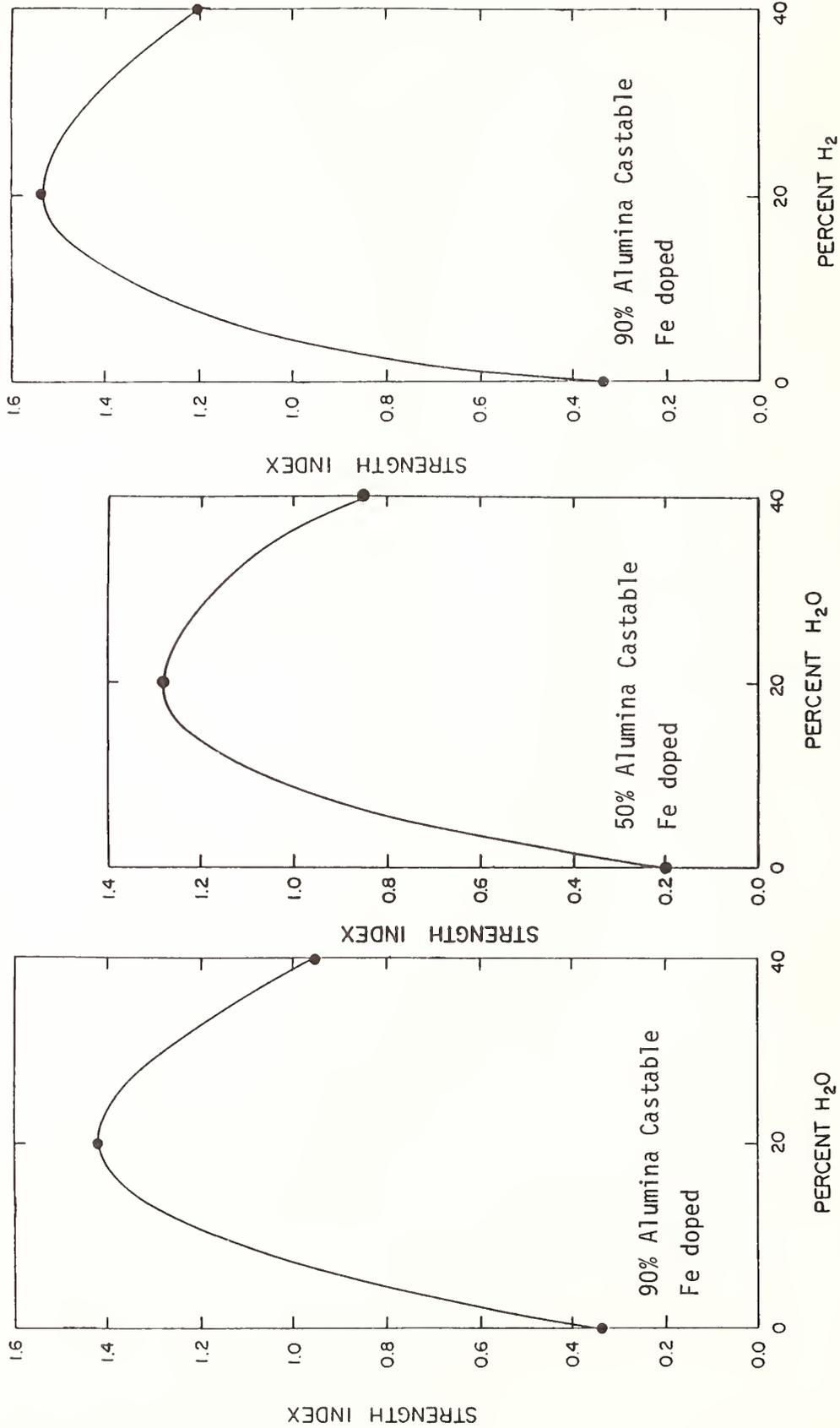
EFFECT OF SPECIFIC COMPONENTS OF GAS MIXTURES[a] ON THE COMPRESSIVE STRENGTH[b] OF IRON-DOPED CASTABLE REFRACTORIES[c][27], Continued

Footnotes

[a] Refractories were exposed to the following feed gas compositions for 100 hours at 500 °C and 1 atmosphere pressure:

99.9% CO	99.2% CO-0.8% H_2S
95% CO-5% CO_2	80% CO-20% H_2O
85% CO-15% CO_2	60% CO-40% H_2O
99.8% CO-0.2% NH_3	80% CO-20% H_2
99.2% CO-0.8% NH_3	60% CO-40% H_2
99.8% CO-0.2% H_2S	

[b] Before and after exposure the compressive strength was measured on a universal testing machine with a crosshead speed of 0.1 in./min. See Section B.3.2.54 for the strength data on which these figures are based. The strength index plotted on the figures is obtained by averaging strength fractions at all dopant levels for a refractory in a specific gas composition. (The strength fraction is the ratio of the doped strength to the undoped strength.) If the combined fraction, call a strength index, is zero, there is no retained strength. A value of one is the average undoped strength index.

[c] The refractories were DOE-specified generic preparations. The refractories were doped with 0.5, 1.0, 1.5, and 2.0 wt% metallic Fe. The 90% alumina castable was prepared from 65 wt% tabular alumina (25 wt% 6 to 10 mesh, 20 wt% 10 to 20 mesh, 20 wt% less than 20 mesh), 10 wt% calcined alumina (less than 325 mesh), 25 wt% calcium aluminate cement (Casting Grade CA-25 cement), all Alcoa materials. Materials were dry mixed for 2 min, then water added with continued mixing to ball-in-hand consistency (~605 ml); refractory was then poured into 2 in. x 2 in. x 2 in. aluminum molds, vibrated for 5 min, sealed in plastic for 24 h, then fired in air for 5 h at 1100 °C. The 50% alumina castable was prepared from 75 wt% calcined kaolin (25 wt% 6 to 10 mesh, 20 wt% 10 to 20 mesh, 15 wt% less than 20 mesh, 20 wt% less than 200 mesh), 25 wt% calcium aluminate cement (Casting Grade CA-25, Alcoa). Sample preparation was the same as the 90% castable except that the water used was ~700 ml.
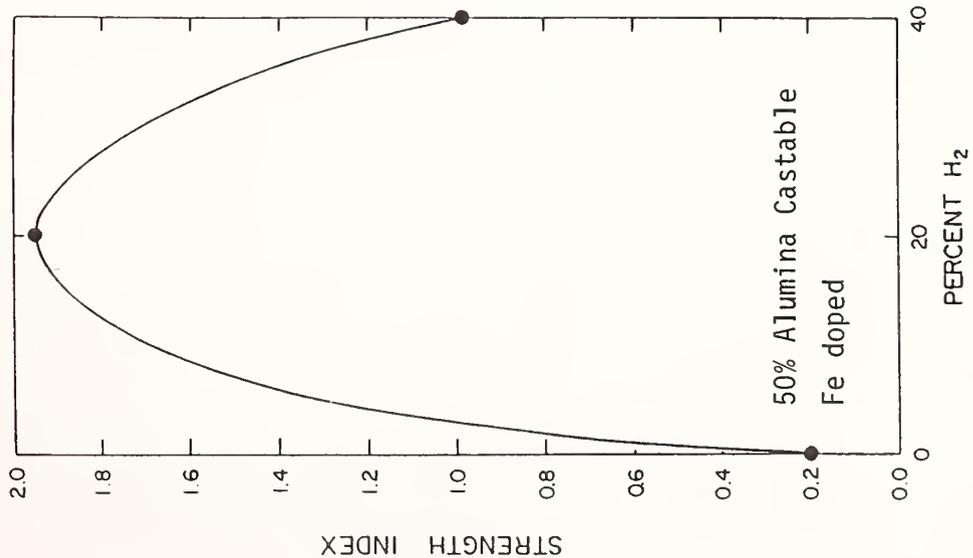
50% Alumina Castable Fe doped

STRENGTH INDEX vs PERCENT H_2

EFFECT OF SPECIFIC COMPONENTS OF GAS MIXTURES[a] ON THE COMPRESSIVE STRENGTH[b] OF IRON-DOPED ALUMINA RAMMING MIX[c][27]

(Data Continued)

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EFFECT OF SPECIFIC COMPONENTS OF GAS MIXTURES[a] ON THE COMPRESSIVE STRENGTH[b] OF IRON-DOPED
ALUMINA RAMMING MIX[c][27], Continued

(Data Continued)

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EFFECT OF SPECIFIC COMPONENTS OF GAS MIXTURES[a] ON THE COMPRESSIVE STRENGHT[b] OF IRON-DOPED ALUMINA RAMMING MIX[c][27], Continued

(Data Continued)

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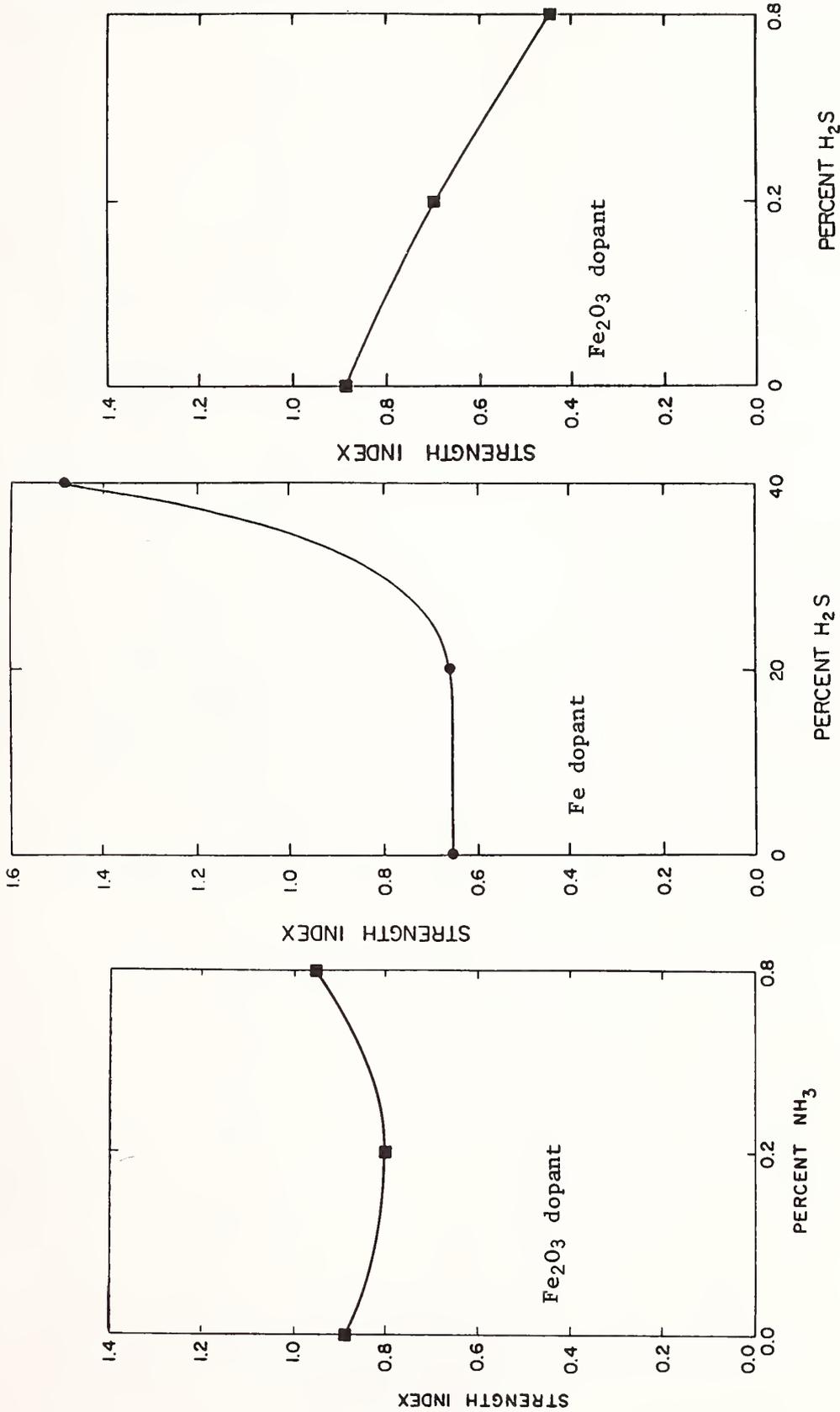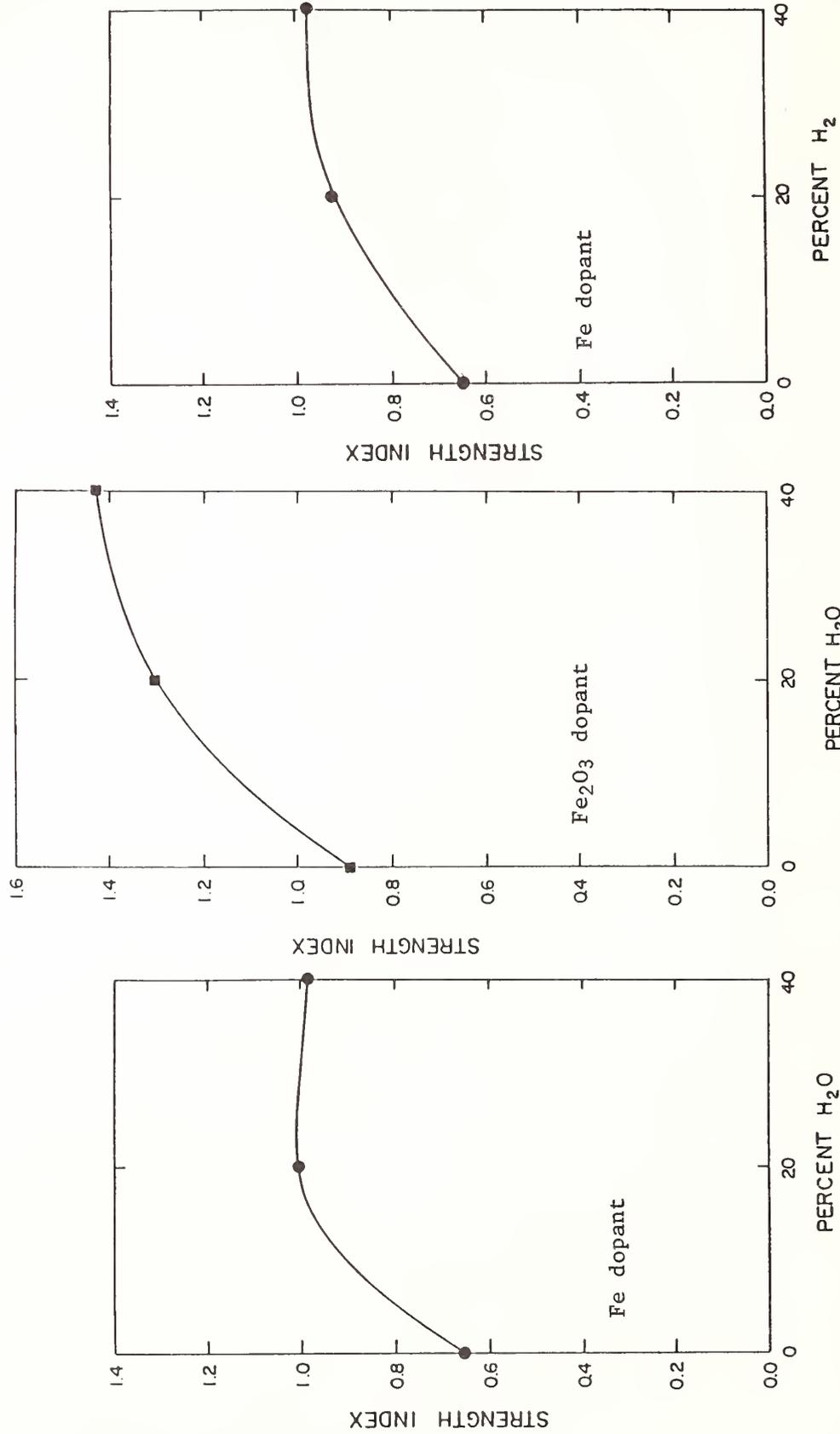
EFFECT OF SPECIFIC COMPONENTS OF GAS MIXTURES[a] ON THE COMPRESSIVE STRENGTH[b] OF IRON-DOPED ALUMINA RAMMING MIX[c][27], Continued

Footnotes

[a]The ramming mix was exposed to the following feed gas composi-
tions for 100 hours at 500 °C and 1 atmosphere pressure:

99.9% CO	99.2% CO–0.8% H_2S
95% CO–5% CO_2	80% CO–20% H_2O
85% CO–15% CO_2	60% CO–40% H_2O
99.8% CO–0.2% NH_3	80% CO–20% H_2
99.2% CO–0.8% NH_3	60% CO–40% H_2
99.8% CO–0.2% H_2S	

[b]Before and after exposure the compressive strength was mea-
sured on a universal testing machine with a crosshead speed
of 0.1 in./min. See Section B.3.2.54 for the strength data
on which these figures are based. The strength index plotted
on these figures is obtained by averaging strength fractions
at all dopant levels for a specific dopant in a given gas com-
position. (The strength fraction is the ratio of the doped
strength to the undoped strength.) If the combined fraction,
called a strength index, is zero, there is no retained
strength. A value of one is the average undoped strength
index.

[c]The 90% alumina ramming mix is a DOE-specified generic prepa-
ration. The ramming mix was doped with 0.5, 1.0, 1.5, and
2.0 wt% iron either as metallic Fe or as hematite (Fe_2O_3).
The mix was prepared from 82 wt% tabular alumina (30 wt% 6 to
10 mesh, 20 wt% 10 to 20 mesh, 15 wt% less than 20 mesh, 17
wt% less than 48 mesh), 15 wt% calcined alumina (less than
325 mesh), 3 wt% bentonite. To this mix was added 1 wt%
hydrated alumina, 6 wt% phosphoric acid (85%) and 0.25 wt%
water. Dry materials were mixed and then hand-mixed in plastic
bags with the liquids; mixture was aged for 18 h; samples were
formed by die pressing at 500 psi, cured at 250 °C in air for
12 h, then fired at 1100 °C for 5 h. Samples were 2 in. x
2 in. x 2 in.

Fe_2O_3 dopant

STRENGTH INDEX

PERCENT H_2

==
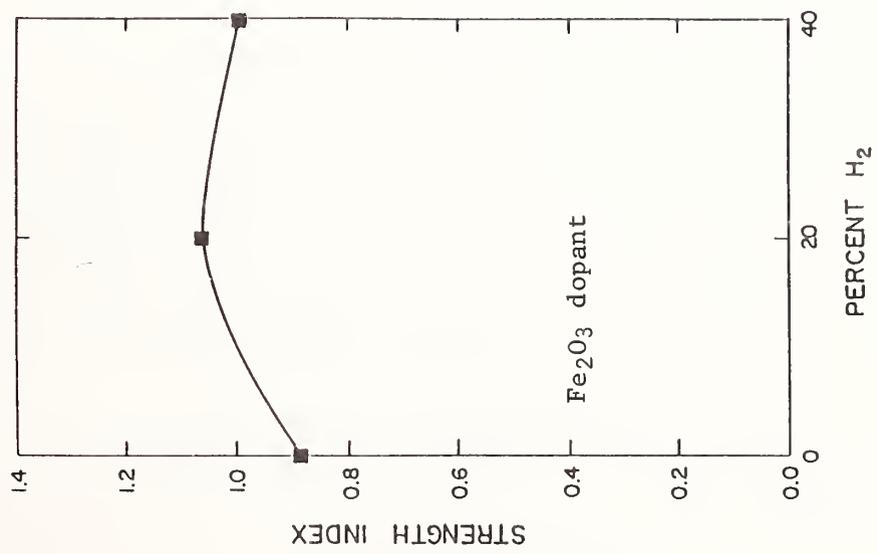
EFFECT OF IRON DOPANT PARTICLE SIZE[a] ON THE STRENGTH[b] OF 90% ALUMINA CASTABLE[c] EXPOSED TO CARBON MONOXIDE[d][27]

Fe_2O_3 Mesh Size	Appearance of Castable	Compressive Strength, psi	No. Samples Tested
12 - 35	Fractures occurred as large cracks generated at dopant sites and propagated through the matrix. Samples completely disintegrated.	0 ± 0	4
35 - 80	Light spalling occurred at corners. Matrix appeared to flake away.	3,950 ± 575	4
80 - 150	Light spalling occurred at corners. Matrix appeared to flake away.	3,500 ± 775	4
<150	Light to moderated spalling occurred at corners and edges. Matrix appeared to flake away.	3,100 ± 775	4
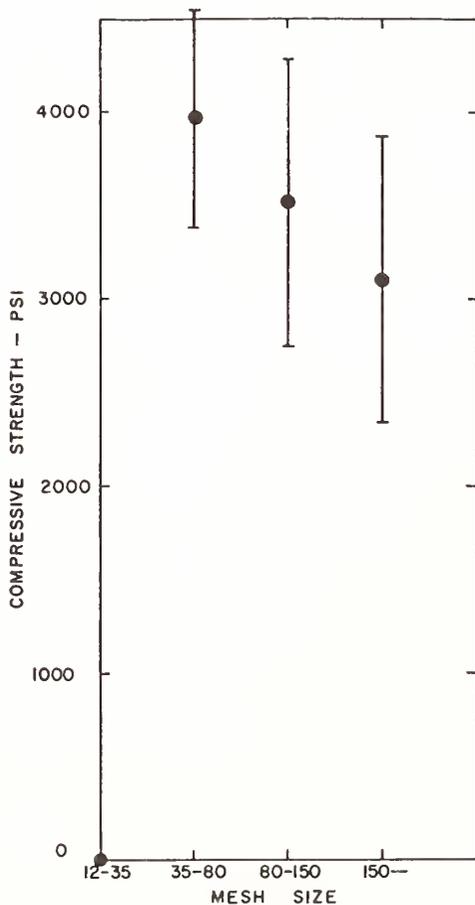
[a] Refractory was doped with 2.0 wt% Fe in the form of hematite (Fe_2O_3). Specimens were prepared with 4 different particle sizes. The Fe_2O_3 particles were obtained by sintering hematite in air at 1250 °C, crushing and hand-screening to 4 ranges of particle size: 12-35 mesh, 35-80 mesh, 80-150 mesh, <150 mesh.

[b] After exposure to CO, compressive strength was measured on a universal testing machine with a crosshead speed of 0.1 in./ min.

[c] DOE 90 generic castable: 65 wt% tabular alumina (25 wt% 6 to 10 mesh, 20 wt% 10 to 20 mesh, 20 wt% <20 mesh), 10 wt% calcined alumina (<325 mesh), 25 wt% calcium aluminate cement (Casting Grade CA-25 cement), all Alcoa materials. Materials were dry mixed for 2 min, then water added with continued mixing to ball-in-hand consistency (∿605 ml); refractory was then poured into 2 in. x 2 in. x 2 in. aluminum molds, vibrated for 5 min, sealed in plastic for 24 h, then fired for 5 h in air at 1100 °C.

[d] Exposure to 99.9% CO was for 100 h at 500 °C and 1 atmosphere gas pressure.

EFFECT OF PRESSURE ON CO DISINTEGRATION[a] TIME OF IRON-DOPED[b] 50% ALUMINA CASTABLE[c][27]
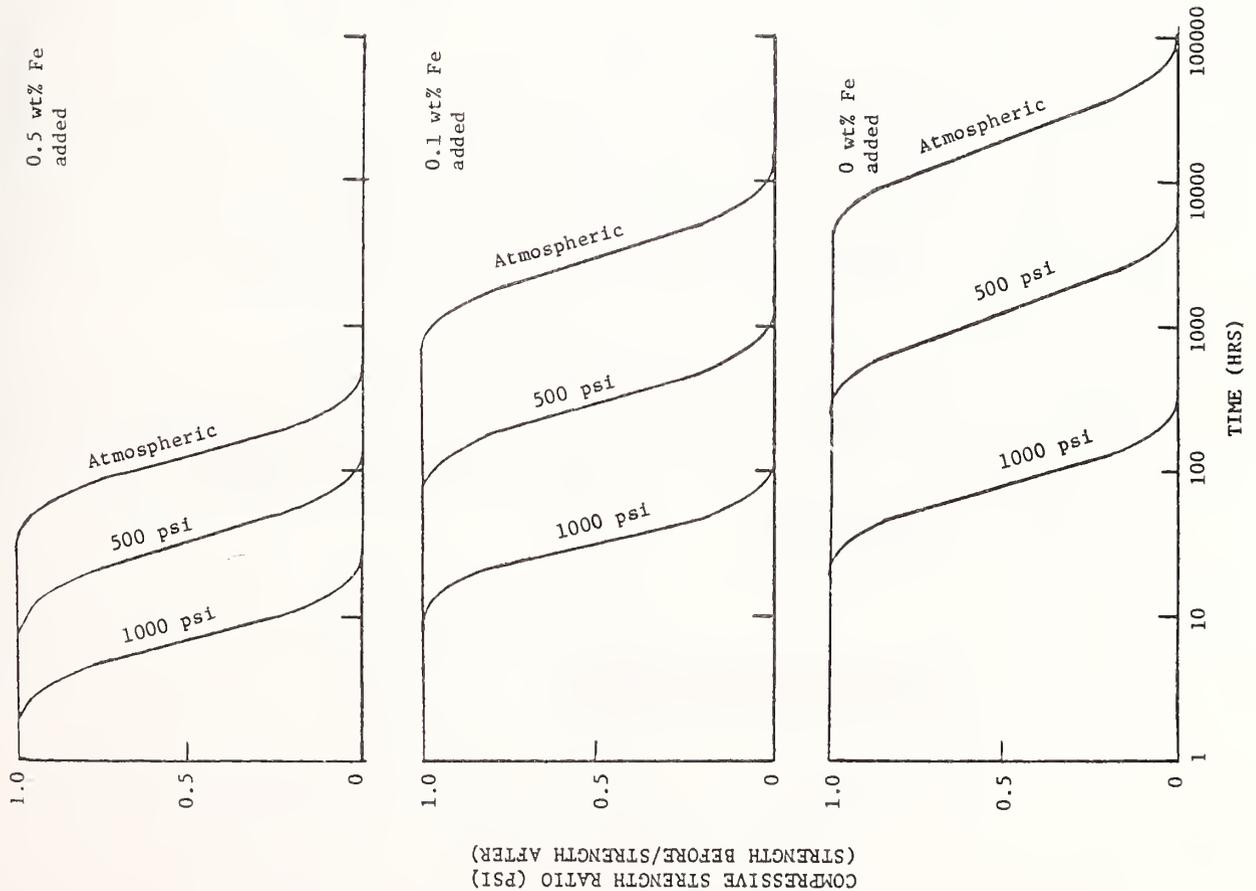
[a]Castable was subjected to 99.9% CO at 500 °C at three pressures for varying times. Compressive strength was measured before and after exposure on a universal testing machine with a crosshead speed of 0.1 in./min.

[b]Castable was doped with 0.1 and 0.5 wt% Fe.

[c]The castable was a DOE generic composition: 75 wt% calcined kaoling (25 wt% 6 to 10 mesh, 20 wt% 10 to 20 mesh, 15 wt% less than 20 mesh, 20 wt% less than 200 mesh), 25 wt% calcium aluminate cement (Casting Grade CA-25, Alcoa). Materials were dry mixed for 2 min, then water added with continued mixing to ball-in-hand consistency (~700 ml); refractory was then poured into 2 in. x 2 in. x 2 in. aluminum molds, vibrated for 5 min, sealed in plastic for 24 h, then fired in air for 5 h at 1100 °C.

COMPRESSIVE STRENGTH RATIO (PSI)
(STRENGTH BEFORE/STRENGTH AFTER)

TIME (HRS)

0.5 wt% Fe added — Atmospheric, 500 psi, 1000 psi

0.1 wt% Fe added — Atmospheric, 500 psi, 1000 psi

0 wt% Fe added — Atmospheric, 500 psi, 1000 psi

==

COMPRESSIVE STRENGTH DATA[a] FOR ALUMINA CASTABLES EXPOSED TO CARBON MONOXIDE[b]
AT 1000 PSI FOR 50 HOURS[27]

Exposure Temperature[b] °C	No. of Samples	Mean Compressive Strength psi	Strength Retained %
- - - - - - - - - - - - - - - -90% Alumina Castable[c]- - - - - - - - - - - - - -
un-exposed	5	4054 ± 511	
400	5	2765 ± 878	68
450	5	2880 ± 529	71
500	5	3602 ± 1108	89
550[d]	5	2195 ± 615	54
- - - - - - - - - - - - - - -50% Alumina Castable[e]- - - - - - - - - - - - - -
un-exposed	5	2473 ± 81	
400	4	1210 ± 365	49
450	5	2216 ± 547	90
500	3	1812 ± 296	73
550	5	1674 ± 545	68

[a] Compressive strength was measured on a universal testing machine with a crosshead speed of 0.1 in./min.

[b] Specimens were exposed to 99.9% CO at four temperatures at 1000 psi for 50 hour periods.

[c] A DOE generic preparation: 65 wt% tabular alumina (25 wt% 6 to 10 mesh, 20 wt% 10 to 20 mesh, 20 wt% less than 20 mesh), 10 wt% calcined alumina (less than 325 mesh), 25 wt% calcium aluminate cement (Casting Grade CA-25 cement), all Alcoa materials. Materials were dry mixed for 2 min, then water was added with continued mixing to ball-in-hand consistency (~605 ml); refractory was then poured into 2 in. x 2 in. x 2 in. aluminum molds, vibrated for 5 min, sealed in plastic for 24 h, then fired for 5 h in air at 1100 °C.

[d] Final temperature was 280 °C; the furnace had shut off.

[e] A DOE generic preparation: 75 wt% calcined kaolin (25 wt% 6 to 10 mesh, 20 wt% 10 to 20 mesh, 15 wt% less than 20 mesh, 20 wt% less than 200 mesh), 25 wt% calcium aluminate cement (Casting Grade CA-25, Alcoa). Samples were prepared in the same way as the 90% alumina castable except that the water used was ~700 ml.

==

EFFECT OF TEMPERATURE ON THE COMPRESSIVE STRENGTH[a] OF ALUMINA CASTABLES
EXPOSED TO CARBON MONOXIDE [27]

50^+ wt.% Al_2O_3

90^+ wt.% Al_2O_3

% Retained Compressive Strength

Temperature in °C

50^+ wt.% Al_2O_3

90^+ wt.% Al_2O_3

[control set]

∇ — difference in strength statistically significant (vs the control set)

Temperature in °C

Compressive Strength (psi)
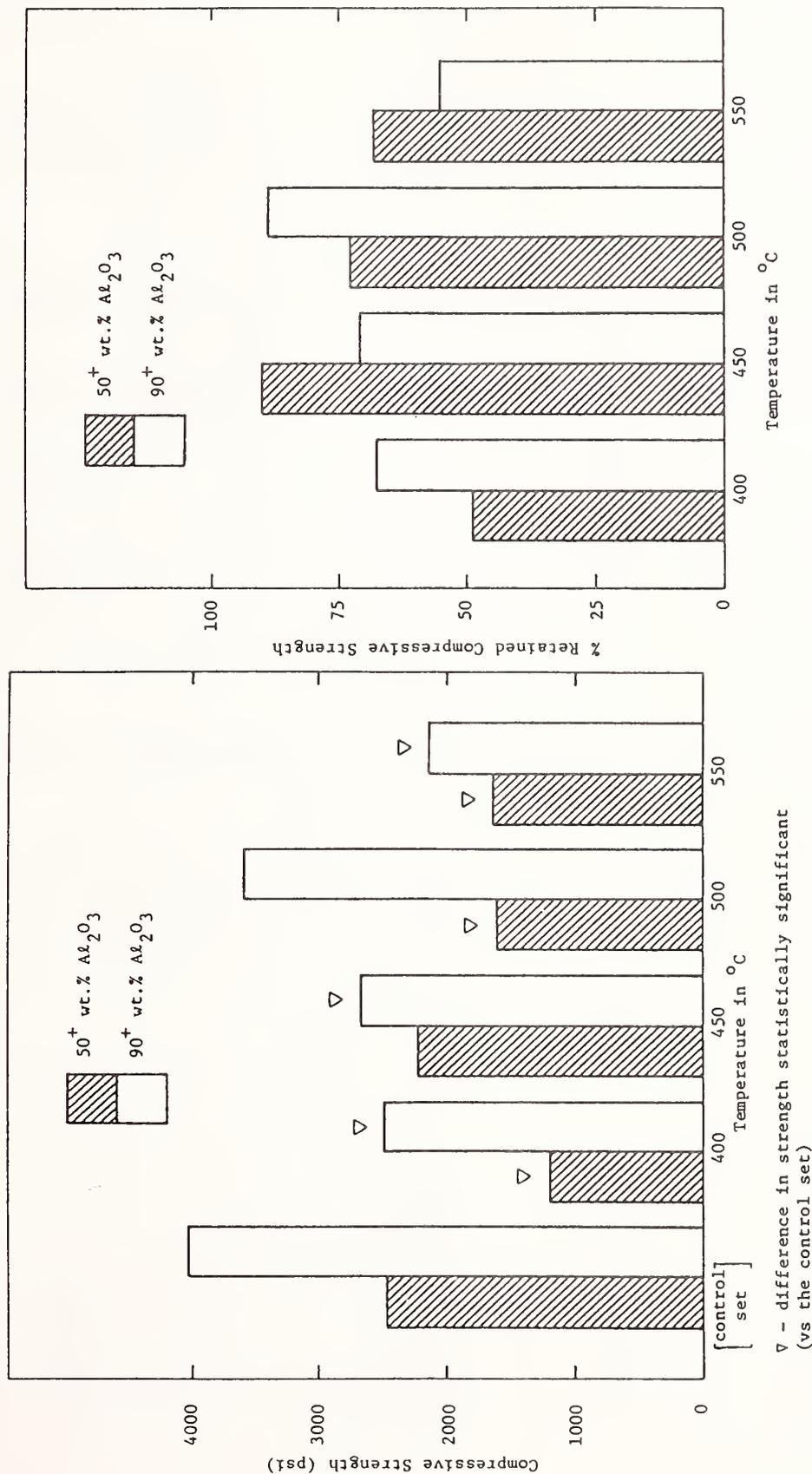
[a] See Section B.3.2.75 for the compressive strength data plotted above as well as the sample preparation and exposure conditions.

COMPRESSIVE STRENGTH[a] OF IRON-DOPED ALUMINA CASTABLES EXPOSED[b] TO CARBON MONOXIDE ATMOSPHERES AT HIGH PRESSURE[c][91]

Gas Composition (vol %)	Wt% Dopant	Exposure Time, h	Compressive Strength[a], psi		Strength Ratio[d]
			Prefired	Exposed	
-90% Alumina Castable[e]					
99.9 CO	0	50	4054 ± 511	3602 ± 1108	0.89
	0.1 Fe[f]	3	completely disintegrated		
	0.1 Fe2O3[f]	3	--	3097 ± 2490	0.720[g]
99.2 CO-0.8 H2S	0	50	4054 ± 511	3871 ± 303	0.95
	0.1 Fe	50	completely disintegrated		
	0	25	--	4929 ± 40	0.815
	0.1 Fe	25	completely disintegrated		
	0	10	--	4302 ± 130	1.051
	0.1 Fe	10	completely disintegrated		
	0	3	--	5516 ± 339	1.012
	0.1 Fe	3	--	5061 ± 349	0.945
79.36 CO-20 H2O-0.64 H2S	0	50	4054 ± 511	5329 ± 334	1.31
78.37 CO-20 H2O-1 NH3-0.63 H2S	0	50	4054 ± 511	4594 ± 141	1.13
-50% Alumina Castable[h]					
99.2 CO-0.8 H2S	0	50	--	5025 ± 56	--
	0.1 Fe	50	completely disintegrated		
60 CO-40 H2O	0	50	--	3826 ± 59	--
	0.1 Fe2O3	50	--	3440 ± 436	--
	0	50	--	3761 ± 334	--
	0.1 Fe	50	--	0	--
60 CO-40 H2	0	50	3334 ± 188	3144 ± 194	0.943
	0.1 Fe2O3	50	3382 ± 119	3249 ± 140	0.961
	0	50	3690 ± 409	2814 ± 190	0.763
	0.1 Fe	50	3954 ± 597	1354 ± 12	0.342
59.5 CO-40 H2O-0.5 H2S	0	50	--	5341 ± 530	--
	0.1 Fe	50	--	4904 ± 42	--

(Table Continued)

[a] Compressive strength was measured on a universal testing machine with a crosshead speed of 0.1 in./min.

===

COMPRESSIVE STRENGTH[a] OF IRON-DOPED ALUMINA CASTABLES EXPOSED[b] TO CARBON MONOXIDE
ATMOSPHERES AT HIGH PRESSURE[c][91] , Continued

Footnotes continued

[b]Castables were exposed to the gas mixtures indicated at 500 °C for the indicated times.

[c]Gas pressure was 1000 psi.

[d]Strength Ratio = $\dfrac{\text{Strength of Exposed Samples}}{\text{Strength of Prefired Samples}}$. Strength of prefired samples was not always measured.

[e]A DOE generic castable: 65 wt% tabular alumina (25 wt% 6 to 10 mesh, 20 wt% 10 to 20 mesh, 20 wt% less than 20 mesh), 10 wt% calcined alumina (less than 325 mesh), 25 wt% calcium aluminate cement (Casting Grade CA-25 cement), all Alcoa materials. Materials were dry mixed for 2 min, then water was added with continued mixing to ball-in-hand consistency ($\sim$605 ml); refractory was then poured into 2 in. x 2 in. x 2 in. aluminum molds, vibrated for 5 min, sealed in plastic for 24 h, then fired in air for 5 h at 1100 °C.

[f]These samples were prefired at 500 °C instead of 1100 °C.

[g]This ratio was calculated using the value for samples prefired at 500 °C.

[h]A DOE generic castable: 75 wt% calcined kaolin (25 wt% 6 to 10 mesh, 20 wt% 10 to 20 mesh, 15 wt% less than 20 mesh, 20 wt% less than 200 mesh), 25 wt% calcium aluminate cement (Casting Grade CA-25, Alcoa). Samples were prepared as described in footnote e except that the water used was $\sim$700 ml.

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EFFECT OF CARBON MONOXIDE ATMOSPHERES[a] ON STAINLESS STEEL FIBER-
REINFORCED[b] ALUMINA CASTABLES AT HIGH PRESSURE[c][91]

Gas Composition (vol %)	Fiber Type[b]	Exposure Time, h	Compressive Strength[d] psi
- - - - - - - - - - - - - -90% Alumina Castable[e]- - - - - - - - - - - - -
N$_2$	446	50	5620 ± 470
	30-20	50	5600 ± 640
	310	50	3740 ± 400
CO	446	50	3790 ± 290
	30-20	50	3260 ± 240
	310	50	2650 ± 430
40 CO-40 N$_2$-20 H$_2$O	446	50	2760 ± 280
	446	100	3154 ± 170
	30-20	50	3760 ± 480
	30-20	100	3720 ± 60
	310	50	2180 ± 210
	310	100	1700 ± 130
80 CO-20 H$_2$O	310	50	2250 ± 210
- - - - - - - - - - - - - -50% Alumina Castable[e]- - - - - - - - - - - - -
N$_2$	446	50	6340 ± 90
	30-20	50	6070 ± 320
	310	50	3530 ± 340
CO	446	50	5250 ± 190
	30-20	50	5460 ± 480
	310	50	2000 ± 370
40 CO-40 N$_2$-20 H$_2$O	446	50	5150 ± 210
	446	100	5380 ± 280
	30-20	50	5770 ± 300
	30-20	100	6380 ± 340
	310	50	3460 ± 400
	310	100	4120 ± 320
80 CO-20 H$_2$O	310	50	3800 ± 550

[a]Castables were exposed to the indicated gases at 500 °C.

[b]Four weight percent of the stainless steel fibers was added to the mix
before samples were casted. The 30-20 steel is the same alloy as the
310 but with an additional 5% chromium.

[c]Gas pressure was 1000 psi.

[d]Compressive strength was measured on a universal testing machine with a
crosshead speed of 0.1 in./min.

[e]These are the same castables as are described in footnotes e and h of
Section B.3.2.77 except that they were not fired at 1100 °C. These
specimens were held at 500 °C for 12 hours before the test exposure. Pre-
firing of these reinforced materials has a deleterious effect on them.

==

COMPRESSIVE STRENGTH DATA[a] ON INSULATING CASTABLES[b] AFTER EXPOSURE
TO VARIOUS TEST GASES[c][91]

Castable[b]	Degree of Disintegration	Compressive Strength[a] psi		Strength Ratio[d]
- - - - - - - - - - - - - - - - - - -N_2 Exposure- - - - - - - - - - - - - - - -
35% Al_2O_3, 2200 °F	Unaffected	366	35.0	1.00
41% Al_2O_3, 2300 °F	Unaffected	278	49.2	1.00
60% Al_2O_3, 2800 °F	Unaffected	946	271	1.00
94% Al_2O_3, 3300 °F	Unaffected	438	106	1.00
- - - - - - -20% CO-21.5% CO_2-49% H_2-7.5% CH_4-2% H_2S Exposure- - - - - - -
35% Al_2O_3, 2200 °F	Affected, pop-outs	327	43.0	0.893
41% Al_2O_3, 2300 °F	Unaffected	245	39.0	0.881
60% Al_2O_3, 2800 °F	Unaffected	973	297	1.03
94% Al_2O_3, 3300 °F	Unaffected	224	74.5	0.511
- - - - - - - - - - - 49% CO-50% N_2-1% H_2S Exposure- - - - - - - - - - - -
35% Al_2O_3, 2200 °F	Destroyed	0		0
41% Al_2O_3, 2300 °F	Unaffected	262	86.4	0.942
60% Al_2O_3, 2800 °F	Affected, pop-outs	865	377	0.914
94% Al_2O_3, 3300 °F	Unaffected	392	95.2	0.895
- - - - - - - - - - - - - - - CO Exposure- - - - - - - - - - - - - - - - -
35% Al_2O_3, 2200 °F	4 samples affected, cracked	164 (4 samples)	71	0.224 (8 samples)
	4 samples destroyed	0		
41% Al_2O_3, 2300 °F	Affected, pop-outs	254	47.2	0.914
60% Al_2O_3, 2800 °F	4 samples affected, cracked	236 (4 samples)	105	0.125 (8 samples)
	4 samples destroyed	0		
94% Al_2O_3, 3300 °F	Affected, pop-outs	446	190	1.02

[a] Data are averages for 8 samples, except where noted otherwise. Compressive
strength was measured on a universal testing machine with a crosshead speed
of 0.1 in./min.

[b] Castables are designated by alumina content and the manufacturers' maximum
recommended use temperature. Manufacturers' data follow, analysis in wt%:
    For 35% alumina: SiO_2 36, Fe_2O_3 5.6, TiO_2 1.2, CaO 21.1, MgO 0.2, Na_2O
                0.8, fired density 58 lb/ft^3.
    For 41% alumina: SiO_2 37, Fe_2O_3 0.9, TiO_2 1.7, CaO 18.6, MgO 0.4, Na_2O
                0.3, fired density 58 lb/ft^3.
    For 60% alumina: SiO_2 33, Fe_2O_3 0.4, TiO_2 0.7, CaO 5.0, MgO 0.1, Na_2O
                0.8, fired density 85 lb/ft^3.
    For 94% alumina: SiO_2 0.5, Fe_2O_3 0.2, CaO 4.6, MgO 0.1, Na_2O 0.5, fired
                density 68 lb/ft^3.
Thermal conductivity values (Btu-in./h-ft^2-°F): 35% alumina, 1.6, 41% alumina,
1.6, 60% alumina, 3.4, 94% alumina, 4.5.
- -

(Table Continued)

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COMPRESSIVE STRENGTH DATA[a] ON INSULATING CASTABLES[b] AFTER EXPOSURE
TO VARIOUS TEST GASES[c][91], Continued

Footnotes continued

Cold crushing strength, in psi:

Condition	35% Alumina	41% Alumina	60% Alumina	94% Alumina
dried 18–24h, 220 °F	700	500	1180	900
fired 5h at 1000 °F	600	450	950	850
1500 °F	500	450	900	700
2200 °F	600	500	1390	800
2800 °F	–	–	4150	1400
3200 °F	–	–	–	2200

[c] Samples were tested in the specified gas compositions at 500 °C and 1000
psig for 50 hours. The tests were run in a vessel which permitted a flow-
through gas rate of 0.16 1/h which was sufficient to renew the reactor
atmosphere every 15 hours.

[d] Strength ratio = $\dfrac{\text{Strength after test gas exposure}}{\text{Strength after } N_2 \text{ exposure}}$.

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EFFECT OF CARBON MONOXIDE LEVEL OF TEST GASES ON THE STRENGTH[a] OF
INSULATING CASTABLES[91]
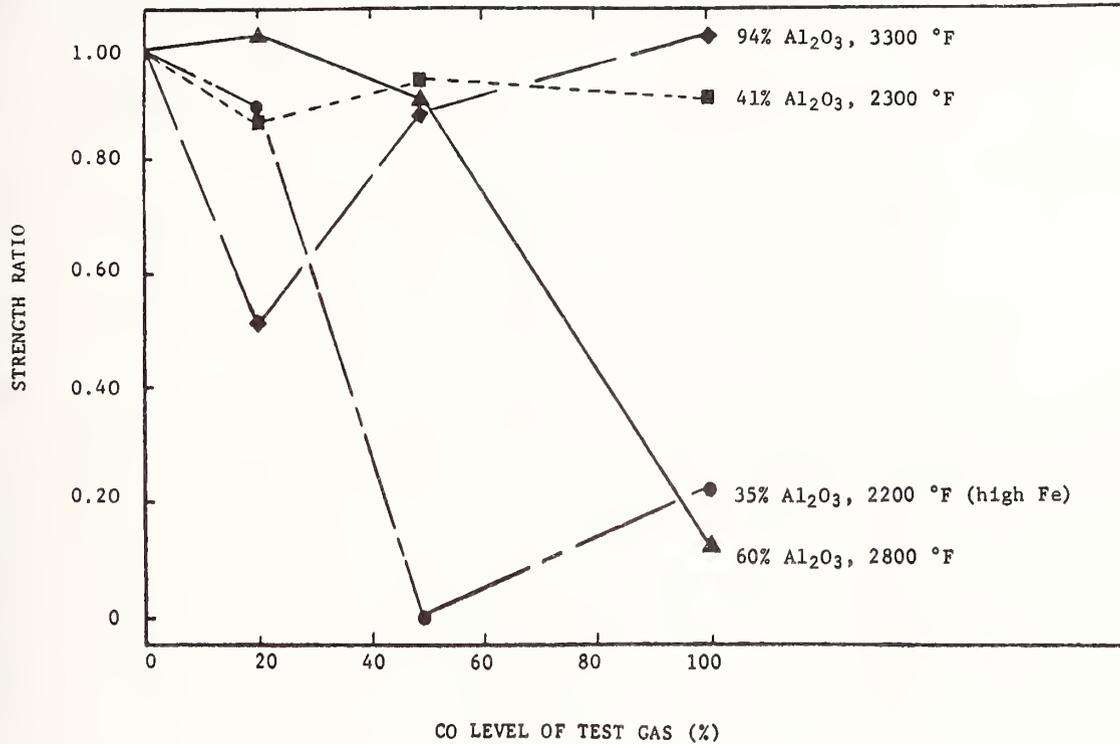
^{a}See Section B.3.2.79 for the strength data plotted in this figure, and
for the identification of the castables as well as the test gases and
test conditions.

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EFFECT OF TEMPERATURE ON THE ELASTIC PROPERTIES[a] OF SEVERAL ALUMINA
REFRACTORIES[b][16]

Curing and Firing Temperature[b] °C	Ambient Temperature Measurements[c]			Elevated Temperature Measurements[c]		
	Young's Modulus GPa	Shear Modulus GPa	Poisson's Ratio	Young's Modulus GPa	Shear Modulus GPa	Poisson's Ratio
Tabular Alumina/Calcium Aluminate Cement[d] -						
Ambient	49.8	21.8	0.141			
100	39.3	17.4	0.126	39.0	17.3	0.127
200	42.4	18.9	0.122	41.4	18.4	0.128
400	46.6	20.8	0.119	43.8	19.6	0.116
600	43.9	21.7	0.128	44.5	19.8	0.126
800	50.3	22.8	0.104	43.3	19.7	0.100
1000	34.4	15.5	0.110	30.3	13.5	0.121
1200	20.9	10.1	0.036	15.0	0.71	0.048
1350	50.2	21.9	0.147	--	--	---
Tabular Alumina/Calcium Aluminate Cement[e] -						
Ambient	53.6	23.4	0.14			
100	38.3	16.9	0.13	38.0	16.8	0.13
200	31.1	14.4	0.08	30.6	14.0	0.09
400	19.6	9.4	0.04	18.5	8.7	0.07
600	20.8	9.6	0.09	18.9	8.6	0.10
800	20.6	9.4	0.10	18.5	8.5	0.09
1000	14.0	6.5	0.08	11.7	5.4	0.08
1200	15.1	6.8	0.12	11.4	5.1	0.11
1350	34.0	14.7	0.16	--	--	--
Calcined Bauxite/Calcium Aluminate Cement[f] -						
Ambient	45.7	20.0	0.14			
100	33.1	14.7	0.13	32.6	14.4	0.13
200	32.0	16.7	0.14	31.0	16.5	0.14
400	30.1	13.4	0.12	28.5	12.6	0.13
600	28.9	12.6	0.15	26.7	11.6	0.15
800	30.5	13.3	0.15	26.9	11.7	0.15
1000	32.7	14.6	0.12	25.9	11.4	0.13
1200	36.0	15.6	0.15	25.8	11.1	0.17
1350	45.0	19.5	0.16	--	--	--
Calcined Bauxite/Calcium Aluminate Cement[g] -						
Ambient	39.4	16.9	0.17			
100	33.0	16.7	0.17	32.5	13.9	0.17
200	25.6	10.9	0.17	24.8	10.7	0.16
400	13.0	5.8	0.12	12.2	5.4	0.12
600	11.6	5.5	0.07	10.1	4.6	0.09
800	11.6	5.4	0.07	9.9	4.6	0.08
1000	10.4	5.0	0.04	8.5	4.0	0.07
1200	13.1	6.3	0.06	10.0	4.5	0.10
1350	21.7	9.8	0.10	--	--	--

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(Table Continued)

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EFFECT OF TEMPERATURE ON THE ELASTIC PROPERTIES[a] OF SEVERAL ALUMINA
REFRACTORIES[b][16], Continued

Curing and Firing Temperature °C	Ambient Temperature Measurements[c]			Elevated Temperature Measurements[c]		
	Young's Modulus GPa	Shear Modulus GPa	Poisson's Ratio	Young's Modulus GPa	Shear Modulus GPa	Poisson's Ratio
Calcined Kaolin/Calcium Aluminate Cement[h]						
Ambient	42.9	18.9	0.15			
100	36.1	15.8	0.14	35.8	15.7	0.14
200	35.2	15.9	0.11	33.6	15.1	0.11
400	35.1	15.6	0.12	32.6	14.4	0.13
600	34.1	15.0	0.14	31.5	13.9	0.13
800	34.0	15.3	0.10	29.4	13.2	0.11
1000	33.9	15.3	0.11	26.9	12.0	0.12
1200	23.8	10.8	0.10	18.7	3.8	0.12
1350	--	--	0.13	--	--	--
Calcined Kaolin/Calcium Aluminate Cement[i]						
Ambient	34.9	15.5	0.12			
100	27.8	12.3	0.12	27.4	12.2	0.12
200	32.1	14.3	0.12	29.1	13.0	0.12
400	31.9	14.0	0.14	27.6	12.1	0.14
600	31.8	14.0	0.14	29.5	13.0	0.14
800	34.2	15.1	0.13	28.2	12.5	0.13
1000	33.4	14.9	0.12	17.9	8.0	0.12
1200	17.9	7.9	0.12	6.9	3.1	0.13
1350	24.7	11.1	0.11	--	--	--
57% Alumina Insulating Castable[j]						
Ambient	9.2	4.1	0.11			
100	6.2	2.9	0.08	6.1	2.8	0.08
200	4.6	3.4	0.04	4.2	2.0	0.07
400	4.8	2.2	0.08	4.2	2.0	0.07
600	5.2	2.4	0.07	4.6	2.1	0.08
800	3.3	1.5	0.06	2.4	1.1	0.10
1000	3.1	1.5	0.04	2.1	0.9	0.09
1200	5.0	2.3	0.10	--	--	0.14
50% Alumina Insulating Castable[k]						
Ambient	-0.3[sic][l]	4.11[m]	0.13			
100	10.0	4.4	0.14	9.9[l]	4.4	0.12
200	9.4	4.1	0.14	9.2	4.0	0.15
400	8.4	3.9	0.09	7.9	3.6	0.11
600	7.8	3.5	0.12	7.2	3.2	0.12
800	6.7	3.1	0.10	5.8	2.6	0.12
1000	6.1	2.8	0.11	4.8	2.2	0.11
1200	6.5	3.6	0.10	--	--	0.11

[a]Elastic moduli were determined by dynamic mechanical resonance method. Specimens 0.65 cm x 2.54 cm x 17.78 cm were saw cut from larger cast specimens. Two specimens of each refractory at each curing and firing temperature were measured. Ambient temperature and elevated

- -

(Table Continued)

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EFFECT OF TEMPERATURE ON THE ELASTIC PROPERTIES[a] OF SEVERAL ALUMINA
REFRACTORIES[b][16], Continued

Footnotes continued

temperature data were obtained on the same specimens. Flexural and torsional resonance frequencies were obtained using an acoustic spectrometer and the elastic moduli were then calculated. See Sections B.3.2.33 and B.3.2.83 for plotted versions of some of these data.

[b]Samples were cast, vibrated one minute, cured sealed in plastic 24 hours at ambient temperature, cured in air 24 hours at ambient temperature, dried in air 24 hours at 100 °C, fired at 60 °C/h to 200, 400, 600, 800, 1000, 1200, and 1350 °C, and soaked at temperature 5 hours, and furnace cooled to ambient temperature.

[c]Elevated temperature measurements were made at the same temperatures as those used for curing and firing. After ambient temperature measurements were made, the specimens were heated at a rate of about 300 °C/h and flexural and torsional resonance frequencies obtained.

[d]75% T-61 alumina (Alcoa) aggregate, 25% Casting Grade CA-25 cement (Alcoa), water to ball-in-hand consistency 10.4% of aggregate and cement.

[e]75% T-61 alumina (Alcoa) aggregate, 25% Secar 71 cement (Lone Star Lafarge), water to ball-in-hand consistency 12.7% of aggregate and cement.

[f]75% Mulcoa M-60 (C-E Minerals) aggregate, 25% Casting Grade CA-25 (Alcoa) cement, water to ball-in-hand consistency 10.6% of aggregate and cement.

[g]75% Mulcoa M-60 (C-E Minerals) aggregate, 25% Secar 71 cement (Lone Star Lafarge), water to ball-in-hand consistency 14.6% of aggregate and cement.

[h]75% Mulcoa M-47 (C-E Minerals) aggregate, 25% Casting Grade CA-25 (Alcoa) cement, water to ball-in-hand consistency 11.3% of aggregate and cement.

[i]75% Mulcoa M-47 (C-E Minerals) aggregate, 25% Refcon cement (Universal Atlas), water to ball-in-hand consistency 13.5% of aggregate and cement.

[j]A commercial lightweight insulating castable (Cer-Lite #75, C-E Refractories), water to ball-in-hand consistency 22% of mix.

[k]A commercial lightweight insulating castable (Litecast 75-28, General Refractories), water to ball-in-hand consistency 24% of mix.

[l]The ambient temperature value is obviously in error. All of these tabulated Young's modulus differ from the values plotted in the same set of reports. See Sections B.3.2.33 and B.3.2.83 for the plotted data.

[m]All of these ambient temperature measurements tabulated differ from the values plotted in the same set of reports. See Section B.3.2.83 for the plotted data.

===

EFFECT OF TEMPERATURE ON THE FRACTURE PROPERTIES[a] OF SEVERAL ALUMINA REFRACTORIES[b][16]

Curing/Firing Temperature °C	Ambient Temperature Measurements[a]				Elevated Temperature Measurements[a]			
	Fracture Toughness K_{IC}, MN/m$^{3/2}$	Notched Beam Fracture Energy γ_{NBT}, J/m^2	Work of Fracture γ_{WOF}, J/m^2	Strain Energy Release Rate GIC, J/m^2	Fracture Toughness K_{IC}, MN/m$^{3/2}$	Notched Beam Fracture Energy γ_{NBT}, J/m^2	Work of Fracture γ_{WOF}, J/m^2	Strain Energy Release Rate GIC, J/m^2
TABULAR ALUMINA/CALCIUM ALUMINATE CEMENT[c]								
Ambient	0.67±0.07	4.58±0.99	46.52± 8.42	9.17± 1.98				
100	0.68±0.09	5.95±1.39	46.02±12.25	11.89± 2.77	0.45±0.03	2.62±0.37	54.19± 8.45	5.24± 0.73
200	0.70±0.14	6.53±0.74	53.45± 8.18	13.05± 1.48	0.58±0.04	4.07±0.53	77.85±15.18	8.15± 1.06
400	0.68±0.08	5.50±0.83	51.02± 8.80	10.99± 1.66	0.47±0.03	2.51±0.31	69.39± 3.84	5.02± 0.63
600	0.65±0.08	4.35±1.01	61.05±12.83	8.70± 2.18[d]	0.47±0.04	2.53±0.43	73.74±13.61	5.05± 0.86
800	0.65±0.09	4.77±0.65	66.25± 9.06	9.53± 1.30[d]	0.63±0.03	4.62±0.48	88.11±12.09	9.23± 0.96
1000	0.77±0.11	8.78±2.53	55.24± 6.95	17.56± 5.07	0.51±0.06	4.38±1.03	110.69±22.54	8.75± 2.06
1200	0.53±0.10	7.14±2.76	44.22± 5.61	14.28± 5.52	0.44±0.07	6.52±2.08	118.72±14.05	13.04± 4.16
1350	1.02±0.09	10.46±1.98	66.14±11.96	20.92± 3.96	--	--	--	--
TABULAR ALUMINA/CALCIUM ALUMINATE CEMENT[e]								
Ambient	0.67±0.03	4.42±0.37	43.80± 2.49	8.84± 0.74				
100	0.69±0.02	6.20±0.34	40.04± 1.58	12.40± 0.64	0.55±0.11	4.20±0.52	40.4 ± 6.8	8.40± 1.04
200	0.46±0.04	3.36±0.51	47.22± 3.29	6.72± 1.02	0.45±0.27	3.32±0.39	60.7 ± 7.7	6.64± 0.78
400	0.33±0.02	2.79±0.25	48.02± 5.44	5.58± 0.50	0.31±0.03	2.56±0.44	67.5 ± 7.0	5.12± 0.88
600	0.30±0.02	2.24±0.32	48.96± 5.82	4.48± 0.64	0.28±0.01	2.15±0.09	63.1 ± 7.8	4.30± 0.18
800	0.42±0.03	2.44±0.33	44.41± 4.70	4.88± 0.66	0.39±0.04	2.39±0.45	88.8 ± 2.2	4.78± 0.90
1000	0.56±0.04	11.31±1.67	42.97± 2.87	22.62± 3.34	0.53±0.00	11.93±0.16	63.0 ± 7.1	23.86± 0.32
1200	0.47±0.01	7.35±0.50	64.50± 6.66	14.70± 1.00	0.34±0.02	11.08±1.10	106.0 ± 9.81	22.16± 2.20
1350	0.54±0.04	4.39±0.67	58.62± 6.09	8.78± 1.34	--	--	--	--
CALCINED BAUXITE/CALCIUM ALUMINATE CEMENT[f]								
Ambient	0.60±0.02	3.91±0.22	53.05± 5.12	7.82± 0.44				
100	0.60±0.01	5.44±0.27[g]	57.73± 8.40	10.88± 0.54	0.57±0.04	5.01±0.71	62.24± 7.67	10.02± 1.42
200	0.51±0.04	4.07±0.59[g]	51.34± 6.26	6.86± 1.18[g]	0.50±0.02	4.05±0.29[g]	70.73± 6.78	6.76± 0.58[g]
400	0.49±0.02	4.05±0.36	62.89± 5.50	8.10± 0.72	0.44±0.03	3.48±0.42	90.69± 1.38	6.96± 0.84
600	0.46±0.01	3.63±0.12	51.99± 6.61	7.36± 0.24	0.43±0.06	3.82±0.65	90.99± 8.99	7.64± 1.30
800	0.51±0.03	4.23±0.48[h]	44.95± 4.26	8.46± 0.96	0.47±0.02	4.25±0.35	92.25± 8.01	8.50± 0.70
1000	0.76±0.02	4.86±0.56[h]	56.11± 6.76	9.72± 1.08	0.54±0.03	5.46±0.50	96.23± 1.47	10.92± 1.00
1200	0.82±0.03	9.40±0.68	60.80± 8.55	18.80± 1.36	0.41±0.04	3.32±0.52	66.52± 8.21	6.64± 0.52
1350	1.26±0.05	17.74±1.51	62.82± 2.13	35.48± 3.02	--	--	--	--
CALCINED BAUXITE/CALCIUM ALUMINATE CEMENT[i]								
Ambient	0.55±0.03	3.85±0.40	41.51± 8.01	7.70± 0.80				
100	0.37±0.01	2.06±0.15	50.04± 5.0	4.12± 0.30	0.36±0.02	2.03±0.21	50.62± 2.00	4.06± 0.42
200	0.36±0.03	2.40±0.26	49.56± 7.06	4.80± 0.52	0.30±0.01	1.85±0.12	51.41± 8.41	3.70± 0.24
400	0.24±0.01	2.58±0.52	48.63± 8.12	5.16± 1.04	0.19±0.01	1.55±0.15	49.24± 8.20	3.10± 0.30
600	0.24±0.01	2.57±0.30	47.78± 8.63	5.14± 0.60	0.17±0.00	1.46±0.08	51.61± 5.63	2.92± 0.16
800	0.24±0.04	2.48±0.76	40.93± 5.78	4.96± 1.52	0.24±0.03	2.69±0.77	80.42± 8.00	5.38± 1.54
1000	0.54±0.02	14.13±1.03	38.82± 2.03	28.26± 2.06	0.38±0.04	8.32±1.61	133.71± 9.34	16.64± 3.22
1200	0.41±0.01	6.37±0.33	43.92± 7.51	12.74± 0.66	0.18±0.03	1.77±0.09	105.57±13.55	3.54± 0.18
1350	0.53±0.05	6.55±0.64	40.64± 5.61	13.10± 1.28	--	--	--	--
CALCINED KAOLIN/CALCIUM ALUMINATE CEMENT[j]								
Ambient	0.77±0.02	6.94±0.32	41.99± 6.57	13.88± 0.64				
100	0.58±0.04	4.64±0.64	51.27± 4.83	9.28± 1.28	0.55±0.03	4.24±0.44	53.87± 9.11	8.48± 0.88
200	0.59±0.05	4.95±0.75	51.62± 7.96	9.90± 1.50	0.53±0.02	4.23±0.29	56.65± 4.50	8.46± 0.58
400	0.58±0.04	4.78±0.49	57.63± 4.34	9.56± 0.98	0.51±0.03	4.06±0.50	70.68± 7.90	8.12± 1.00
600	0.54±0.02	4.52±0.24	45.87± 3.72	9.04± 0.48	0.50±0.02	4.19±0.29	88.87± 7.9	8.38± 0.58
800	0.58±0.02	5.00±0.59[k]	50.50± 9.30	10.00± 1.18[k]	0.47±0.06	3.52±0.92	113.93± 5.52	7.04± 1.84
1000	0.63±0.02	6.30±0.49[k]	54.9 ± 6.00	15.74± 0.98[k]	0.41±0.02	3.08±0.30	131.84±10.75	6.16± 0.60
1200	0.61±0.01	7.85±0.28	53.92± 5.10	15.70± 0.56	0.22±0.03	1.27±0.54	51.49± 4.89	2.54± 1.08
1350	0.84±0.03	3.75±0.23	52.93± 5.20	7.50± 0.46	--	--	--	--

(Table Continued)

EFFECT OF TEMPERATURE ON THE FRACTURE PROPERTIES[a] OF SEVERAL ALUMINA REFRACTORIES[b][16], Continued

Curing/Firing Temperature °C	Ambient Temperature Measurements[a]				Elevated Temperature Measurements[a]			
	Fracture Toughness K_{IC}, $MN/m^{3/2}$	Notched Beam Fracture Energy γ_{NBT}, J/m^2	Work of Fracture γ_{WOF}, J/m^2	Strain Energy Release Rate G_{IC}, J/M^2	Fracture Toughness K_{IC}, $MN/m^{3/2}$	Notched Beam Fracture Energy γ_{NBT}, J/m^2	Work of Fracture γ_{WOF}, J/m^2	Strain Energy Release Rate G_{IC}, J/m^2
CALCINED KAOLIN/CALCIUM ALUMINATE CEMENT[l]								
Ambient	0.91±0.05	11.85±1.35	48.20± 1.72	23.70± 2.70	--	--	--	--
100	0.68±0.02	8.32±0.67	51.84± 4.17	16.64± 1.34	--	--	--	--
200	0.53±0.01	4.36±0.33	46.48± 3.00	8.72± 0.66	0.45±0.05[m]	3.56±1.13[n]	50.00± 8.7	7.12± 2.26
400	0.46±0.04	4.00±0.86	57.20± 2.36	8.00± 1.72	0.38±0.01	2.69±0.19	58.9 ± 3.6	5.38± 0.38
600	0.54±0.01	4.57±0.45	52.23± 0.62	9.14± 0.90	0.39±0.03	2.60±0.35	99.7 ±14.1	5.20± 0.70
800	0.54±0.01	4.44±0.59	54.08± 1.90	8.88± 1.18	0.43±0.05	3.31±0.84	125.9 ±16.5	6.62± 1.68
1000	0.67±0.03	6.74±1.26	65.60± 6.70	13.48± 2.52	0.30±0.04	2.62±0.70	106.9 ±11.4	5.24± 1.40
1200	0.47±0.03	6.19±0.94	51.96± 3.50	12.38± 1.88	0.23±0.01	3.83±0.50	104.0 ±19.0	7.66± 1.00
1350	0.63±0.01	8.06±0.98	34.56± 3.24	16.12± 1.96	--	--	--	--
57% ALUMINA INSULATING CASTABLE[o]								
Ambient	0.29±0.02[p]	4.52±0.32	15.82± 3.08	9.04± 0.64	0.23±0.03[p]	5.11±0.70	20.62± 1.81	10.22± 1.40[q]
100	0.25±0.02	4.78±0.41	16.91± 3.11	9.56± 0.82	0.17±0.02	3.29±0.62	22.55± 2.71	6.58± 1.24
200	0.17±0.01	3.94±0.45	15.53± 0.51	7.88± 0.90	0.19±0.02	5.09±0.40	24.43± 4.25	10.18± 0.80
400	0.21±0.01	4.67±0.21	17.87± 1.54	9.34± 0.42	0.23±0.02	5.25±0.64	31.87± 5.62	10.50± 1.28
600	0.22±0.01	4.68±0.22	15.62± 1.54	9.36± 0.44	0.21±0.02	9.34±0.83	55.23± 6.87	18.68± 1.66
800	0.22±0.01	7.61±0.43	16.35± 1.08	15.22± 0.86	0.14±0.02	4.74±0.66	4.91± 1.51	9.48± 1.32
1000	0.17±0.01	4.75±0.52	7.37± 1.51	9.50± 1.04	--	2.54± 0.37	--	--
1200	0.26±0.01	6.84±0.74	12.34± 1.46	13.68± 1.48				
50% ALUMINA INSULATING CASTABLE[r]								
Ambient	0.27±0.01[s]	4.02±0.24	24.46± 3.27	8.04± 0.49	0.18±0.01	3.24±0.11[g]	27.00± 2.10	3.36± 0.22[g]
100	0.27±0.02	3.65±0.81[g]	20.43± 1.54	6.48± 1.45[g]	0.20±0.01	2.21±0.28	24.47± 1.52	4.42± 0.56
200	0.22±0.04	2.48±0.93	17.26± 1.30	4.96± 1.56[g]	0.25±0.01	2.54±0.35[g]	28.43± 6.40	8.02± 0.70[g]
400	0.21±0.01	2.63±0.24[g]	21.10± 1.99	3.92± 0.58[g]	0.20±0.01	2.94±0.28	33.53± 1.71	5.88± 0.56
600	0.24±0.01	3.69±0.45[g]	17.98± 3.06	9.52± 0.96[g]	0.20±0.02	3.60±0.73	64.67± 7.98	7.20± 1.46
800	0.25±0.02	4.67±0.44[g]	18.04± 1.93	9.42± 0.98[g]	0.18±0.01	3.42±0.53	39.47± 1.13	6.84± 1.06
1000	0.22±0.01	3.96±0.52	22.30± 1.59	7.92± 0.98	--	--	--	--
1200	0.23±0.02	4.10±0.58	11.66± 0.62	8.2 ± 1.03				

a Fracture toughness was measured by the single edge notched beam method; notch width was saw cut to a depth of 1/2 the specimen thickness, average notch width was ~0.032 in. Five to seven samples for each set of conditions were tested in three-point bending, crosshead speed 0.05 in./min. One set of samples was tested at ambient temperature, a second set was heated in a globar furnace and tested at the curing/firing temperatures when the samples reached thermal equilibrium. Samples were 1 in. x 1 in. x 7 in. [See Sections B.3.2.34-B.3.2.35 for plotted version for some of these data.]

b Samples were cast, vibrated one minute, cured sealed in plastic 24 hours at ambient temperature, cured in air 24 hours at ambient temperature, dried in air 24 hours at 100 °C, fired at 60 °C/h to 200, 400, 600, 800, 1000, 1200, and 1350 °C, soaked at temperature 5 hours and furnace cooled.

c 75% T-61 alumina (Alcoa) aggregate, 25% Casting Grade CA-25 cement (Alcoa), water to ball-in-hand consistency 10.4% of aggregate and cement.

d Misprinted as 0.53±1.30 in table in the original report.

e 75% T-61 alumina (Alcoa) aggregate, 25% Secar 71 cement (Lone Star Lafarge), water to ball-in-hand consistency 12.7% of aggregate and cement.

f 75% Mulcoa M-60 (C-E Minerals) aggregate, 25% Casting Grade CA-25 (Alcoa) cement, water to ball-in-hand consistency 10.6% of aggregate and cement.

g Values of γ_{NBT} and G_{IC} are not consistent with each other.

h Apparently misprinted as 8.86±0.56 in the original report.

i 75% Mulcoa M-60 (C-E Minerals) aggregate, 25% Secar 71 cement (Lone Star Lafarge), water to ball-in-hand consistency 14.6% of aggregate and cement.

j 75% Mulcoa M-47 (C-E Minerals) aggregate, 25% Casting Grade CA-25 (Alcoa) cement, water to ball-in-hand consistency 11.3% of aggregate and cement.

k Values of γ_{NBT} and G_{IC} are not consistent. See Section B.3.2.36 for plotted data. Two different γ_{NBT} figures were given in the set of reports, one agreeing with the tabulated γ_{NBT} value here and the other agreeing with the tabulated G_{IC} (= $2\gamma_{NBT}$) value here.

l 75% Mulcoa M-47 (C-E Minerals) aggregate, 25% Refcon cement (Universal Atlas), water to ball-in-hand consistency 13.5% of aggregate and cement.

m All these tabulated values of fracture toughness for this refractory do not agree with the figure given in Section B.3.2.34 taken from the same set of reports.

n All these tabulated values of notch beam fracture surface energy do not agree with the figure given in Section B.3.2.36 taken from the same reports.

o A commercial lightweight insulating castable (Cer-Lite #75, C-E Refractories), water to ball-in-hand consistency 22% of mix.

p All these tabulated values of fracture toughness for this refractory do not agree with the figure in Section B.3.2.34 from the same set of reports. If these values are correct then probably the points plotted in the figure were mistakenly plotted on the ksi√in. scale.

q In the original report the values in this column are mistakenly placed down one line and the ambient temperature data value inserted on the 100 °C line.

r A commercial lightweight insulating castable (Litecast 75-28, General Refractories), water to ball-in-hand consistency 24% of mix.

s All these tabulated values of fracture toughness for this refractory do not agree with the figure in Section B.3.2.34 taken from the same set of reports or with another figure also given in the reports but not included in B.3.2.34.

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ELASTIC MODULI[a] AS A FUNCTION OF FIRING TEMPERATURES FOR SEVERAL ALUMINA REFRACTORIES[b][16]
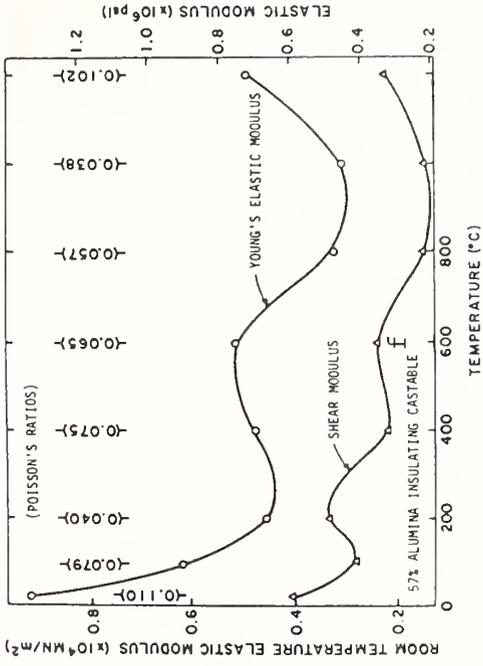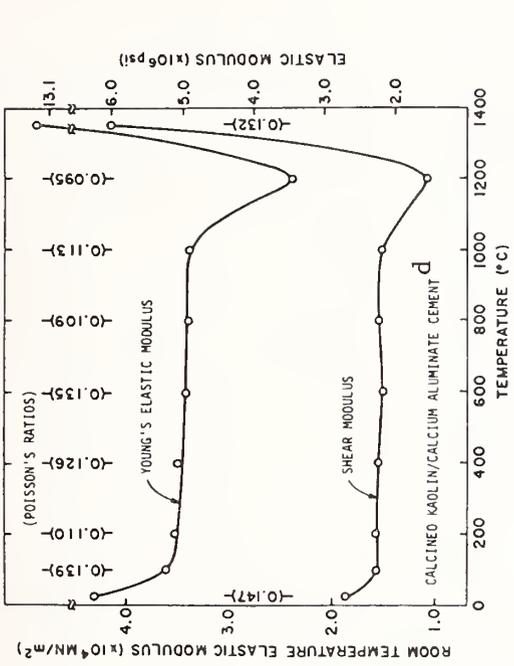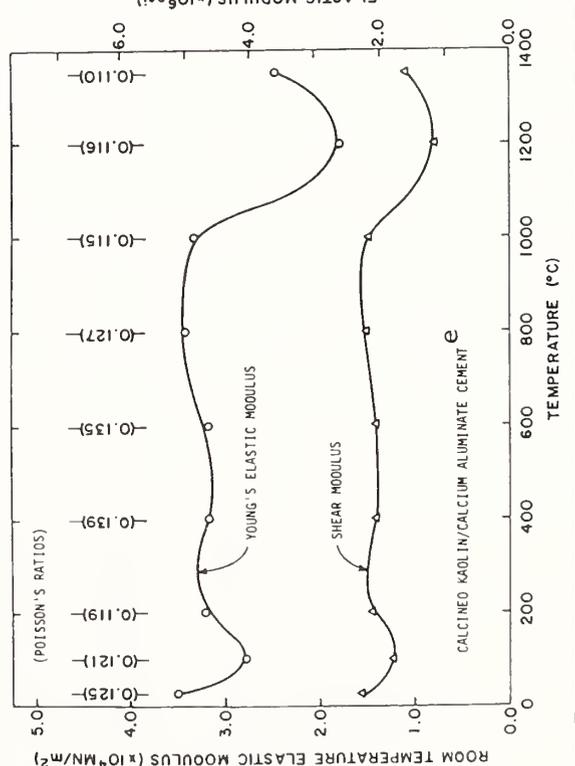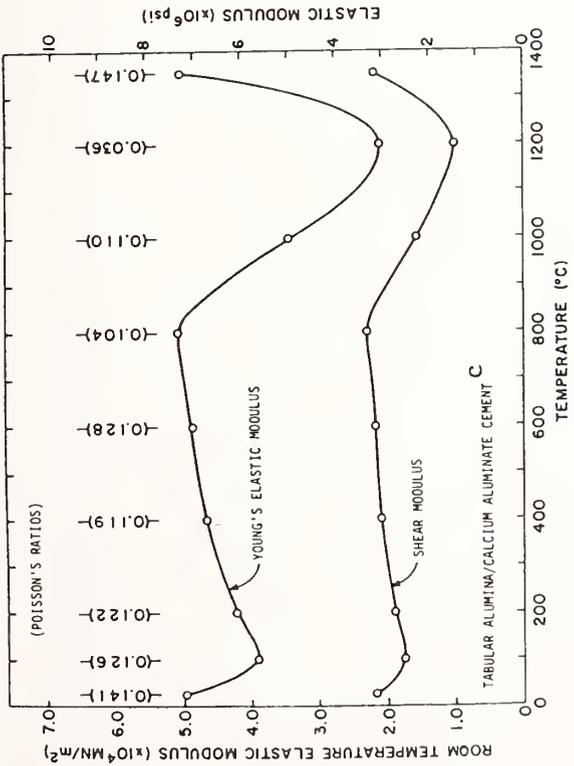
(Data Continued)

ELASTIC MODULI[a] AS A FUNCTION OF FIRING TEMPERATURES FOR SEVERAL ALUMINA REFRACTORIES[b][16], Continued
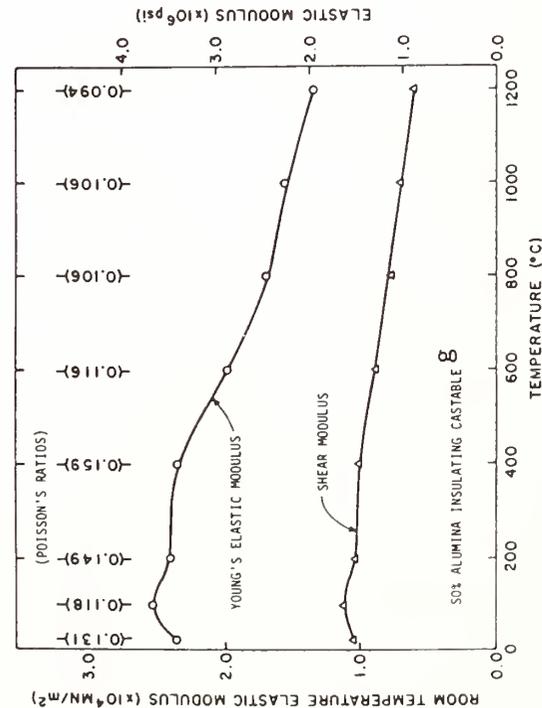
[a]Elastic moduli were determined by dynamic mechanical resonance method. Specimens 0.65 cm x 2.54 cm x 17.78 cm were saw cut from larger cast specimens. Two specimens of each refractory at each curing/firing temperature were measured. Measurements were made at ambient temperature. Flexural and torsional resonance frequencies were obtained using an acoustic spectrometer and the elastic moduli were then calculated. See Section B.3.2.81 for these data tabulated and Section B.3.2.33 for other figures of the Young's moduli.

[b]Samples were cast, vibrated one minute, cured sealed in plastic 24 hours at ambient temperature, cured in air 24 hours at ambient temperature, dried in air 24 hours at 100 °C, fired at 60 °C/h to 200, 400, 600, 800, 1000, 1200, and 1350 °C, soaked at temperature 5 hours, and furnace cooled to ambient temperature.

[c]75% T-61 alumina (Alcoa) aggregate, 25% Casting Grade CA-25 cement (Alcoa), water to ball-in-hand consistency 10.4% of aggregate and cement.

[d]75% Mulcoa M-47 (C-E Minerals) aggregate, 25% Casting Grade CA-25 (Alcoa) cement, water to ball-in-hand consistency 11.3% of aggregate and cement.

[e]75% Mulcoa M-47 (C-E Minerals) aggregate, 25% Refcon cement (Universal Atlas), water to ball-in-hand consistency 13.5% of aggregate and cement.

[f]A commercial lightweight insulating castable (Cer-Lite #75, C-E Refractories), water to ball-in-hand consistency 22% of mix.

[g]A commercial lightweight insulating castable (Litecast 75-28, General Refractories), water to ball-in-hand consistency 24% of mix.

ELASTIC MODULUS (x10^6 psi)

ROOM TEMPERATURE ELASTIC MODULUS (x10^4 MN/m^2)

TEMPERATURE (°C)

(POISSON'S RATIOS)

YOUNG'S ELASTIC MODULUS

SHEAR MODULUS

50% ALUMINA INSULATING CASTABLE [g]

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FLEXURAL STRENGTHS[a] OF SEVERAL ALUMINA REFRACTORIES[b] AT VARIOUS TEMPERATURES[16]

Curing/Firing Temperature °C	Flexural Strength,[a] MN/m² (=MPa)	
	At Ambient Temperature	At Elevated Temperatures
TABULAR ALUMINA/CALCIUM ALUMINATE CEMENT[c] - - - - - - - - - - - - - - -		
Ambient	10.06 ± 1.34	
100	12.06 ± 0.56	8.88 ± 0.91
200	9.49 ± 0.98	7.70 ± 0.45
400	11.14 ± 1.11	9.34 ± 0.99
600	10.27 ± 0.99	8.83 ± 0.73
800	11.23 ± 0.50	9.69 ± 0.65
1000	7.52 ± 1.20	7.92 ± 0.96
1200	6.23 ± 2.04	6.86 ± 0.90
1350	16.62 ± 1.70	--
TABULAR ALUMINA/CALCIUM ALUMINATE CEMENT[d] - - - - - - - - - - - - - - -		
Ambient	9.50 ± 0.20	
100	9.27 ± 0.63	8.08
200	5.94 ± 0.28	5.62
400	5.66 ± 0.55	5.44
600	4.72 ± 0.20	3.69
800	5.48 ± 0.24	5.25
1000	3.64 ± 0.14	3.09
1200	3.40 ± 0.41	3.34
1350	7.66 ± 0.36	--
CALCINED BAUXITE/CALCIUM ALUMINATE CEMENT[e] - - - - - - - - - - - - - -		
Ambient	11.03 ± 0.43	
100	7.87 ± 0.35	7.39 ± 0.64
200	7.83 ± 0.55	7.54 ± 0.25
400	8.12 ± 0.48	7.66 ± 0.47
600	8.20 ± 0.61	7.50 ± 0.14
800	8.11 ± 0.34	7.66 ± 0.64
1000	8.02 ± 0.40	6.59 ± 0.34
1200	11.23 ± 0.86	5.26 ± 0.29
1350	19.77 ± 0.62	--
CALCINED BAUXITE/CALCIUM ALUMINATE CEMENT[f] - - - - - - - - - - - - - -		
Ambient	8.51 ± 0.58	
100	7.98 ± 0.68	7.71 ± 0.32
200	4.94 ± 0.32	4.51 ± 0.20
400	2.20 ± 0.11	2.09 ± 0.13
600	2.79 ± 0.38	2.37 ± 0.08
800	2.77 ± 0.43	2.47 ± 0.40
1000	3.03 ± 0.20	2.36 ± 0.17
1200	3.88 ± 0.37	1.52 ± 0.33
1350	8.13 ± 0.48	--

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(Table Continued)

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FLEXURAL STRENGTHS[a] OF SEVERAL ALUMINA REFRACTORIES[b] AT VARIOUS
TEMPERATURES[16], Continued

Curing/Firing Temperature °C	Flexural Strength, [a] MN/m^2 (=MPa)	
	At Ambient Temperature	At Elevated Temperatures
CALCINED KAOLIN/CALCIUM ALUMINATE CEMENT[g] – – – – – – – – – – – – – – – – –		
Ambient	8.51 ± 1.06	
100	9.74 ± 0.74	8.71 ± 0.70
200	7.86 ± 0.77	8.07 ± 0.39
400	7.94 ± 0.99	8.00 ± 0.45
600	8.03 ± 0.48	6.70 ± 0.62
800	8.35 ± 0.60	5.29 ± 0.18
1000	7.94 ± 0.56	4.17 ± 0.52
1200	9.01 ± 0.78	3.53 ± 0.48
1350	17.15 ± 1.03	--
CALCINED KAOLIN/CALCIUM ALUMINATE CEMENT[h] – – – – – – – – – – – – – – – –		
Ambient	9.74 ± 1.08	
100	9.58 ± 1.31	9.31 ± 0.83
200	7.88 ± 0.63[i]	8.20 ± 0.50
400	6.42 ± 0.30	6.17 ± 0.28[i]
600	6.51 ± 0.93	5.24 ± 0.51
800	6.84 ± 0.48	4.96 ± 0.32[i]
1000	6.57 ± 0.42[i]	4.44 ± 0.47[i]
1200	6.20 ± 0.10[i]	1.88 ± 0.19
1350	10.24 ± 0.73	--
57% ALUMINA INSULATING CASTABLE[j] – – – – – – – – – – – – – – – – – –		
Ambient	4.54 ± 0.14	
100	4.77 ± 0.20	4.78 ± 0.36
200	2.76 ± 0.09	2.71 ± 0.15
400	4.24 ± 0.18	3.64 ± 0.09
600	3.56 ± 0.13	3.71 ± 0.09
800	3.11 ± 0.26	2.97 ± 0.26
1000	1.21 ± 0.15	0.78 ± 0.06
1200	2.39 ± 0.59	1.17 ± 0.36
50% ALUMINA INSULATING CASTABLE[k] – – – – – – – – – – – – – – – – – –		
Ambient	4.56 ± 0.19	
100	4.50 ± 0.45	4.16 ± 0.39
200	2.68 ± 0.38	2.60 ± 0.15
400	2.94 ± 0.13	2.98 ± 0.16
600	3.07 ± 0.18	3.37 ± 0.20
800	2.86 ± 0.16	3.68 ± 0.21
1000	2.34 ± 0.11	1.57 ± 0.13
1200	3.06 ± 0.35	0.68 ± 0.10

[a]Flexural strength was measured in three-point bending, crosshead speed
0.05 in./min over a span of 6 in. One set of samples was tested at am-
- -
(Table Continued)

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FLEXURAL STRENGTHS[a] OF SEVERAL ALUMINA REFRACTORIES[b] AT VARIOUS
TEMPERATURES[16], Continued

Footnotes continued

bient temperature, the second set was heated in a globar furnace and tested
at the firing temperatures when the samples reached thermal equilibrium.
Samples were 1 in. x 1 in. x 7 in. See Section B.3.2.32 for plotted versions
of some of the data given here.

[b]Samples were cast, vibrated one minute, cured sealed in plastic 24 hours at
ambient temperature, cured in air 24 hours at ambient temperature, dried in
air 24 hours at 100 °C, fired at 60 °C/h to 200, 400, 600, 800, 1000, 1200,
and 1350 °C, soaked at temperature 5 hours, and furnace cooled to ambient
temperature.

[c]75% T-61 alumina (Alcoa) aggregate, 25% Casting Grade CA-25 cement (Alcoa),
water to ball-in-hand consistency 10.4% of aggregate and cement.

[d]75% T-61 alumina (Alcoa) aggregate, 25% Secar 71 cement (Lone Star Lafarge),
water to ball-in-hand consistency 12.7% of aggregate and cement.

[e]75% Mulcoa M-60 (C-E Minerals) aggregate, 25% Casting Grade CA-25 (Alcoa)
cement, water to ball-in-hand consistency 10.6% of aggregate and cement.

[f]75% Mulcoa M-60 (C-E Minerals) aggregate, 25% Secar 71 cement (Lone Star
Lafarge), water to ball-in-hand consistency 14.6% of aggregate and cement.

[g]75% Mulcoa M-47 (C-E Minerals) aggregate, 25% Casting Grade CA-25 (Alcoa)
cement, water to ball-in-hand consistency 11.3% of aggregate and cement.

[h]75% Mulcoa M-47 (C-E Minerals) aggregate, 25% Refcon cement (Universal
Atlas), water to ball-in-hand consistency 13.5% of aggregate and cement.

[i]These values do not agree with those plotted in the figures given in the
same set of reports. See Section B.3.2.32.

[j]A commercial lightweight insulating castable (Cer-Lite #75, C-E Refractories),
water to ball-in-hand consistency 22% of mix.

[k]A commercial lightweight insulating castable (Litecast 75-28, General Re-
fractories), water to ball-in-hand consistency 24% of mix.

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EFFECT OF TEMPERATURE[a] AND AGGREGATE SIZE DISTRIBUTION[b] ON THE FLEXURAL STRENGTH[c] OF A HIGH-ALUMINA CASTABLE[d][16]

Curing/Firing Temperature °C	Flexural Strength,[c] MN/m^2			
	Generic Castable[b]	Continuous Aggregate Formulation[b]		
		20% Cement	25% Cement	30% Cement
Ambient	10.06±1.34	10.47±1.56	10.29±1.32	10.78±1.40
100	8.88±0.91	7.65±1.86	9.23±1.09	9.95±0.68
200	7.70±0.45	7.64±0.50	9.70±1.34	8.62±0.42
400	9.34±0.99	7.95±0.61	8.17±0.52	9.19±0.91
600	8.83±0.73	7.94±0.48	9.55±0.95	8.88±0.30
800	9.69±0.65	8.31±0.66	9.65±1.02	10.50±0.80
1000	7.92±0.96	7.90±1.43	10.66±1.09	10.60±0.74
1200	6.86±0.90	7.65±0.89	8.88±1.11	10.72±0.97

	Gap-sized Aggregate Formulation[b]		
	20% Cement	25% Cement	30% Cement
Ambient	10.70±0.64	11.40±2.61	13.82±1.50
100	9.18±1.17	12.10±2.07	11.29±1.59
200	8.60±1.04	7.21±0.60	9.61±0.44
400	5.16±0.54	6.82±0.78	8.57±1.36
600	5.95±0.62	8.51±1.96	10.67±1.49
800	6.33±0.75	9.38±2.82	9.94±0.83
1000	6.62±1.01	7.81±1.29	6.67±0.90
1200	3.96±0.84	6.49±0.82	5.34±0.80

[a] Tests were performed on the samples at the curing/firing temperatures. Samples were heated in a globar furnace and tested when they reached thermal equilibrium.

[b] Various samples were prepared to study the effect of aggregate particle size distribution on the strength of the castable. The generic castable has a size distribution typical of a standard industrial product and is the as-received material. The cement content is 25%. The other two formulations are designed for maximum density of the aggregate and the distributions were calculated according to formulas for each case. The continuous formula contains aggregate continuously graded so that the difference between successive particle sizes is a factor of $\sqrt{2}$, with the largest particles at U.S. Sieve Number 4 x 6 and the smallest -325. Gap-sized packing provides sizes and proportions so that the larger voids created between larger particles are filled by smaller particles and new voids thus created filled by even smaller particles. The formulation used here is 61.5% of 4x6, 24.5% of 16 x 20, 9.8% of 70 x 100, and 4.2% of -325 mesh.

[c] Flexural strength was measured in three-point bending, crosshead speed 0.05 in/min over a 6 in. span. Samples were 1 in. x 1 in. x 7 in. See Section B.3.2.86 for plotted versions of these data.

[d] 75% T-61 alumina (Alcoa) aggregate, 25% Casting Grade CA-25 (Alcoa) cement. Water to ball-in-hand consistency: generic 10.3%; continuous 20% cement 9.9%, 25% cement 10.6%, 30% cement 11.1%; gap-sized 20% cement 8.0%, 25% cement 9.1%, 30% cement 9.8% of aggregate and cement.

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FLEXURAL STRENGTHS[a] OF A HIGH-ALUMINA CASTABLE WITH VARIOUS AGGREGATE PARTICLE SIZE DISTRIBUTIONS[16]
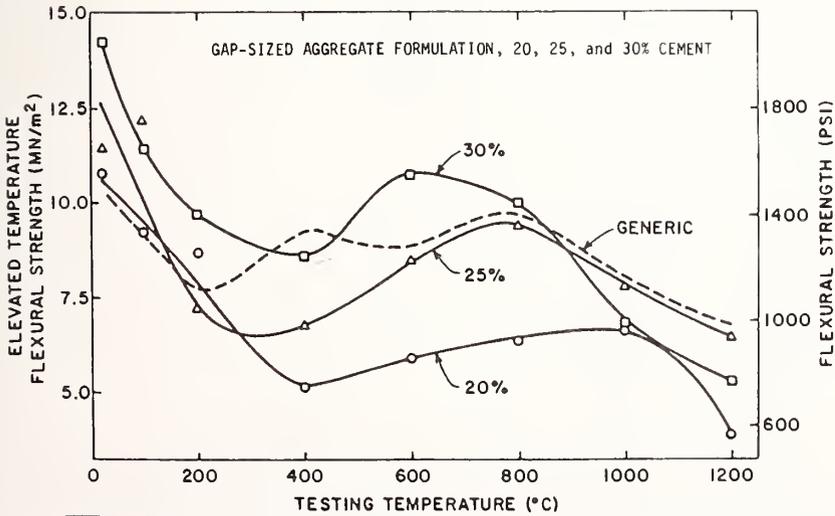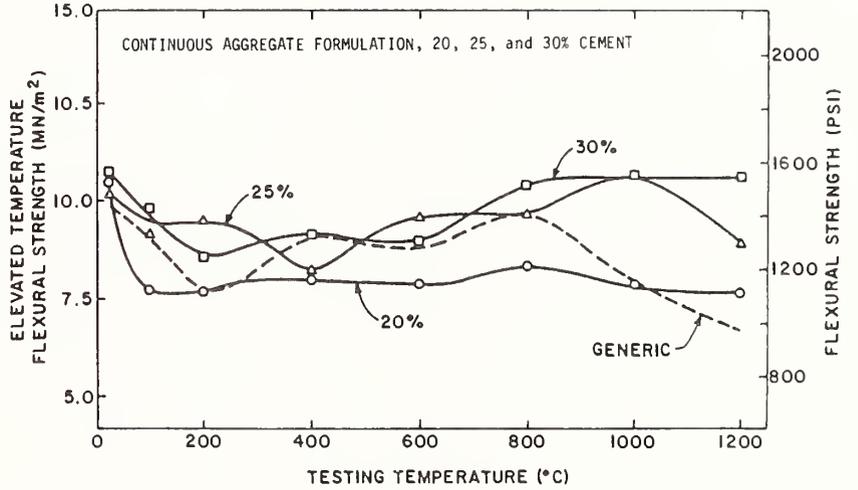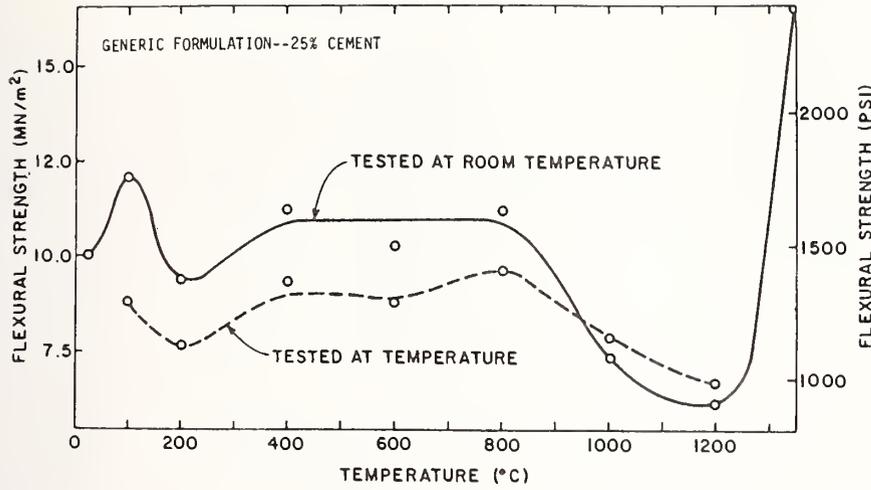
[a]See Section B.3.2.85 for the data plotted here.

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EFFECT OF TEMPERATURE[a] AND AGGREGATE SIZE DISTRIBUTION[b] ON THE ELASTIC
PROPERTIES[c] OF A HIGH-ALUMINA CASTABLE[d][16]

Curing/Firing Temperature °C	Young's Modulus (x 10^4 MN/m^2)	Shear Modulus	Poisson's Ratio	Young's Modulus (x 10^4 MN/m^2)	Shear Modulus	Poisson's Ratio
	GENERIC FORMULATION[b] - - - - -			CONTINUOUS FORMULATION, 20%[b] -		
Ambient	4.98	2.18	0.141	6.66	2.80	0.191
100	3.90	1.73	0.127	4.93	2.13	0.157
200	4.14	1.84	0.128	4.37	1.88	0.163
400	4.38	1.96	0.116	4.70	1.87	0.168
600	4.45	1.98	0.126	4.28	1.84	0.164
800	4.33	1.97	0.100	4.11	1.79	0.150
1000	3.03	1.35	0.121	4.32	1.93	0.122
1200	1.50	0.71	0.048	2.47	1.13	0.090
	CONTINUOUS FORMULATION, 25%[b] -			CONTINUOUS FORMULATION, 30%[b] -		
Ambient	6.40	2.73	0.169	6.49	2.74	0.184
100	5.01	2.15	0.163	5.89	2.49	0.181
200	4.52	1.98	0.142	4.68	2.01	0.161
400	4.07	1.81	0.125	4.32	1.88	0.147
600	4.45	2.00	0.113	3.88	1.67	0.160
800	4.45	1.94	0.148	3.78	1.67	0.128
1000	4.08	1.85	0.102	4.08	1.77	0.152
1200	2.00	0.91	0.090	1.83	0.86	0.063
	GAP-SIZED FORMULATION, 20%[b] -			GAP-SIZED FORMULATION, 25%[b] -		
Ambient	8.36	3.65	0.143	7.03	3.10	0.131
100	7.41	3.29	0.125	6.98	3.10	0.126
200	7.02	3.10	0.131	5.82	2.65	0.097
400	6.36	2.87	0.107	5.94	2.65	0.121
600	6.05	2.81	0.084	6.25	2.79	0.121
800	5.97	2.63	0.133	5.93	2.57	0.150
1000	7.98	3.44	0.157	6.79	2.96	0.146
1200	4.71	2.23	0.053	4.60	2.16	0.065
	GAP-SIZED FORMULATION, 30%[b] -					
Ambient	7.54	3.29	0.1444			
100	6.83	2.98	0.1444			
200	5.90	2.66	0.109			
400	5.11	2.33	0.095			
600	5.28	2.38	0.110			
800	4.96	2.23	0.109			
1000	3.62	1.71	0.056			
1200	3.37	1.51	0.113			

[a]Tests were performed on the samples at the curing/firing temperatures. Samples
were heated in a globar furnace and tested when they reached thermal equilibrium.

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(Table Continued)

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EFFECT OF TEMPERATURE[a] AND AGGREGATE SIZE DISTRIBUTION[b] ON THE ELASTIC
PROPERTIES[c] OF A HIGH-ALUMINA CASTABLE[d][16], Continued

Footnotes continued

[b]Various samples were prepared to study the effect of aggregate particle size
distribution on the properties of the castable. The generic castable has a
size distribution typical of a standard industrial product and is the as-
received material. The cement content is 25%. The other two formulations
are designed for maximum density of the aggregate and the distributions were
calculated according to formulas for each case. The continuous formula con-
tains aggregate continuously graded so that the difference between successive
particle sizes is a factor of $\sqrt{2}$, with the largest particles sized at U.S.
Sieve Number 4 x 6 and the smallest -325. Gap-sized packing provides sizes
and proportions so that the larger voids created between larger particles are
filled by smaller particles and new voids thus created filled by even smaller
particles. The formulation used here is 61.5% of 4x6, 24.5% of16x20, 9.8% of
70x100, and 4.2% of -325 mesh.

[c]Elastic moduli were determined by dynamic mechanical resonance method. Spec-
imens 0.65 cm x 2.54 cm x 17.78 cm were saw cut from larger cast specimens.
Flexural and torsional resonance frequencies were obtained using an acoustic
spectrometer and the elastic moduli were then calculated. See Sections
B.3.2.88 and B.3.2.89 for plotted versions of the data.

[d]T-61 tabular alumina (Alcoa) aggregate, Casting Grade CA-25 (Alcoa) cement.
Generic formulation 75% aggregate, 25% cement, water to ball-in-hand consis-
tency 10.3% of aggregate and cement. Continuous formula, 20% cement 9.9% water,
25% cement 10.6% water, 30% cement 11.1% water; gap-sized, 20% cement 8.0%
water, 25% cement 9.1% water, 30% cement 9.8% water.

ELASTIC PROPERTIES[a] OF A HIGH-ALUMINA CASTABLE WITH VARIOUS AGGREGATE PARTICLE SIZE DISTRIBUTIONS[16]
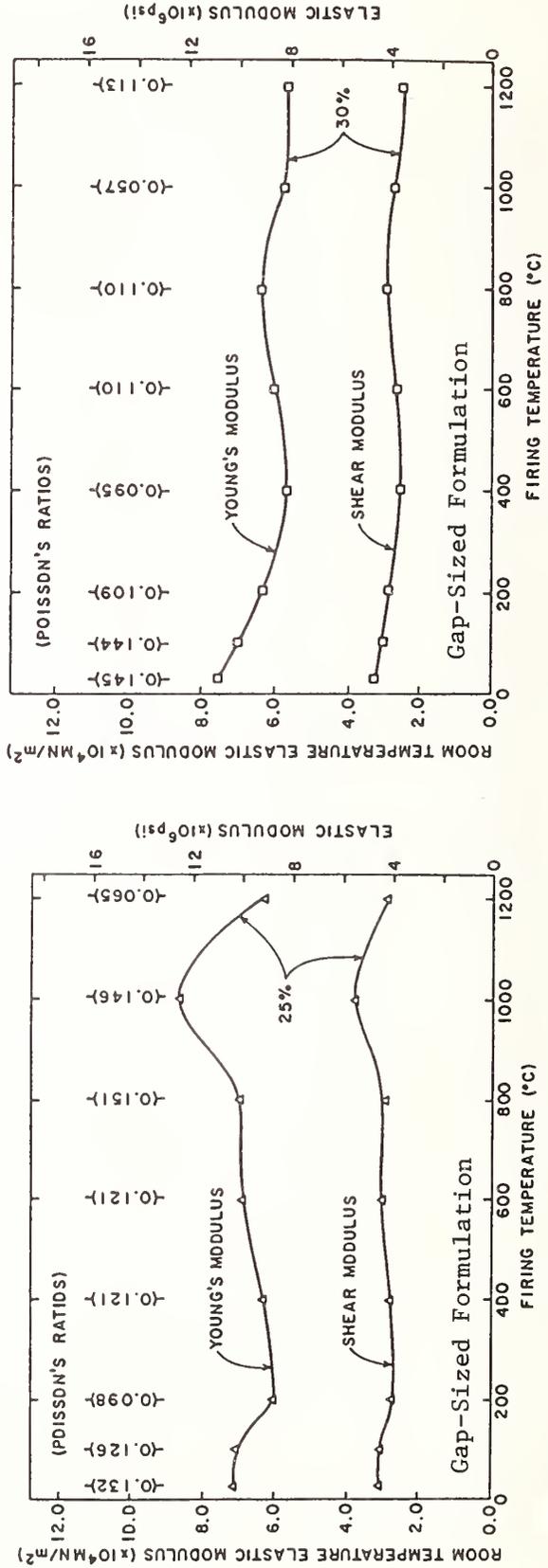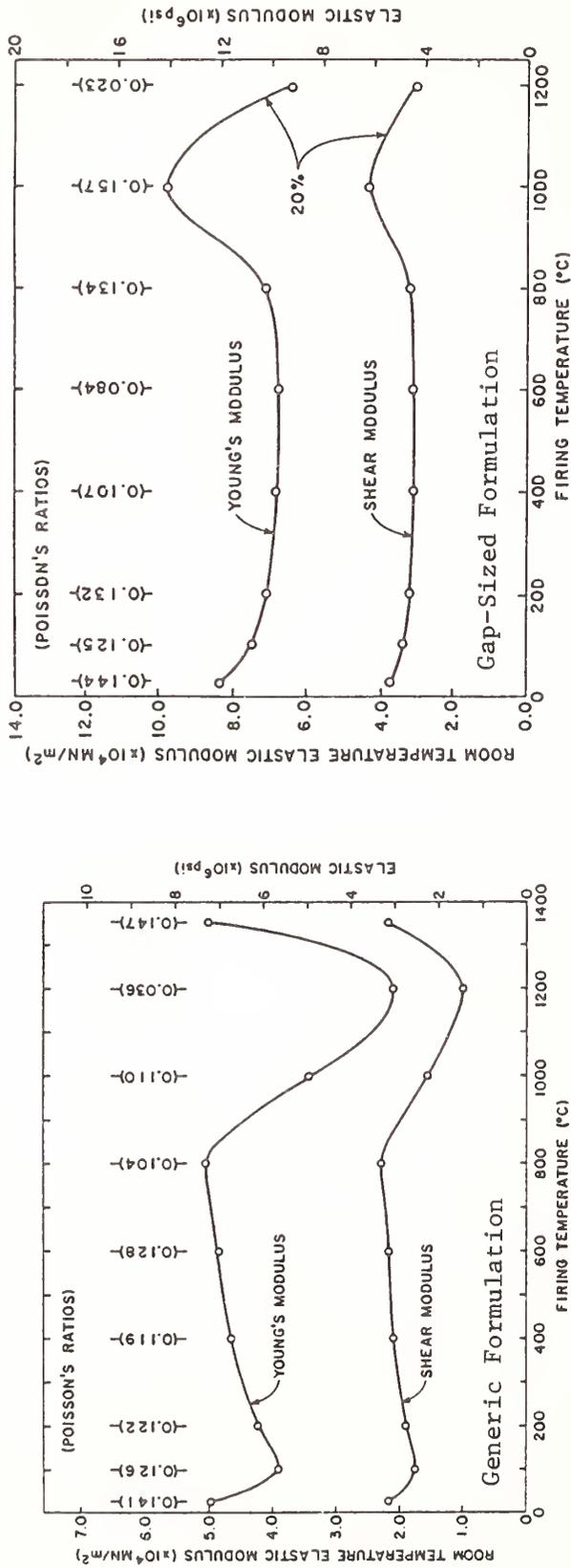
[a]See Section B.3.2.87 for data plotted here.

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YOUNG'S MODULUS[a] OF A HIGH-ALUMINA CASTABLE WITH VARIOUS AGGREGATE PARTICLE SIZE DISTRIBUTIONS[16]
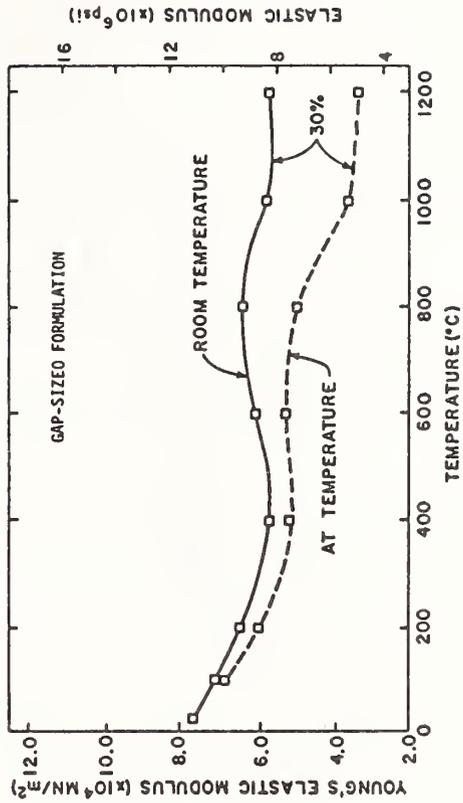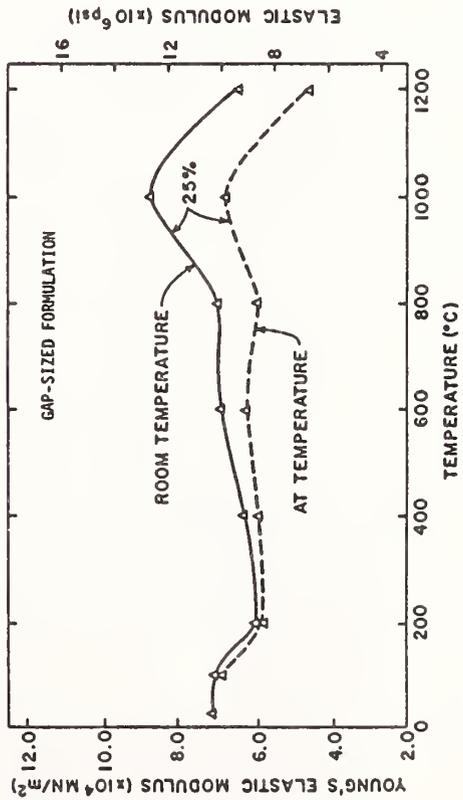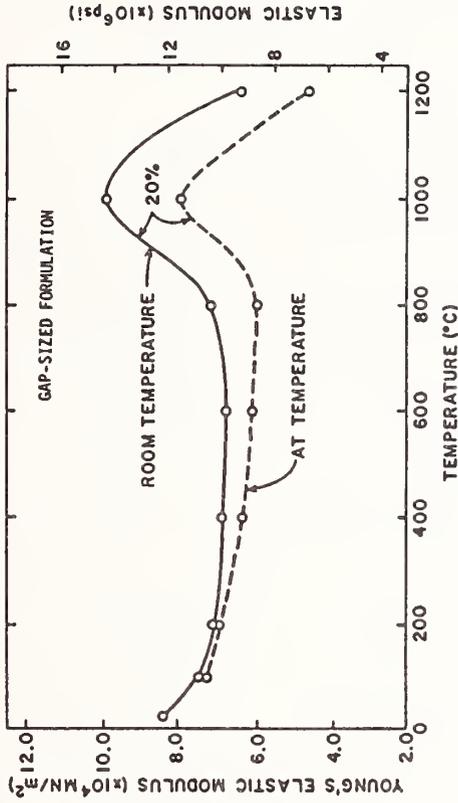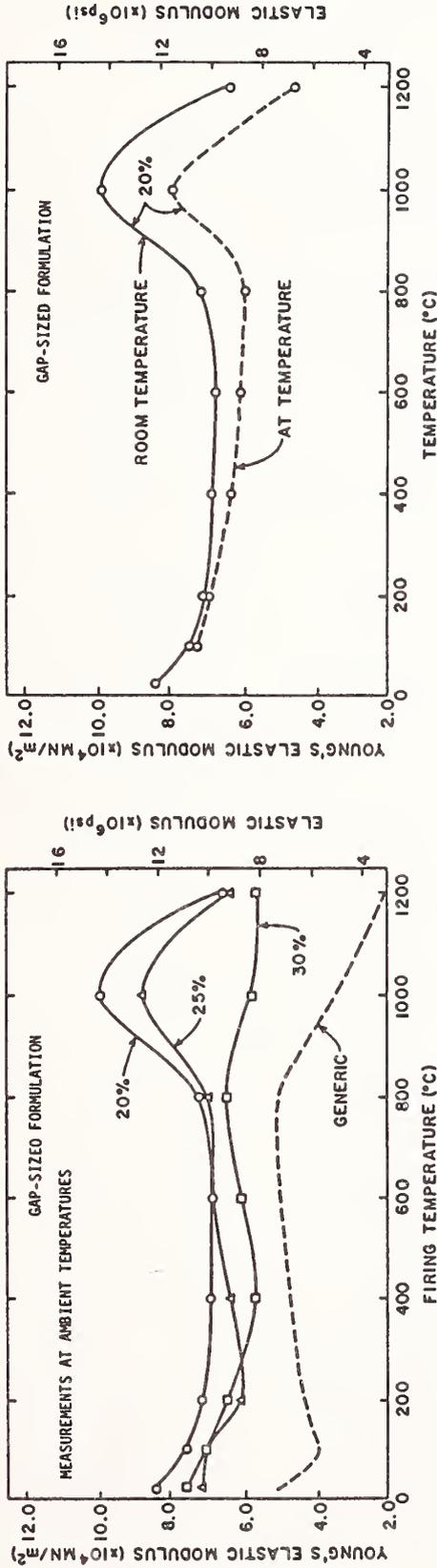
(Data Continued)

YOUNG'S MODULUS[a] OF A HIGH-ALUMINA CASTABLE WITH VARIOUS AGGREGATE PARTICLE SIZE DISTRIBUTIONS[16]
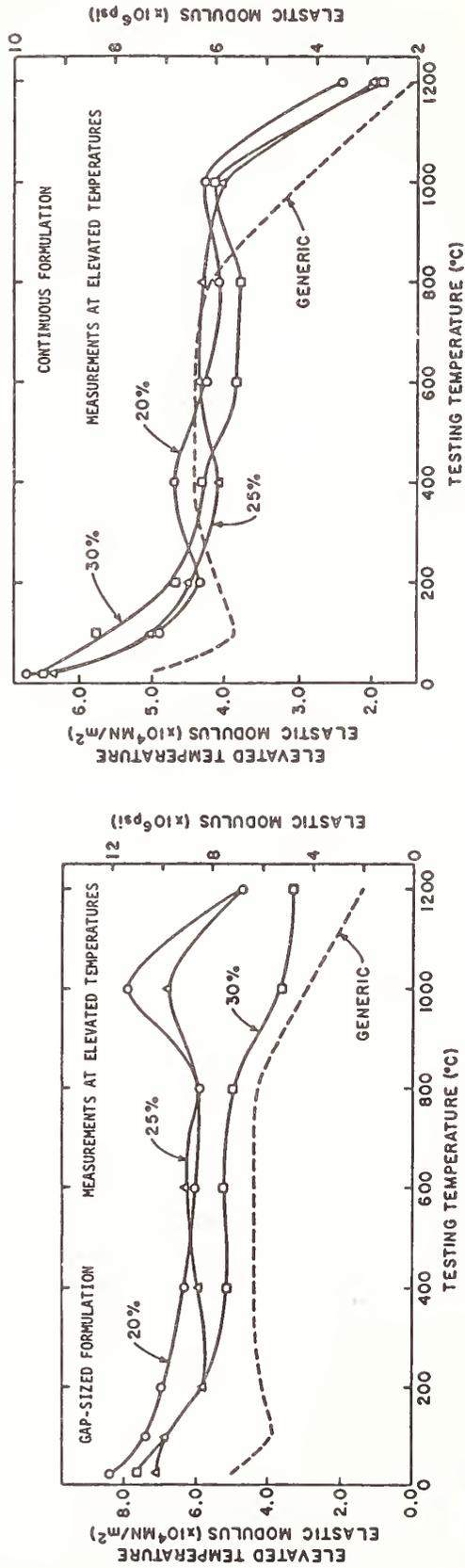
[a] See Section B.3.2.87 for the data plotted here.

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EFFECT OF TEMPERATURE[a] AND AGGREGATE PARTICLE SIZE DISTRIBUTION[b] ON THE FRACTURE
PROPERTIES[c] OF A HIGH-ALUMINA CASTABLE[d][16]

Curing/Firing Temperature °C	Fracture Toughness K_{IC}, MN/m$^{3/2}$	Notched Beam Fracture Energy γ_{NBT}, J/m^2	Work of Fracture γ_{WOF}, J/m^2	Strain Energy Release Rate G_{IC}, J/m^2	J-Integral J_{IC}, J/m^2
GENERIC FORMULATION[b] MEASURED AT AMBIENT TEMPERATURE					
Ambient	0.67±0.07	4.58±0.99	46.52± 8.42	9.17±1.98	11.24± 2.07
100	0.68±0.09	5.95±1.39	46.02±12.25	11.89±2.77	12.55± 1.34
200	0.70±0.14	6.53±0.74	53.45± 8.18	13.05±1.48	16.11± 1.93
400	0.68±0.08	5.50±0.83	51.02± 8.80	10.99±1.66	14.69± 1.84
600	0.65±0.08	4.35±1.01	61.05±12.83	8.70±2.18	16.08± 1.76
800	0.65±0.09	4.77±0.65	66.25± 9.06	9.53±1.30[e]	16.64± 2.42
1000	0.77±0.11	8.78±2.53	55.24± 6.95	17.56±5.07	21.79± 2.78
1200	0.53±0.10	7.14±2.76	44.22± 5.61	14.28±5.52	16.56± 1.53
1350	1.02±0.09	10.46±1.98	66.14±11.96	20.92±3.96	27.27± 2.46
GENERIC FORMULATION MEASURED AT CURING/FIRING TEMPERATURE					
100	0.45±0.03	2.62±0.37	54.19± 8.45	5.24±0.73	18.55± 3.03
200	0.58±0.04	4.07±0.53	77.85±15.18	8.15±1.06	27.56± 2.40
400	0.47±0.03	2.51±0.31	69.39± 3.84	5.02±0.63	22.31± 3.72
600	0.47±0.04	2.53±0.43	73.74±13.61	5.05±0.86	24.51± 4.55
800	0.63±0.03	4.62±0.48	88.11±12.09	9.23±0.96	31.99± 3.82
1000	0.51±0.06	4.38±1.03	110.69±22.54	8.75±2.06	43.28± 8.47
1200	0.44±0.07	6.52±2.08	118.72±14.05	13.04±4.16	47.61± 4.33
CONTINUOUS FORMULATION[b] WITH 20% CEMENT MEASURED AT CURING/FIRING TEMPERATURE					
Ambient	0.60±0.10	2.76±0.88	56.09±17.06	5.52±1.77	18.53± 3.57
100	0.53±0.05	2.81±0.52	49.67±17.27	5.61±1.03	15.39± 3.50
200	0.53±0.08	3.31±0.90	56.74± 4.63	6.61±1.79	24.34± 3.10
400	0.51±0.05	2.74±0.52	65.19±13.97	5.48±1.03	23.51± 4.80
600	0.46±0.05	2.49±0.51	56.55± 8.16	4.97±1.01	22.70± 2.57
800	0.61±0.04	4.54±0.63	116.61±45.75	9.07±1.25	39.17± 3.32
1000	0.75±0.20	6.69±3.21	100.12±12.27	13.38±6.41	36.41± 4.57
1200	0.51±0.07	5.31±1.38	196.51±43.31	10.63±2.77	51.22± 5.57
CONTINUOUS FORMULATION WITH 25% CEMENT MEASURED AT CURING/FIRING TEMPERATURE					
Ambient	0.78±0.07	4.72±0.85	67.54±15.54	9.45±1.71	25.19±11.47
100	0.56±0.14	3.44±1.71[f]	43.16± 6.86	6.88±3.43	12.43± 1.19
200	0.58±0.08	3.68±1.01	65.78±10.55	7.37±2.02	19.70± 4.02
400	0.52±0.04	3.28±0.50	62.24±10.88	6.56±1.00	26.25± 3.28
600	0.57±0.04	3.60±0.57	73.02±19.91	7.21±1.14	30.56± 5.42
800	0.59±0.11	3.93±1.56	110.17±22.29	7.86±3.12	38.23± 3.02
1000	0.85±0.12	9.03±2.48	108.82±32.93	18.06±4.97	45.29± 4.01
1200	0.52±0.07	6.86±1.86	205.67±44.41	19.73±3.73[g]	58.54± 7.56
CONTINUOUS FORMULATION WITH 30% CEMENT MEASURED AT CURING/FIRING TEMPERATURE					
Ambient	0.86±0.17	5.80±2.34	70.86±11.52	11.60±4.68	25.53± 8.86
100	0.72±0.06	4.42±0.75	44.07± 7.30	8.85±1.50	20.93± 4.14
200	0.56±0.10	3.34±1.18	61.82±10.42	6.68±2.37	19.77± 3.74
400	0.55±0.05	3.44±0.59	66.02±10.46	6.89±1.18	27.04± 2.58
600	0.55±0.02	3.82±0.28	68.86±26.41	7.64±0.57	28.91± 4.13
800	0.67±0.06	5.99±1.05	97.80±12.57	11.98±2.11	41.99± 3.22
1000	0.78±0.14	7.60±2.42	98.41±13.66	15.20±4.84	38.47± 6.73
1200	0.60±0.07	9.78±2.41	233.50±24.55	19.57±4.82	57.56±10.79

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(Table Continued)

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EFFECT OF TEMPERATURE[a] AND AGGREGATE PARTICLE SIZE DISTRIBUTION[b] ON THE FRACTURE
PROPERTIES[c] OF A HIGH-ALUMINA CASTABLE[d][16], Continued

Curing/Firing Temperature °C	Fracture Toughness K_{IC}, $MN/m^{3/2}$	Notched Beam Fracture Energy γ_{NBT}, J/m^2	Work of Fracture γ_{WOF}, J/m^2	Strain Energy Release Rate G_{IC}, J/m^2	J-Integral J_{IC}, J/m^2
GAP-SIZED FORMULATION[b] WITH 20% CEMENT MEASURED AT CURING/FIRING TEMPERATURE - - - - - - - - -					
Ambient	0.73±0.05	3.22±0.49	102.40±13.17	6.45±0.98	23.81± 3.31
100	0.53±0.05	1.92±0.36	82.65±24.50	3.85±0.73	25.55± 5.94
200	0.68±0.06	3.34±0.65	85.69±21.74	6.68±1.30	31.50± 5.12
400	0.43±0.05	1.43±0.36	126.14±36.27	2.86±0.72	34.26± 8.88
600	0.51±0.09	2.20±0.88	130.26±29.59	4.40±1.77	34.32± 4.79
800	0.53±0.06	2.37±0.56	141.88±34.13	4.75±1.12	40.89±11.48
1000	0.54±0.06	1.83±0.44	154.42±36.94	3.66±0.89	46.12± 7.48
1200	0.30±0.04	0.98±0.26	110.02±28.64	1.97±0.53	35.88± 3.44
GAP-SIZED FORMULATION WITH 25% CEMENT MEASURED AT CURING/FIRING TEMPERATURE - - - - - - - - - -					
Ambient	0.80±0.17	4.66±1.87	71.89±14.03	9.33±3.75	28.00± 5.15
100	0.63±0.10	2.89±0.89	102.40±27.04	5.78±1.79	32.00± 5.48
200	0.59±0.09	2.98±0.91	106.88±22.12	5.96±1.83	32.90± 6.97
400	0.53±0.07	2.34±0.66	129.39±22.17	4.68±1.32	39.15± 4.26
600	0.63±0.11	3.25±1.02	110.17±28.55	6.50±2.04	38.69± 4.50
800	0.67±0.12	3.88±1.37	161.87±34.61	7.76±2.74	47.62± 8.92
1000	0.58±0.04	2.47±0.29	125.52±32.19	4.94±0.59	42.72± 6.10
1200	0.43±0.03	2.00±0.29	251.49±56.75	4.01±0.59	66.17± 3.67
GAP-SIZED FORMULATION WITH 30% CEMENT MEASURED AT CURING/FIRING TEMPERATURE - - - - - - - - - -					
Ambient	0.89±0.07	5.29±0.89	89.27±24.81	10.59±1.79	24.15± 5.79
100	0.72±0.08	3.82±0.91	91.76±24.06	7.65±1.83	27.50± 5.24
200	0.76±0.11	5.02±1.45	96.88±12.35	10.04±2.90	32.52± 5.61
400	0.57±0.02	3.25±0.28	132.19±30.50	6.51±0.56	43.71± 5.11
600	0.66±0.06	4.12±0.81	106.89±23.45	8.24±1.62	38.85± 5.74
800	0.69±0.10	4.84±1.40	167.25±25.35	9.68±2.81	61.67± 6.34
1000	0.56±0.05	4.47±0.91	153.42±31.80	8.94±1.82	59.39± 6.51
1200	0.43±0.04	2.77±0.59	234.60±41.27	5.55±1.18	69.16± 5.36

[a] Samples were heated in a globar furnace to the curing/firing temperatures and tested when the
samples reached thermal equilibrium.

[b] Various samples were prepared to study the effect of aggregate particle size distribution on the
properties of the castable. The generic castable has a size distribution typical of a standard
industrial product and is the as-received material. The cement content is 25%. The other two
formulations are designed for maximum density of the aggregate and the distributions were calcu-
lated according to formulas for each case. The continuous formula contains aggregate continuous-
ly graded so that the difference between successive particle sizes is a factor of $\sqrt{2}$, with the
largest particles sized at U.S. Sieve Number 4 x 6 and the smallest -325. Gap-sized packing
provides sizes and proportions so that the larger voids created between larger particles are
filled by smaller particles and new voids thus created filled by even smaller particles. The
formulation used here is 61.5% of 4x6, 24.5% of 16x20, 9.8% of 70x100, and 4.2% of -325 mesh.

[c] Fracture toughness was measured by single edge notched beam method; notch was saw cut to a depth
of 1/2 the specimen thickness, average notch width was about 0.032 in. Specimens were tested in
three-point bending, crosshead speed was 0.127 cm/min. Five to seven samples for each set of con-
ditions were tested. The J-Integral can be considered as a parameter which describes the total
energy of the crack tip stress-strain field or as a measure of the total energy dissipated during
fracture initiation. It includes non-linear elastic and plastic behavior. In the case is per-
fectly linear elastic then $G_{IC} = J_{IC}$.

- -

(Table Continued)

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EFFECT OF TEMPERATURE[a] AND AGGREGATE PARTICLE SIZE DISTRIBUTION[b] ON THE FRACTURE

PROPERTIES[c] OF A HIGH-ALUMINA CASTABLE[d][16], Continued

Footnotes continued
_____

[d]T-61 tabular alumina (Alcoa) aggregate, Casting Grade CA-25 (Alcoa) cement. Generic formulation 75% aggregate, 25% cement, water to ball-in-hand consistency 10.3% of aggregate and cement. Continuous formulation: 20% cement has 9.9% water, 25% cement has 10.6% water, 30% cement has 11.1% water. Gap-sized formulation: 20% cement has 8.0% water, 25% cement has 9.1% water, 30% cement has 9.8% water. Samples were cast, vibrated one minute, cured sealed in plastic 24 hours at ambient temperature, cured in air 24 hours at ambient temperature, dried in air 24 hours at 100 °C, fired at 60 °C/h to 200, 400, 600, 800, 1000, 1200 °C, soaked at temperature 5 hours, and furnace cooled to ambient temperature.

[e]Misprinted as 0.53±1.30 in table in the original report.

[f]Misprinted (apparently) as 0.44±1.71 in the original reports. This value agrees with graphical data from the same reports. See Section B.3.2.94.

[g]This value disagrees with the value plotted in Section B.3.2.94 and also with the γ_{NBT} value.

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EFFECT OF TEMPERATURE ON THE FRACTURE TOUGHNESS[a] OF A HIGH-ALUMINA
CASTABLE WITH VARIOUS AGGREGATE PARTICLE SIZE DISTRIBUTION[16]
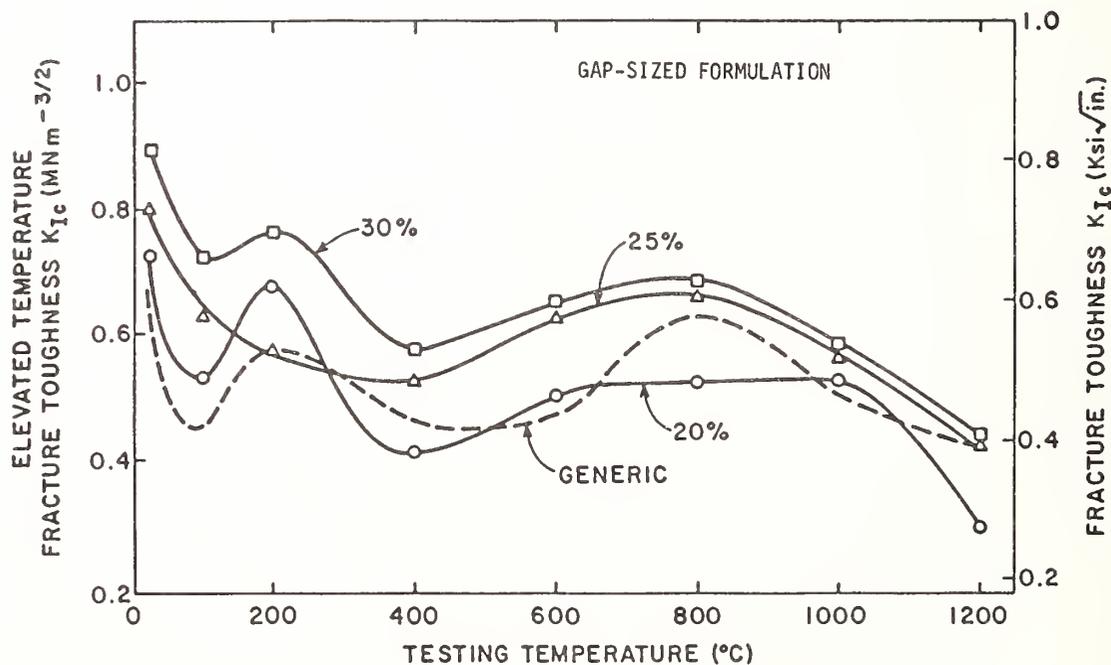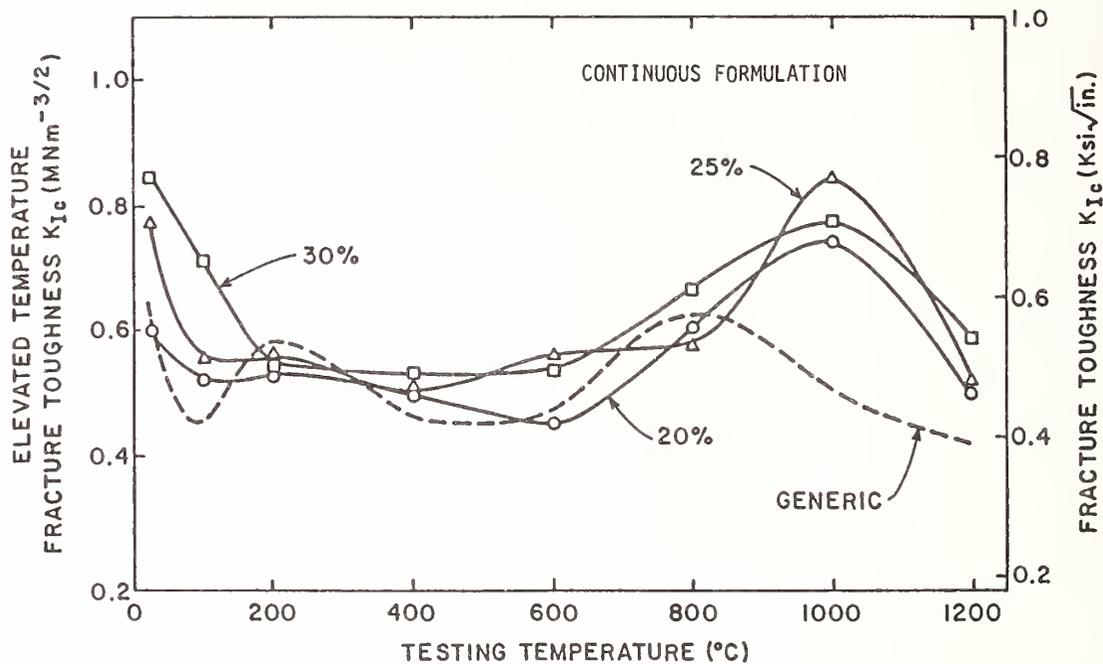
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[a]See Section B.3.2.90 for the data plotted here.

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EFFECT OF TEMPERATURE ON NOTCHED BEAM TEST RESULTS[a] OF A HIGH-ALUMINA CASTABLE
WITH VARIOUS AGGREGATE PARTICLE SIZE DISTRIBUTIONS[16]
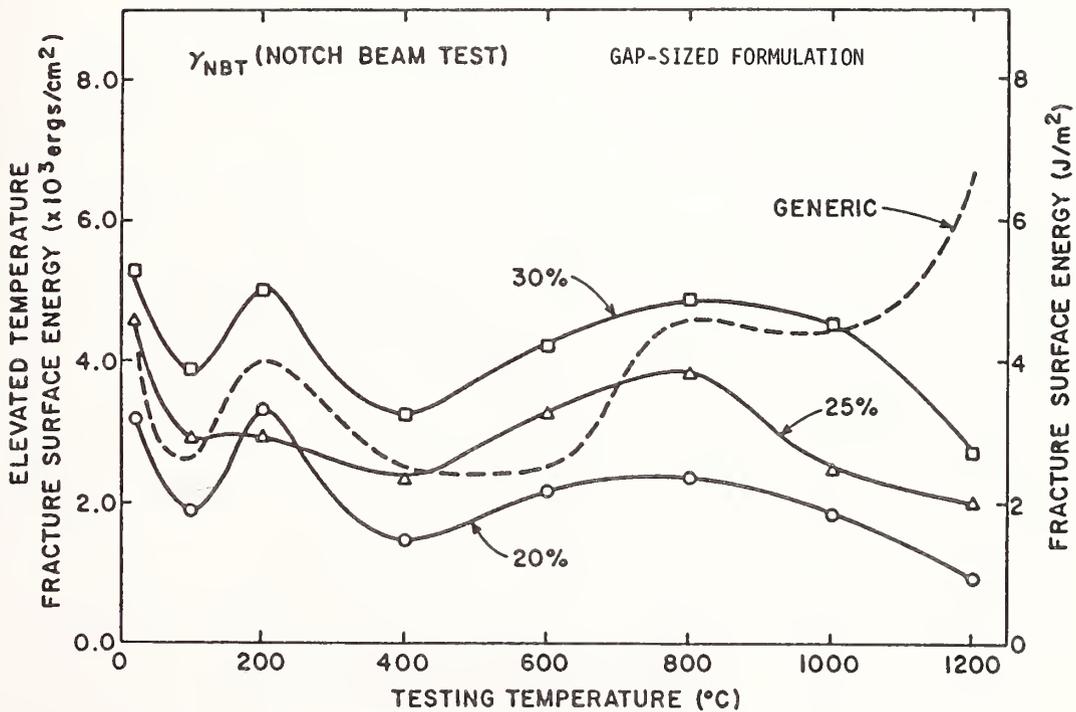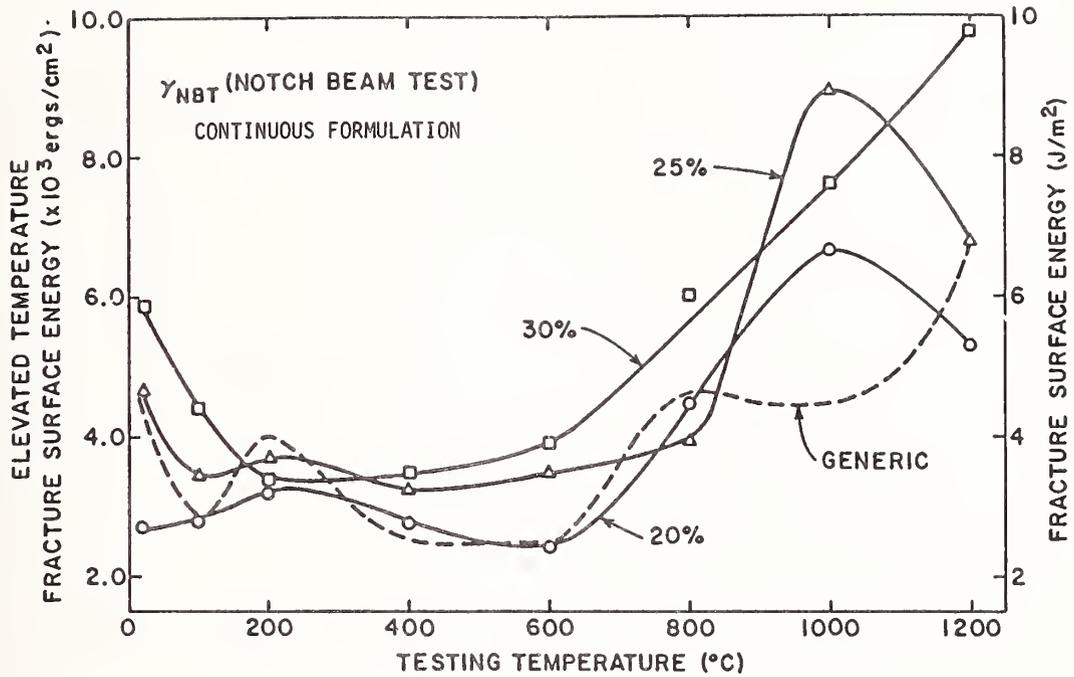
[a]See Section B.3.2.90 for data plotted here.

EFFECT OF TEMPERATURE ON THE WORK OF FRACTURE[a] OF A HIGH-ALUMINA CASTABLE
WITH VARIOUS AGGREGATE PARTICLE SIZE DISTRIBUTIONS[16]
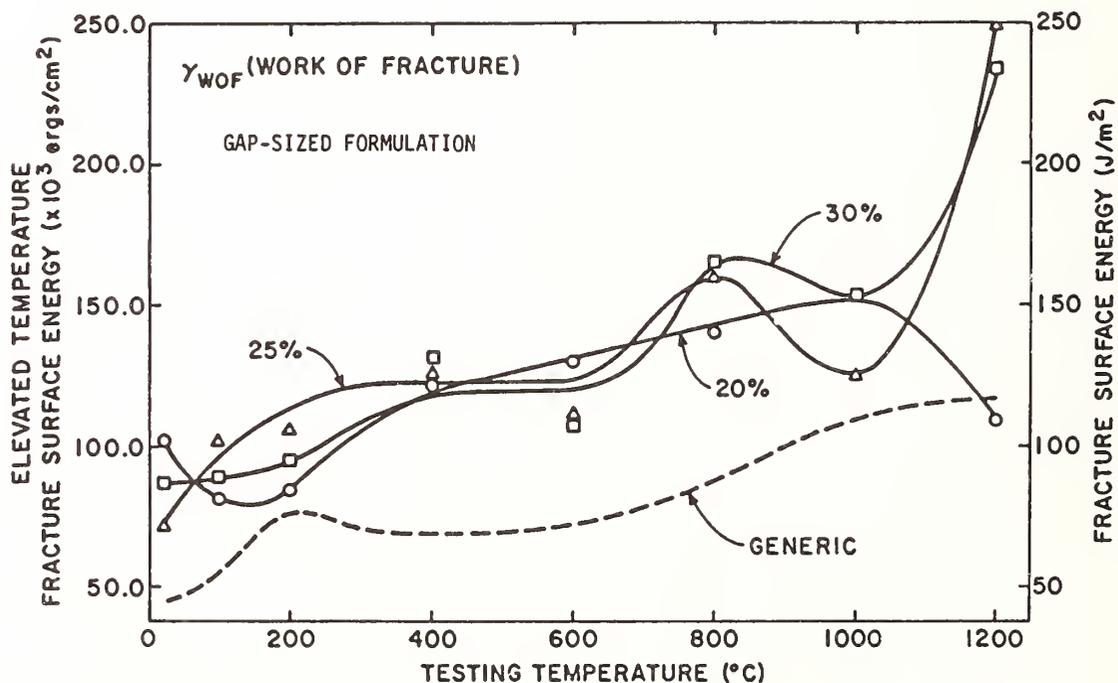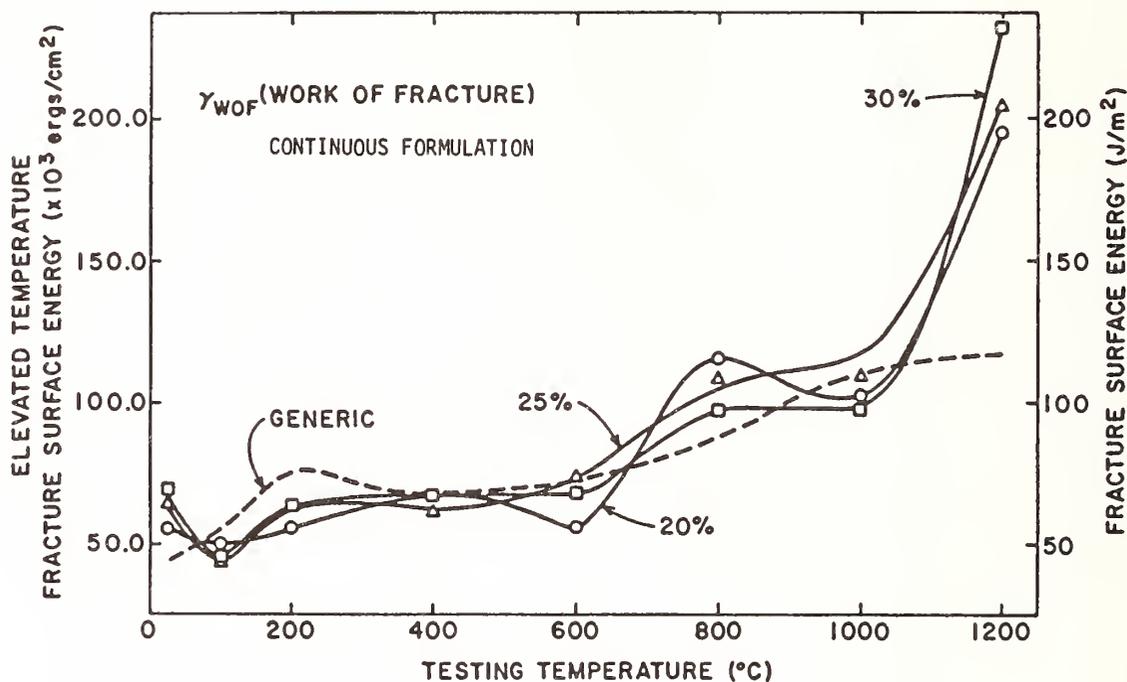
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[a]See Section B.3.2.90 for the data plotted here.

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EFFECT OF TEMPERATURE ON STRAIN ENERGY RELEASE RATE (G_{IC}) AND J-INTEGRAL (J_{IC}) OF A HIGH-ALUMINA CASTABLE WITH VARIOUS AGGREGATE PARTICLE SIZE DISTRIBUTIONS[a][16]
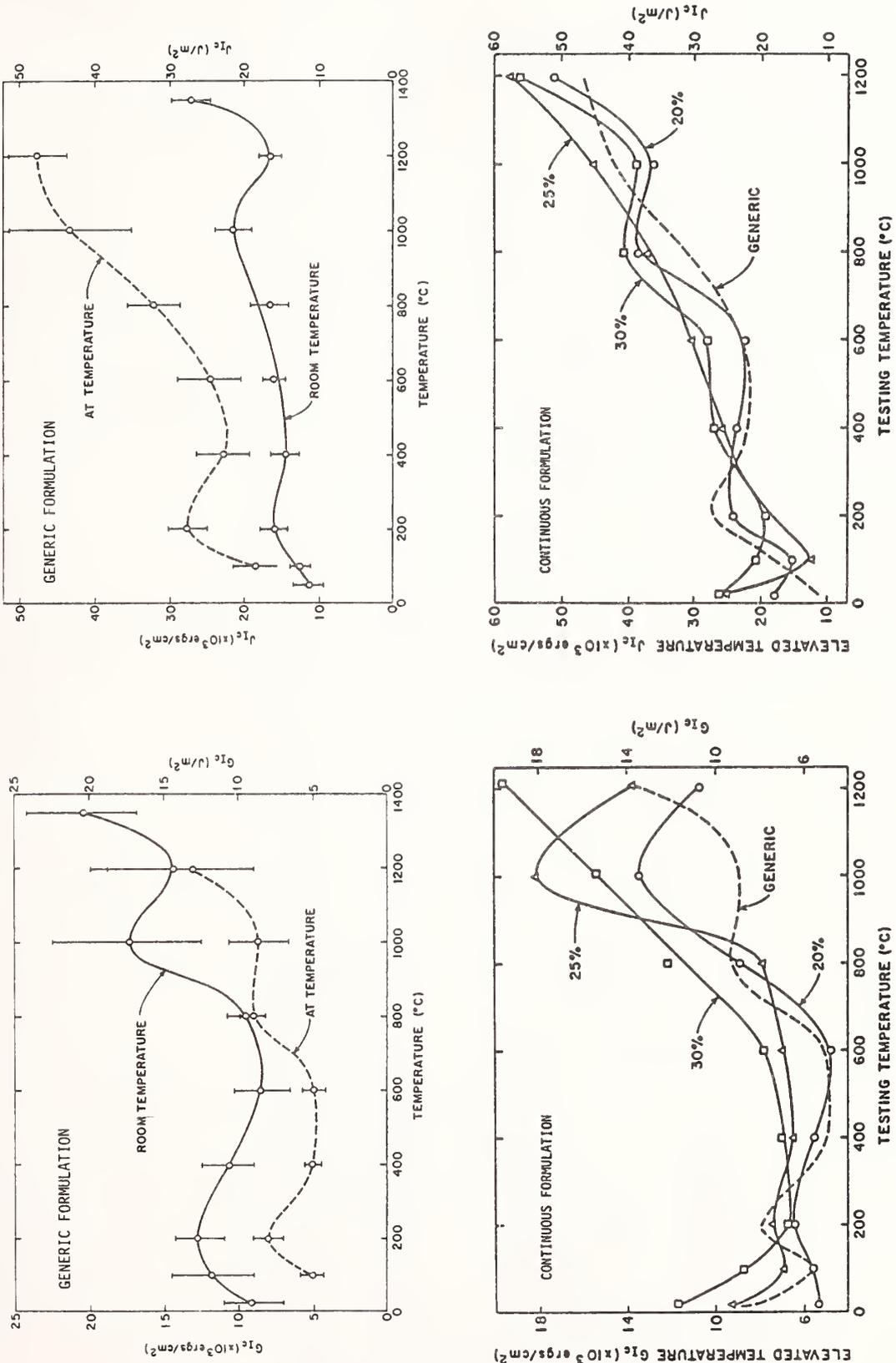
(Data Continued)

EFFECT OF TEMPERATURE ON STRAIN ENERGY RELEASE RATE (G_{IC}) AND J-INTEGRAL (J_{IC}) OF A HIGH-ALUMINA CASTABLE WITH VARIOUS AGGREGATE PARTICLE SIZE DISTRIBUTIONS[a][16], Continued

J_{IC} (J/m^2)

GAP-SIZED FORMULATION

25%

30%

20%

GENERIC

TESTING TEMPERATURE (°C)

ELEVATED TEMPERATURE J_{IC} ($\times 10^3$ ergs/cm^2)

G_{IC} (J/m^2)

GENERIC

25%

30%

20%

GAP-SIZED FORMULATION

TESTING TEMPERATURE (°C)

ELEVATED TEMPERATURE G_{IC} ($\times 10^3$ ergs/cm^2)
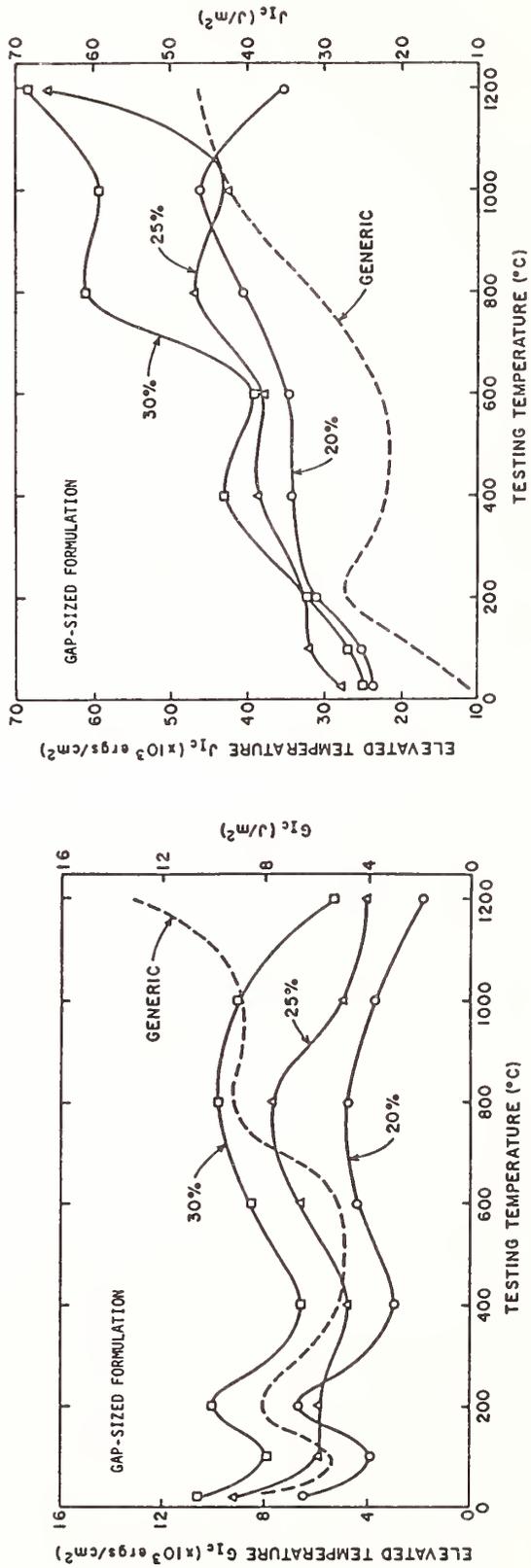
a See B.3.2.90 for data plotted here.

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EFFECT OF SINGLE QUENCH THERMAL SHOCK[a] ON THE FLEXURAL STRENGTH[b] OF
SEVERAL ALUMINA CASTABLES[c][16]

Quench ΔT (°C)	Flexural Strength (MPa)	% Reduction in Strength
Tabular alumina/calcium aluminate cement[d]		
Furnace Cooled	3.40 ± 0.41	--
200	3.14 ± 0.18	7.65
400	3.01 ± 0.16	11.45
600	3.02 ± 0.19	11.17
800	3.05 ± 0.17	10.29
1000	3.04 ± 0.19	10.59
1200	2.95 ± 0.15	13.23
Calcined bauxite/calcium aluminate cement[e]		
Furnace Cooled	11.23 ± 0.86	--
200	10.44 ± 0.81	7.03
400	7.03 ± 0.58	37.39
600	6.74 ± 0.52	39.98
800	5.86 ± 0.729	47.82
1000	5.08 ± 0.33	54.76
1200	4.27 ± 0.29	61.98
Calcined bauxite/calcium aluminate cement[f]		
Furnace Cooled	3.88 ± 0.48	--
200	3.73 ± 0.18	3.87
400	2.99 ± 0.16	22.94
600	2.74 ± 0.07	29.38
800	2.59 ± 0.16	33.25
1000	2.51 ± 0.21	35.31
1200	2.39 ± 0.19	38.40
Calcined kaolin/calcium aluminate cement[g]		
Furnace Cooled	9.01 ± 0.78	--
200	7.55 ± 0.35	16.26
400	5.18 ± 0.15	42.53
600	4.99 ± 0.33	44.54
800	4.62 ± 0.36	48.76
1000	3.98 ± 0.44	55.87
1200	2.81 ± 0.22	68.82
Calcined kaolin/calcium aluminate cement[h]		
Furnace Cooled	7.14 ± 0.10	--
200	5.10 ± 0.36	28.49
400	4.00 ± 0.16	43.97
600	3.63 ± 0.16	49.16
800	3.29 ± 0.30	53.92
1000	3.24 ± 0.19	54.46
1200	2.87 ± 0.25	59.80

(Table Continued)

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EFFECT OF SINGLE QUENCH THERMAL SHOCK[a] ON THE FLEXURAL STRENGTH[b] OF
SEVERAL ALUMINA CASTABLES[c][16], Continued

Quench ΔT (°C)	Flexural Strength (MPa)	% Reduction in Strength
57% Alumina insulating castable[i]		
Furnace Cooled	1.21 ± 0.15	--
200	1.19 ± 0.11	1.65
400	1.07 ± 0.05	11.57
600	0.97 ± 0.12	19.83
800	0.86 ± 0.11	28.92
1000	0.59 ± 0.06	51.24
50% Alumina insulating castable[j]		
Furnace Cooled	2.34 ± 0.11	--
200	2.24 ± 0.13	4.27
400	1.84 ± 0.06	21.37
600	1.63 ± 0.16	30.03
800	1.59 ± 0.13	32.05
1000	1.34 ± 0.14	42.47

[a]Thermal shock damage resistance was studied by monitoring the percent change in flexural strength as a function of a single quench from various elevated temperatures. Specimens, 2.54 cm x 2.54 cm x 17.78 cm, were heated on end in a globar furnace and quenched into air. They were heated to 225, 425, 625, 825, 1025, 1225 °C and held at temperature ∿15 minutes; removed from furnace and rapidly cooled in air (∿25 °C); therefore the ΔT = 200, 400, etc.

[b]Flexural strength measured in three-point bending, crosshead speed 0.05 in./min, span 6 in., at ambient temperature.

[c]Samples were cast, vibrated one minute, cured sealed in plastic 24 hours at ambient temperature, cured in air 24 hours at ambient temperature, dried in air 24 hours at 100 °C, fired at 60 °C/h to 1200 °C, soaked at temperature 5 hours and furnace cooled to ambient temperature.

[d]75% T-61 alumina (Alcoa) aggregate, 25% Secar 71 cement (Lone Star Lafarge), water to ball-in-hand consistency 12.7% of aggregate and cement.

[e]75% Mulcoa M-60 (C-E Minerals) aggregate, 25% Casting Grade CA-25 (Alcoa) cement, water to ball-in-hand consistency 10.6% of aggregate and cement.

[f]75% Mulcoa M-60 (C-E Minerals) aggregate, 25% Secar 71 cement (Lone Star Lafarge), water to ball-in-hand consistency 14.6% of aggregate and cement.

[g]75% Mulcoa M-47 (C-E Minerals) aggregate, 25% Casting Grade CA-25 (Alcoa) cement, water to ball-in-hand consistency 11.3% of aggregate and cement.

[h]75% Mulcoa M-47 (C-E Minerals) aggregate, 25% Refcon cement (Universal Atlas), water to ball-in-hand consistency 13.5% of aggregate and cement.

[i]A commercial lightweight insulating castable (Cer-Lite #75, C-E Refractories), water to ball-in-hand consistency 22% of mix.

[j]A commercial lightweight insulating castable (Litecast 75-28, General Refractories), water to ball-in-hand consistency 24% of mix.

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SINGLE QUENCH THERMAL SHOCK EFFECT[a] ON ALUMINA CASTABLES[16]

(Data Continued)

SINGLE QUENCH THERMAL SHOCK EFFECT[a] ON ALUMINA CASTABLES[16], Continued

[a]See Section B.3.2.95 for the data plotted here.

[b]75% T-61 alumina (Alcoa) aggregate, 25% Secar 71 cement (Lone Star Lafarge), water to ball-in-hand consistency 12.7% of aggregate and cement.

[c]75% Mulcoa M-60 (C-E Minerals) aggregate, 25% Casting Grade CA-25 (Alcoa) cement, water to ball-in-hand consistency 10.6% of aggregate and cement.

[d]75% Mulcoa M-60 (C-E Minerals) aggregate, 25% Secar 71 cement (Lone Star Lafarge), water to ball-in-hand consistency 14.6% of aggregate and cement.

[e]75% Mulcoa M-47 (C-E Minerals) aggregate, 25% Casting Grade CA-25 (Alcoa) cement, water to ball-in-hand consistency 11.3% of aggregate and cement.

[f]75% Mulcoa M-47 (C-E Minerals) aggregate, 25% Refcon cement (Universal Atlas), water to ball-in-hand consistency 13.5% of aggregate and cement.

[g]A commercial lightweight insulating castable (Cer-Lite #75, C-E Refractories), water to ball-in-hand consistency 22% of mix.

[h]A commercial lightweight insulating castable (Litecast 75-28, General Refractories), water to ball-in-hand consistency 24% of mix.

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EFFECT OF MULTIPLE QUENCH THERMAL SHOCK[a] ON THE FLEXURAL STRENGTH[b] OF
SEVERAL ALUMINA CASTABLES[c][16]

Number of Quench Cycles	Flexural Strength (MPa)	% Reduction in Strength
Tabular alumina/calcium aluminate cement[d] - - - - - - - - - - - - - - - -		
Furnace Cooled	3.40 ± 0.41	--
1 ⎫	2.95 ± 0.15	13.23
3 ⎪	2.76 ± 0.12	18.82
5 ⎬ $\Delta T = 1200\ °C$	2.55 ± 0.20	25.00
7 ⎪	2.48 ± 0.18	27.71
10 ⎭	2.42 ± 0.23	28.82
Calcined bauxite/calcium aluminate cement[e] - - - - - - - - - - - - - - -		
Furnace Cooled	11.23 ± 0.86	--
1 ⎫	4.27 ± 0.29	61.98
3 ⎪	3.07 ± 0.30	72.66
5 ⎬ $\Delta T = 1200\ °C$	2.66 ± 0.38	76.31
7 ⎪	2.28 ± 0.40	79.69
10 ⎭	2.04 ± 0.22	81.83
Calcined bauxite/calcium aluminate cement[f] - - - - - - - - - - - - - -		
Furnace Cooled	3.88 ± 0.48	--
1 ⎫	2.31 ± 0.19	40.46
3 ⎪	2.17 ± 0.26	44.72
5 ⎬ $\Delta T = 1200\ °C$	1.69 ± 0.34	56.44
7 ⎪	1.38 ± 0.15	64.43
10 ⎭	1.36 ± 0.11	64.95
Calcined kaolin/calcium aluminate cement[g] - - - - - - - - - - - - - - -		
Furnace Cooled	9.01 ± 0.78	--
1 ⎫	2.81 ± 0.22	68.82
3 ⎪	2.66 ± 0.24	70.48
5 ⎬ $\Delta T = 1200\ °C$	1.99 ± 0.30[h]	77.92
7 ⎪	1.93 ± 0.23[h]	78.56
10 ⎭	1.61 ± 0.28[h]	82.12
Calcined kaolin/calcium aluminate cement[i] - - - - - - - - - - - - - - -		
Furnace Cooled	7.14 ± 0.10	--
1 ⎫	2.87 ± 0.25	59.80
3 ⎪	2.48 ± 0.17	65.27
5 ⎬ $\Delta T = 1200\ °C$	2.01 ± 0.17	71.18
7 ⎪	1.98 ± 0.13	72.27
10 ⎭	1.70 ± 0.17	76.19
57% Alumina insulating castable[j] - - - - - - - - - - - - - - - - - - -		
Furnace Cooled	1.21 ± 0.15	--
1 ⎫	0.59 ± 0.06	51.24
3 ⎪	0.47 ± 0.05	61.15
5 ⎬ $\Delta T = 1000\ °C$	0.43 ± 0.05	64.45
7 ⎪	0.27 ± 0.05	77.68
10 ⎭	0.19 ± 0.03	84.29

- - - - - - - - - - - - - - - (Table Continued) - - - - - - - - - - - - - - -

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EFFECT OF MULTIPLE QUENCH THERMAL SHOCK[a] ON THE FLEXURAL STRENGTH[b] OF
SEVERAL ALUMINA CASTABLES[c][16], Continued

Number of Quench Cycles	Flexural Strength (MPa)	% Reduction in Strength
50% Alumina insulating castable[k]		
Furnace Cooled	2.34 ± 0.11	--
1	1.34 ± 0.14[l]	42.47
3	0.88 ± 0.11	62.39
5 $\Delta T = 1000$ °C	0.67 ± 0.11	71.14
7	0.64 ± 0.06	72.26
10	0.58 ± 0.05[m]	75.21

[a]Thermal shock damage resistance was studied by monitoring the percent change
in flexural strength as a function of multiple quenching from high temper-
ature. Specimens, 2.54 cm x 2.54 cm x 17.78 cm, were heated on end in a
globar furnace and quenched into air. The samples were heated to 1225 or
1025 °C, held at temperature 15 minutes, removed from the furnace and cooled
in air ($\sim$25 °C) for 15 minutes, therefore the ΔT = 1200 or 1000. The heat-
ing-cooling cycle was repeated 1, 3, 5, 7, and 10 times.

[b]Flexural strength measured in three-point bending, crosshead speed 0.05
in./min, span 6 in., at ambient temperature.

[c]Samples were cast, vibrated one minute, cured sealed in plastic 24 hours at
ambient temperature, cured in air 24 hours at ambient temperature, dried in
air 24 hours at 100 °C, fired at 60 °C/h to 1200 °C, soaked at temperature
5 hours and furnace cooled to ambient temperature.

[d]75% T-61 alumina (Alcoa) aggregate, 25% Secar 71 cement (Lone Star Lafarge),
water to ball-in-hand consistency 12.7% of aggregate and cement.

[e]75% Mulcoa M-60 (C-E Minerals) aggregate, 25% Casting Grade CA-25 (Alcoa)
cement, water to ball-in-hand consistency 10.6% of aggregate and cement.

[f]75% Mulcoa M-60 (C-E Minerals) aggregate, 25% Secar 71 cement (Lone Star
Lafarge), water to ball-in-hand consistency 14.6% of aggregate and cement.

[g]75% Mulcoa M-47 (C-E Minerals) aggregate, 25% Casting Grade CA-25 (Alcoa)
cement, water to ball-in-hand consistency 11.3% of aggregate and cement.

[h]Somewhat higher values seem to be plotted in the figure from the same reports
which is given in Section B.3.2.98.

[i]75% Mulcoa M-47 (C-E Minerals) aggregate, 25% Refcon cement (Universal
Atlas), water to ball-in-hand consistency 13.5% of aggregate and cement.

[j]A commercial lightweight insulating castable (Cer-Lite #75, C-E Refracto-
ries), water to ball-in-hand consistency 22% of mix.

[k]A commercial lightweight insulating castable (Litecast 75-28, General
Refractories), water to ball-in-hand consistency 24% of mix.

[l]For this data point a value greater than 1.5 was plotted on the figure
shown in Section B.3.2.98.

[m]Misprinted in the original reports as 1.58 ±0.05.

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MULTIPLE QUENCH THERMAL SHOCK EFFECT[a] ON ALUMINA CASTABLES[16]

(Data Continued)

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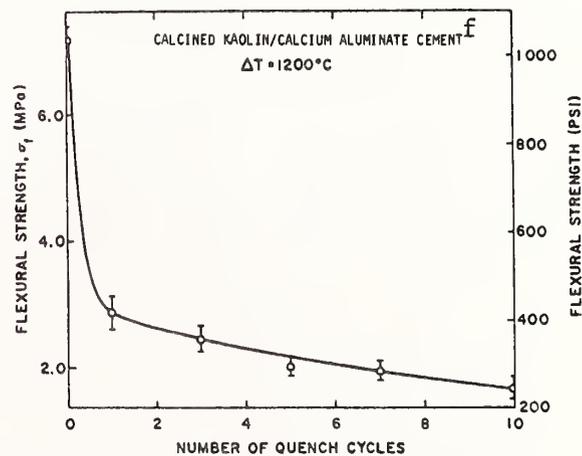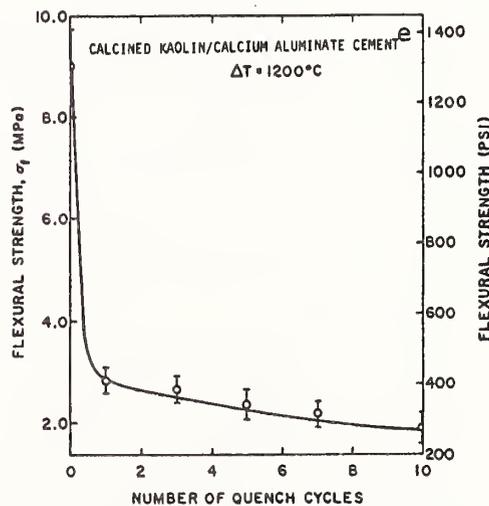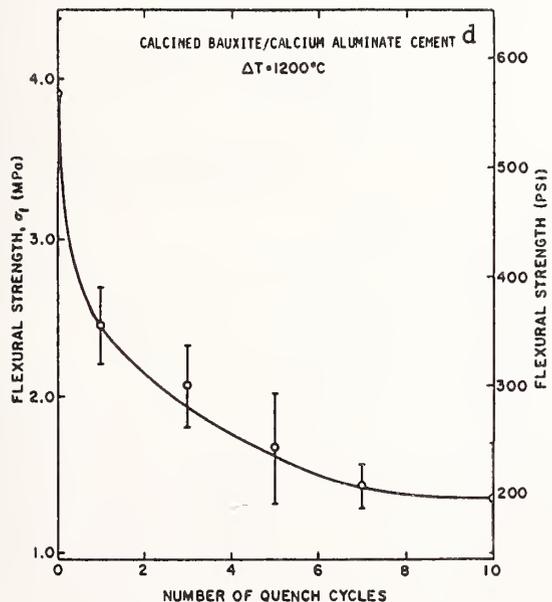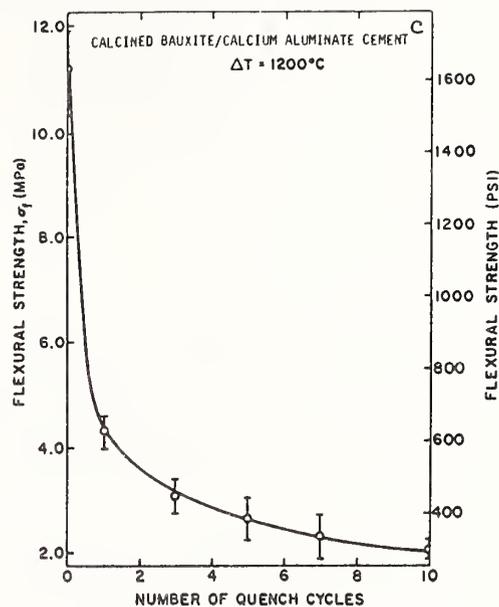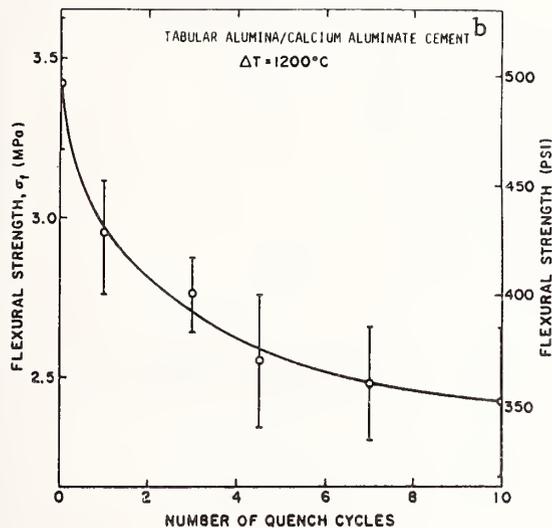
MULTIPLE QUENCH THERMAL SHOCK EFFECT[a] ON ALUMINA CASTABLES[16], Continued
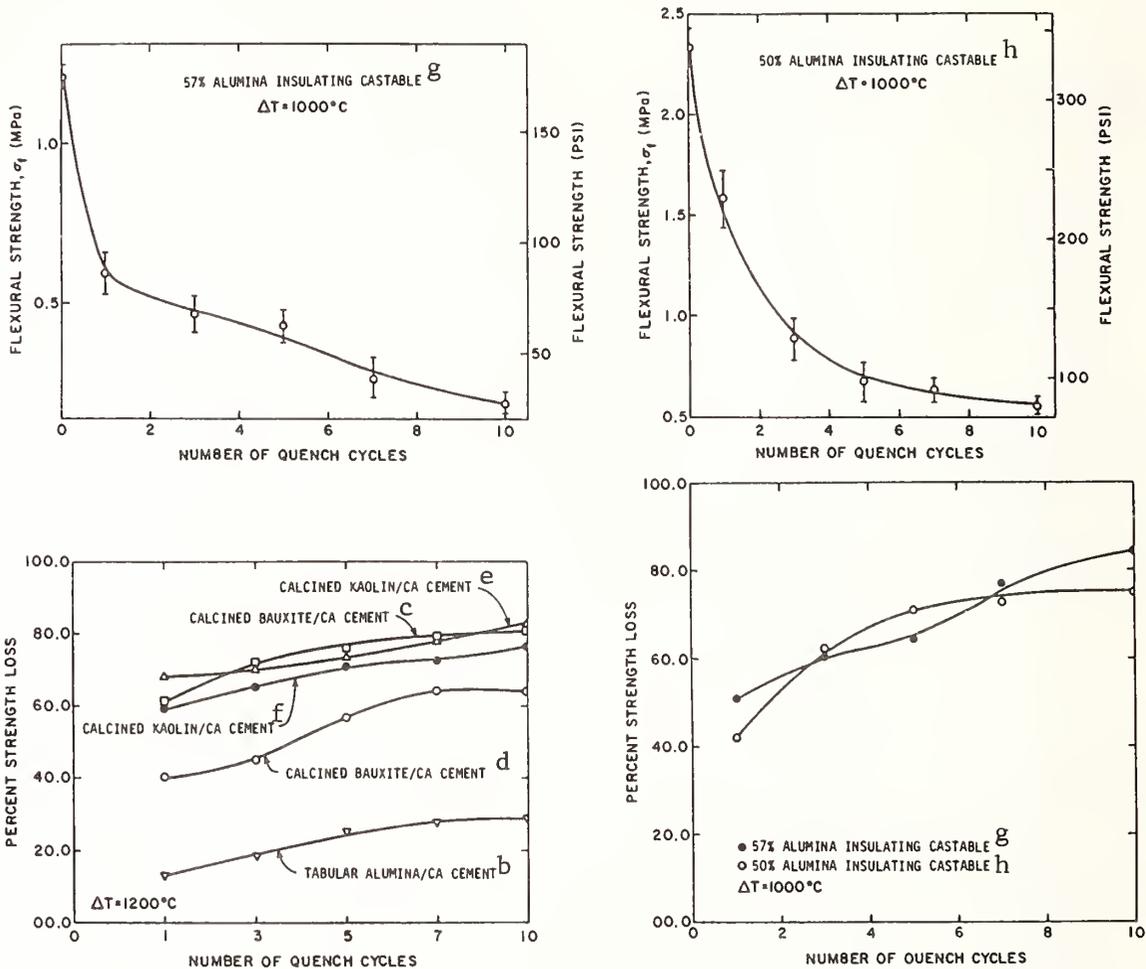
[a]See Section B.3.2.97 for the data plotted here.

[b]75% T-61 alumina (Alcoa) aggregate, 25% Secar 71 cement (Lone Star Lafarge), water to ball-in-hand consistency 12.7% of aggregate and cement.

[c]75% Mulcoa M-60 (C-E Minerals) aggregate, 25% Casting Grade CA-25 (Alcoa) cement, water to ball-in-hand consistency 10.6% of aggregate and cement.

[d]75% Mulcoa M-60 (C-E Minerals) aggregate, 25% Secar 71 cement (Lone Star Lafarge), water to ball-in-hand consistency 14.6% of aggregate and cement.

[e]75% Mulcoa M-47 (C-E Minerals) aggregate, 25% Casting Grade CA-25 (Alcoa) cement, water to ball-in-hand consistency 11.3% of aggregate and cement.

[f]75% Mulcoa M-47 (C-E Minerals) aggregate, 25% Refcon cement (Universal Atlas), water to ball-in-hand consistency 13.5% of aggregate and cement.

[g]A commercial lightweight insulating castable (Cer-Lite #75, C-E Refractories), water to ball-in-hand consistency 22% of mix.

[h]A commercial lightweight insulating castable (Litecast 75-28, General Refractories), water to ball-in-hand consistency 24% of mix.

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SINGLE QUENCH THERMAL SHOCK EFFECT[a] ON AN ALUMINA CASTABLE[b] WITH
VARIOUS AGGREGATE PARTICLE SIZE DISTRIBUTIONS[c][16]

(Data Continued)

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SINGLE QUENCH THERMAL SHOCK EFFECT[a] ON AN ALUMINA CASTABLE[b] WITH
VARIOUS AGGREGATE PARTICLE SIZE DISTRIBUTIONS[c][16], Continued

[a] Thermal shock damage resistance was studied by monitoring the percent change
in flexural strength as a function of a single quench from various elevated
temperatures. Specimens, 2.54 cm x 2.54 cm x 17.78 cm, were heated on end
in a globar furnace and quenched into air. They were heated to 225, 425,
625, 825, 1025, 1225 °C and held at temperature ∿15 minutes; removed from
furnace and rapidly cooled in air (∿25 °C); therefore the ΔT = 200, 400, etc.
Flexural strength was measured in three-point bending, crosshead speed 0.05
in. min, span 6 in., at ambient temperature. Five to eight samples were
tested at each quench level.

[b] T-61 tabular alumina (Alcoa) aggregate, Casting Grade CA-25 calcium alumi-
nate cement (Alcoa). Generic formulation 75% aggregate, 25% cement, water
to ball-in-hand consistency 10.3% of aggregate and mix. Continuous formula-
tion: 20% cement has 9.9% water, 25% cement has 10.6% water, 30% cement has
11.1% water. Gap-sized formulation: 20% cement has 8.0% water, 25% cement
has 9.1% water, 30% cement has 9.8% water. Samples were cast, vibrated one
minute, cured sealed in plastic 24 hours at ambient temperature, cured in
air 24 hours at ambient temperature, dried in air 24 hours at 100 °C, fired
at 60 °C/h to 1200 °C, soaked at temperature 5 hours, and furnace cooled to
ambient temperature.

[c] Various samples were prepared to study the effect of aggregate particle size
distribution on the properties of the castable. The generic castable has a
size distribution typical of a standard industrial product and is the as-
received material. The other two formulations are designed for maximum den-
sity of the aggregate and the distributions were calculated according to
formulas for each case. The continuous formula contains aggregate continu-
ously graded so that the difference between successive particle sizes is a
factor of $\sqrt{2}$, with the largest particles sized at U.S. Sieve Number 4 x 6
and the smallest -325. Gap-sized packing provides sizes and proportions so
that the larger voids created between larger particles are filled by smaller
particles and new voids thus created filled by even smaller particles. The
formulation used here is 61.5% of 4x6, 24.5% of 16x20, 9.8% of 70x100, and
4.2% of -325 mesh.

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MULTIPLE QUENCH THERMAL SHOCK EFFECT[a] ON AN ALUMINA CASTABLE[b] WITH VARIOUS
AGGREGATE PARTICLE SIZE DISTRIBUTIONS[c][16]

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(Data Continued)

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MULTIPLE QUENCH THERMAL SHOCK EFFECT[a] ON AN ALUMINA CASTABLE[b] WITH VARIOUS AGGREGATE PARTICLE SIZE DISTRIBUTIONS[c][16], Continued

[a]Thermal shock damage resistance was studied by monitoring the percent change in flexural strength as a function of multiple quenching from high tempera- ture. Specimens, 2.54 cm x 2.54 cm x 17.78 cm, were heated on end in a globar furnace and quenched into air. The samples were heated to 1225 or 1025 °C, held at temperature 15 minutes, removed from the furnace and cooled in air (∿25 °C) for 15 minutes, therefore the ΔT = 1200 or 1000. The heat- ing-cooling cycle was repeated 1, 3, 5, 7, and 10 times. Flexural strength was measured in three-point bending, crosshead speed 0.05 in./min, span 6 in., at ambient temperature. Five to eight samples were tested at each quench level.

[b]T-61 tabular alumina (Alcoa) aggregate, Casting Grade CA-25 calcium alumi- nate cement (Alcoa). Generic formulation 75% aggregate, 25% cement, water to ball-in-hand consistency 10.3% of aggregate and cement. Continuous formula- tion: 20% cement has 9.9% water, 25% cement has 10.6% water, 30% cement has 11.1% water. Gap-sized formulation: 20% cement has 8.0% water, 25% cement has 9.1% water, 30% cement has 9.8% water. Samples were cast, vibrated one minute, cured sealed in plastic 24 hours at ambient temperature, cured in air 24 hours at ambient temperature, dried in air 24 hours at 100 °C, fired at 60 °C/h to 1200 °C, soaked at temperature 5 hours, and furnace cooled to ambient temperature.

[c]Various samples were prepared to study the effect of aggregate particle size distribution on the properties of the castable. The generic castable has a size distribution typical of a standard industrial product and is the as- received material. The other two formulations are designed for maximum den- sity of the aggregate and the distributions were calculated according to formulas for each case. The continuous formula contains aggregate continu- ously graded so that the difference between successive particle sizes is a factor of $\sqrt{2}$, with the largest particles sized at U.S. Sieve Number 4 x 6 and the smallest - 325. Gap-sized packing provides sizes and proportions so that the larger voids created between larger particles are filled by smaller particles and new voids thus created filled by even smaller particles. The formulation used here is 61.5% of 4x6, 24.5% of 16x20, 9.8% of 70x100, and 4.2% of -325 mesh.

SLOW CRACK GROWTH DATA[a] FOR A MEDIUM-ALUMINA CASTABLE[b][16]

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SLOW CRACK GROWTH DATA[a] FOR A MEDIUM-ALUMINA CASTABLE[b][16], Continued

[a]The stress intensity (K_I) and the crack velocity (V_I) were calculated from load relaxation curves. N, the slope of the line, is a measure of resistance to slow crack growth. A high N indicates good crack resistance. Slow crack growth was determined by a double torsion test that uses a constant displacement procedure. Specimens, 20.32 cm x 10.16 cm x 1.27 cm thick, were prepared with a saw-cut center groove 0.635 cm deep over the length of the specimen. Specimens were loaded by four-point bending applied at one end. A constant crosshead speed was applied to a maximum load, the crosshead displacement stopped, and the specimen allowed to relax. At ambient temperatures (RT on the figures) the crosshead speed was 0.005 cm/min and the load relaxation was carried out for 20 minutes. At elevated temperatures the specimen was first heated for 30 minutes, placed in test position, held at temperature 20 minutes and then the load relaxations were measured for about 5 minutes. Two specimens were tested in each test condition.

[b]75% Calcined kaolin (Mulcoa M-47, C-E Minerals) aggregate, 25% calcium aluminate cement (Casting Grade CA-25, Alcoa), water to ball-in-hand consistency 11.3% of aggregate and cement. Samples were cast, vibrated one minute, cured sealed in plastic 24 hours at ambient temperature, cured in air 24 hours at ambient temperature, dried in air 24 hours at 100 °C, fired at 60 °C/h to 200, 400, 600, 800, 1000, 1200, and 1350 °C, soaked at temperature 5 hours, and furnace cooled to ambient temperature.

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SLOW CRACK GROWTH DATA[a] FOR SEVERAL DENSE ALUMINA CASTABLES[b][16]

(Data Continued)

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SLOW CRACK GROWTH DATA[a] FOR SEVERAL DENSE ALUMINA CASTABLES[b][16], Continued

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SLOW CRACK GROWTH DATA[a] FOR SEVERAL DENSE ALUMINA CASTABLES[b][16], Continued

[a]The stress intensity (K_I) and the crack velocity (V_I) were calculated from load relaxation curves. N, the slope of the line, is a measure of resistance to slow crack growth. A high N indicates good crack resistance. Slow crack growth was determined by a double torsion test that uses a constant displacement procedure. Specimens, 20.32 cm x 10.16 cm x 1.27 cm thick, were prepared with a saw-cut center groove 0.635 cm deep over the length of the specimen. Specimens were loaded by four-point bending applied at one end. A constant crosshead speed was applied to a maximum load, the crosshead displacement stopped, and the specimen allowed to relax. At ambient temperatures (RT on the figures) the crosshead speed was 0.005 cm/min and the load relaxation was carried out for 20 minutes. At elevated temperatures the specimen was first heated for 30 minutes, placed in test position, held at temperature 20 minutes and then the load relaxations were measured for about 5 minutes. Two specimens were tested in each test condition.

[b]Calcined kaolin/CA cement = 75% Mulcoa M-47 (C-E Minerals) aggregate, 25% Refcon calcium aluminate cement (Universal Atlas), water to ball-in-hand consistency 13.5% of aggregate and cement. Calcined bauxite/CA cement-1 = 75% Mulcoa M-60 (C-E Minerals), 25% Secar 71 calcium aluminate cement (Lone Star Lafarge), water to ball-in-hand consistency 14.6% of aggregate and cement. Calcined bauxite/CA cement-2 = 75% Mulcoa M-60 (C-E Minerals), 25% Casting Grade CA-25 calcium aluminate cement (Alcoa), water to ball-in-hand consistency 10.6% of aggregate and cement. Tabular alumina/CA cement = 75% T-61 alumina (Alcoa) aggregate, 25% Secar 71 calcium aluminate cement (Lone Star Lafarge), water to ball-in-hand consistency 12.7% of aggregate and cement. Samples were cast, vibrated one minute, cured sealed in plastic 24 hours at ambient temperature, cured in air 24 hours at ambient temperature, dried in air 24 hours at 100 °C, fired at 60 °C/h to 200, 400, 600, 800, 1000, 1200, and 1350 °C, soaked at temperature 5 hours, and furnace cooled to ambient temperature.

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SLOW CRACK GROWTH DATA[a] FOR TWO INSULATING CASTABLES[b][16]

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SLOW CRACK GROWTH DATA[a] FOR TWO INSULATING CASTABLES[b][16], Continued

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SLOW CRACK GROWTH DATA[a] FOR TWO INSULATING CASTABLES[b][16], Continued

[a]The stress intensity (K_I) and the crack velocity (V_I) were calculated from load relaxation curves. N, the slope of the line, is a measure of resistance to slow crack growth. A high N indicates good crack resistance. Slow crack growth was determined by a double torsion test that uses a constant displacement procedure. Specimens, 20.32 cm x 10.16 cm x 2.54 cm thick, were prepared with a saw-cut center groove 1.27 cm deep over the length of the specimen. Specimens were loaded by four-point bending applied at one end. A constant crosshead speed was applied to a maximum load, the crosshead displacement stopped, and the specimen allowed to relax. At ambient temperatures (RT on the figures) the crosshead speed was 0.005 cm/min and the load relaxation was carried out for 20 minutes. At elevated temperatures the specimen was first heated for 30 minutes, placed in test position, held at temperature 20 minutes and then the load relaxations were measured for about 5 minutes. Two specimens were tested in each test condition.

[b]50% Alumina castable = a commercial lightweight insulating castable (Litecast 75-28, General Refractories), water to ball-in-hand consistency 24% of mix. 57% Alumina castable = a commercial lightweight insulating castable (Cer-Lite #75, C-E Refractories), water to ball-in-hand consistency 22% of mix. Samples were cast, vibrated one minute, cured sealed in plastic 24 hours at ambient temperature, cured in air 24 hours at ambient temperature, dried in air 24 hours at 100 °C, fired at 60 °C/h to 200, 400, 600, 800, 1000, and 1200 °C, soaked at temperature 5 hours, and furnace cooled to ambient temperature.

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SLOW CRACK GROWTH DATA[a] FOR A HIGH-ALUMINA CASTABLE[b] WITH VARIOUS
AGGREGATE PARTICLE SIZE DISTRIBUTIONS[c][16]

(Data Continued)

SLOW CRACK GROWTH DATA[a] FOR A HIGH-ALUMINA CASTABLE[b] WITH VARIOUS
AGGREGATE PARTICLE SIZE DISTRIBUTIONS[c][16], Continued

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SLOW CRACK GROWTH DATA[a] FOR A HIGH-ALUMINA CASTABLE[b] WITH VARIOUS
AGGREGATE PARTICLE SIZE DISTRIBUTIONS[c][16], Continued

(Data Continued)

SLOW CRACK GROWTH DATA[a] FOR A HIGH-ALUMINA CASTABLE[b] WITH VARIOUS
AGGREGATE PARTICLE SIZE DISTRIBUTIONS[c][16], Continued

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(Data Continued)

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SLOW CRACK GROWTH DATA[a] FOR A HIGH-ALUMINA CASTABLE[b] WITH VARIOUS
AGGREGATE PARTICLE SIZE DISTRIBUTIONS[c][16], Continued

Footnotes

[a]The stress intensity (K_I) and the crack velocity (V_I) were calculated from
load relaxation curves. N, the slope of the line, is a measure of resistance
to slow crack growth. A high N indicates good crack resistance. Slow crack
growth was determined by a double torsion test that uses a constant dis-
placement procedure. Specimens, 20.32 cm x 10.16 cm x 1.27 cm thick, were
prepared with a saw-cut center groove 0.635 cm deep over the length of the
specimen. Specimens were loaded by four-point bending applied at one end.
A constant crosshead speed was applied to a maximum load, the crosshead dis-
placement stopped, and the specimen allowed to relax. At ambient tempera-
tures (RT on the figures) the crosshead speed was 0.005 cm/min and the load
relaxation was carried out for 20 minutes. At elevated temperatures the
specimen was first heated for 30 minutes, placed in test position, held at
temperature 20 minutes and then the load relaxations were measured for about
5 minutes. Two specimens were tested in each test condition.

[b]T-61 tabular alumina (Alcoa) aggregate, Casting Grade CA-25 calcium aluminate
cement (Alcoa). Generic formulation: 75% aggregate, 25% cement, water to ball-
in-hand consistency 10.3% of aggregate and cement. Continuous formulation:
20% cement has 9.9% water, 25% cement has 10.6% water, 30% cement has 11.1%
water. Gap-sized formulation: 20% cement has 8.0% water, 25% cement has 9.1%
water, 30% cement has 9.8% water. Samples were cast, vibrated one minute,
cured sealed in plastic 24 hours at ambient temperature, cured in air 24 hours
at ambient temperature, dried in air 24 hours at 100 °C, fired at 60 °C/h to
200, 400, 600, 800, 1000, 1200, and 1350 °C, soaked at temperature 5 hours,
and furnace cooled to ambient temperature.

[c]Various samples were prepared to study the effect of aggregate particle size
distribution on the properties of the castable. The generic castable has a
size distribution typical of a standard industrial product and is the as-
received material. The other two formulations are designed for maximum density
of the aggregate and the distributions were calculated according to formulas
for each case. The continuous formula contains aggregate continuously graded
so that the difference between successive particle sizes is a factor of $\sqrt{2}$,
with the largest particles sized at U.S. Sieve Number 4 x 6 and the smallest
-325. Gap-sized packing provides sizes and proportions so that the larger
voids created between larger particles are filled by smaller particles and new
voids thus created filled by even smaller particles. The formulation used
here is 61.5% of 4x6, 24.5% of 16x20, 9.8% of 70x100, and 4.2% of -325 mesh.

MODULUS OF RUPTURE[a] OF ALUMINA CASTABLES[b] AS A FUNCTION OF BOEHMITE CONTENT[c][39]

Atmosphere[e]	Temperature °C / °F	Pressure MPa/psi	Time days	93% Alumina[f]		57% Alumina[g]	
				Modulus of Rupture, psi	Boehmite[d] %	Modulus of Rupture, psi	Boehmite[d] %
Air (fired)	260/ 500	ambient	3/4	2220	0	1570	0
CGA, 30% saturated	260/ 500	3.4/500	30	2860	13	2130	14
CGA with 1% H_2S, 100% saturated	231/ 447	6.9/1000	30	4870	29	3920	15
CGA with 1% H_2S, 100% saturated	231/ 447	6.9/1000	60	4600	32	3560	18

[a] Modulus of Rupture determined in 3-point bending tests, ASTM C268-70, span of 2 inches, crosshead speed 0.5 cm/min. See Sections B.3.2.55, B.3.2.58, B.1.2.17, and B.1.2.18 for the tests and the data summarized here.

[b] Refractories were cast in molds, cured 24 hours in air at 100% humidity at ambient temperature, and dried at 230 °F for 24 hours. All specimens were stored at 230 °F until tested.

[c] See Sections B.1.2.21, B.3.2.56, B.4.2.10, and B.4.2.11 for testing relative to boehmite formation.

[d] Determined by DTA-TGA analyses.

[e] CGA = coal gasification atmosphere; composition (in vol %): 18 CO, 12 CO_2, 24 H_2, 5 CH_4, 41 H_2O. Tests designated as 100% saturated were conducted in a steam generator; the 30% saturated test took place in another pressure vessel.

[f] 93% Alumina dense castable, UMR-1 generic preparation (70% tabular alumina + 30% calcium aluminate cement) (T-61 alumina, Alcoa; CA-25 Calcium Aluminate Cement, 79% alumina-18% calcia, Alcoa).

[g] 57% Alumina, 34% silica castable, calcium aluminate bonded (RC-3, General Refractories; CA-25 Calcium Aluminate Cement, Alcoa).

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MODULUS OF RUPTURE[a] FOR CALCIUM ALUMINATE CEMENT[b] EXPOSED TO STEAM-SATURATED ATMOSPHERES[c] AS A FUNCTION OF SATURATION[d] AND BOEHMITE FORMATION[e][39]
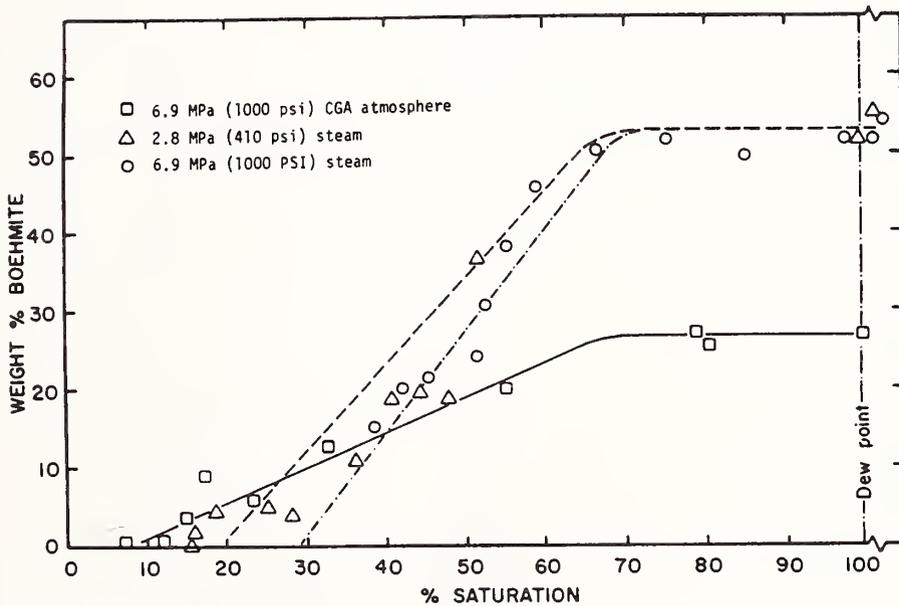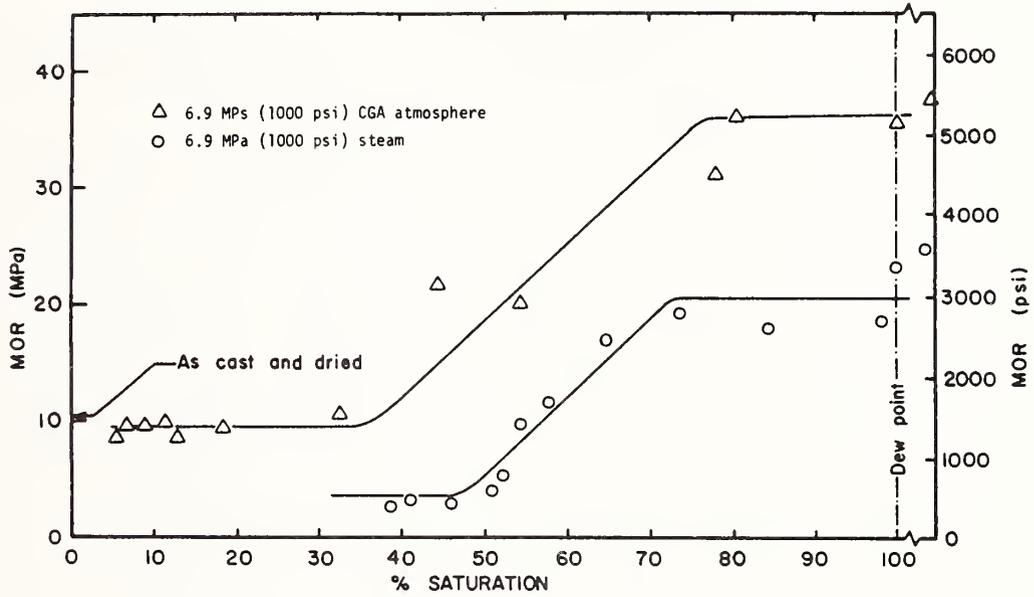
[a]The 18-inch long specimen was broken in 3-point bending in an Instron machine using a 2-inch span so that fracture loads were measured for every inch corresponding to a different percent saturation. The modulus of rupture was calculated from the fracture load.

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(Data Continued)

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MODULUS OF RUPTURE[a] FOR CALCIUM ALUMINATE CEMENT[b] EXPOSED TO STEAM-SATURATED ATMOSPHERES[c] AS A FUNCTION OF SATURATION[d] AND BOEHMITE FORMATION[e][39], Continued

Footnotes continued

[b]A 79% alumina–18% calcia cement (CA–25 Calcium Aluminate Cement, Alcoa). Neat cement specimens were cast, water/cement ratio = 0.3, which were 18 x 1/4 x 1/4 inches.

[c]CGA = coal gasification atmosphere. Composition (in vol %): 18 CO, 12 CO_2, 24 H_2, 5 CH_4, 40 H_2O, and 1 H_2S. The atmosphere was 100% steam saturated. Exposures to CGA or steam only were for ten days.

[d]A pressure vessel (18 inches long and 1/2 inch diameter) was heated in a horizontal temperature gradient furnace. During heating to the test temperature (545 °F) the vessel was opened to permit escape of air and water of hydration. At the test temperature, the vessel was closed and steam or steam-saturated CGA was introduced from a steam generator. Test vessel temperature was maintained so as to provide 100% steam saturation at the cool end (540 °F) and 0% at the hot end (900 °F) with the degree of steam saturation following the temperature gradient along the pressure vessel length. Test temperature for CGA was 447 °F.

[e]Other tests showed that boehmite formation occurred in steam-saturated atmospheres and resulted in very large increases in the flexural strength of calcium aluminate bonded refractories.

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COMPARISON OF COMPRESSIVE STRENGTHS[a] OF CASTABLE REFRACTORY[b] BONDED WITH BARIUM[c]
AND CALCIUM[d] ALUMINATE CEMENTS [9]

Cement Used	Compressive Strength,[a] psi		
	After drying	After firing to 1000 °C for 8 hours	After 24 thermal cycles to 1220 °C[e]
Calcium aluminate	4,950	4,220	4,870
Barium aluminate	3,300	3,030	2,440

[a] Compressive strengths measured following ASTM C133-72.

[b] Tabular alumina aggregate used; 80 vol % aggregate and 20 vol % cement. Water added, 11 ml for every 100 g of dry mix. Refractory was cast into 1-in. cubes, cured at 20 °C and 100 % relative humidity for 24 hours, then dried at 120 °C for 24 hours.

[c] Barium aluminate was prepared by firing equimolar amounts of barium carbonate and alumina at 1460 °C for 20 hours.

[d] Commercial cement, CA-25 (Alcoa).

[e] One cycle consisted of heating samples from 420 °C to 1220 °C in 15 minutes followed by a 45 minute cooling to 420 °C.

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EFFECT OF SIMULATED CATALYTIC COAL GASIFICATION ATMOSPHERE[a] ON THE COMPRESSIVE STRENGTH[b] OF SOME ALUMINA REFRACTORIES[c][9]

Refractory Composition[c]	Brand Name	Compressive Strength[b]		
		Air-Prefired[d]		Exposed[a]
95% Al_2O_3 dense castable[e]	Castolast G	9,110 psi	↔[f]	4,375 psi
45% Al_2O_3 dense castable[e]	H.S. Brikcast BF	4,275	↔	10,355
45% Al_2O_3 dense castable[e] + 304 SS fibers	H.S. Brikcast BF	4,095	↔	12,025
45% Al_2O_3 fired super duty brick	KX-99	9,500		7,515
90% Al_2O_3 ramming mix, phosphate bonded	Brikram 90R	11,585		9,600

[a] A reducing atmosphere: 21.6 H_2, 32.2 H_2O, 6.1 CO, 14.2 CO_2, 22 CH_4, 2.1 N_2, 1.2 NH_3, and 1.0 H_2S (mole %). The exposure was for 2000 hours at 730 °C and 500 psig. The test chamber contained crucibles with molten KOH to provide an alkali vapor. See Section B.1.2.33.

[b] Tests were performed on 1-inch cubes, following ASTM C133-72.

[c] Fired brick was taken as received. Castables were thoroughly mixed, oven dried for 24 h at 230 °F, cooled in a moisture-proof container, stored at 75 °F for 24 h, mixed at 144 rpm with 25 °C water to give ball-in-hand consistency, troweled into molds and vibrated for 3 minutes, cured covered with plastic for 24 h at 90 °F and 100% humidity, and dried 24 h at 230 °F. The ramming mix, as-received, was pressed at 1000 psi into mold, dried at 250 °F for 28 h, heated at 50 °F/h to 500 °F, held 24 h, heated at 50 °F/h to 1000 °F, held 24 h, and cooled slowly to ambient temperature.

[d] Prefired in air at 730 °C for 24 hours.

[e] Castables are all calcium aluminate bonded.

[f] Symbol ↔ denotes difference significant at 99% level of confidence [indicated this way in the original report].

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EFFECT OF VARIOUS HIGH-BTU GAS COMPOSITIONS[a] ON THE COMPRESSIVE STRENGTHS[b]
OF VARIOUS ALUMINA CASTABLES[c] [95]

| Castable tested[c] | Cold Compressive Strength, psi | | | | | |
	Air 1 1100 °C 1 atm 6 h	Gas A 500 °C 1000 psi 58 h	Air 2 500 °C 1 atm 1000 h	Gas A 500 °C 1000 psi 1000 h	Gas B 500 °C 1000 psi 1000 h	Gas C 500 °C 1000 psi 1000 h
DENSE CASTABLES:						
90% Al_2O_3	5,430	6,570	5,510	2,268[d]	2,306[d]	1,840[d]
90% Al_2O_3 + 0.1% Fe	2,390	4,100	5,781	2,771[d]	1,120[def]	2,515[de]
90% Al_2O_3 + 310 SS[g]	2,890	1,750				
90% Al_2O_3 + alkali[h]	4,520	4,060				
50% Al_2O_3	3,800	2,530	6,970	7,993[i]	9,283[d]	10,135[di]
50% Al_2O_3 + 0.1% Fe	5,140	4,630				
50% Al_2O_3 + 310 SS[g]	2,680	2,470				
50% Al_2O_3 + alkali[h]	5,140	6,080				
INSULATING CASTABLES:						
95% Al_2O_3			547	353[d]	360[d]	341[d]
95% Al_2O_3 + rust			534	393	418	314
55% Al_2O_3			1,026	1,258[k]	1,688[dek]	1,157[e]
55% Al_2O_3 + rust			1,069	1,508[dj]	1,195[j]	1,272
40% Al_2O_3			1,090	1,706[d]	1,521[d]	1,499
40% Al_2O_3 + rust			814	1,270[d]	1,582[de]	940[e]

[a] Gas compositions in mole percent:
A = 38 H_2O, 24 H_2, 18 CO, 12 CO_2, 5 CH_4, 2 NH_3, 1 H_2S
B = 40 H_2O, 25 H_2, 18 CO, 12 CO_2, 5 CH_4
C = 40 H_2O, 25 H_2,   12 CO_2, 5 CH_4, 18 N_2.
Temperature, pressure, and exposure time are given in the table for each exposure.
Specimens were prefired before exposure according to the conditions given for
Air 1 or Air 2.

[b] Compressive strength was tested according to ASTM C133-72.

[c] No preparative information or other characterization was given in the reports and
no commercial brand names were included. All castables are calcium aluminate
bonded. No information was given about the manner of the Fe addition or the rust
addition. See sections for reference [9] for earlier reports from the same organi-
zation.

[d] The reports indicated a difference significant at the 99% level of confidence be-
tween the results for this exposure and for the Air 2 firing.

[e] Values for Gas B and Gas C reported to show a difference significant at the 99%
confidence level.

[f] In one of the original reports this value is given as 1,842.

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(Table Continued)

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EFFECT OF VARIOUS HIGH-BTU GAS COMPOSITIONS[a] ON THE COMPRESSIVE STRENGTHS
OF VARIOUS ALUMINA CASTABLES[b][95], Continued

Footnotes continued

[g]310 stainless steel fibers (4 wt%) were added to the mix before casting.

[h]Alkali-containing specimens were prepared by soaking for 6 hours in a 50% Na_2CO_3-50% K_2CO_3 saturated solution prior to firing.

[i]Values for Gas A and Gas C reported to show a difference significant at the 99% confidence level.

[j]Values for Gas A and Gas B reported to show a difference significant at the 99% confidence level.

EFFECT OF UNSATURATED STEAM AND CO/STEAM ATMOSPHERES[a] ON THE COMPRESSIVE STRENGTH[b] OF ALUMINA REFRACTORIES[c][95]

Refractory[c]	Brand Name/Manufacturer	Compressive Strength,[b] psi				
		Oven-Dried 110 °C 24 h	Air-Fired 1000 °C 250 h	Steam 1000 °C 250 h 105 psig	CO/H$_2$O=3.5 1000 °C 250 h 105 psig	CO/H$_2$O=7.0 1000 °C 250 h 105 psig
High-Al$_2$O$_3$ dense castable	Plicast L-22-84/Plibrico	12,940[d]	12,670[d]	11,190[e]	15,910[e]	14,630[e]
High-Al$_2$O$_3$ dense castable	Greenkon-33/A.P. Green	15,620	6,410	5,660	NA[f]	3,270[g]
High-Al$_2$O$_3$ dense castable	H-W 9/79/[Harbison-Walker]	9,320	12,300	10,060	13,110	15,920[g]
90+% Al$_2$O$_3$ brick	Kricor/Kaiser	15,400	6,040	13,010[g]	13,830[g]	14,640[g]
High-Al$_2$O$_3$ chrome phosphate-bonded ramming mix	Lavalox X-8/Lava Crucible Refractories	6,550	9,050	11,230	8,250	10,990
Intermediate-Al$_2$O$_3$ dense castable	Resco Cast RS-17E/Resco Products	8.290	6,070	12,180[g]	7,660[g]	6,680
Fireclay dense castable	Plicast HyMOR 3000/Plibrico	4,120	5,570	7,710[g]	NA[f]	6,630[g]
Fireclay castable	H-W 8/80/[Harbison-Walker]	6,050	4,760	8,130[g]	NA[f]	4,520

a Refractories were exposed under the indicated conditions to unsaturated atmospheres of steam and carbon monoxide/steam. The two CO/steam atmospheres had two different ratios of CO to steam, 3.5 and 7.0.

b [Compressive strength was probably tested according to ASTM C133-72.]

c Specimens were 2-inch cubes prepared and supplied by the manufacturers. Before testing the specimens were oven-dried at 110 °C for 24 hours.

d Values in this column are the average of five samples.

e Values in this column are the average of three samples.

(Table Continued)

EFFECT OF UNSATURATED STEAM AND CO/STEAM ATMOSPHERES[a] ON THE COMPRESSIVE STRENGTH[b] OF

ALUMINA REFRACTORIES[c][95], Continued

Footnotes continued

[f] Samples not available for compressive strength testing due to damage in the exposure test vessel.

[g] Value is significantly different at the 99% level of confidence from the value for samples fired in air at 1000 °C.

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EFFECT OF UNSATURATED ATMOSPHERES[a] OF STEAM AND CO/STEAM ON THE MODULUS
OF RUPTURE[b] OF ALUMINA REFRACTORIES[c][95]

Refractory[c]	Brand Name/ Manufacturer	Modulus of Rupture,[b] psi		
		Air-Fired	Steam	$CO/H_2O=3.5$
High-Al_2O_3 dense castable	Plicast L-22-84/ Plibrico	3,450	1,670	3,350
High-Al_2O_3 dense castable	Greenkon-33/ A.P. Green	1,670	1,110	750
94% Al_2O_3 dense castable	Greencast-94/ A.P. Green	1,510	1,170	1,220
High-Al_2O_3 insulating castable	B&W Kaolite 3300/ Babcock & Wilcox	130	160	NA[d]
90+% Al_2O_3 brick	Kricor/Kaiser	3,220	3,890	3,690
High-Al_2O_3 phosphate bonded ramming mix	Kritab/Kaiser	3,270	3,760	3,820
High-Al_2O_3 phosphate bonded ramming mix	Lavalox X-8/Lava Crucible Refractories	5,710	5,920	NA[d]
93% Al_2O_3 phosphate bonded castable	B&W KaoPhos 93/ Babcock & Wilcox	1,680	1,870	1,850
90% Al_2O_3 phosphate bonded ramming mix	Brikram 90R/ General Refractories	3,010	2,970	3,490
Intermediate-Al_2O_3 dense castable	Resco Cast RS-17E/ Resco Products	950	2,680	2,810
Intermediate-Al_2O_3 dense castable	Blast Furnace Back-up Castable/C-E Refractories	1,150	1,590	1,350
Intermediate-Al_2O_3 dense castable	Lo-Erode/Kaiser	440	1,040	1,260
Intermediate-Al_2O_3 gunning mix	BF Castable 122-N/ General Refractories	270	640	690
Fireclay dense castable	Plicast HyMOR 3000/ Plibrico	1,110	1,270	1,300
Intermediate-Al_2O_3 insulating castable	Plicast LWI 206I/ Plibrico	100	120	NA[d]
59% Al_2O_3 insulating castable	Kast-O-Lite 30/ A.P. Green	190	440	NA[d]
35% Al_2O_3 insulating castable	VSL-50/A.P. Green	40	110	NA[d]
29% Al_2O_3 insulating castable	Greencast-29-L/ A.P. Green	20	20	NA[d]

- -

(Table Continued)

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EFFECT OF UNSATURATED ATMOSPHERES[a] OF STEAM AND CO/STEAM ON THE MODULUS

OF RUPTURE[b] OF ALUMINA REFRACTORIES[c][95], Continued

Refractory[c]	Brand Name/ Manufacturer	Modulus of Rupture,[b] psi		
		Air-Fired	Steam	CO/H_2O=3.5
54% Al_2O_3 insulating castable	Litecast 75-28/ General Refractories	400	540	640
Intermediate-Al_2O_3 gunning mix	72 Gunite Grade/ Sauereisen Cements	NA[d]	NA[d,e]	NA[d,e]

[a]Refractories were exposed to the indicated unsaturated atmospheres for 250 hours at 1000 °C. The firing took place at atmospheric pressure, the other exposures at 105 psig.

[b][Test method not specified.]

[c]Specimens may have been 2-inch cubes prepared and supplied by the manufacturers. Before testing the specimens were oven-dried at 110 °C for 24 hours.

[d]NA = not available.

[e]This material fused during steam exposure.

===

CREEP RATES[a] FOR REFRACTORY RESEMBLING SLAG-PENETRATED REFRACTORY[b][96]

Temperature °C	Applied Stress MPa	Specimen Number	Secondary Strain Rate s^{-1}
754	30	15	4.4×10^{-10}
		16	4.8×10^{-10}
		17	6.9×10^{-10}
805	10	8	3.1×10^{-10}
		9	1.4×10^{-10}
809	20	1	3.9×10^{-9}
		2	4.8×10^{-9}
		3	1.4×10^{-9}
793	30	4	3.2×10^{-9}
		5	2.2×10^{-9}
		6	4.0×10^{-9}
804[c]	50	10	3.5×10^{-8}
		11	2.3×10^{-8}
		12	2.0×10^{-8}
854[c]	30	21	1.0×10^{-7}
		22	3.9×10^{-8}
		23	1.1×10^{-7}
		18	3.5×10^{-7}
		19	9.7×10^{-8}
		20	1.3×10^{-7}

[a] Test specimens ∿10 mm wide and 5 mm high were tested in 4-point bending. The
outer and inner loading points were 40 and 10 mm respectively. Dead weight
loading was applied via a frictionless pneumatic bellows system. Displacement
during creep was measured by a linear-voltage-displacement-transducer attached
to the upper ram. Load and displacement were both monitored continuously.

[b] The commercial fused-cast refractory used in testing has a microstructure
which closely resembles that of refractories which have been penetrated by
slag. Composition (wt %): 14.25 SiO_2, 50.1 Al_2O_3, 34.2 ZrO_2 (Monofrax CS-3,
Carborundum). X-ray diffraction analysis shows that the refractory consists
of α-alumina and monoclinic zirconia embedded in an aluminosilicate glass
matrix. Test specimens were cut from the center portion of a large block of
the refractory. The scatter in the data is attributed to a lack of uniform
microstructure from point to point within the block.

[c] All test specimens at this temperature and the corresponding applied stress
failed. See Section B.3.2.113 for the graphical creep data corresponding
to the above data.

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CREEP DATA[a] FOR REFRACTORY RESEMBLING SLAG-PENETRATED REFRACTORY[b][96]
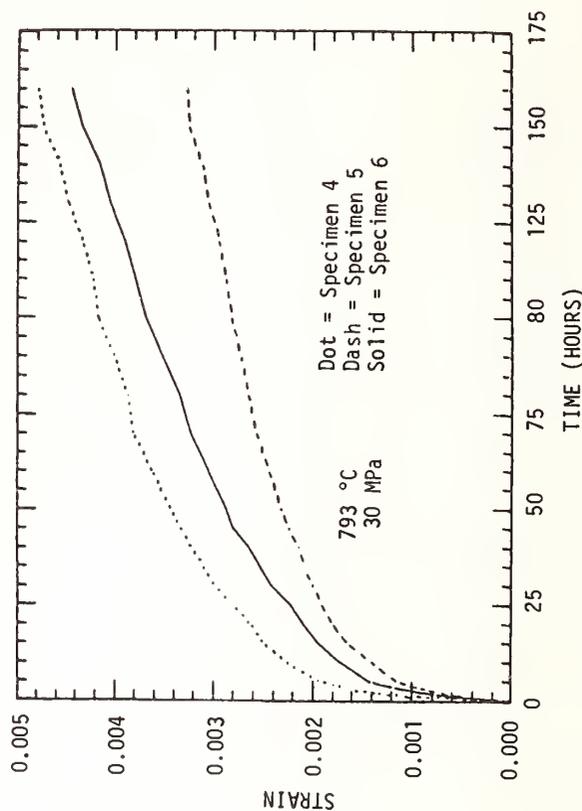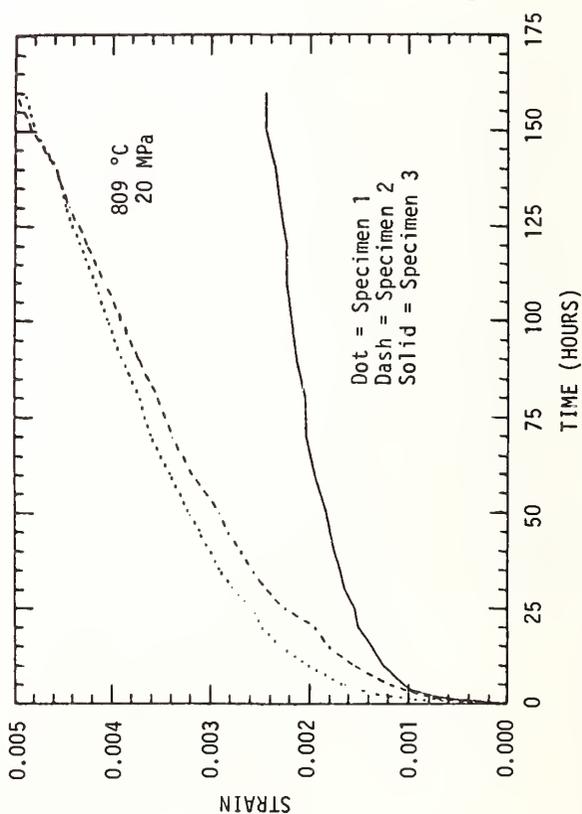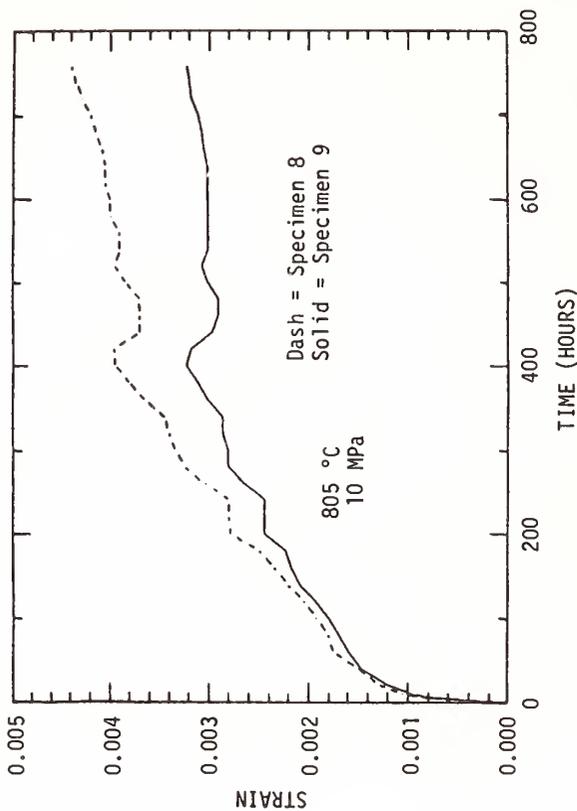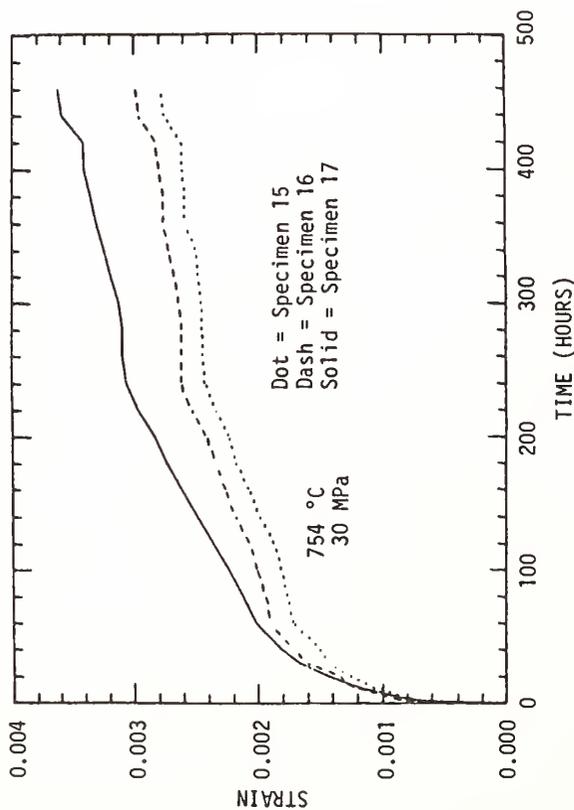
(Data Continued)

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CREEP DATA[a] FOR REFRACTORY RESEMBLING SLAG-PENETRATED REFRACTORY[b][96], Continued
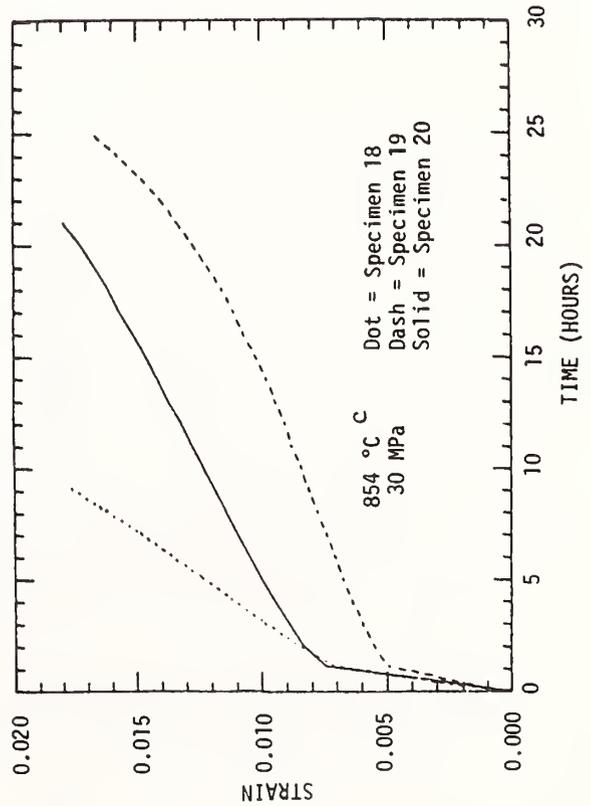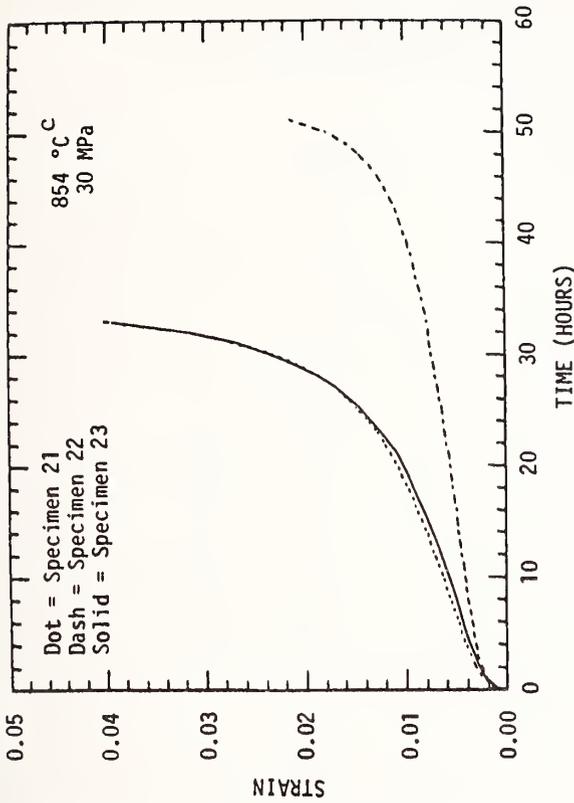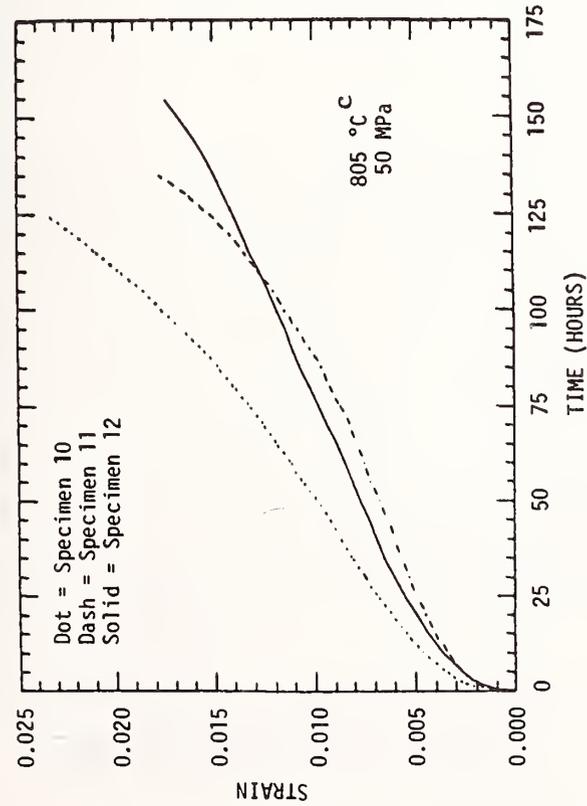
[a]See Section B.3.2.112 for the test information and for the secondary creep rates calculated from these data.

[b]A commercial fused-cast refractory (Monofrax CS-3, Carborundum) with a microstructure which resembles that of refractory which has been penetrated by slag. See Section B.3.2.112 for composition and other details.

[c]All the specimens tested under these conditions failed.

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TIME,[a] STRESS,[b] AND TEMPERATURE[c] DEPENDENCE OF CREEP DATA FOR REFRACTORY
RESEMBLING SLAG-PENETRATED REFRACTORY[d][96]

Figure 1

Figure 2

- -

(Data Continued)

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TIME,[a] STRESS,[b] AND TEMPERATURE[c] DEPENDENCE OF CREEP DATA FOR REFRACTORY
RESEMBLING SLAG-PENETRATED REFRACTORY[d][96]

Figure 3

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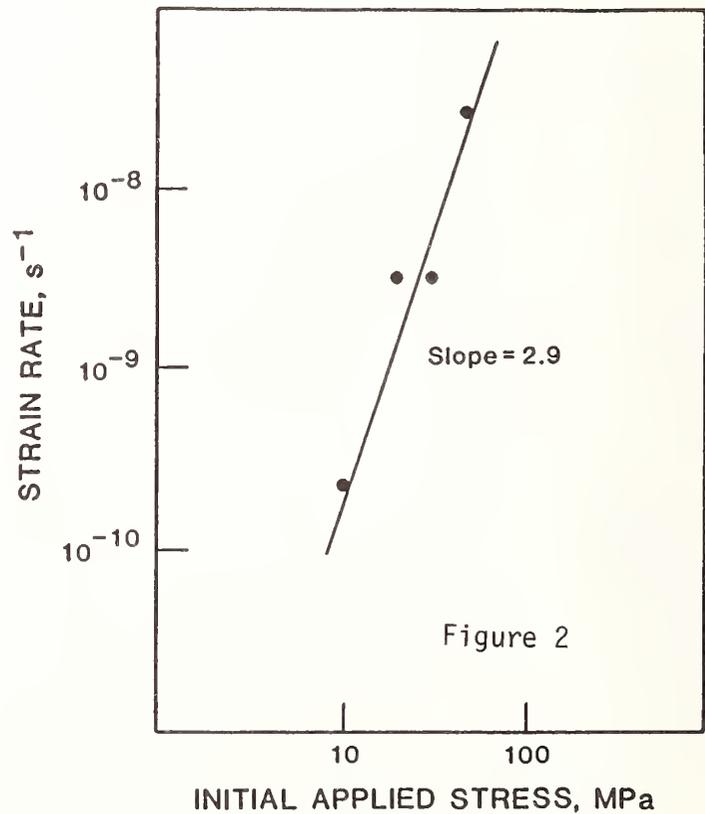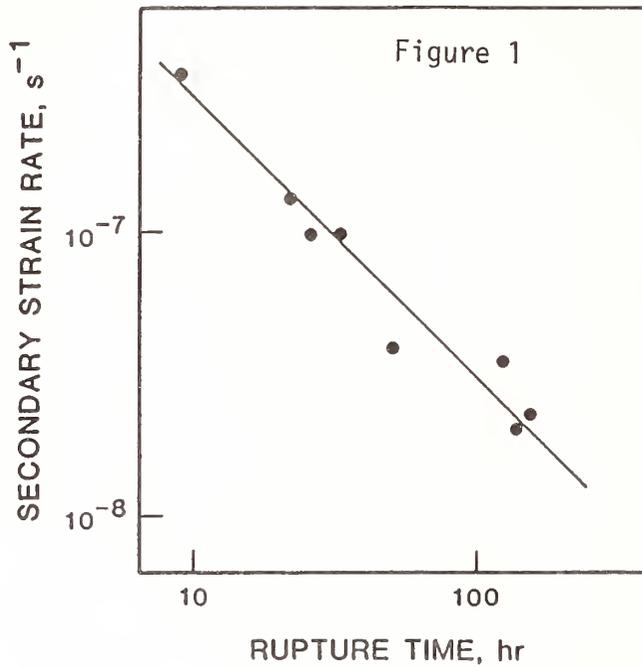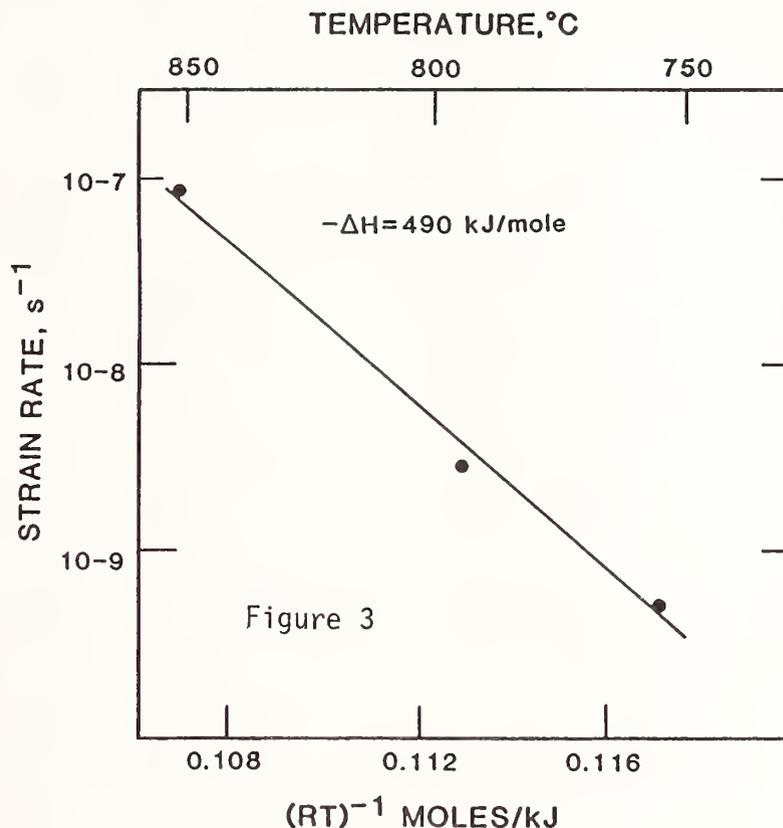
[a]Secondary creep rate and time to failure plotted in Figure 1 was found to
fit the Monkman-Grant Relation, for which the time to failure (t) is in-
versely proportional to the creep rate ($\dot{\varepsilon}$): $t\dot{\varepsilon}$ = constant. The slope of
the line is -1. Data are plotted for the specimens which failed in the
tests reported in Sections B.3.2.112 and B.3.2.113.

[b]Data plotted in Figure 2 are those reported in Sections B.3.2.112 and
B.3.2.113 collected at a nominal temperature of 800 °C (tests at 793, 804,
805, and 809 °C). Slope = 2.9, suggestive of cavitation within the vitreous
phase during the creep process.

[c]Data from Sections B.3.2.112 and B.3.2.113 for constant applied stress
(30 MPa) are plotted in Figure 3 to show temperature dependence. Creep is
shown to be an activated process with an activation energy of ∿490 kJ/mole.
This value is similar to that for the viscous creep of glass at elevated
temperatures.

[d]A commercial fused-cast refractory (Monofrax CS-3, Carborundum) with a
microstructure which resembles that of refractory which has been pene-
trated by slag. See footnote b, Section B.3.2.112 for the details of
composition and microstructure.

STEPPED-TEMPERATURE CREEP TESTS[a] FOR ALUMINA WITH VARYING GLASS CONTENT [96]
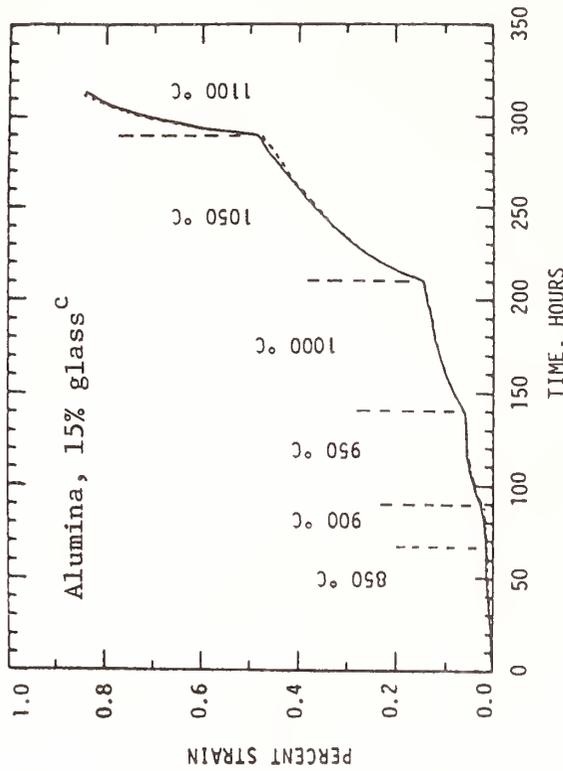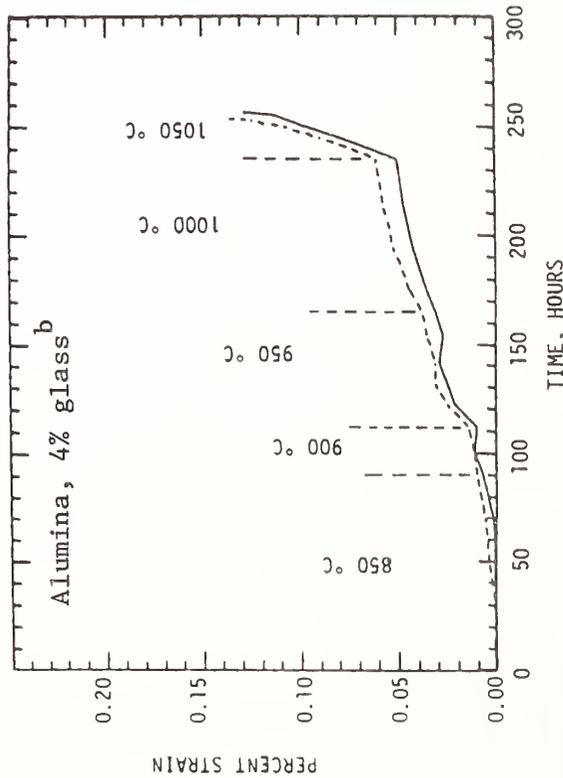
[a] Stepped-temperature creep testing was performed in which a constant load was applied to the specimen and the temperature increased at regular intervals until either failure occurred or the deformation was so severe that further deformation was pointless. Specimens were 5 mm wide and 3 mm high and were tested in 4-point bending. The outer and inner loading points were 40 and 10 mm respectively. Dead weight loading was applied via a frictionless pneumatic bellows system. Displacement during creep was measured by a linear-voltage-displacement-transducer attached to the upper ram. Load and displacement were both monitored continuously. Applied stress was 60 MPa. Dashed and solid lines indicate duplicate specimens.

[b] High-density alumina with a fine-grain microstructure, low porosity, 12 μm grain size, and 4% glass content (AD-96, Coors Porcelain).

[c] High-density alumina with a fine-grain microstructure, low porosity, 6 μm grain size, and 15% glass content (AD-85, Coors Porcelain).

CREEP DATA[a] AND TEMPERATURE DEPENDENCE[b] OF CREEP FOR A 4% GLASS-CONTENT ALUMINA[c][96]
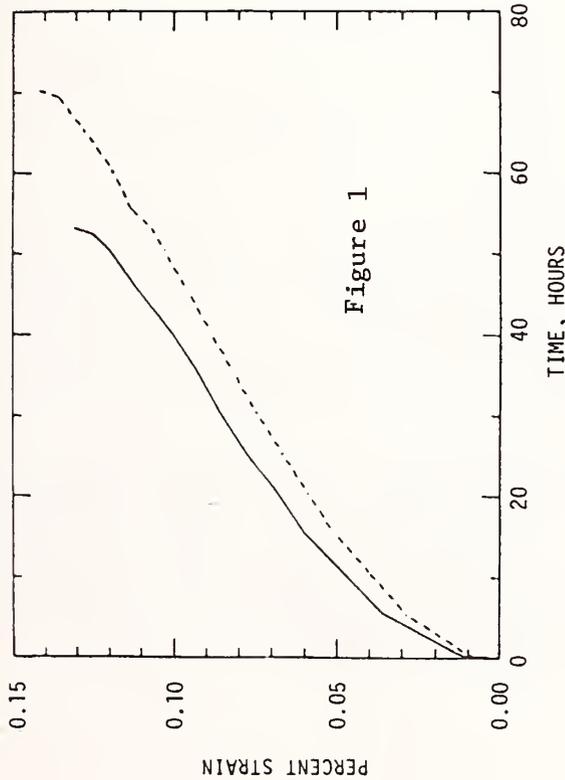
Figure 1
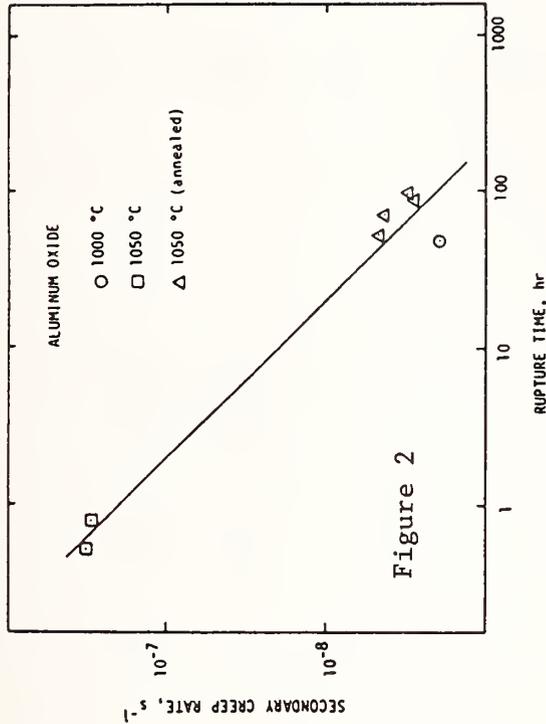
Figure 2

[a]Figure 1 shows typical creep data for this material at 1050 °C with an initial outer fiber stress of 60 MPa applied. The total strain to failure was ~0.14%. This material showed far less scatter in the data than did another glass-containing refractory (see Sections B.3.2.112 and B.3.2.113). Specimens 5mm wide and 3 mm high were tested in 4-point bending. The outer and inner loading points were 40 and 10 mm respectively. Dead weight loading was applied via a frictionless pneumatic bellows system. Displacement during creep was measured by a linear-voltage-displacement-transducer attached to the upper ram. Load and displacement were both monitored continuously. Dashed and solid lines indicate duplicate specimens.

[b]Figure 2 shows secondary strain rate versus time to failure for creep tests at the indicated temperatures and 60 MPa applied stress. This material seems to show Monkman-Grant behavior (see Section B.3.2.114).

[c]High-density alumina with a fine-grain microstructure, low porosity, 12 μm grain size, and 4% glass content (AD-96, Coors Porcelain).

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UNIT CREEP[a] FOR A HIGH-ALUMINA CASTABLE REFRACTORY[b][97]
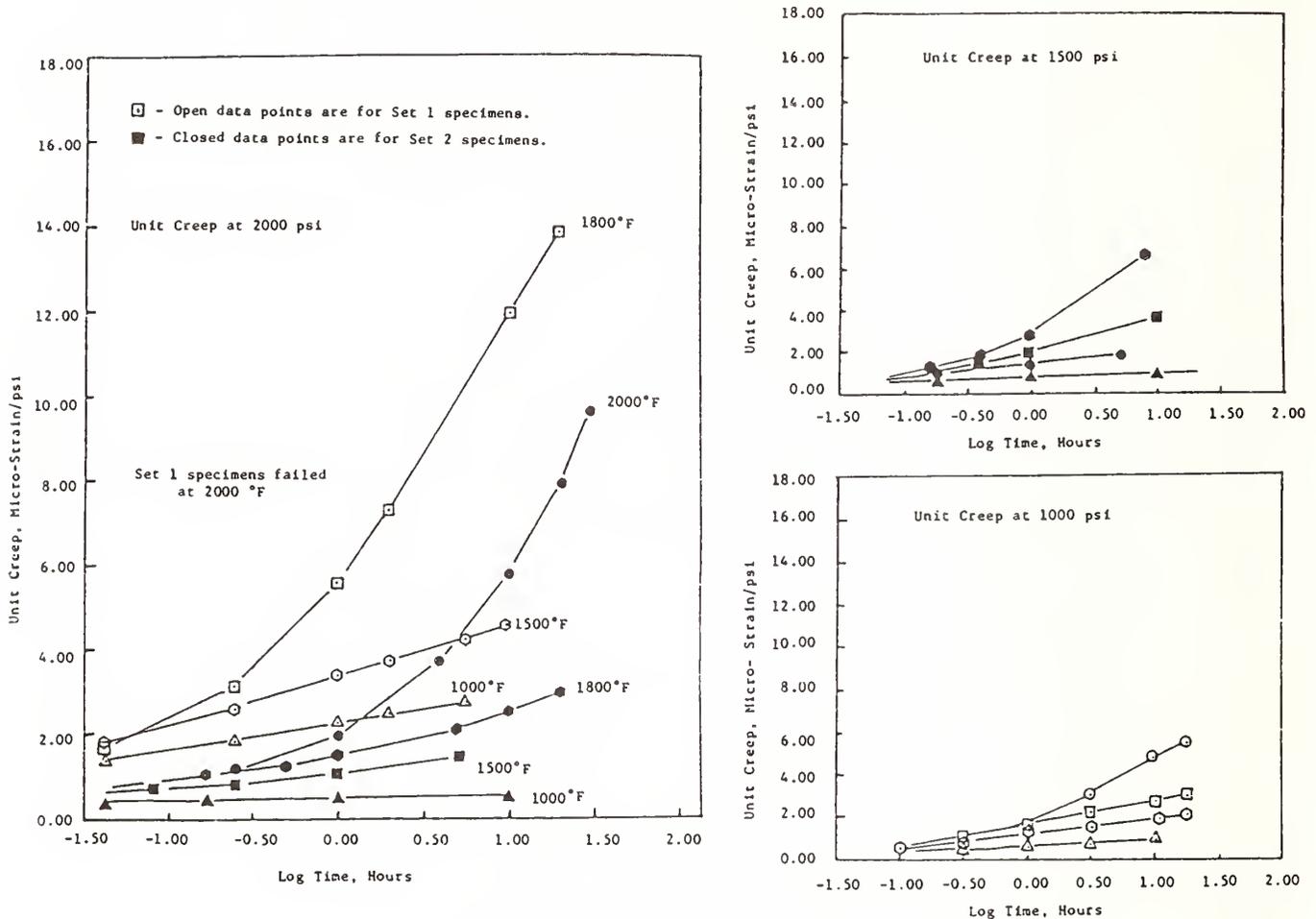
[a]Specimens were tested in air in creep furnaces under compressive loading. The heat-up period is about 5 hours. Specimens were heated up to the desired temperature and then the load was applied. After the run the load was released, the specimen heated to the next temperature where the load was applied again, etc. Creep strain was monitored with a linear voltage displacement transducer.

[b]Castable is a dense generic formulation, cast in brick in accordance with ASTM C862-77. The 90+% alumina refractory consisted of 70% tabular alumina (-6 mesh + fines) (T-61, Alcoa), 5% calcined alumina (-325 mesh) (A-2, Alcoa), and 25% calcium aluminate cement (CA-25C, Alcoa), with 8.0% water. Set 1 specimens were cast in the form of the 1 in. x 1 in. x 6 in. specimens by another laboratory, Set 2 were cast in 9 in. molds and cut to size at the Iowa State University laboratory doing this testing.

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EFFECT OF INITIAL HEATING RATE ON THE CREEP[a] OF A HIGH-ALUMINA CASTABLE[b][97]
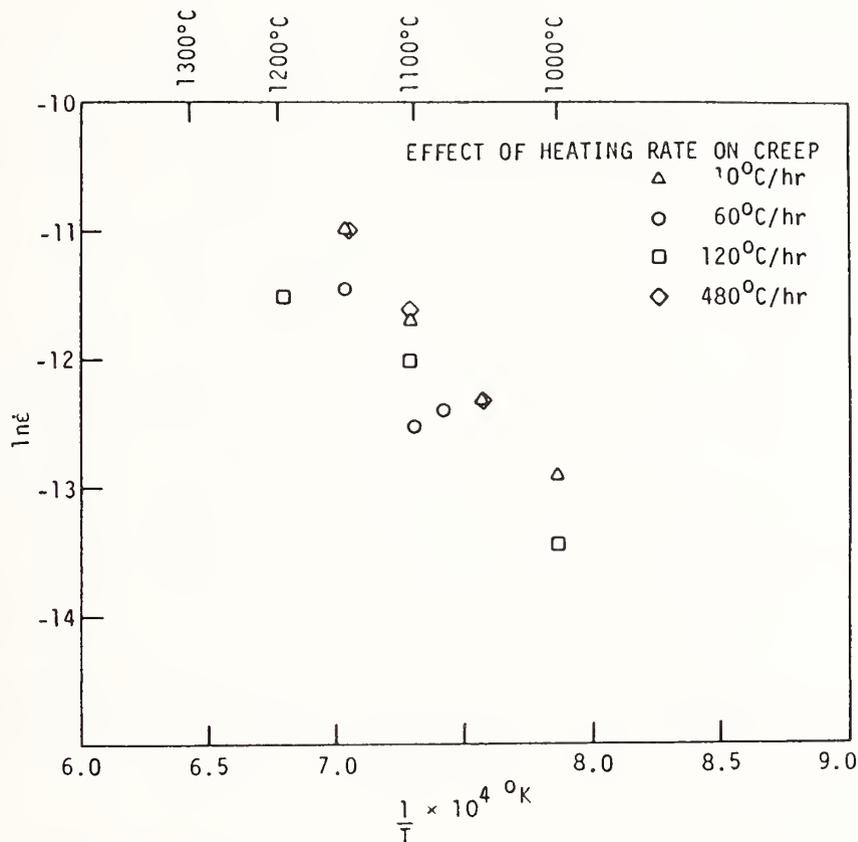
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[a]Creep deformation was measured using specimens 3 cm in diameter by 5 cm in
length. Specimens were tested in air in creep furnaces under compressive
loading. The heat-up period was 5 hours. Specimens were heated up to the
desired temperature and then the load was applied. After the run the load
was released, the specimen heated to the next temperature where the load was
applied again, etc. Creep strain was monitored with a linear voltage dis-
placement transducer. An empirical model was developed in an Arrhenius
form for steady-state strain rates: $\dot{\varepsilon} = A\sigma^n \exp(-\Delta H_c/RT)$ where $\dot{\varepsilon}$ is creep
rate, A is a constant, σ is stress to the power n, ΔH_c is the activation
energy for creep, R is the gas constant, and T is the absolute temperature.

[b]Castable is a generic formulation, cast in brick in accordance with ASTM
C862-77. Specimens were core drilled from the brick. The 90+% alumina re-
fractory consisted of 70% tabular alumina (-6 mesh + fines) (T-61, Alcoa),
5% calcined alumina (-325 mesh) (A-2, Alcoa), and 25% calcium aluminate
cement (CA-25C, Alcoa), with 9.0-9.5% water.

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EFFECT OF CEMENT CONTENT[a] ON THE MODULUS OF RUPTURE[b] OF A HIGH-ALUMINA CASTABLE[c][97]

MOR VS % CEMENT
WATER CONTENT = 8.5%

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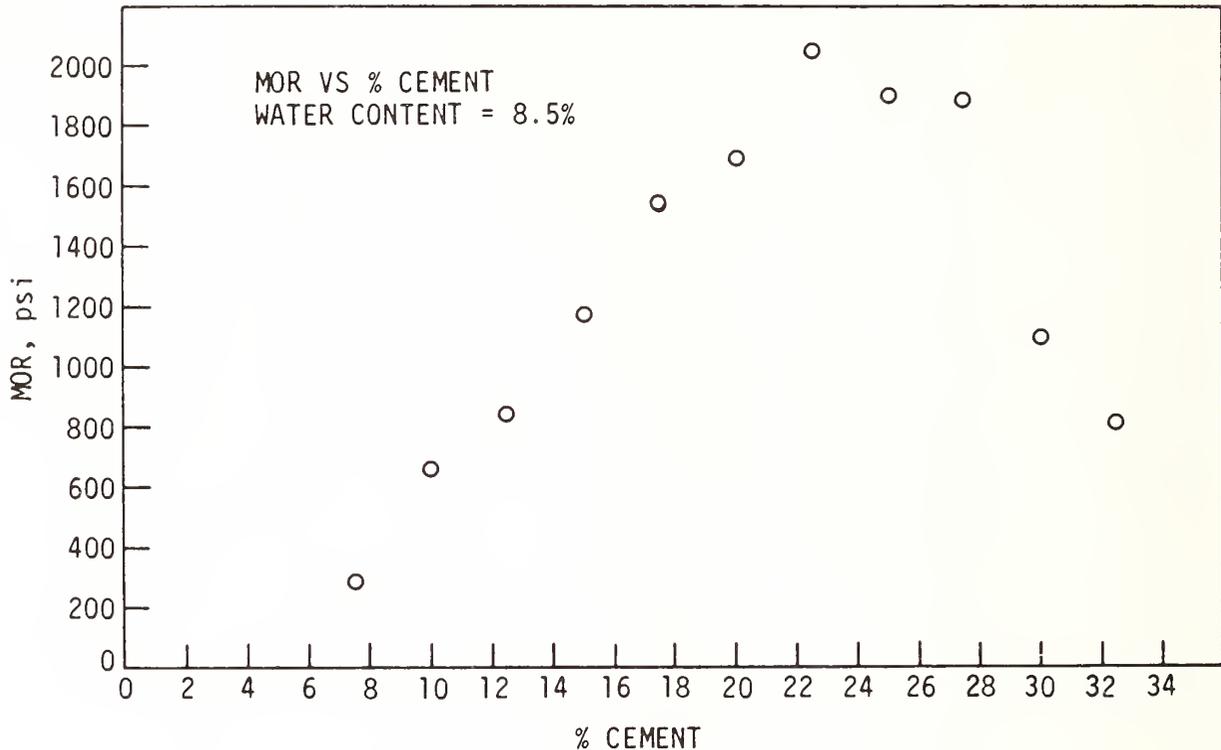
[a]The calcium aluminate cement content varied for different specimens from 7.5% to 32.5% of the total dry weight material. The water content was held constant at 8.5% of the dry mix.

[b]Modulus of rupture measurements were made on the as-cast and cured material. [No information was given in the reports concerning the test methods.]

[c]A 90+% alumina generic castable, cast in bricks according to ASTM C862-77. The aggregate consisted of tabular alumina (-6 mesh + fines) (T-61, Alcoa) and calcined alumina (-325 mesh) (A-2, Alcoa). The cement used was CA-25C calcium aluminate cement (Alcoa). The specimen with 7.5% cement failed immediately upon loading.

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ACTIVATION ENERGIES FOR CREEP[a] AND STRESS EXPONENTS FOR ALUMINA CASTABLES[b]
AFTER VARIOUS TREATMENTS[c] AND WITH VARIOUS CEMENTS[97]

Material[b]	Test Conditions[c]	Activation Energy, H_c kcal/mol (stress)	Stress Exponent n (temperature)
90+% Alumina with 80% alumina cement	Cured, initial heat-up	41 (2000 psi)	0.5 (2050 °F)
	Cured, cycled	149 (2000)	3.4 (2050)
	Prefired, 5 h, 2200 °F	64 (2000)	-
	Prefired, cycled	190[d] (2000)	-
	Prefired, 1000 h, 1800 °F	42 (2000)	-
	Gasifier test, initial heat	46 (2000)	-
	Gasifier test, cycled	155 (2000)	-
90+% Alumina with 81% alumina cement	Cured, initial heat-up	24 (1600)	-0.9 (2000)
	Cured, cycled	120 (1600)	2.6 (2000)
90+% Alumina with 71% alumina cement	Cured, initial heat-up	34 (1600)	-0.6 (2000)
	Cured, cycled	133 (1600)	not given
90+% Alumina with 58% alumina cement	Cured, initial heat-up	44 (1600)	-0.2 (2000)
	Cured, cycled	159 (1600)	not given
90+% Alumina with 39% alumina cement	Cured, initial heat-up	57 (1600)	0.7 (2000)
	Cured, cycled	133 (1600)	2.9 (2000)
50% Alumina with 80% alumina cement	Cured, initial heat-up	30[e] (2000)	0.7 (2012)
	Cured, cycled	179[e] (2000)	3.4 (2012)
	Prefired, 1000 h, 1800 °F	56 (2000)	-
	Gasifier test, initial heat	83 (2000)	-
50% Alumina with 81% alumina cement	Cured, initial heat-up	19 (1600)	0.8 (2000)
	Cured, cycled	101 (1600)	2.2 (2000)
50% Alumina with 71% alumina cement	Cured, initial heat-up	41 (1600)	1.0 (2000)
	Cured, cycled	failed at 1900 °F	failed at 2000 psi
50% Alumina with 58% alumina cement	Cured, initial heat-up	45 (1600)	1.3 (2000)
	Cured, cycled	failed at 1900 °F	failed at 2500 psi
50% Alumina with 39% alumina cement	Cured, initial heat-up	27 (1600)	0.9 (2000)
	Cured, cycled	154 (1600)	failed at 2000 psi
80% Alumina cement[f]	Cured, initial heat-up	33 (2000)	0.8 (1900)
	Cured, cycled	170 (2000)	2.9 (1900)

[a]Creep deformation was measured using specimens 3 cm in diameter by 5 cm in
length. Specimens were tested in air in creep furnaces under compressive load-
ing over ranges 1000-2200 °F (538-1204 °C) and 1000-2500 psi (6.9-17.2 MPa).
Heat-up periods were 5 hours. For constant stress tests, specimens were heated
to the first test temperature, the load applied, and deformation recorded. The
load was then released and the specimen heated to the next temperature, the same
load reapplied and deformation recorded. Thus the total strain at the higher
temperatures included the strain at lower temperatures. Constant temperature
tests involved increasing the stress incrementally from 500 psi (3.4 MPa) to

- -

(Table Continued)

==

ACTIVATION ENERGIES FOR CREEP[a] AND STRESS EXPONENTS FOR ALUMINA CASTABLES[b]
AFTER VARIOUS TREATMENTS[c] AND WITH VARIOUS CEMENTS[97]

Footnotes continued

3000 psi (20.7 MPa) by 500 psi (3.4 MPa) increments. Stress level was cycled
down and then up. Creep strain was monitored with a linear voltage displace-
ment transducer until steady state creep was obtained. Constant stress-
variable temperature data were found to have a linear dependence on inverse
absolute temperature. Constant temperature-variable stress data were found
to have a linear dependence on the ln of the stress. An empirical model
for steady-state strain rates was developed in an Arrhenius form:
$\dot{\varepsilon} = AS\sigma^n \exp(-\Delta H_c/RT)$ where $\dot{\varepsilon}$ is creep rate, A is a constant, S is a structure-
dependent term, σ is stress to the power n, ΔH_c is the activation energy for
creep, R is the gas constant, and T is the absolute temperature.

[b]Two generic castables were tested, cast as brick in accordance with ASTM
C862-77. Test specimens were core drilled from the brick. The 90+% alumina
dense castable consisted of 70% tabular alumina (-6 mesh + fines) (T-61,
Alcoa), 5% calcined alumina (-325 mesh) (A-2, Alcoa), and 25% calcium alumi-
nate cement, with 9.0-9.5% water. The 50+% alumina dense castable consisted
of 75% calcined kaolin (27.5% -6 + 10 mesh, 22.5% -10 + 20 mesh, 20% -20
mesh, 5% ball milled fines 5% of which was <325 mesh) (Mulcoa 47, C-E Miner-
als) and 25% cement. The calcium aluminate cements used were--
80% alumina: 79.7% Al_2O_3, 18.4% CaO (CA-25C, Alcoa),
81% alumina: 80.9% Al_2O_3, 17.0% CaO (SECAR 80, Lone Star Lafarge),
71% alumina: 70.6% Al_2O_3, 27.5% CaO (SECAR 71, Lone Star Lafarge),
58% alumina: 58.0% Al_2O_3, 33.5% CaO, 5.6% SiO_2, 1.5% Fe_2O_3 (Refcon, Universal
          Atlas Cement),
39% alumina: 39.0% Al_2O_3, 38.5% CaO, 4.5% SiO_2, 12.0% Fe_2O_3 (FONDU, Lone Star
          Lafarge).
The castable specimens failed in shear, the neat cement merely slumped, at the
highest temperatures and pressures.

[c]For tests where cycling is indicated, the specimens were put through the iden-
tical procedures more than once. Specimens designated prefired were fired in
air at the stated temperature for the given time prior to creep testing. The
specimens exposed to a gasifier atmosphere prior to creep testing were sub-
jected to the conditions at the DoE IITRI facility for 1000 hours. [Condi-
tions were probably 982 °C (1800 °F), 1000 psi, gas composition (vol %):
24 H_2, 18 CO, 12 CO_2, 5 CH_4, 1 NH_3, 0-1.0 H_2S, balance H_2O.]

[d]Some of the reports of the series gave this value as 170 kcal/mole.

[e]Earlier reports of the series gave these values as 44 and 151 kcal/mole.

[f]Neat cement specimens were cast to specimen size, with water/cement ratio =
0.3.

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COMPOSITE CREEP CURVE[a] FOR A HIGH-ALUMINA CASTABLE[b][97]

COMPOSITE CREEP CURVE

σ 2200 °F
◇ 2100 °F
▽ 2000 °F
○ 1900 °F
△ 1750 °F
□ 1500 °F
○ 1250 °F

STRAIN

TIME, h
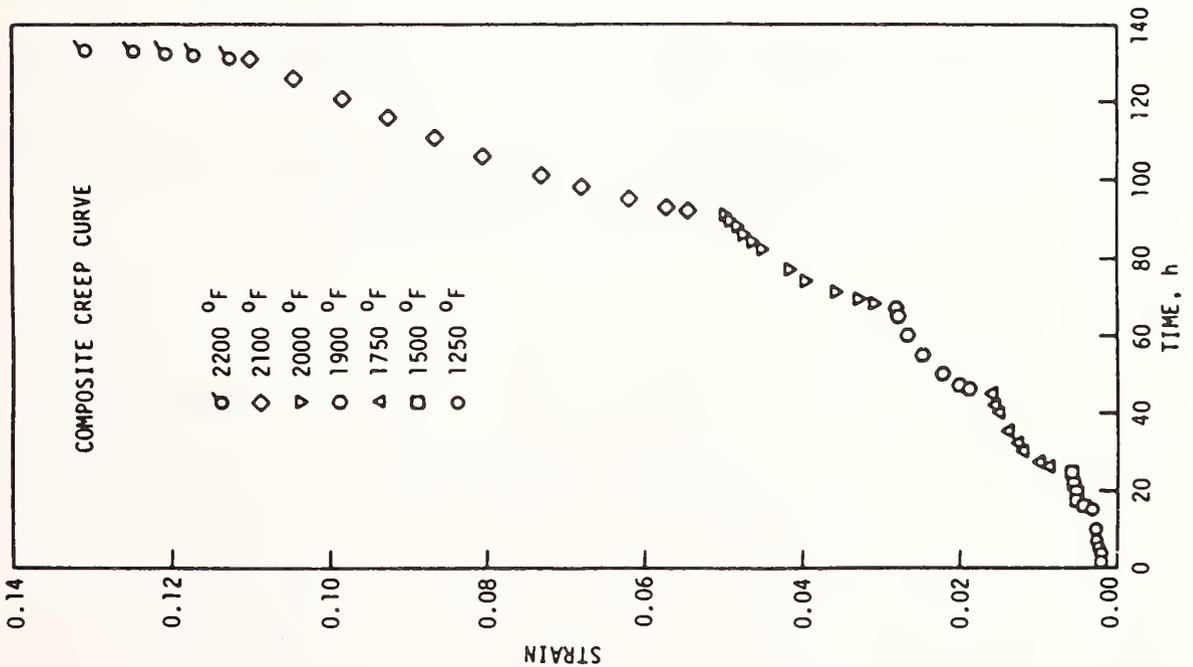
[a]Applied stress was 2000 psi (13.8 MPa). Creep deformation was measured using specimens 3 cm in diameter by 5 cm in length. Specimens were tested in air in creep furnaces under compressive loading. Heat-up periods were 5 hours. For constant stress tests, specimens were heated to the first test temperature, the load applied, and deformation recorded. The load was then released and the specimen heated to the next temperature, the same load reapplied and deformation recorded. Thus the total strain at the higher temperatures included the strain at lower temperatures. Creep strain was monitored with a linear voltage displacement transducer until steady state creep was obtained. Constant stress-variable temperature data were found to have a linear dependence on inverse absolute temperature.

[b]The 90+% alumina generic dense castable consisted of 70% tabular alumina (-6 mesh + fines) (T-61, Alcoa), 5% calcined alumina (-325 mesh) (A-2, Alcoa), and 25% calcium aluminate cement (CA-25C, Alcoa), with 9.0-9.5% water. Bricks were cast in accordance with ASTM C862-77 and test specimens were core drilled from the bricks.

COMPARISON OF CREEP DATA[a] FOR A HIGH-ALUMINA CASTABLE[b] AND NEAT CEMENT[c][97]
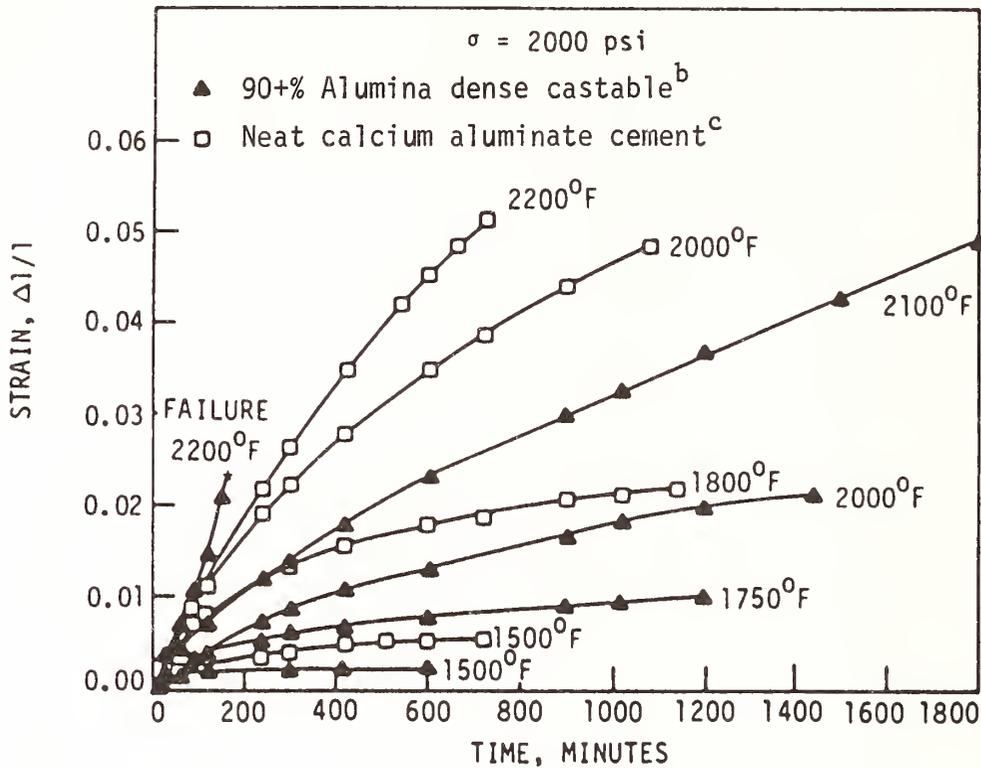
[a] See Section B.3.2.121, footnote a, for the details of the creep testing procedure.

[b] See Section B.3.2.121, footnote b, for the composition of the 90+% alumina dense generic castable tested.

[c] Cement is 79.7% alumina, 18.4% calcia (CA-25C, Alcoa). Neat cement specimens were cast to specimen size, with water/cement ratio = 0.3.

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EFFECT OF TEMPERATURE AND TEMPERATURE CYCLING ON CREEP DATA[a] FOR A HIGH-ALUMINA CASTABLE[b] WITH VARIOUS CEMENTS[c] [97]

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EFFECT OF TEMPERATURE AND TEMPERATURE CYCLING ON CREEP DATA[a] FOR A HIGH-
ALUMINA CASTABLE[b] WITH VARIOUS CEMENTS[c][97], Continued

===

EFFECT OF TEMPERATURE AND TEMPERATURE CYCLING ON CREEP DATA[a] FOR A HIGH-
ALUMINA CASTABLE[b] WITH VARIOUS CEMENTS[c][97], Continued

===

EFFECT OF TEMPERATURE AND TEMPERATURE CYCLING ON CREEP DATA[a] FOR A HIGH-ALUMINA CASTABLE[b] WITH VARIOUS CEMENTS[c][97], Continued

[a] Creep deformation was measured using specimens 3 cm in diameter by 5 cm in length. Specimens were tested in air in creep furnaces under compressive loading. Heat-up periods were 5 hours. For constant stress tests, speci-mens were heated to the first test temperature, the load applied, and de-formation recorded. The load was then released and the specimen heated to the next temperature, the same load reapplied and deformation recorded. Creep strain was monitored with a linear voltage displacement transducer until steady state creep was obtained. Constant stress-variable temperature data were found to have a linear dependence on inverse absolute temperature. For tests where cycling is indicated, the specimens were put through the identical procedures more than once.

[b] The 90+% alumina dense generic castable was cast as brick in accordance with ASTM C862-77 and test specimens were core drilled from the brick. Composition: 70% tabular alumina (-6 mesh + fines) (T-61, Alcoa), 5% cal-cined alumina (-325 mesh) (A-2, Alcoa), and 25% calcium aluminate cement, with 9.0-9.5% water.

[c] The calcium aluminate cements used were--
80% alumina: 79.7% Al_2O_3, 18.4% CaO (CA-25C, Alcoa),
81% alumina: 80.9% Al_2O_3, 17.0% CaO (SECAR 80, Lone Star Lafarge),
71% alumina: 70.6% Al_2O_3, 27.5% CaO (SECAR 71, Lone Star Lafarge),
58% alumina: 58.0% Al_2O_3, 33.5% CaO, 5.6% SiO_2, 1.5% Fe_2O_3 (Refcon, Uni-
               versal Atlas Cement).

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TEMPERATURE DEPENDENCE OF THE STRAIN RATE IN CREEP TESTING[a] OF ALUMINA CASTABLES[b][97]

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TEMPERATURE DEPENDENCE OF THE STRAIN RATE IN CREEP TESTING[a] OF ALUMINA
CASTABLES[b][97], Continued

===

TEMPERATURE DEPENDENCE OF THE STRAIN RATE IN CREEP TESTING[a] OF ALUMINA
CASTABLES[b][97], Continued

---

[a]Creep deformation was measured using specimens 3 cm in diameter by 5 cm in
length. Specimens were tested in air in creep furnaces under compressive
loading. Heat-up periods were 5 hours. For constant stress tests, specimens
were heated to the first test temperature, the load applied, and deformation
recorded. The load was then released and the specimen heated to the next
temperature, the same load reapplied and deformation recorded. Creep strain
was monitored with a linear voltage displacement transducer until steady
state creep was obtained. Constant stress-variable temperature data were
found to have a linear dependence on inverse absolute temperature. An em-
pirical model for steady-state strain rates was developed in an Arrhenius
form: $\dot{\varepsilon} = AS\sigma^n exp(-\Delta H_c/RT)$ where $\dot{\varepsilon}$ is creep rate, A is a constant, S is a
structure-dependent term, σ is stress to the power n, ΔH_c is the activation
energy for creep, R is the gas constant, and T is the absolute temperature.
For tests where cycling is indicated, the specimens were put through the
identical procedures more than once. Specimens designated prefired were
fired in air at the stated temperature for the given time prior to creep
testing. The specimens exposed to a gasifier atmosphere prior to creep test-
ing were subjected to the conditions at the DoE IITRI facility for 1000 hours.
[Conditions were probably 982 °C (1800 °F), 1000 psi, gas composition (vol %):
24 H_2, 18 CO, 12 CO_2, 5 CH_4, 1 NH_3, 0-1.0 H_2S, balance H_2O.] See B.3.2.120.

[b]Two generic castables were tested, cast as brick in accordance with ASTM
C862-77. Test specimens were core drilled from the brick. The 90+% alumina
dense castable consisted of 70% tabular alumina (-6 mesh + fines) (T-61,
Alcoa), 5% calcined alumina (-325 mesh) (A-2, Alcoa), and 25% calcium alumi-
nate cement (CA-25C, Alcoa), with 9.0-9.5% water. The 50% alumina dense
castable consisted of 75% calcined kaolin (27.5% -6 + 10 mesh, 22.5% -10
+ 20 mesh, 20% -20 mesh, 5% ball milled fines 5% of which was <325 mesh)
(Mulcoa 47, C-E Minerals) and 25% calcium aluminate cement (CA-25C, Alcoa).

TEMPERATURE DEPENDENCE OF THE CREEP STRAIN RATE[a] FOR A HIGH-ALUMINA CASTABLE[b] WITH VARIOUS CEMENTS[c] [97]

(Data Continued)

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TEMPERATURE DEPENDENCE OF THE CREEP STRAIN RATE[a] FOR A HIGH-ALUMINA CASTABLE[b] WITH VARIOUS CEMENTS[c][97], Continued

[a]See Section B.3.2.120 for details of the creep testing. The activation energy for creep is given on the figures.

[b]A generic dense castable consisting of 70% tabular alumina (-6 mesh + fines) (T-61, Alcoa), 5% calcined alumina (-325 mesh) (A-2, Alcoa), and 25% calcium aluminate cement, with 9.0-9.5% water. Bricks were cast in accordance with ASTM C862-77 and test specimens were core drilled from the bricks. Neat cement specimens were cast to specimen size, with water/cement ratio = 0.3.

[c]See footnote b, Section B.3.2.120 for compositions of the cements used.

TEMPERATURE DEPENDENCE OF THE CREEP STRAIN RATE[a] FOR A MEDIUM-ALUMINA CASTABLE[b] WITH VARIOUS CEMENTS[c] [97]

(Data Continued)

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TEMPERATURE DEPENDENCE OF THE CREEP STRAIN RATE[a] FOR A MEDIUM-ALUMINA CASTABLE[b] WITH VARIOUS CEMENTS[c][97], Continued

[a]See Section B.3.2.120 for details of the creep testing. The activation energy for creep is given on the figures.

[b]A generic dense castable consisting of 75% calcined kaolin (27.5% -6 + 10 mesh, 22.5% -10 + 20 mesh, 20% -20 mesh, 5% ball milled fines 5% of which was <325 mesh) (Mulcoa 47, C-E Minerals) and 25% calcium aluminate cement, with 9.0-9.5% water. Bricks were cast in accordance with ASTM C862-77 and test specimens were core drilled from the bricks.

[c]See footnote b, Section B.3.2.120 for compositions of the cements used.

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EFFECT OF STRESS AND STRESS CYCLING ON CREEP DATA[a] FOR A HIGH-ALUMINA CASTABLE[b] WITH
VARIOUS CEMENTS[c][97]

(Data Continued)

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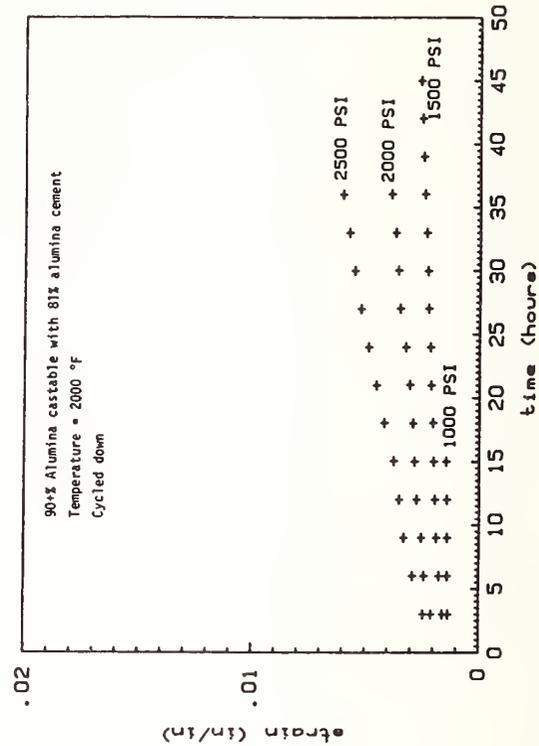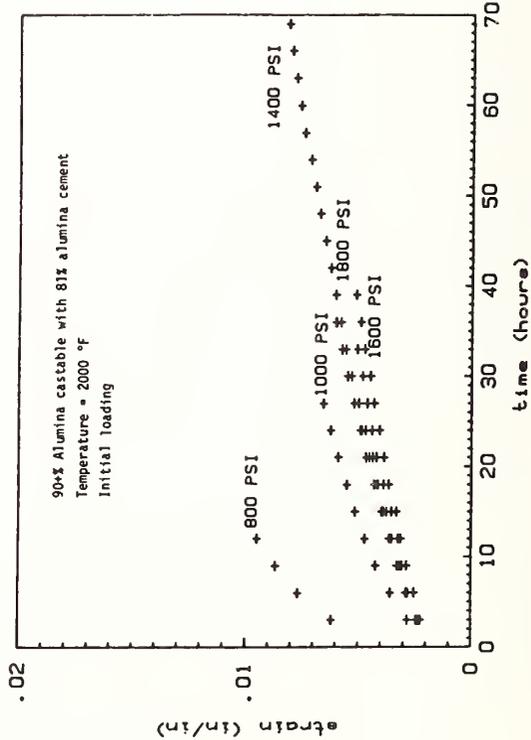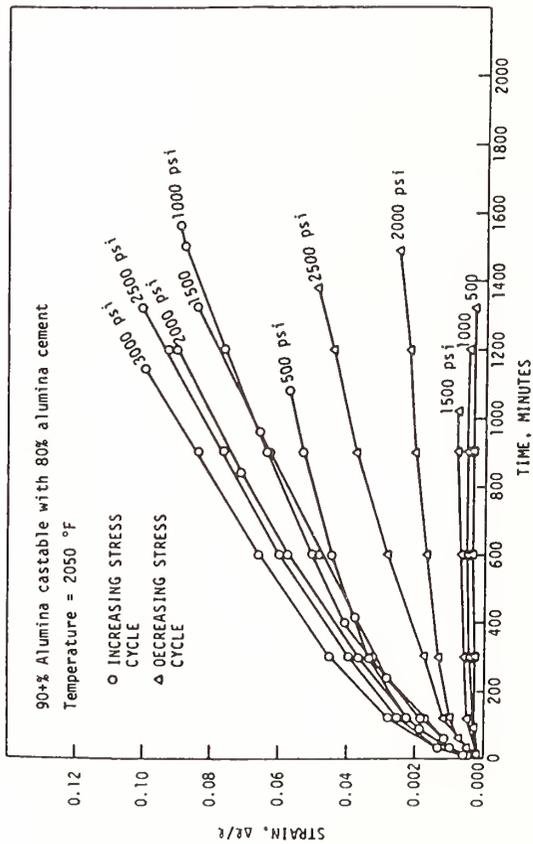
EFFECT OF STRESS AND STRESS CYCLING ON CREEP DATA[a] FOR A HIGH-ALUMINA CASTABLE[b] WITH

VARIOUS CEMENTS[c][97], Continued
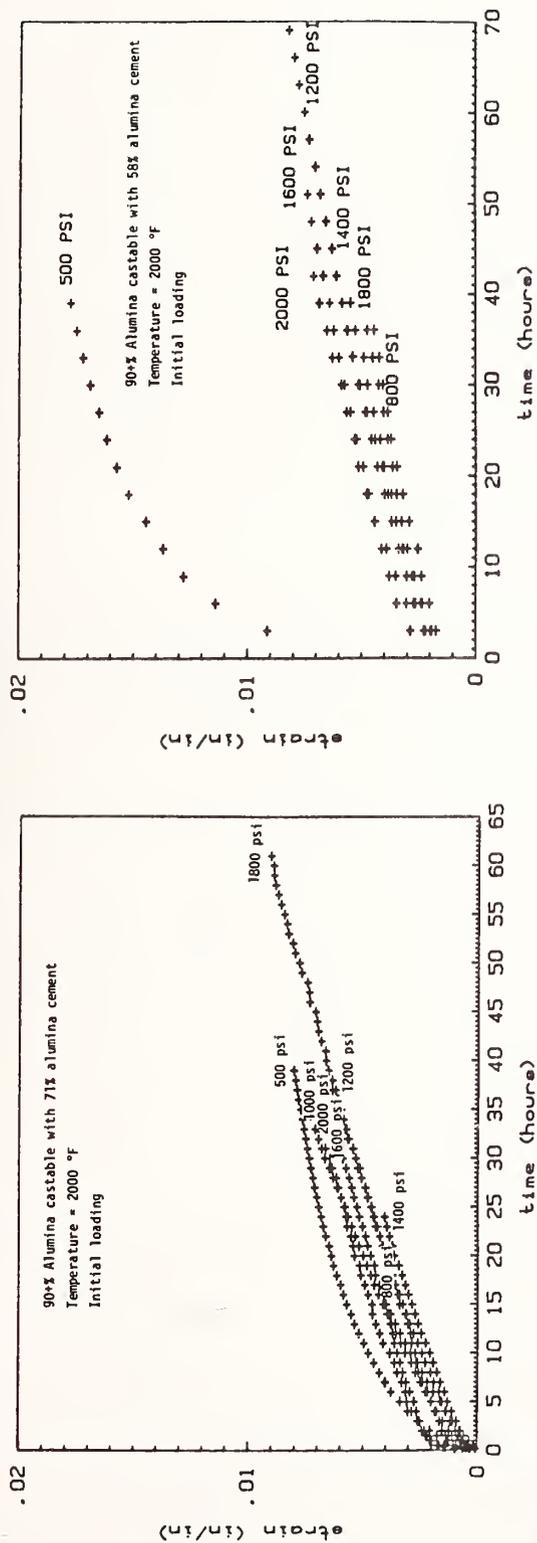
[a]Creep deformation was measured using specimens 3 cm in diameter by 5 cm in length. Specimens were tested in air in creep furnaces under compressive loading. Heat-up periods were 5 hours. Constant temperature tests involved increasing the stress incrementally from 500 psi (3.4 (MPa) to 3000 psi (20.7 MPa) by 500 psi (3.4 MPa) increments. Stress level was cycled down and then up. Creep strain was monitored with a linear voltage displacement transducer until steady state creep was obtained. Constant temperature-variable stress data were found to have a linear dependence on the ln of the stress.

[b]See Section B.3.2.123, footnote b, for the composition of the generic dense castable.

[c]See Section B.3.2.123, footnote c, for the compositions of the cements used.

STRESS DEPENDENCE OF THE CREEP STRAIN RATE[a] FOR A HIGH-ALUMINA CASTABLE[b] WITH VARIOUS CEMENTS[c] [97]

(Data Continued)

STRESS DEPENDENCE OF THE CREEP STRAIN RATE[a] FOR A HIGH-ALUMINA CASTABLE[b] WITH VARIOUS CEMENTS[c][97], Continued

90+% Alumina castable with 39% alumina cement
Temperature = 2000 °F

n = 0.7 (LOADING)

n = 2.9 (UNLOADING)

90+% Alumina castable with 58% alumina cement
Temperature = 2000 °F

n = -0.2 (LOADING)

[a] See Section B.3.2.120 for details of the creep testing. The stress exponent, n, is given on the figures.

[b] A generic dense castable consisting of 70% tabular alumina (-6 mesh + fines) (T-61, Alcoa), 5% calcined alumina (-325 mesh) (A-2, Alcoa), and 25% calcium aluminate cement, with 9.0-9.5% water. Bricks were cast in accordance with ASTM C862-77 and test specimens were core drilled from the bricks. Neat cement specimens were cast to specimen size, with water/cement ratio = 0.3.

[c] See footnote b, Section B.3.2.120, for compositions of the cements used.

STRESS DEPENDENCE OF THE CREEP STRAIN RATE[a] FOR A MEDIUM-ALUMINA CASTABLE[b] WITH VARIOUS CEMENTS[c][97]

(Data Continued)

STRESS DEPENDENCE OF THE CREEP STRAIN RATE[a] FOR A MEDIUM-ALUMINA CASTABLE[b] WITH VARIOUS CEMENTS[c][97], Continued

[a]See Section B.3.2.120 for details of the creep testing. The stress exponent, n, is given on the figures.

[b]A generic dense castable consisting of 75% calcined kaolin (27.5% -6 + 10 mesh, 22.5% -10 + 20 mesh, 20% -20 mesh, 5% ball milled fines 5% of which was <325 mesh) (Mulcoa 47, C-E Minerals) and 25% calcium aluminate cement, with 9.0-9.5% water. Bricks were cast in accordance with ASTM C862-77 and test specimens were core drilled from the bricks.

[c]See footnote b, Section B.3.2.120, for compositions of the cements used.

===

TOTAL CREEP STRAIN[a] AT CONSTANT TEMPERATURE AND AT CONSTANT STRESS FOR
ALUMINA CASTABLES[b] WITH DIFFERENT CEMENTS[c][97]

At Constant Temperature

At Constant Stress

- -

(Data Continued)

==

TOTAL CREEP STRAIN[a] AT CONSTANT TEMPERATURE AND AT CONSTANT STRESS FOR
ALUMINA CASTABLES[b] WITH DIFFERENT CEMENTS[c][97], Continued

At Constant Temperature

( ) = hours
25 = 2500 psi
50+% Alumina castable

80% Al$_2$O$_3$ 81% Al$_2$O$_3$ 71% Al$_2$O$_3$ 58% Al$_2$O$_3$ 39% Al$_2$O$_3$

At Constant Stress

( ) = hours
19 = 1900 °F
50+% Alumina castable

80% Al$_2$O$_3$ 81% Al$_2$O$_3$ 71% Al$_2$O$_3$ 58% Al$_2$O$_3$ 39% Al$_2$O$_3$

- -

(Data Continued)

==

TOTAL CREEP STRAIN[a] AT CONSTANT TEMPERATURE AND AT CONSTANT STRESS FOR
ALUMINA CASTABLES[b] WITH DIFFERENT CEMENTS[c][97], Continued

---

[a]Creep deformation was measured using specimens 3 cm in diameter by 5 cm in
length. Specimens were tested in air in creep furnaces under compressive
loading. Heat-up periods were 5 hours. For constant stress tests, speci-
mens were heated to the first test temperature, the load applied, and de-
formation recorded. The load was then released and the specimen heated
to the next temperature, the same load reapplied and deformation recorded.
Thus the total strain at the higher temperatures included the strain at
lower temperatures. Constant temperature tests involved increasing the
stress incrementally from 500 psi (3.4 MPa) to 3000 psi (20.7 MPa) by 500
psi (3.4 MPa) increments. Stress level was cycled down and then up.
Creep strain was monitored with a linear voltage displacement transducer
until steady state creep was obtained.

[b]Two generic castables were tested, cast as brick in accordance with ASTM
C862-77. Test specimens were cored drilled from the brick. The 90+%
alumina dense castable consisted of 70% tabular alumina (-6 mesh + fines)
(T-61, Alcoa), 5% calcined alumina (-325 mesh) (A-2, Alcoa), and 25% cal-
cium aluminate cement, with 9.0-9.5% water. The 50+% alumina dense castable
consisted of 75% calcined kaolin (27.5% -6 + 10 mesh, 22.5% -10 + 20 mesh,
20% -20 mesh, 5% ball milled fines 5% of which was <325 mesh) (Mulcoa 47,
C-E Minerals) and 25% calcium aluminate cement.

[c]The percent alumina in each cement forms the horizontal axis of the above
figures. The calcium aluminate cements used were--
80% alumina: 79.7% Al_2O_3, 18.4% CaO (CA-25C, Alcoa),
81% alumina: 80.9% Al_2O_3, 17.0% CaO (SECAR 80, Lone Star Lafarge),
71% alumina: 70.6% Al_2O_3, 27.5% CaO (SECAR 71, Lone Star Lafarge),
58% alumina: 58.0% Al_2O_3, 33.5% CaO, 5.6% SiO_2, 1.5% Fe_2O_3 (Refcon, Univer-
              sal Atlas Cement),
39% alumina: 39.0% Al_2O_3, 38.5% CaO, 4.5% SiO_2, 12.0% Fe_2O_3 (FONDU, Lone
              Star Lafarge).

===

COMPARISON OF CREEP STRAIN RATES[a] FOR TWO ALUMINA CASTABLES[b] AND A CALCIUM
ALUMINATE CEMENT[c] AFTER VARIOUS TREATMENTS[d][97]

STRAIN RATE (minutes^{-1})

LN STRAIN RATE (minutes^{-1})

STRAIN RATE (minutes^{-1})

LN STRAIN RATE (minutes^{-1})

- -

(Data Continued)

==

COMPARISON OF CREEP STRAIN RATES[a] FOR TWO ALUMINA CASTABLES[b] AND A CALCIUM
ALUMINATE CEMENT[c] AFTER VARIOUS TREATMENTS[d][97], Continued

---

[a]Creep deformation was measured using specimens 3 cm in diameter by 5 cm in
length. Specimens were tested in air in creep furnaces under compressive
loading. Heat-up periods were 5 hours. Creep strain was monitored with a
linear voltage displacement transducer until steady state creep was obtained.
Compare Sections B.3.2.120 and B.3.2.124.

[b]Two generic dense castables were tested, cast as brick in accordance with
ASTM C862-77. Test specimens were core drilled from the brick. The 90+%
alumina castable consisted of 70% tabular alumina (-6 mesh + fines) (T-61,
Alcoa), 5% calcined alumina (-325 mesh) (A-2, Alcoa), and 25% calcium alu-
minate cement, with 9.0-9.5% water. The 50% alumina castable consisted of
75% calcined kaolin (27.5% -6 + 10 mesh, 22.5% -10 + 20 mesh, 20% -20 mesh,
5% ball milled fines 5% of which was <325 mesh) (Mulcoa 47, C-E Minerals)
and 25% calcium aluminate cement. See footnote c for cement composition.

[c]The cement was 79.7% alumina, 18.4% calcia (CA-25C, Alcoa). Neat cement
specimens were cast to specimen size, with water/cement ratio = 0.3.

[d]For tests where cycling is indicated, the specimens were put through the
procedures more than once, heated up, loaded, load released, cooled, heated,
etc. Specimens designated prefired were fired in air at the stated temper-
ature for the given time prior to creep testing. The specimens exposed to
a gasifier atmosphere prior to creep testing were subjected to the conditions
at the DoE IITRI facility for 1000 hours. [Conditions were probably 982 °C
(1800 °F), 1000 psi, gas composition (vol %): 24 H_2, 18 CO, 12 CO_2, 5 CH_4,
1 NH_3, 0-1.0 H_2S, balance H_2O.]

===

EFFECT OF CEMENT CONTENT[a] ON THE CREEP[b] OF A HIGH-ALUMINA CASTABLE[c][97]

(Data Continued)

EFFECT OF CEMENT CONTENT[a] ON THE CREEP[b] OF A HIGH-ALUMINA CASTABLE[c][97], Continued

(Data Continued)

==

EFFECT OF CEMENT CONTENT[a] ON THE CREEP[b] OF A HIGH-ALUMINA CASTABLE[c][97], Continued

(G)

[a] Figures A and B compare the temperature dependence of the strain rate for the castable prepared with 25% cement and with 12.5% of the same cement.
Figures C and D compare the stress dependence of the strain rate for the castable prepared with 25% and 12.5% cement.
Figures E and F show the dependence of strain rate on the percent of cement in the castable under two different creep test conditions. The castable was prepared with varying amounts of cement, from 7.5% to 32.5%. The 7.5% cement specimen failed on loading.
Figure G shows the total strain to failure as a function of the cement content of the castable.

[b] See Section B.3.2.120, footnote a, for the conditions of the creep testing.

[c] A 90+% generic dense castable, cast as brick in accordance with ASTM C962-77. Test specimens were core drilled from the brick. Composition for 25% cement material: 70% tabular alumina (-6 mesh + fines) (T-61, Alcoa), 5% calcined alumina (-325 mesh) (A-2, Alcoa), and 25% calcium aluminate cement (79.7% alumina, 18.4% calcia; CA-25C, Alcoa). Cement content varied from 7.5% to 32.5% for various samples but details of the adjustment of the amounts of the other components are not given in the original reports.

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EFFECT OF CEMENT COMPOSITION[a] ON THE CREEP[b] OF A HIGH-ALUMINA CASTABLE[c][97]
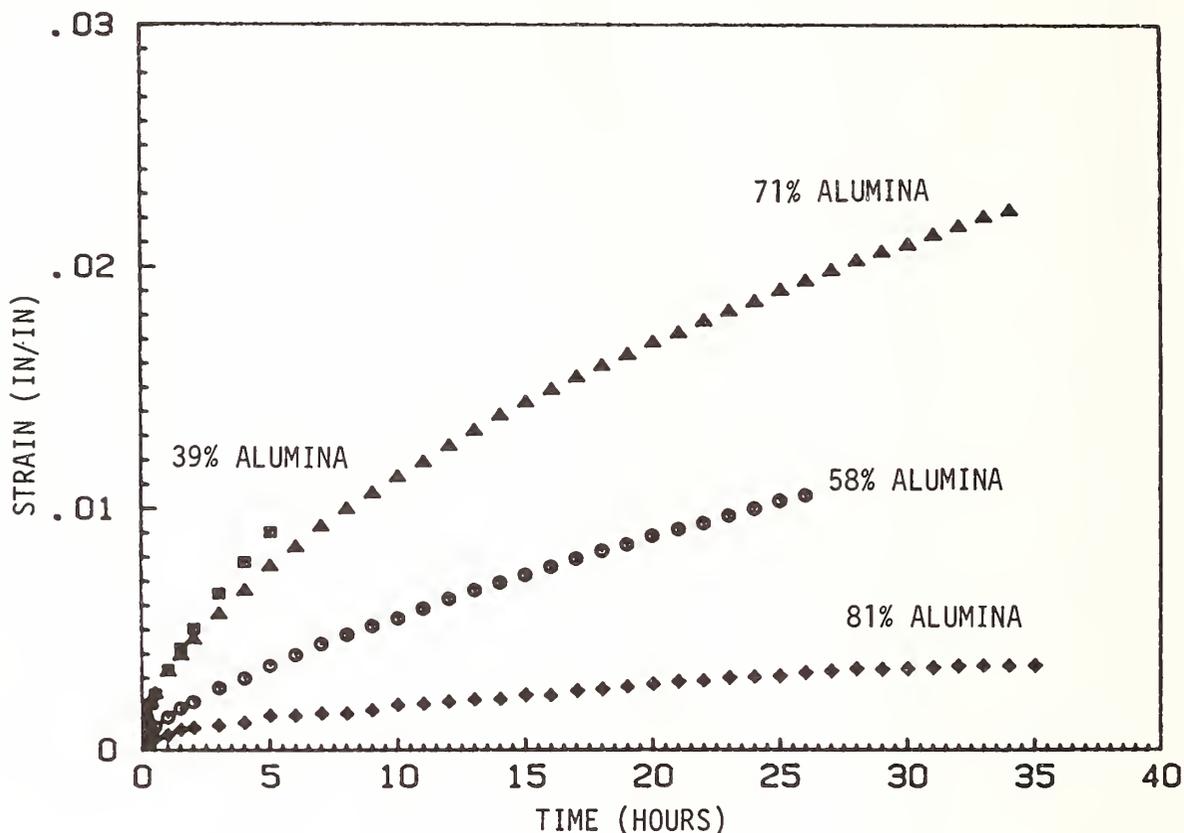
[a]The calcium aluminate cements used were--
  81% alumina: 80.9% Al_2O_3, 17.0% CaO (SECAR 80, Lone Star Lafarge);
  71% alumina: 70.6% Al_2O_3, 27.5% CaO (SECAR 71, Lone Star Lafarge);
  58% alumina: 58.0% Al_2O_3, 33.5% CaO, 5.6% SiO_2, 1.5% Fe_2O_3 (Refcon,
                Universal Atlas Cement);
  39% alumina: 39.0% Al_2O_3, 38.5% CaO, 4.5% SiO_2, 12.0% Fe_2O_3 (FONDU,
                Lone Star Lafarge).

[b]See Sections B.3.2.120, and B.3.2.125 through B.3.2.130 for details of
  creep testing and for other creep data for castables using these cements.

[c]The 90+% generic dense castable consisted of 70% tabular alumina (-6 mesh
  + fines) (T-61, Alcoa), 5% calcined alumina (-325 mesh) (A-2, Alcoa), and
  25% cement, with 9.0-9.5% water. Materials were cast as brick in
  accordance with ASTM C862-77 and specimens were core drilled from the
  bricks.

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CREEP[a] OF THE CALCIUM ALUMINATE BONDING PHASE[b] OF A HIGH-ALUMINA CASTABLE[c][97]
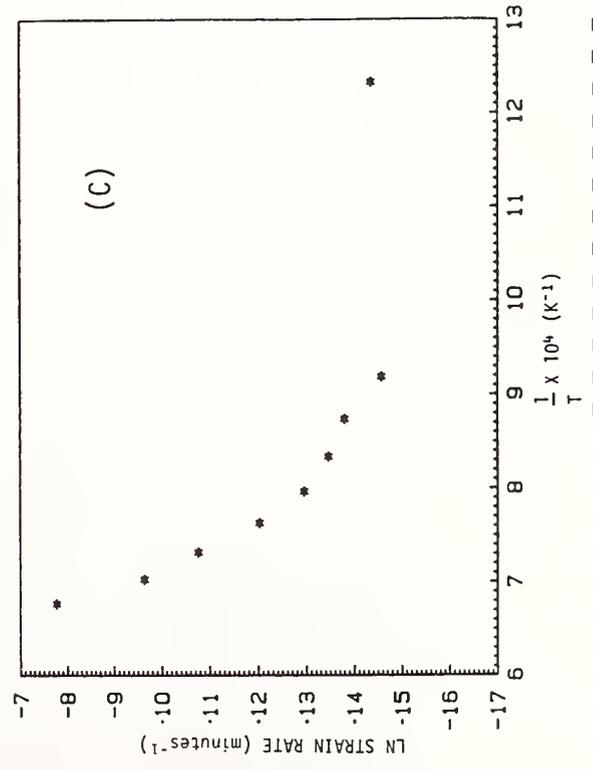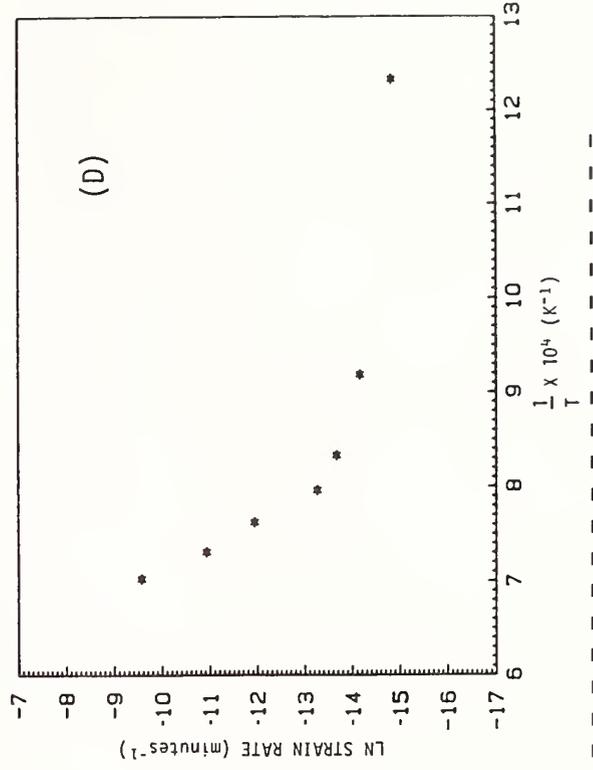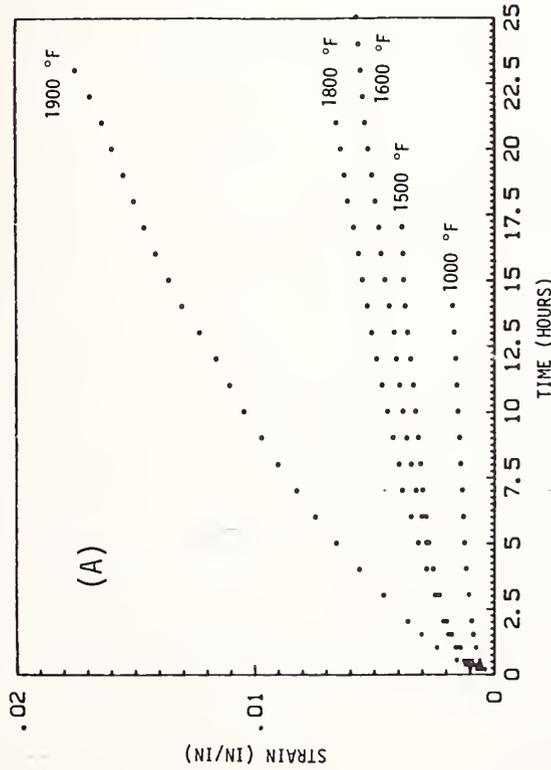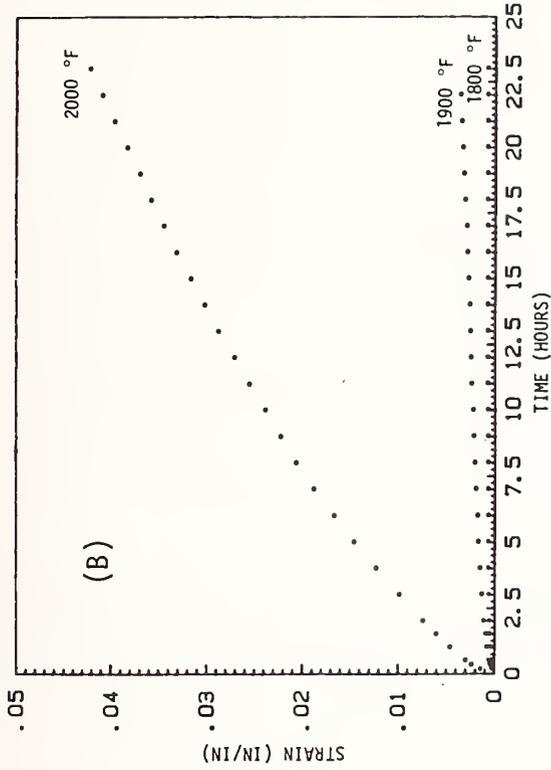
(Data Continued)

CREEP[a] OF THE CALCIUM ALUMINATE BONDING PHASE[b] OF A HIGH-ALUMINA CASTABLE[c][97], Continued

[a]See Section B.3.2.120, footnote a, for details of the creep testing.
Figure A shows "typical results" of creep testing at a stress of 2000 psi on initial loading at the given temperatures for specimens with the water/cement ratio varying from 0.28 to 0.34.
Figure B shows "typical results" of creep testing at a stress of 2000 psi after first testing at 2000 °F for the same material with water/cement ratio varying from 0.28 to 0.34.
Figure C shows the temperature dependence of the strain rate at 2000 psi based on 12 specimens of varying water/cement ratios (0.28 to 0.34). The activation energy for creep based on the six high temperature points is 78 kcal/mole.
Figure D shows the temperature dependence of the strain rate at 2000 psi for material with a water/cement ratio = 0.28. The activation energy from the slope at high temperature is 63 kcal/mole.

[b]Specimens were cast of fine calcined alumina aggregate (-325 mesh) (A-2, Alcoa) and calcium aluminate cement (79.7% alumina, 18.4% calcia) (CA-25C, Alcoa). The cement/aggregate ratio was 5:1 by weight and the water/cement ratios varied from 0.28 to 0.34 in increments of 0.01.

[c]The castable is the 90+% generic dense refractory used in the testing reported in Sections B.3.2.120 through B.3.2.133.

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CREEP DATA[a] FOR SOME CHROMIA-CONTAINING REFRACTORIES[b][97]

(Data Continued)

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CREEP DATA[a] FOR SOME CHROMIA-CONTAINING REFRACTORIES[b][97], Continued

(E)

78% Chromia-18% MgO (CR 78-12 in Section B.4.2.41)
Constant stress = 1000 psi

Total strain before failure at 2300 °F = 0.016 in/in.

(F)

78% Chromia-18% Magnesia
(CR 78-12 in Section B.4.2.41)
Constant stress = 1000 psi

Failed at 2300 °F

(G)

78% Chromia-18% Magnesia (CR 78-12 in Section B.4.2.41)
Constant temperature = 2600 °F (1427 °C)

(H)

60% Chromia-20% Alumina-12% Zirconia-7% Silica
Constant stress = 500 psi

(Data Continued)

===

CREEP DATA[a] FOR SOME CHROMIA-CONTAINING REFRACTORIES[b][97], Continued

(Data Continued)

CREEP DATA[a] FOR SOME CHROMIA-CONTAINING REFRACTORIES[b][97], Continued

(M) 90% Alumina-10% Chromia (CR 10-3 in Section B.4.2.41)
Constant temperature = 2460 °F (1349 °C)

(N) 90% Alumina-10% Chromia
Constant stress = 300 psi
Total strain before failure at 2500 °F = 0.029 in/in.

(O) 90% Alumina-10% Chromia
Constant temperature = 2460 °F (1350 °C)
Total creep strain = 0.0451 in/in.

(P) 90% Alumina-10% Chromia
Constant temperature = 2200 °F (1200 °C)
Creep strain at 500 psi = 0.0054 in/in.
Total creep strain = 0.0321 in/in.
Specimen failed at 1200 psi after 82 hours under load.

(Data Continued)

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CREEP DATA[a] FOR SOME CHROMIA-CONTAINING REFRACTORIES[b][97], Continued

Footnotes

[a]All creep tests were performed in air under compressive loading. Creep tests were conducted under two sets of conditions: cycled temperature with constant applied stress and constant temperature with cycled stress. Deformation was recorded for about 10 hours after reaching steady state conditions. All results are based on steady state creep.

[b]Compositions of the refractories are given on the figures. Neither brand names nor other characterization were given in the original reports. Specimens were prepared by core drilling cylinders ∿1 in. in diameter which were sectioned to lengths of ∿2 in. with the parallel faces perpendicular to the axis of the cylinder.

Figures A and B are for the same material. The material is probably CR 80-17 in Section B.4.2.41, a fine-grained high-purity spinel.

Figures C and D are for the same material. It is unclear from the original reports whether this material is included among those reported in Section B.4.2.41. It is described as a high-purity magnesia-chromia spinel.

Figures E, F, and G are apparently for the same material. The original reports are unclear.

Figures H, I, J and K are for the same material described as a coarse rebonded refractory that is an inhomogeneous mixture of chromia, zirconia, and alumina.

Figures L and M are for the same material.

Figures N, O, and P are for the same material.

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STRESS-STRAIN DATA[a] FOR TWO DENSE ALUMINA CASTABLES[99]

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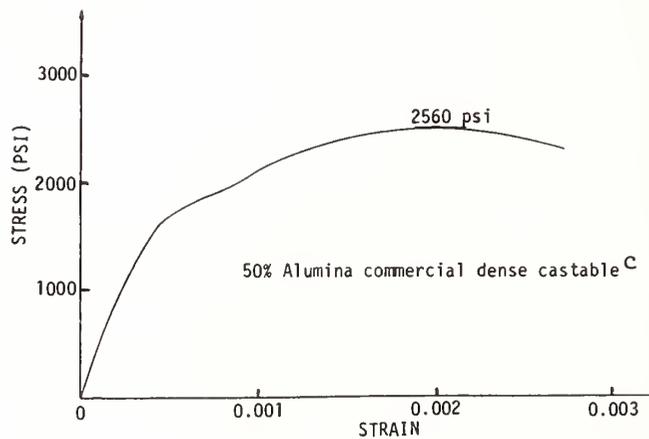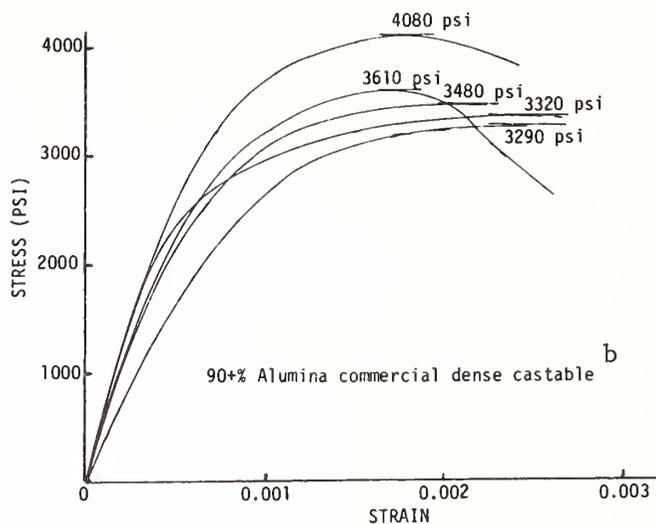
[a]Specimens were tested under uniaxial compression.

[b]A Babcock and Wilcox material, no other information given.

[c]Mix 36-C, a Babcock and Wilcox refractory.

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BIAXIAL STRESS-STRAIN DATA[a] FOR TWO ALUMINA CASTABLES[b][99]
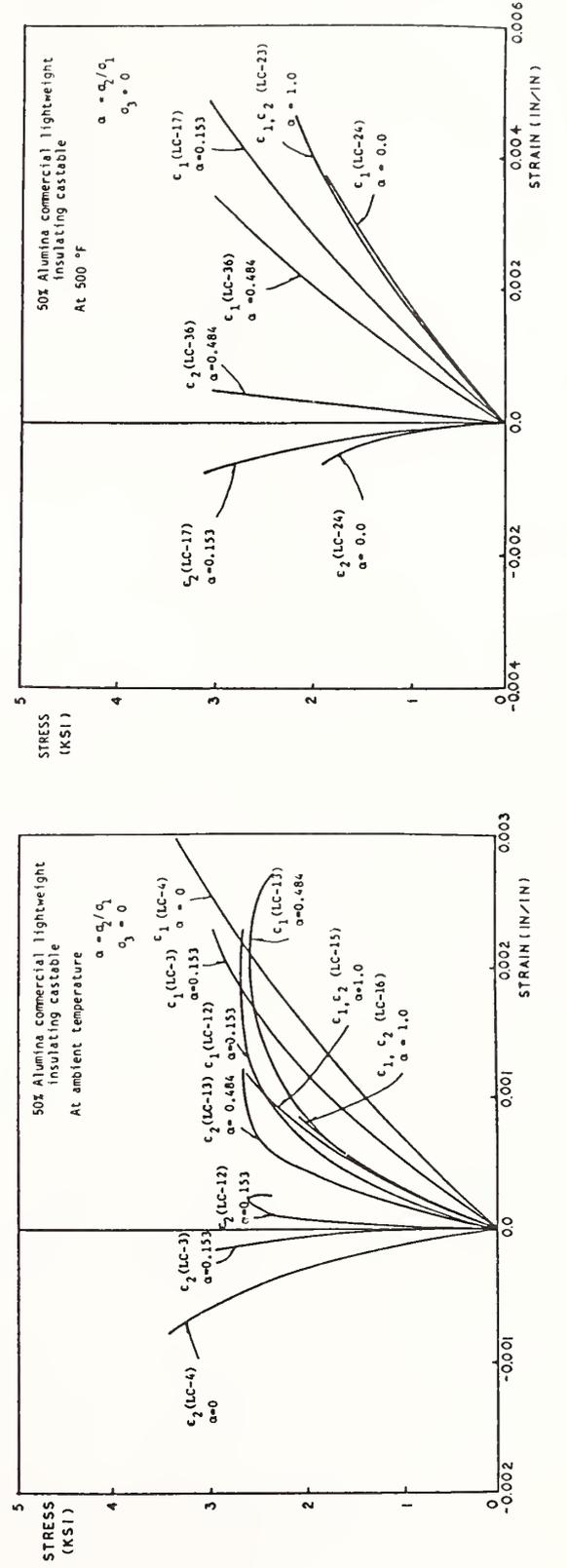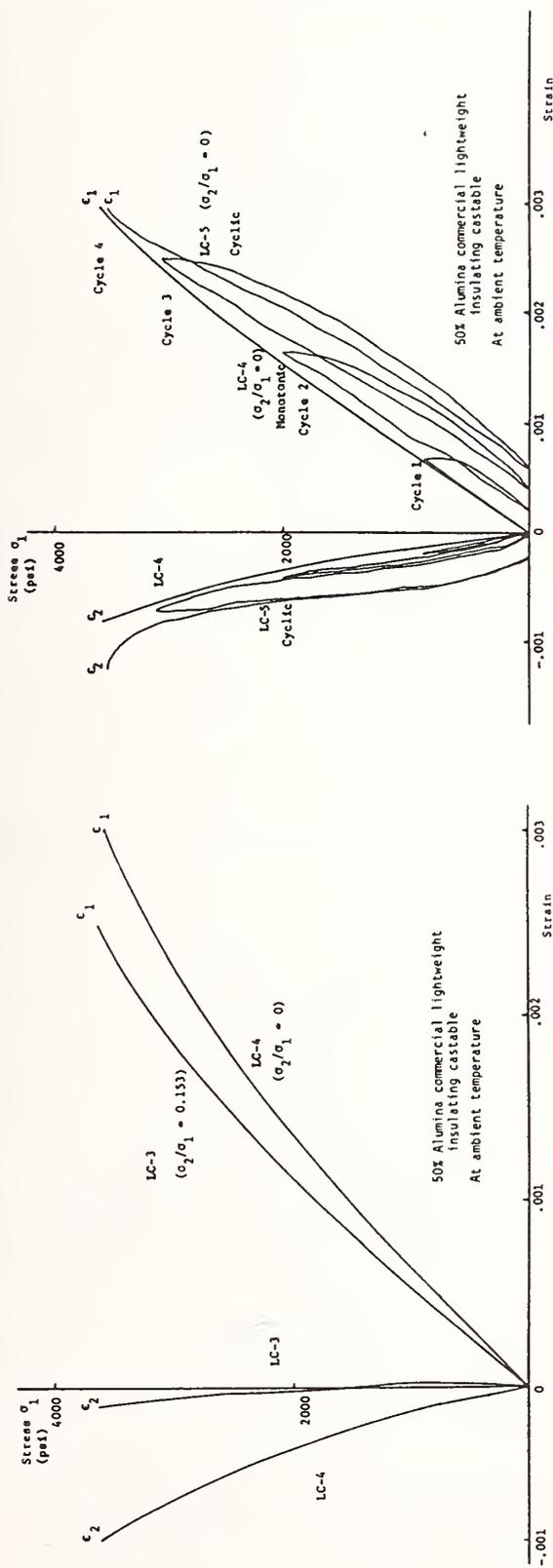
(Data Continued)

BIAXIAL STRESS-STRAIN DATA[a] FOR TWO ALUMINA CASTABLES[b][99], Continued
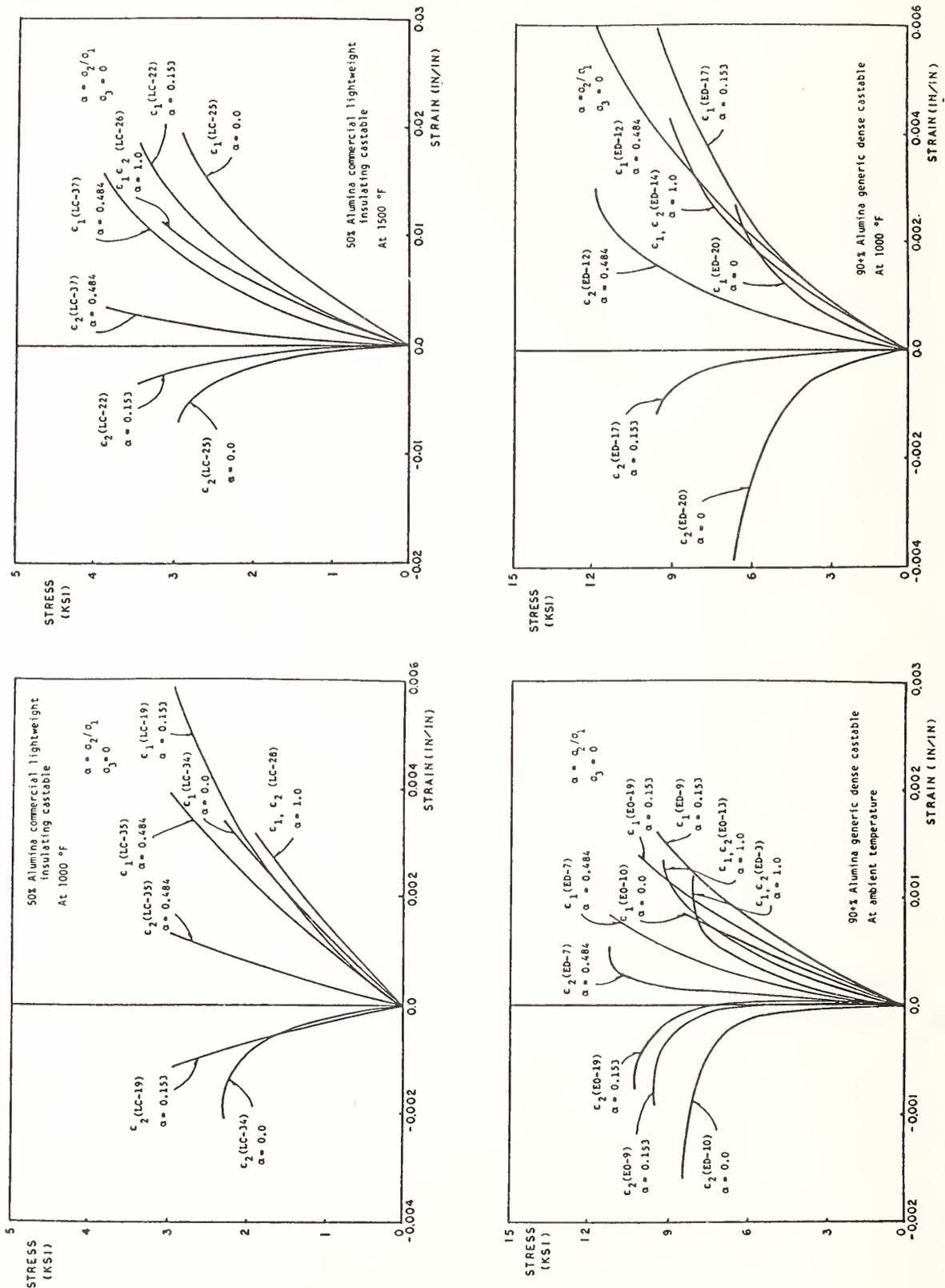
(Data Continued)

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BIAXIAL STRESS-STRAIN DATA[a] FOR TWO ALUMINA CASTABLES[b][99], Continued
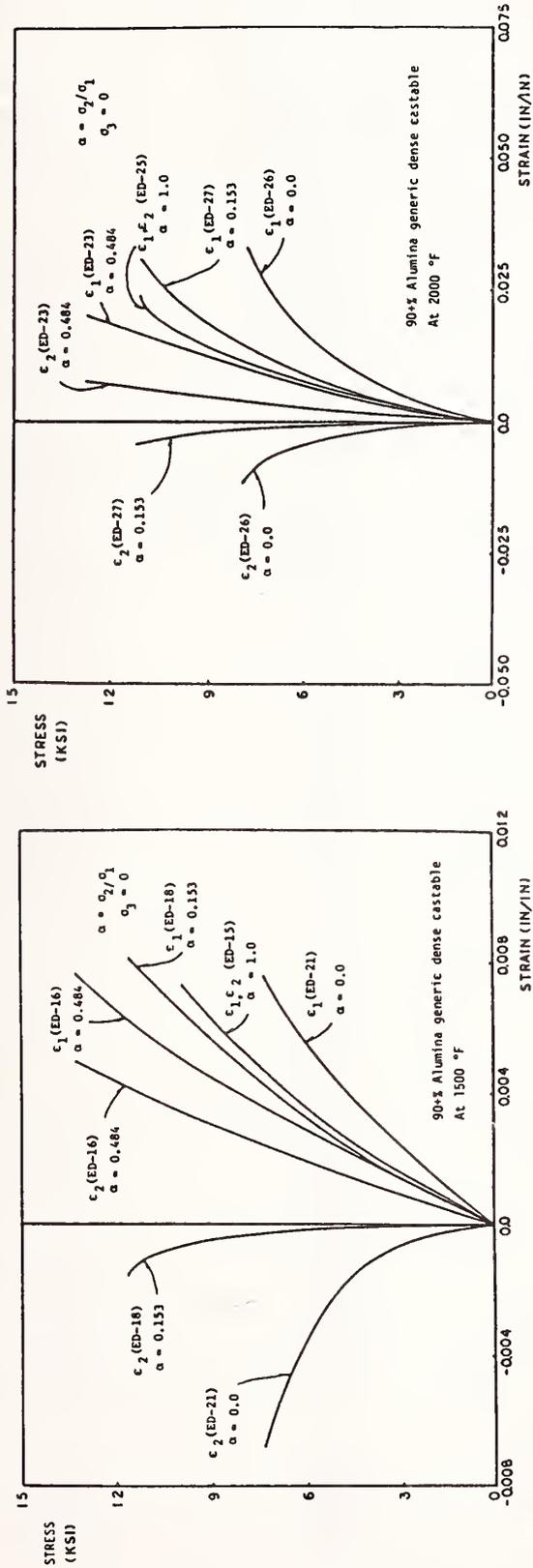
^{a}Data collected with flat square specimens in biaxial compression. Compressive stresses were applied to specimens in two perpendicular principal directions. Strain was detected by attaching slide rods to pins cast into the specimens and measuring the movement of the slide rods by extensometers. On the figures, ε_1 is the strain for the main loading direction, ε_2 is the strain for the lateral stress confinement direction. The stress ratio, $\sigma_2/\sigma_1 = \alpha$, is kept constant throughout a given test. The stress ratios used were 0 (uniaxial stress), 0.153, 0.484, and 1.00 (100% biaxial loading). The labels, LC-3, LC-36, ED-12, ED-25, etc., are specific specimen identification codes used by the original investigators.

^{b}The 50% alumina refractory is Litecast 75-28 (General Refractories); 21% casting water; 5 x 5 x 1 in. specimens used. The 90+% castable is DOE (ERDA) 90 consisting of 70% tabular alumina (-6 mesh + fines; T-61, Alcoa), 5% calcined alumina (325 mesh; A-2, Alcoa), 25% calcium aluminate cement (CA-25C, Alcoa); 8% casting water; 5 x 5 x 3/4 in. specimens. Materials were cast in molds and vibrated into place. Pins cut from high-purity 1/8-in. diameter Al thermocouple insulation were cast in samples for strain detection. Cast samples were cured first in sealed plastic bags overnight, then in air at 225 °F overnight. Samples were then heated and held at temperature for a minimum of 1 hour, the 90% alumina material at 2000 °F and the 50% alumina castable at 1500 °F.

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BIAXIAL COMPRESSION TEST DATA[a] FOR TWO ALUMINA CASTABLES[b][99]

Specimen Code[c]	Test Mode[d]	Temperature °F	Stress Ratio σ_2/σ_1	Maximum Stress in Main Loading Direction σ_1 (max) (psi)	Peak Strain in Main Loading Direction ε_1 (peak)
50% ALUMINA COMMERCIAL LIGHTWEIGHT INSULATING CASTABLE - - - - - - - - - - -					
LC-4	M	ambient	0.0	3200	0.0030
LC-5	C	ambient	0.0	3130	0.0028
LC-11	C	ambient	0.0	2840	0.0017
LC-2	M	ambient	0.153	3020	0.0019
LC-3	M	ambient	0.153	3220	0.0023
LC-12	M	ambient	0.153	2600	0.0023
LC-13	M	ambient	0.484	2660	0.0027
LC-15	M	ambient	1.0	2570	0.0023
LC-16	M	ambient	1.0	2070	0.0009
LC-24	M	500	0.0	1880	0.0036
LC-17	M	500	0.153	3050	0.0049
LC-36	M	500	0.484	3020	0.0034
LC-23	M	500	1.0	2140	0.0046
LC-34	M	1000	0.0	2240	0.0033
LC-19	M	1000	0.153	2930	0.0059
LC-35	M	1000	0.484	2960	0.0040
LC-28	M	1000	1.0	1860	0.0031
LC-25	M	1500	0.0	2900	0.0194
LC-22	M	1500	0.153	3480	0.0185
LC-37	M	1500	0.484	3850	0.0157
LC-26	M	1500	1.0	3380	0.0113
90+% ALUMINA GENERIC DENSE CASTABLE - - - - - - - - - - - - - - - - - - -					
ED-10	M	ambient	0.0	8600	0.0008
ED-22	M	ambient	0.0	6470	--
ED-11	C	ambient	0.0	9140	--
ED-9	M	ambient	0.153	9700	0.0016
ED-19	M	ambient	0.153	10200	0.0014
ED-7	M	ambient	0.484	11500	0.0008
ED-3	M	ambient	1.0	8170	0.0011
ED-24	M	ambient	1.0	5800	0.0006
ED-20	M	1000	0.0	6620	0.0027
ED-17	M	1000	0.153	9530	0.0059
ED-12	M	1000	0.484	11940	0.0060
ED-13	M	1000	1.0	9300	0.0013
ED-14	M	1000	1.0	9230	0.0043
ED-21	M	1500	0.0	7430	0.0075
ED-18	M	1500	0.153	11470	0.0081
ED-16	M	1500	0.484	13080	0.0076
ED-15	M	1500	1.0	10030	0.0072

- -

(Table Continued)

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BIAXIAL COMPRESSION TEST DATA[a] FOR TWO ALUMINA CASTABLES[b][99], Continued

Specimen Code[c]	Test Mode[d]	Temperature °F	Stress Ratio σ_2/σ_1	Maximum Stress in Main Loading Direction σ_1 (max) (psi)	Peak Strain in Main Loading Direction ε_1 (peak)
ED-26	M	2000	0.0	7710	0.033
ED-27	M	2000	0.153	11050	0.031
ED-23	M	2000	0.484	12650	0.020
ED-25	M	2000	1.0	11010	0.024

[a] See Section B.3.2.137 for the stress-strain figures corresponding to these data. See also footnote a of the same section for test information.

[b] See Section B.3.2.137, footnote b, for composition and sample preparation.

[c] Specific specimen identification codes used by the original investigators. The same codes are used on the figures in Section B.3.2.137.

[d] M = monotonic testing, C = cyclic testing.

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FAILURE ENVELOPE FOR TWO ALUMINA CASTABLES IN BIAXIAL COMPRESSION TESTS[a][99]
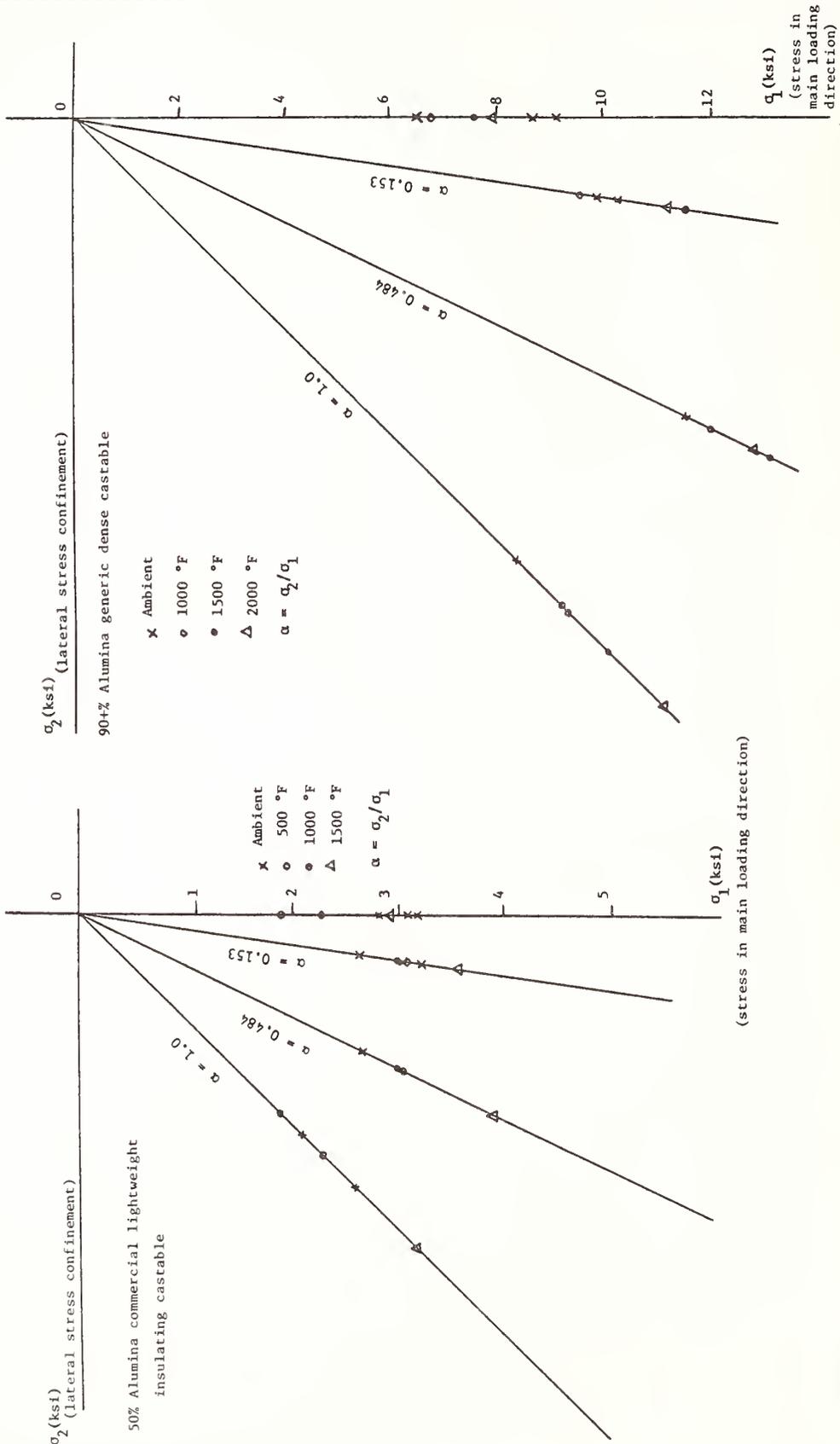
[a]See Sections B.3.2.137 and B.3.2.138 for the data plotted here.

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INITIAL ELASTIC MODULUS[a] OF TWO ALUMINA CASTABLES[b] AS A FUNCTION OF TEMPERATURE[99]

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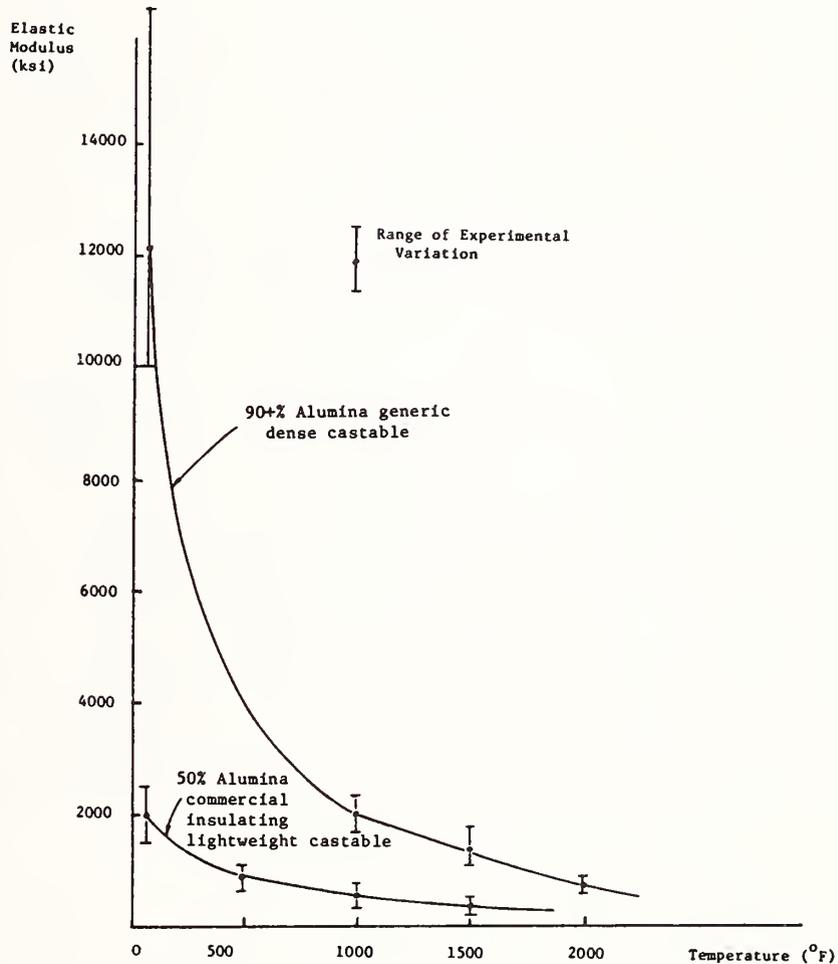
[a]See Section B.3.2.137 for biaxial stress-strain curves for the castables. The initial elastic modulus was drawn from the initial slope of the stress-strain curves.

[b]See footnote b, Section B.3.2.137, for composition and sample preparation.

HARDNESS AND FRACTURE TOUGHNESS DATA[a] FOR SELECTED CERMET MATERIALS SUBJECTED TO COAL SLURRY EROSION TESTING[b][103]

Material	Composition	Source	Hardness (GPa)		Fracture Toughness K_{IC} (MPa√m)	
			10 kg	20 kg	10 kg	20 kg
WC	WC-0.75 Ni	Kennametal (WCX)	12.99	13.09	c	c
WC/TiC	WC-0.5 Co, 3 TaC, 20 TiC, 0.1 NbC	Kennametal (SP-278)	18.37	16.91	d	d
WC-Co	WC-1.7 Co, 10 TaC, 0.2 TiC, 0.1 NbC	Kennametal (K-602)	20.01	20.46	4.76	5.20
WC-Co	WC-6 Co, 0.5 (TaC, TiC, NbC)	Kennametal (K-6T)	12.29	12.34	c	c
WC-Co	not given	Kennametal (SPZ-313)	18.81	18.56	d	d
WC-Co	WC-2.8 Co	Kennametal (K-11)	17.46	18.74	8.96	8.02
WC-Co	WC-5.8 Co	Kennametal (K-68)	17.48	17.71	11.24	10.62
WC-Co	WC-7.8 Co	Kennametal (K-3406)	13.36	13.09	17.28	18.46
WC-Co	WC-9.5 Co	Kennametal (K-3560)	10.49	10.74	c	c
WC-Co	WC-12.2 Co	Kennametal (K-3109)	11.64	11.58	c	c
WC-Co	WC-3 Co	GTE (WA-4)	17.89	17.70	7.86	7.58
WC-Co	WC-4.3 Co	GTE (WA-3)	17.03	16.53	10.10	8.70
WC-Co	WC-6 Co	GTE (WA-2)	15.72	16.25	11.09	11.47
WC-Co	WC-9 Co	GTE (WA-41)	13.89	13.50	15.66	15.53
WC-Co	submicron carbide	GTE (WA-110)	15.17	15.14	d	d
WC-Co,Cr	WC-6 Co, 0.9 Cr, 4.2 TaC, 4 TiC, 0.9 NbC	Kennametal (K-714)	16.49	16.53	13.29	12.74
WC-Co,Cr	WC-8.5 Co, 4.5 Cr	Kennametal (K-701)	15.98	16.16	7.47	12.74
WC-Co,Cr	WC-6 Co, 1.0 Cr	Kennametal (K-703)	17.04	16.91	10.98	9.46
WC-Co-Cr	WC-6 Co, 1.0 Cr	Kennametal (K-703)	17.03	16.53	11.22	9.92
TiB₂-Ni	not given	ORNL	26.19	24.38	d	d
Al₂O₃	hot isostatically pressed	Battelle	21.87	-	d	-

[a] No details given for measurement procedures. Test results are given for two different loads, 10 and 20 kg. Values for fracture toughness are given only where elastic moduli are known.

[b] See Sections B.2.2.30 and B.2.2.31 for erosion test data.

[c] No cracks observed around indentations.

[d] Elastic modulus value unknown.

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DEPENDENCE OF FRACTURE TOUGHNESS ON BINDER CONTENT OF CEMENTED
TUNGSTEN CARBIDES[a][103]
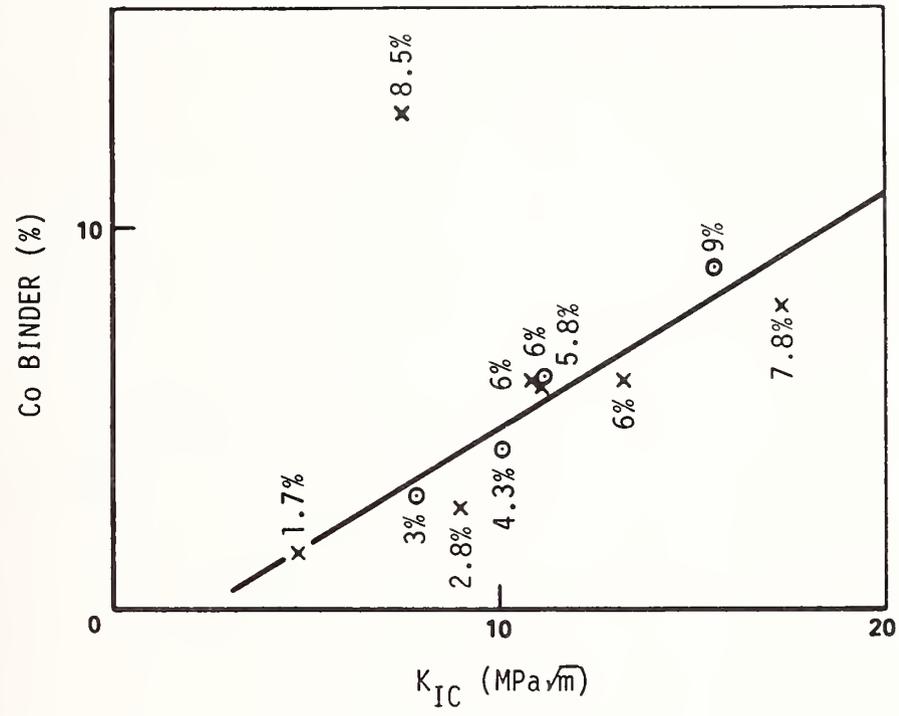
^{a}See Section B.3.2.141 for the data plotted here.

MECHANICAL PROPERTIES[a] FOR VARIOUS MATERIALS SUBJECTED TO SILICA SLURRY EROSION TESTING[b][103]

Material	Source	Young's Modulus E (GPa)	Hardness H (GPa)	Fracture Toughness K_{IC} (MPa√m)
WC-10% Co-4% Cr	Kennametal (K701)	533	16.2	7.8
B₄C, hot-pressed	Norton (Norbide)	448	31.0	---
SiC, sintered	Carborundum (α-SiC)	406	26.1	2.7
SiC, hot-pressed	Norton (NC-203)	440	22.7	4.0
SiC, hot-pressed	Ceradyne	---	20.7	5.3
Al₂O₃, sintered	3M Co. (Alsimag 614)	316	9.4	4.0
Al₂O₃, hot-pressed	Avco	413	17.7	2.7
AlN, hot-pressed	Battelle	---	10.9	2.9
Si₃N₄, hot-pressed	Norton (NC-i32)	310	15.9	4.2
Glass-ceramic	Corning (Pyroceram 9606)	116	6.6	2.4
Soda-lime glass	PPG Ind. (Float glass)	72	5.2	0.75

[a]Assessed by Vickers indentation. K_{IC} calculated from Vickers indentation crack lengths.

[b]See Section B.2.2.36 for erosion test data.

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HARDNESS AND FRACTURE TOUGHNESS DATA[a] FOR CEMENTED TUNGSTEN CARBIDES[b] AS A FUNCTION OF THE COBALT BINDER CONTENT[103]

Cobalt Binder Volume Fraction	Hardness (GPa)	Fracture Toughness K_{IC} (MPa$\sqrt{m}$)
0.051	16.72	9.4
0.076	16.33	9.3
0.101	14.93	9.9
0.148	13.63	11.3
0.171	12.19	13.9
0.193	12.16	13.8
0.208	11.81	15.3
0.236	11.50	13.0
0.306	10.35	16.1
0.369	9.75	17.9

[a]No details are given for measurement procedures.

[b]These materials were subjected to erosion testing with silica slurry, see Section B.2.2.37.

THERMAL SHOCK ESTIMATES[a] BASED ON PHYSICAL AND MECHANICAL PROPERTIES OF REFRACTORIES FOR SLAGGING GASIFIERS[b][100]

Refractory Composition[b]	σ_f Tensile Strength MPa	E Young's Modulus 10^5 MPa	α, Coefficient of Expansion 10^{-6} °C^{-1}	Poisson's Ratio ν	γ Work of Fracture J/m^2	Calculated Damage Resistance Parameters[a] R'''' 10^{-2} m	R_{st} $\sqrt{m}$ °C
89.7 Al$_2$O$_3$–10.0 Cr$_2$O$_3$–0.1 Fe$_2$O$_3$– 0.1 SiO$_2$, solid solution	14.8	1.15	8.0	0.15	40.81	2.53	2.35
89.7 Al$_2$O$_3$–10.0 Cr$_2$O$_3$–0.1 Fe$_2$O$_3$–0.1 SiO$_2$, solid solution	30.6	1.65	8.0	0.14	45.74	0.94	2.08
79.7 Cr$_2$O$_3$– 4.7 Al$_2$O$_3$–6.1 Fe$_2$O$_3$–8.1 MgO–1.3 SiO$_2$, direct bond	31.1	1.90	7.5	0.33	49.48	1.45	2.15
79.0 Cr$_2$O$_3$–6.0 Al$_2$O$_3$–5.0 Fe$_2$O$_3$–5.0 MgO–3.0 SiO$_2$–0.2 CaO–1.6 TiO$_2$, Si-Ti-rich glass	115.9	2.23	7.4	0.26	NA[c]	--	--
78.0 Cr$_2$O$_3$–1.0 Al$_2$O$_3$–1.5 Fe$_2$O$_3$–18.0 MgO–0.5 SiO$_2$–0.5 CaO, direct bond	7.3	1.08	7.2	0.25[d]	15.90	4.32	1.73
60.0 Cr$_2$O$_3$–20.0 Al$_2$O$_3$–0.1 Fe$_2$O$_3$–0.1 MgO–12.0 ZrO$_2$–6.5 SiO$_2$–0.1 CaO–0.7 TiO$_2$, Si-rich glass	30.0	1.45	7.8	0.23	32.81	0.69	1.93
72.8 Cr$_2$O$_3$–0.4 Al$_2$O$_3$–0.6 Fe$_2$O$_3$–25.5 MgO–0.6 SiO$_2$–0.1 TiO$_2$, direct bond	11.3	1.02	7.0	0.31	16.06	1.85	1.79
76.0 Cr$_2$O$_3$–9.0 Al$_2$O$_3$–10.5 SiO$_2$–3.0 TiO$_2$, Si-Al-rich glass	27.6	1.14	7.4	0.25	NA[c]	--	--

[a] Quantities assessing thermal shock resistance of refractories are defined as R'''' = $E\gamma/(1-\nu)\sigma_f^2$, and

(Table Continued)

THERMAL SHOCK ESTIMATES[a] BASED ON PHYSICAL AND MECHANICAL PROPERTIES OF REFRACTORIES FOR SLAGGING GASIFIERS[b][100], Continued

Footnotes continued

$R_{st} = (\gamma/\alpha^2 E)^{1/2}$. R'''' is intended to be proportional to the inverse of the crack area per unit volume propagated by thermal shock (for both stable and unstable crack growth). R_{st} is related to the maximum allowable temperature difference required to propagate long cracks under severe thermal stress conditions (for stable crack growth).

[b]A number of the refractories in this table were tested in a slag corrosion testing program conducted at the same laboratory (see Section B.1.2.26).

[c]NA = not available.

[d]Assumed value.

THERMAL SHOCK DATA[a] FOR REFRACTORIES FOR SLAGGING GASIFIERS[100]
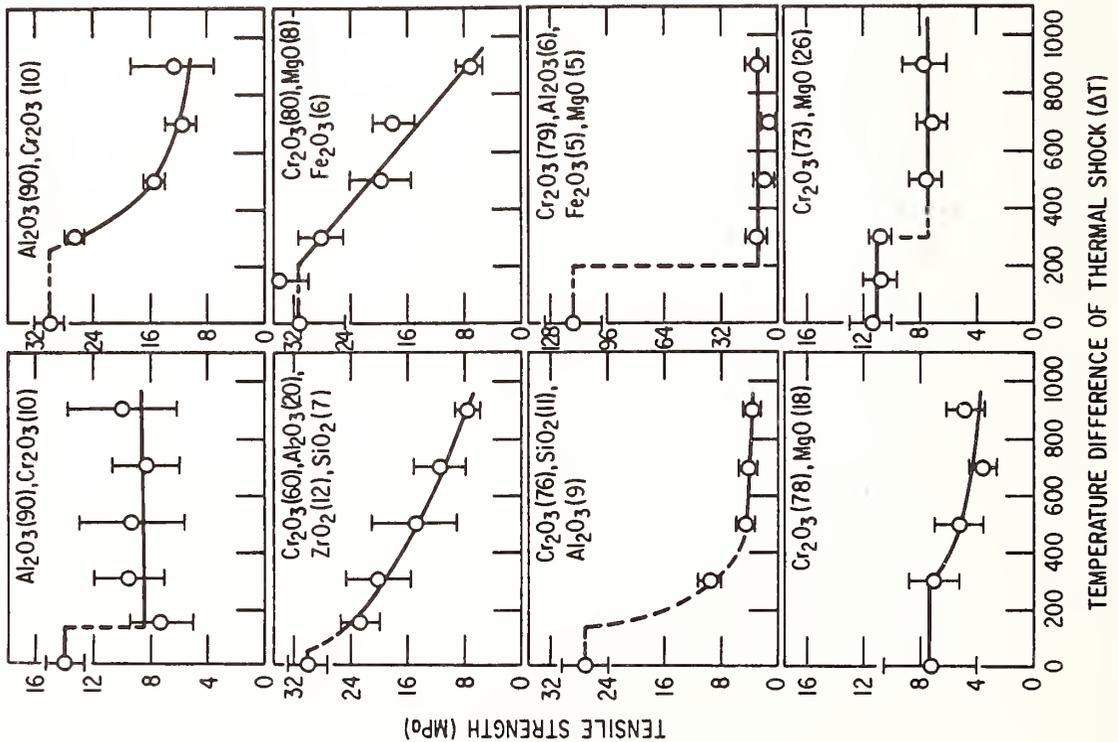
[a]Specimens were thermally shocked by quenching from various temperatures into boiling water at 100 °C. Specimens were 114.3 x 12.7 x 12.7 mm [incorrectly given as cm in the original reports] (4.5 x 0.5 x 0.5 in.) and were arranged in bundles so that upon quenching they received cooling from one side only. Tensile strengths were measured after quenching for comparison with as-received values. The data are for single quenches. Each data point is the average of at least four tests for each temperature difference (ΔT). The data should be compared with the idealized figure given here.

REGION I – Constant Strength No Fracture Nucleation

REGION II – Instantaneous Decrease in Strength Crack Propagation

REGION III – Constant Strength Cracks Subcritical

REGION IV – Gradual Decrease in Strength

ΔT_c (Critical Temperature Difference)

FRACTURE STRENGTH

TEMPERATURE DIFFERENCE OF THERMAL SHOCK (ΔT)
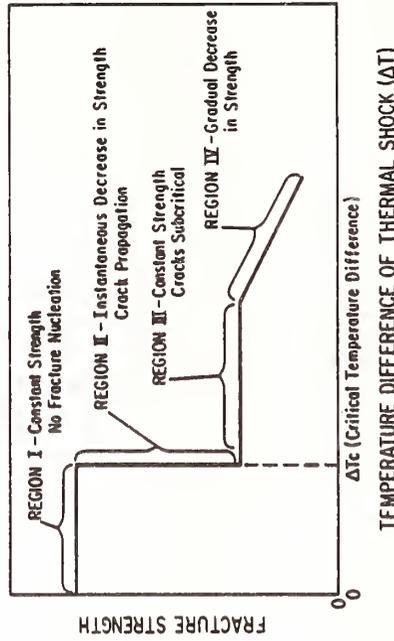
Until the critical difference between the temperature of heating and the temperature of quenching is reached no cracking occurs. When the critical difference (ΔT_c) is reached, cracks are initiated and grow instantaneously.

THERMAL SHOCK DATA FOR REFRACTORIES FOR SLAGGING GASIFIERS

TENSILE STRENGTH (MPa)

TEMPERATURE DIFFERENCE OF THERMAL SHOCK (ΔT)

EFFECT OF MULTIPLE THERMAL SHOCKS[a] ON THE TENSILE STRENGTH[b] OF REFRACTORIES[c] FOR SLAGGING GASIFIERS [100]

Refractory Composition[c]	As-received Tensile Strength MPa	After Thermal Shock[a]					
		One Cycle		Three Cycles		Five Cycles	
		Tensile Strength MPa	Retained Strength %	Tensile Strength MPa	Retained Strength %	Tensile Strength MPa	Retained Strength %
89.7 Al_2O_3-10.0 Cr_2O_3-0.1 Fe_2O_3-0.1 SiO_2, solid solution	14.8±1.3	10.0	67.8	7.2	48.7	5.9	40.2
89.7 Al_2O_3-10.0 Cr_2O_3-0.1 Fe_2O_3-0.1 SiO_2, solid solution	30.6±2.0	12.6	41.1	7.8	25.4	6.2	20.4
79.7 Cr_2O_3-4.7 Al_2O_3-6.1 Fe_2O_3-8.1 MgO-1.3 SiO_2, direct bond	31.1±6.8	6.9	22.4	3.6	11.6	1.7	5.6
79.0 Cr_2O_3-6.0 Al_2O_3-5.0 Fe_2O_3-5.0 MgO-3.0 SiO_2-0.2 CaO-1.6 TiO_2, Si-Ti-rich glass	115.9±15.1	11.3	9.7	3.1	2.7	1.7	1.5
78.0 Cr_2O_3-1.0 Al_2O_3-1.5 Fe_2O_3-18.0 MgO-0.5 SiO_2-0.5 CaO, direct bond	7.3±3.0	5.0	68.3	2.5	34.8	1.8	24.6
60.0 Cr_2O_3-20.0 Al_2O_3-0.1 Fe_2O_3-0.1 MgO-12.0 ZrO_2-6.5 SiO_2-0.1 CaO-0.7 TiO_2, Si-rich glass	30.0±3.4	7.7	25.8	6.8	22.7	2.2	7.5
72.8 Cr_2O_3-0.4 Al_2O_3-0.6 Fe_2O_3-25.5 MgO-0.6 SiO_2-0.1 TiO_2, direct bond	11.3±1.3	7.7	67.6	5.2	46.4	2.9	25.5
76.0 Cr_2O_3-9.0 Al_2O_3-10.5 SiO_2-3.0 TiO_2, Si-Al-rich glass	27.6±3.1	3.6	13.5	2.2	8.0	2.0	7.4

(Table Continued)

EFFECT OF MULTIPLE THERMAL SHOCKS[a] ON THE TENSILE STRENGTH[b] OF REFRACTORIES[c] FOR SLAGGING GASIFIERS[100], Continued

TEMPERATURE DIFFERENCE FOR THERMAL SHOCK: 900°C

Figure A

INITIAL STRENGTH (MPa)

PERCENT RETAINED STRENGTH

○ ONE CYCLE
△ THREE CYCLES
□ FIVE CYCLES

R''''(m)

Figure B

PERCENT RETAINED STRENGTH
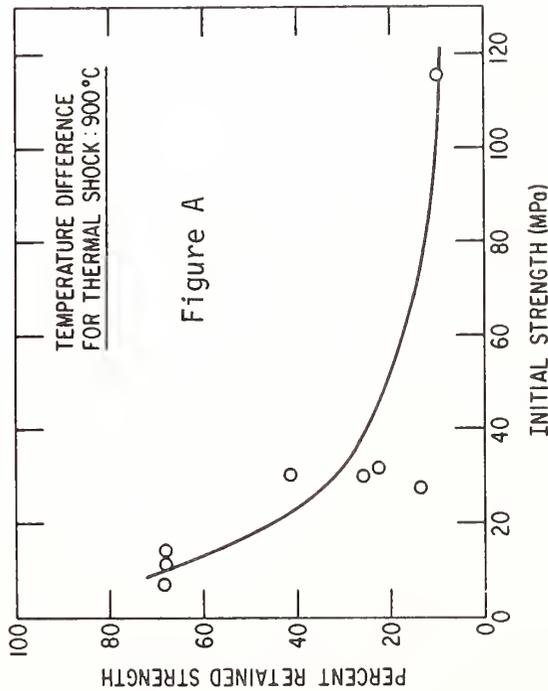
Figure A shows the percent retained strength after one cycle as a function of the initial tensile strength.
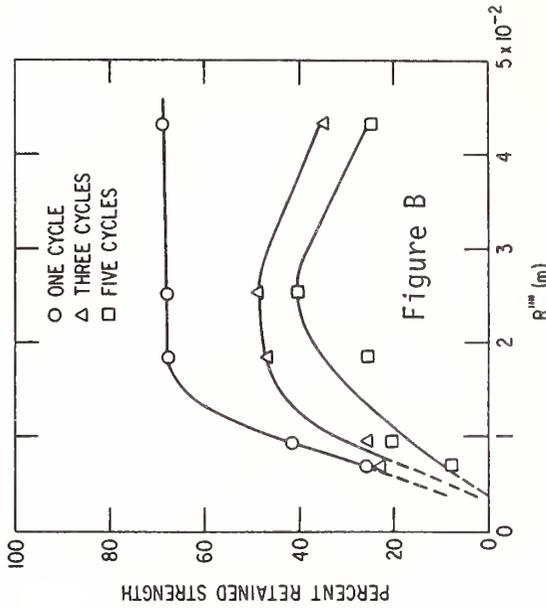
Figure B shows the retained tensile strength for multiple quenchings versus the thermal shock resistance R'''' (see Section B.3.2.145).

[a] Specimens were thermally shocked by quenching from 1000 °C into boiling water at 100 °C. The heating and quenching constituted one cycle. Specimens were arranged in bundles so that upon quenching they received cooling from one side only.

[b] Specimens were 114.3 x 12.7 x 12.7 mm [incorrectly given as cm in the original reports] (4.5 x 0.5 x 0.5 in.) but no details about the methods of tensile testing are given.

[c] A number of refractories in this section were tested in a slag corrosion testing program conducted at the same laboratory (see Section B.1.2.26).

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THERMAL SHOCK ESTIMATES[a] BASED ON PHYSICAL AND MECHANICAL PROPERTIES OF
ALUMINA CASTABLES[16]

| | Calculated Damage Resistance Parameters[a] | | |
| | R'''', 10^{-2} m | | R_{st} |
Refractory	ambient temp.	1200 °C	$10^{-1}\sqrt{m}$ °C
Tabular alumina/CA[b] cement[c]	7.26	---	148.75
Calcined bauxite/CA cement[d]	2.05	---	59.58
Calcined bauxite/CA cement[e]	4.04	---	109.19
Calcined kaolin/CA cement[f]	1.75	---	72.13
Calcined kaolin/CA cement[g]	2.74	---	80.42
57% Alumina insulating castable[h]	1.62	---	59.47
50% Alumina insulating castable[i]	2.78	---	79.57
Tabular alumina/CA cement[j]			
Generic formulation	1.89	3.96	---
Continuous, 20% cement	2.40	9.10	---
Continuous, 25% cement	2.29	5.73	---
Continuous, 30% cement	1.54	3.98	---
Gap-sized, 20% cement	16.02	34.84	---
Gap-sized, 25% cement	18.69	29.38	---
Gap-sized, 30% cement	19.08	31.23	---

[a] Quantities assessing thermal shock resistance of refractories are defined as
$R'''' = E\gamma/(1-\nu)\sigma_f^2$, and $R_{st} = (\gamma/\alpha^2 E)^{1/2}$, where E = Young's modulus, γ = the
work of fracture, ν = Poisson's ratio, σ_f^2 = tensile strength, and α = the
coefficient of thermal expansion. R'''' is intended to be proportional to
the inverse of the crack area per unit volume propagated by thermal shock.
R_{st} is related to the maximum allowable temperature difference required to
propagate long cracks under severe thermal stress conditions.

[b] CA = calcium aluminate.

[c] 75% T-61 alumina (Alcoa) aggregate, 25% Secar 71 cement (Lone Star Lafarge),
water to ball-in-hand consistency 12.7% of aggregate and cement.

[d] 75% Mulcoa M-60 (C-E Minerals) aggregate, 25% Casting Grade CA-25 (Alcoa)
cement, water to ball-in-hand consistency 10.6% of aggregate and cement.

[e] 75% Mulcoa M-60 (C-E Minerals) aggregate, 25% Secar 71 cement (Lone Star
Lafarge), water to ball-in-hand consistency 14.6% of aggregate and cement.

[f] 75% Mulcoa M-47 (C-E Minerals) aggregate, 25% Casting Grade CA-25 (Alcoa)
cement, water to ball-in-hand consistency 11.3% of aggregate and cement.

[g] 75% Mulcoa M-47 (C-E Minerals) aggregate, 25% Refcon cement (Universal
Atlas), water to ball-in-hand consistency 13.5% of aggregate and cement.

[h] A commercial lightweight insulating castable (Cer-Lite #75, C-E Refracto-
ries), water to ball-in-hand consistency 22% of mix.

[i] A commercial lightweight insulating castable (Litecast 75-28, General
Refractories), water to ball-in-hand consistency 24% of mix.

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(Table Continued)

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THERMAL SHOCK ESTIMATES[a] BASED ON PHYSICAL AND MECHANICAL PROPERTIES OF
ALUMINA CASTABLES[16], Continued

Footnotes continued

[j]T-61 tabular alumina (Alcoa) aggregate, Casting Grade CA-25 calcium alumi-
nate cement (Alcoa). Generic formulation 75% aggregate, 25% cement, water
to ball-in-hand consistency 10.3% of aggregate and mix. Various samples
were prepared to study the effect of aggregate particle size distribution
on the properties of the castable. The generic castable has a size distri-
bution typical of a standard industrial product and is the as-received mate-
rial. The other two formulations are designed for maximum density of the
aggregate and the distributions were calculated according to formulas for
each case. The continuous formula contains aggregate continuously graded
so that the difference between successive particle sizes is a factor of $\sqrt{2}$,
with the largest particles sized at U.S. Sieve Number 4 x 6 and the smallest
-325. Gap-sized packing provides sizes and proportions so that the larger
voids created between larger particles are filled by smaller particles and
new voids thus created filled by even smaller particles. For the continuous
formulation: 20% cement has 9.9% water, 25% cement has 10.6% water, 30% ce-
ment has 11.1% water. For the gap-sized formulation: 20% cement has 8.0%
water, 25% cement has 9.1% water, 30% cement has 9.8% water.

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STRENGTH LOSS[a] AGREEMENT WITH ESTIMATED THERMAL SHOCK DAMAGE RESISTANCE[b]
FOR ALUMINA CASTABLES[16]

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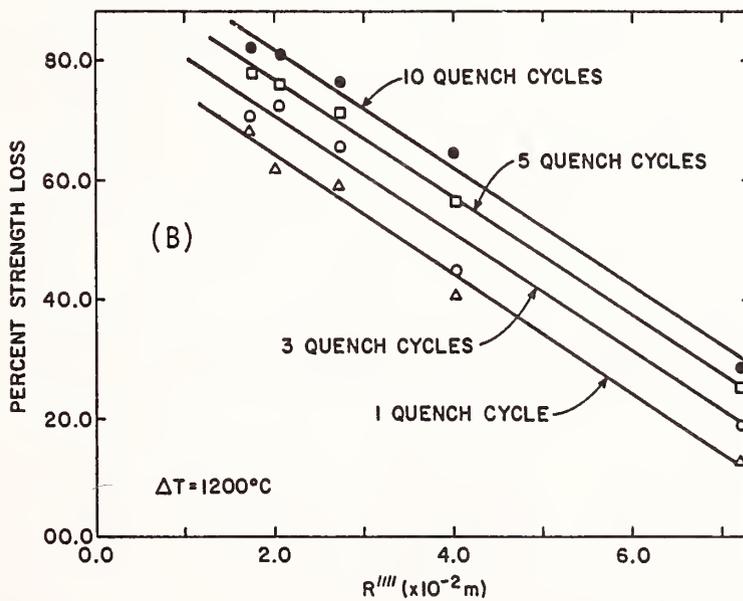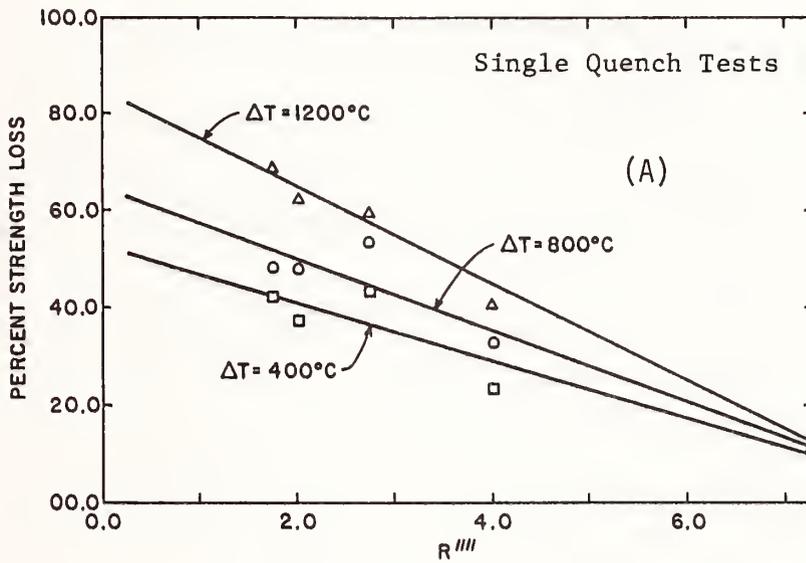
(Data Continued)

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STRENGTH LOSS[a] AGREEMENT WITH ESTIMATED THERMAL SHOCK DAMAGE RESISTANCE[b]
FOR ALUMINA CASTABLES[16], Continued

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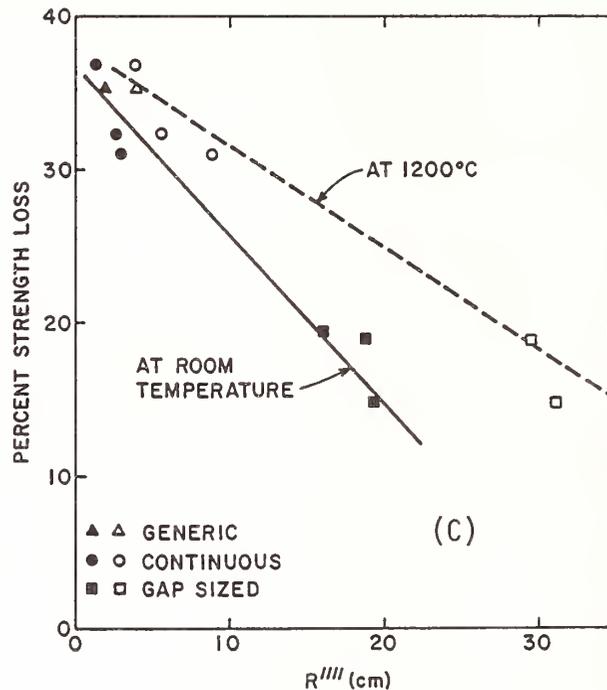
[a]See Sections B.3.2.95, B.3.2.97, and B.3.2.99 for the data plotted here as percent strength loss and for the identification of the castables.

[b]See Section B.3.2.148 for the damage resistance parameter, R'''', plotted here.
    Figure A shows the data from the single quench tests reported in Section
        B.3.2.95.
    Figure B shows the data from the multiple quench tests reported in Section
        B.3.2.97.
    Figure C shows the data from the single quench tests reported in Section
        B.3.2.99 for a generic castable prepared with different particle size
        distribution formulations for the aggregate material.

EFFECT OF SEVERAL FUEL COMBUSTION ENVIRONMENTS[a] ON THE TENSILE STRENGTH[b] OF VARIOUS CERAMIC TUBE MATERIALS[106]

| Material	Tensile Strength,[b] MPa (ksi)		Change[c] %
	As-Received	After Exposure	
TEST EXPOSURE 1[a]			
α-SiC, sintered, pressureless (Carborundum)	408.4±45.6 (59.23±6.61)	287.2±63.0 (41.65±9.14)	− 30
SiC, siliconized (KT-SiC, Carborundum)	201.9±31.2 (29.29±4.52)	277.7±19.7 (40.27±2.85)	+ 37
SiC, siliconized (NC 430, Norton)	168.0±32.4 (24.37±4.70)	241.2±35.7 (34.98±5.17)	+ 44
SiC, siliconized (Refel SiC, Pure Carbon Co.)	179.7±24.8 (26.06±3.60)	265.0±21.8 (38.44±3.17)	+ 48
Al₂O₃, high-purity (AD 998, Coors Porcelain)	209.0±19.3 (30.31±2.79)	140.7±11.7 (20.40±1.69)	− 50
CVD SiC on SiC[d] (Deposits & Composites Inc.)	194.2±26.4 (28.17±3.83)	130.9±53.6 (18.99±7.78)	− 33
CVD SiC (Deposits & Composites Inc.)	256.5±86.1 (37.21±12.5)	248.2±52.0 (36.00±7.54)	− 4
SiC, clay-bonded (Carbofrax A, Carborundum)	17.0± 6.6 (2.47±0.96)	35.8± 3.6 (5.20±0.52)	+110
SiC, clay-bonded (Carbofrax M, Carborundum)	21.3± 4.1 (3.10±0.59)	23.3± 4.2 (3.38±0.61)	+ 9
SiC, Si₃N₄-bonded (Refrax 20, Carborundum)	26.8± 1.7 (3.88±0.25)	61.8±15.2 (8.99±2.20)	+131
Aluminosilicate, high-purity (Mullite, Coors Porcelain)	125.8±22.5 (18.39±3.26)	Tube cracked extensively	
Magnesium aluminosilicate (MAS 8200, GTE Sylvania)	51.4± 0.1 (7.45±0.01)	Tube cracked extensively	
Magnesium aluminosilicate (MAS 8400, GTE Sylvania)	26.2± 4.1 (3.79±0.59)	Tube cracked extensively	
Zirconia plus mullite (Zirmul, GTE Sylvania)	71.2±19.1 (10.32±2.76)	Tube cracked extensively	
TEST EXPOSURE 2[a]			
SiC, siliconized (KT-SiC, Carborundum)	179 ±8.27 (25.9 ±1.2)	198 ±15.2 (28.8 ±2.2)	+ 11
α-SiC, sintered, pressureless (Carborundum)	305 ±52.4 (44.2 ±7.6)	292 ±24.1 (42.4 ±3.5)	− 4
CVD SiC (Deposits & Composites Inc.)	347 ±53.8 (50.3 ±7.8)	335 ±40.7 (48.6 ±5.9)	− 3
Al₂O₃, high-purity (AD 998, Coors Porcelain)	Tubes cracked extensively during exposure		
SiAlON (GE 128, General Electric)	Tubes cracked extensively during exposure		
TEST EXPOSURE 3[a]			
Al₂O₃, high-purity (AD 998, Coors Porcelain)	241.3±21.5 (35.0 ±3.1)	174.3±44.5 (25.3 ±6.5)	− 28
α-SiC, sintered, pressureless (Carborundum)	263.5±30.3 (38.2 ±4.4)	255.2±47.0 (37.0 ±6.8)	− 3
SiC, siliconized (NC 430, Norton)	174.4±17.6 (25.3 ±2.5)	108.6±22.9 (15.8 ±3.3)	− 38
SiC, siliconized (KT-SiC, Carborundum)			
thin wall tube (6.57 mm)	202.9±12.8 (29.4 ±1.8)	107.7±15.3 (15.6 ±2.2)	− 47
thick wall tube (12.67 mm)	178.6± 8.2 (25.9 ±1.2)	72.1±21.7 (10.5 ±3.1)	− 60

(Table Continued)

EFFECT OF SEVERAL FUEL COMBUSTION ENVIRONMENTS[a] ON THE TENSILE STRENGTH[b] OF VARIOUS CERAMIC TUBE MATERIALS[106], Continued

Material	Tensile Strength,[b] MPa (ksi)		Change[c] %
	As-Received	After Exposure	
TEST EXPOSURE 3[a], continued			
SiC, siliconized (SC-2, Coors Porcelain)	238.8±23.2 (34.6 ±3.4)	92.8±51.6 (13.5 ±7.5)	− 61
		One tube fractured on removal	
CVR SiC[e] (experimental material, Syntax)	85.1± 2.6 (12.3 ±3.8)	71.4±20.3 (10.3 ±2.9)	− 16
SiAlON (GE-128, General Electric)		Tubes fractured on removal	
TEST EXPOSURE 4[a,f]			
α-SiC, sintered, pressureless (Carborundum)	263.5±30.3 (38.2 ±4.40)	276.9±44.1 (40.1 ±6.41)	+ 5
SiC, Si$_3$N$_4$-bonded (CS SiC, Norton)	11.58±4.18 (1.68 ±0.61)	75.73±26.2 (11.0 ±3.79)	+ 554
Al$_2$O$_3$, high-purity (AD 998, Coors Porcelain)	241.3±21.5 (35.13±3.12)	268.2±39.90(38.9 ±5.78)	+ 11

[a] Ceramic tubes were exposed in the flue gas of a combustor during 4 tests with 4 different fuels. The parameters for each test are given below. For some of the tests more than one tube of the material was exposed. In Test 4 some tubes were closed on one end and were pressurized to 0.68 MPa (100 psig).
TEST EXPOSURE 1--fired with No. 6 fuel oil with a high vanadium content (~83 weight ppm); tube wall thicknesses varied from 1.6 to 9.5 mm for the different materials; 7 heating-holding-cooling cycles occurred during the test.
TEST EXPOSURE 2--fired with Venezuelan crude-derived No. 6 fuel oil containing 10 wt% powdered bituminous coal, with over 98.9% of particle size <44 μm; ash content was acidic with base to acid ratio = 0.29; slag was viscous; 3 thermal cycles occurred during the test (1200→800→1200 °C).
TEST EXPOSURE 3--fired with Venezuelan crude-derived No. 6 fuel oil containing 20 wt% powdered coal from Hanna Seam, Wyoming, with 99.2% of particle size <44 μm; ash content was basic with base to acid ratio = 1.14; slag was fluid; tube wall thicknesses varied from 5.5 to 8.7 for the different materials. The values given in the table below for this exposure are given for the 222nd hour of the exposure.
TEST EXPOSURE 4--fired with Venezuelan crude-derived No. 6 fuel oil containing 20 wt% powdered coal with refractory ash (fusion temperature >1482 °C; ash was very acidic with base to acid ratio = 0.09; slag formed a sticky particulate buildup; see figures below for temperature excursions during test. Values given in the table for this exposure are for the 506th hour of the exposure.

All temperatures in the following table are in °C. The duration given for the test is the total time of the exposure.

(Table Continued)

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EFFECT OF SEVERAL FUEL COMBUSTION ENVIRONMENTS[a] ON THE TENSILE STRENGTH[b] OF VARIOUS CERAMIC TUBE MATERIALS[106], Continued

Footnotes continued

Test	Time h	Tube Temperature°C	Flue Gas Temperature°C Inlet	Flue Gas Temperature°C Outlet	Air Temperature°C Inlet	Air Temperature°C Outlet	Air Velocity m/s	Heat Transfer Rate, kW	Heat Flux kW/m²
1	500	1100-1250	1410	1385	ambient	340	8.1	23.7	54
2	500	1200-1280	1390	1349	ambient	427	7.6	27.6	13.6
3	240	1220	1418	1378	47.0	353	10.9	23.7	49.4
4	350	see below	1302	1198	36	162	14.4	16.8	166

COMBUSTOR EXPERIENCED EIGHT MAJOR TEMPERATURE EXCURSIONS DURING TEST 4

TUBE TEMPERATURES EXPERIENCED MAJOR FLUCTUATIONS DURING TEST 4

[b] Half-ring (C-ring) compression testing was used. Rings were loaded to failure on an Instron machine, crosshead speed 21.2 μm/s (0.05 in./min). The analytical expression for fracture in the 1/2-ring specimen;

$$\sigma = \frac{P}{A} - \frac{M}{Ay}\left(\frac{y}{R-y}\right)$$

where σ = tensile strength; P = compressive load causing fracture in the 1/2-ring specimen; A = cross section area of 1/2-ring; M = P(r_o + r_i)/2, where r_o = outer radius, r_i = inner radius; R = (r_o + r_i)/ln(r_o/r_i); y = (r_o - r_i)/2-$\bar{y}$, where $\bar{y}$ = (r_o + r_i)/w-R.

[c] Change = (exposed value - as-received value)/as-received value.

[d] Chemically vapor deposited SiC on reaction-sintered SiC substrate.

[e] Chemically vapor reacted SiC; silicon reacted with graphite mandrel at 1800 °C and a CVD SiC over-lay applied.

(Table Continued)

EFFECT OF SEVERAL FUEL COMBUSTION ENVIRONMENTS[a] ON THE TENSILE STRENGTH[b] OF VARIOUS CERAMIC
TUBE MATERIALS[106], Continued

Footnotes continued

[f]Test 4 data presented graphically. Samples were oriented so fracture occurred from the region that ex-
hibited the greatest apparent reaction between the ceramic and the coal slag.
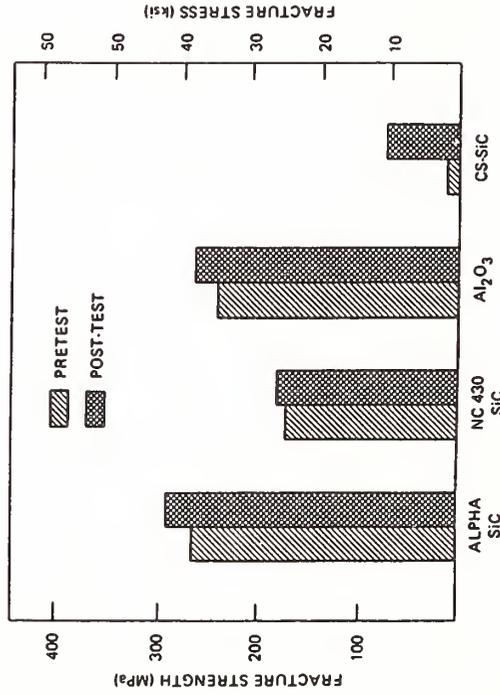
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EFFECT OF A BASIC SLAG[a] IN A HIGH-TEMPERATURE ENVIRONMENT[b] ON HALF-RING
FRACTURE STRENGTH[c] OF SINTERED SILICON CARBIDE TUBES[d][106]

| Exposure Time, h | Fracture Strength,[c] MPa (ksi) | |
	Air Environment	Slag Environment
- - Temperature 1250 °C, no applied stress during exposure - - - - - -		
0	327±55 (47.5±8.0)	--
24	389±55 (56.6±8.0)	389±35 (56.1±5.0)
168 (thin slag deposits)[e]--see Figure A below		
(thick slag deposits)		325±12 (47.1±1.8)
- - Temperature 1175 °C, exposures both with and without applied stress		
168	see Figure B below	
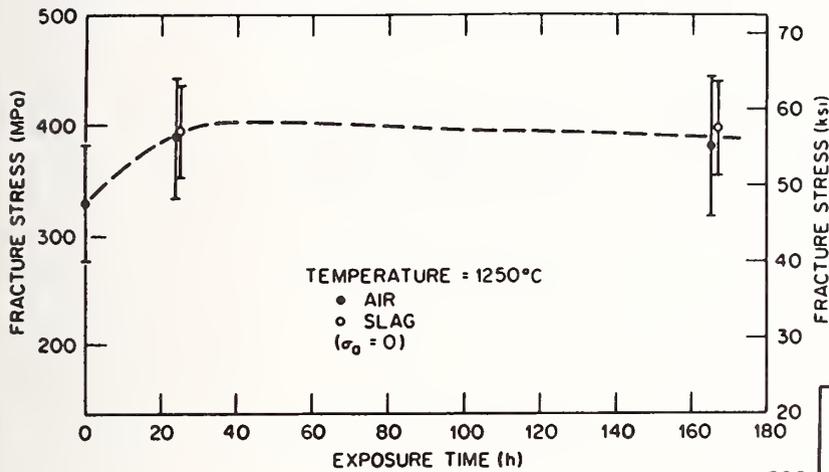
← Figure A

Figure B →
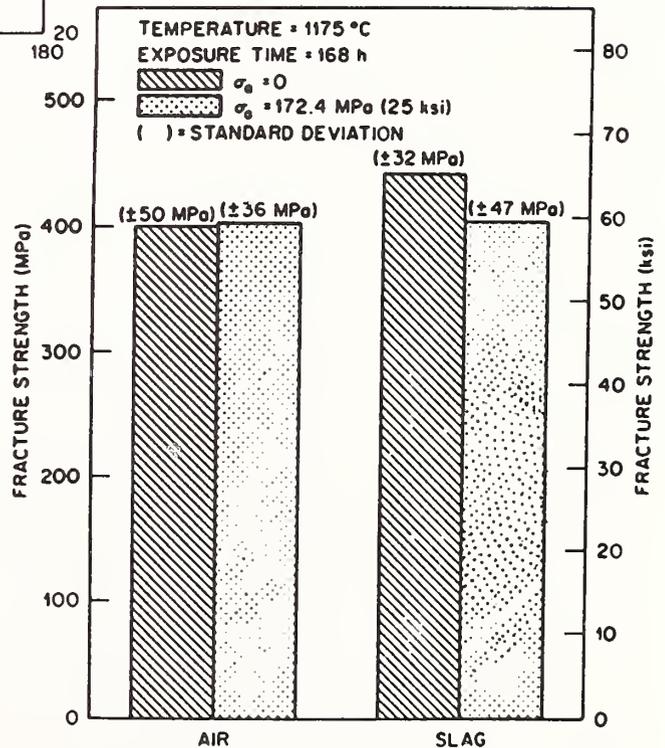
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EFFECT OF A BASIC SLAG[a] IN A HIGH-TEMPERATURE ENVIRONMENT[b] ON HALF-RING
FRACTURE STRENGTH[c] OF SINTERED SILICON CARBIDE TUBES[d][106], Continued

Footnotes

[a]See footnote a, Section B.3.2.150 for information for Test Exposure 3.
The slag in the test in this section is the same slag as used in Test
Exposure 3 in Section B.3.2.150.

[b]The material was exposed in a high-temperature corrosion facility which
permitted exposure to slag environments and also permitted strength
testing at elevated temperatures both in the environment and in air.
Strength testing was performed both at 1250 and 1175 °C. Exposures
were conducted both with and without applied stress on the material.

[c]Half-ring (C-ring) compression testing; see footnote b, Section B.3.2.150,
for the analytical expression for the testing. No details of the test
methods were given for these tests.

[d]Pressureless sintered α-SiC (Carborundum).

[e]The data given in Figure A for the 168-hour exposure were measured with
the samples where the slag layer was less than 300 µm. Where the slag
was thick (greater than 1 mm), the strength value was considerably lower
than the values shown in Figure A.

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EFFECT OF VARIOUS COAL-OIL COMBUSTION PRODUCTS[a] ON THE FLEXURE STRENGTH[b] OF VARIOUS CERAMIC MATERIALS[106]

Material	Flexure Strength, MPa		% Retained Strength
	As-Received	After Exposure	
COMBUSTION TEST EXPOSURE 2, acidic slag, base to acid ratio = 0.29			
Si$_3$N$_4$, hot-pressed (NC 132, Norton)	851.2±54.9(ground)^{c,d}	298.1±14.2(with slag)^{d} 805.3±76.3(ground)^{c,e}	35 95
Si$_3$N$_4$, hot-pressed (NCX 34, Norton)	808.1±69.1(ground)^{c,d}	320.6±34.9(with slag)^{d} 798.6±101.1(ground)^{c,e}	40 99
SiAlON (GE 128, General Electric)	359.5	Sample broke in handling	-
SiC, hot-pressed (NC 203, Norton)	647.2±33.5(ground)^{c,d}	440.5±31.0(with slag)^{d} 638.7±13.8(ground)^{c,e}	68 99
SiC, sintered-α (Hexoloy SA, Carborundum)	369.2±31.1(ground)^{c,d}	208.6±40.0(with slag)^{d} 370.0±46.9(ground)^{c,e}	57 100
SiC, siliconized (KT-SiC, Carborundum)	206.0±37.7(ground)^{c,d}	195.7±28.0(with slag)^{d} 241.0± 6.3(ground)^{c,e}	95 117
COMBUSTION TEST EXPOSURE 3, basic slag, base to acid ratio = 1.14^{f}			
SiC, hot-pressed (NC 203, Norton)	593.0±86.5^{g}	365.9±120.5^{g}	62
SiC, sintered-α (Hexoloy SA, Carborundum)	367.9±52.5^{g}	277.7±26.2^{g}	75
COMBUSTION TEST EXPOSURE 4, acidic slag, base to acid ratio = 0.09			
Si$_3$N$_4$, hot-pressed (NC 132, Norton)	not given	436 ±14^{h}	53
Si$_3$N$_4$, hot-pressed (NCX 34, Norton)	not given	329 ±51^{h}	41
SiC, hot-pressed (NC 203, Norton)	not given	410 ±31^{h}	68
SiC, sintered-α (Hexoloy SA, Carborundum)	not given	283 ±31^{h}	76
SiC, siliconized (KT-SiC, Carborundum)	not given	193 ±17^{h}	100

[a] Bar specimens suitable for flexure testing were exposed to the hot combustion products of the Combustion Test Exposures 2, 3, and 4 reported in Section B.3.2.150. The specimens were subjected to the flue gases and hot particulates produced in those tests. See footnote a, B.3.2.150, for the test conditions.

[b] Specimens were tested in four-point loading. Details are given in footnotes d, e, g, and h.

(Table Continued)

EFFECT OF VARIOUS COAL-OIL COMBUSTION PRODUCTS[a] ON THE FLEXURE STRENGTH[b] OF VARIOUS CERAMIC MATERIALS[106], Continued

Footnotes continued

[c] Diamond abrasive used; surface ground and edge beveled parallel to bar length.

[d] Slag coating on bars was 5-10 mm thick on upstream side, 1-5 mm thick on downstream side. Bars were tested at 22 °C, 37.5 mm outer span and 12.5 mm inner span, strain rate 1×10^{-1}/m.

[e] Surfaces were ground to eliminate effect of surface corrosion, various chemical reactions, and micro-structure changes due to slag and flue gases. Bars were tested at 22 °C, 18.9 mm outer span and 6.3 mm inner span, strain rate 2×10^{-1}/m.

[f] Specimens were greatly thinned by corrosion and fell from the supports into the combustion chamber. They were, therefore, exposed to the slag but not to the combustion gases during the entire test period. Not all the specimens exposed have flexure strength data reported for this exposure. See Section B.3.2.156 for other data for these materials exposed to this same basic slag.

[g] Surface condition not specified, and dimensions and strain rate not given.

[h] Surface condition not specified. Bars were tested at 22 °C in 60% relative humidity, strain rate 1.5×10^{-1}/m. The SiC materials from Test Exposure 4 were also strength tested at 1150 and 1300 °C. The data are presented graphically in the following figure. The siliconized SiC showed no real changes after exposure from the as-received values of strength so there is only one curve given for that material.

AS-RECEIVED | AFTER EXPOSURE 4
Hot-Pressed SiC □ ◁
Sintered α-SiC ▽ ●
Siliconized SiC ○ ▲

Hot-Pressed SiC, as-received
Sintered α-SiC, as-received
Hot-Pressed SiC
Sintered α-SiC
Siliconized SiC

FLEXURE STRENGTH (MPa)
TEST TEMPERATURE (°C)
FOUR POINT FLEXURE, AIR

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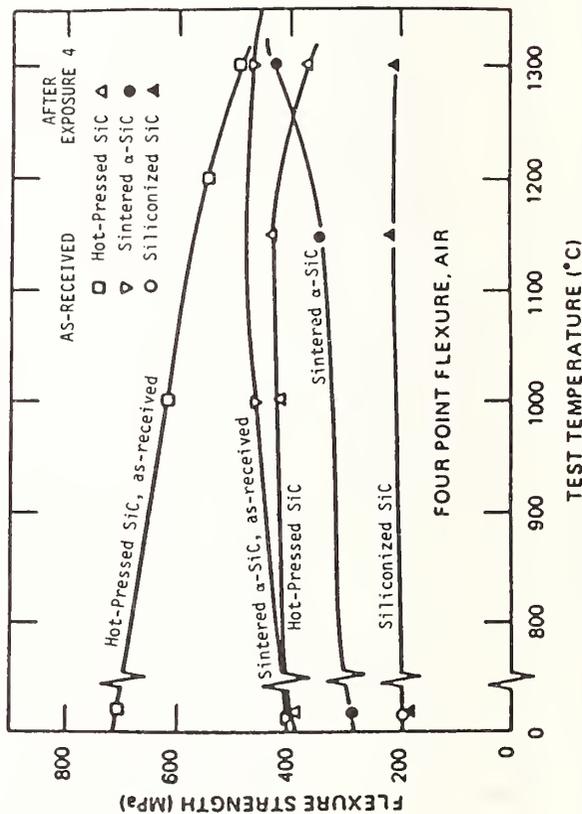
COMPARISON OF FRACTURE STRENGTHS[a] OF SILICON-BASED CERAMICS[b] AFTER
COAL-OIL FUEL COMBUSTION TESTS[c][106]
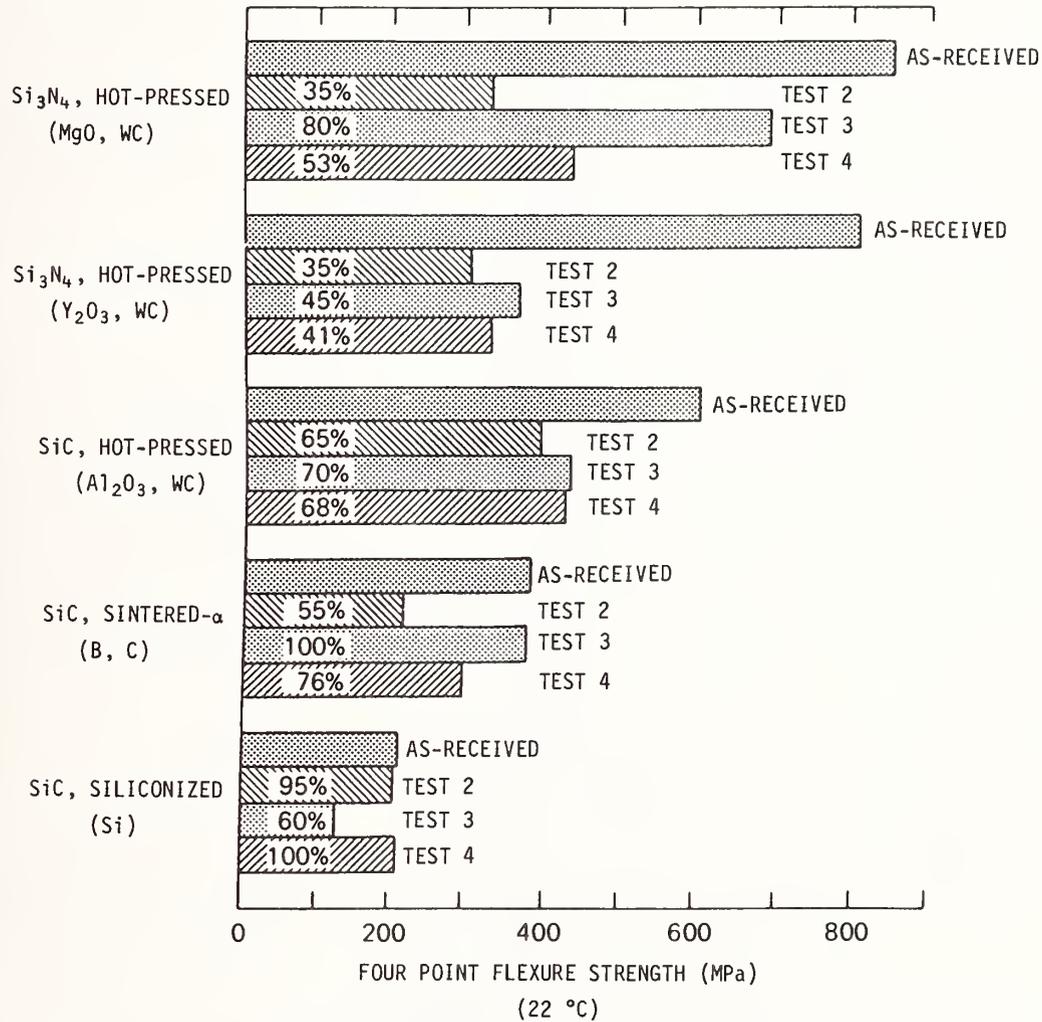
[a]See Sections B.3.2.152 and B.3.2.156 for the data plotted here. The numbers
on the bars of the graph are the percent strength retained by the exposed
specimens with respect to the as-received strength. Note that the data for
Test 3, because of the damage to the specimens by the conditions, are not
from B.3.2.152. Data from B.3.2.156 in which the specimens were subjected
to the same basic slag have been plotted.

[b]The parentheses below the material designations indicate the additives con-
tained in them.

[c]See Section B.3.2.150, footnote a, for the test conditions for the coal-
oil combustion.

FRACTURE STRENGTH[a] CHANGES IN SILICON-BASED CERAMICS EXPOSED TO HIGH-TEMPERATURE OXIDATION[b][106]

Material/(Densification Aid)	Fraction of as-received strength retained		
	After Test Exposure 2, in acidic slag[c] 1220 °C, ~500 h	Oxidation in Air, 1325 °C, 1000 h	
		4 Oxidation Cycles	4 Oxidation Cycles with Surface Removal
SiC, sintered-α (Hexoloy SA, Carborundum)/(B, C)	0.55		0.95
SiC, hot-pressed (NC 203, Norton)/(Al₂O₃, WC)	0.65	0.55	>0.75
Si₃N₄, hot-pressed (NC 132, Norton)/(MgO, WC)[d]	0.35	0.45	0.60

[a]Flexure bars were tested in four-point bending.

[b]Bars were exposed to four oxidation cycles. One cycle consisted of a 250-hour exposure in air at 1325 °C followed by cooling to ambient temperature. One set of specimens was subjected to removal of the surface oxide layers which formed after each of the first three cycles. The oxide layers formed were smaller with each subsequent cycle.

[c]See footnote a, Section B.3.2.150, for conditions of Test Exposure 2 in a coal-oil fuel combustor. Data are included for comparison.

[d]Si_2ON_2 found as an impurity.

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FLEXURE STRENGTH[a] OF SiC CERAMICS AFTER LONG-TERM OXIDATION[b][106]

| Material | Flexure Strength,[a] MPa | | |
	as-received	2000 h oxidation	5000 h oxidation
Siliconized SiC (KT-SiC, Carborundum)	209±40	250±20	290±10
Sintered α-SiC (Hexoloy SA, Carborundum)	368±53	425± 8	
Hot-pressed SiC (NC 203, Norton)	593±87	429±40	

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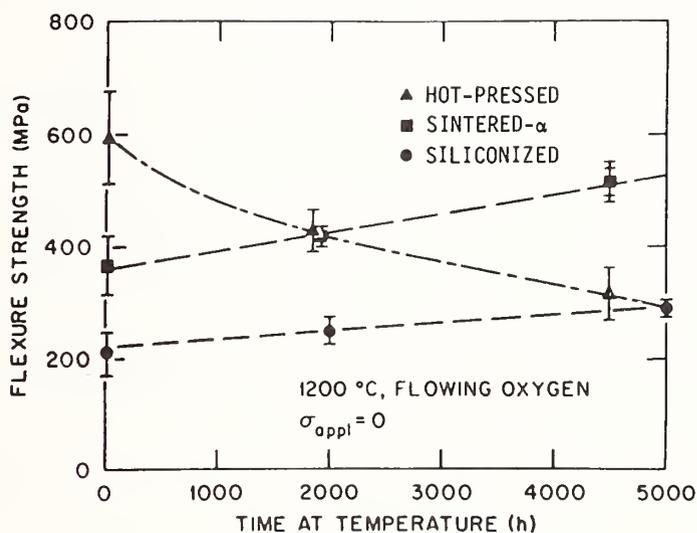
[a]Flexure bars were tested in four-point bending to fracture; inner span 6.35 mm and outer span 19.05 mm, constant stress 100 MPa, at 22 °C in air with 60% relative humidity.

[b]Specimens were heated in a resistance-heated high-purity alumina tube furnace. Test bars were contained in a sintered α-SiC tube through which oxygen passed. Oxidation took place at 1200 °C for 2000 and 5000 hours. No stress was applied during the oxidation.

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FLEXURE STRENGTH[a] OF SILICON-BASED CERAMICS EXPOSED TO BASIC SLAG[b] SLURRY
IN AN OXYGEN ATMOSPHERE[c][106]

| Material | Flexure Strength,[a] MPa | | Retained |
	As-Received	After Exposure	Strength
SiC, sintered-α (Hexoloy SA, Carborundum)	368±53	364±101	99 %
SiC, siliconized (KT-SiC, Carborundum)[d]	209±40	123± 22	59
SiC, hot-pressed (NC 203, Norton)[d]	593±87	428±192	72
Si_3N_4, hot-pressed (NC 132, Norton)[e]	851±55	689± 48	81
Si_3N_4, hot-pressed (NCX 34, Norton)[e]	808±69	358±261	44

[a] Flexure bars tested to fracture in four-point bending.

[b] Slurry was prepared from the basic coal slag from Combustion Test Exposure 3
(see footnote a, Section B.3.2.150). Slag had a base to acid ratio = 1.14.
Test bars were coated with the slurry.

[c] The slurry-coated bars were exposed to flowing oxygen at 1200 °C for 200 hours
in an alumina tube furnace.

[d] These materials showed that corrosion had penetrated the ceramic below the
slag layer.

[e] These materials showed cracking and slag penetration well into the bulk material.

===

CRACK GROWTH DATA[a] FOR A SILICON CARBIDE[b] AND A SILICON NITRIDE[c] TESTED
IN AIR[107]

Crack Growth Mechanism Maps[a] - - - - - - - - - - - - - - - - - - -
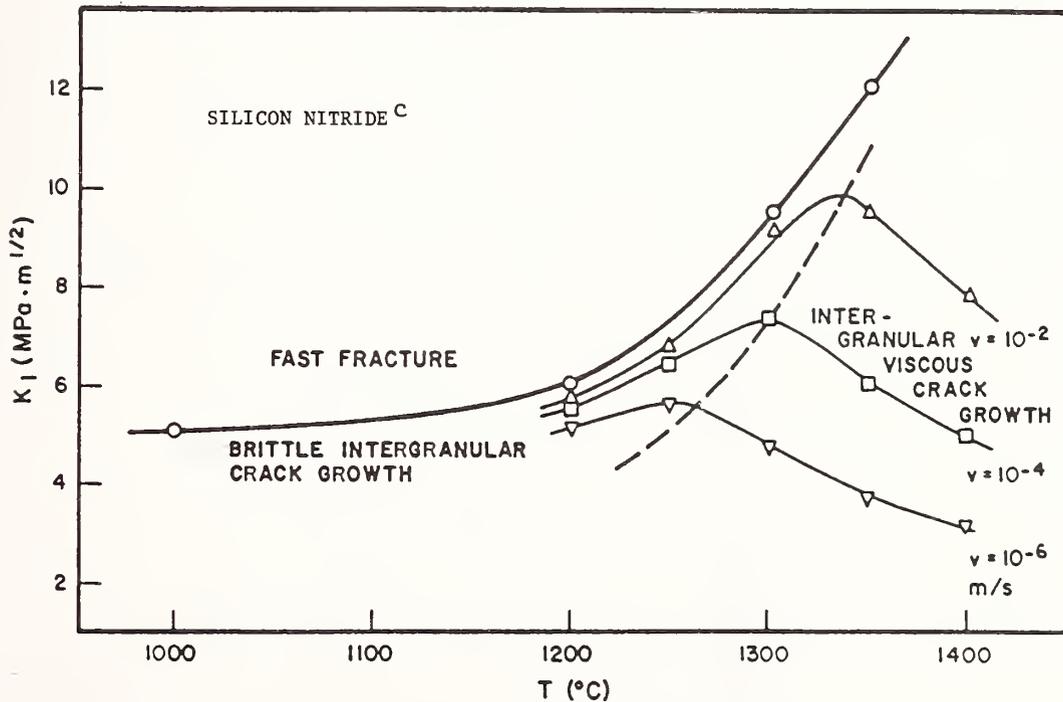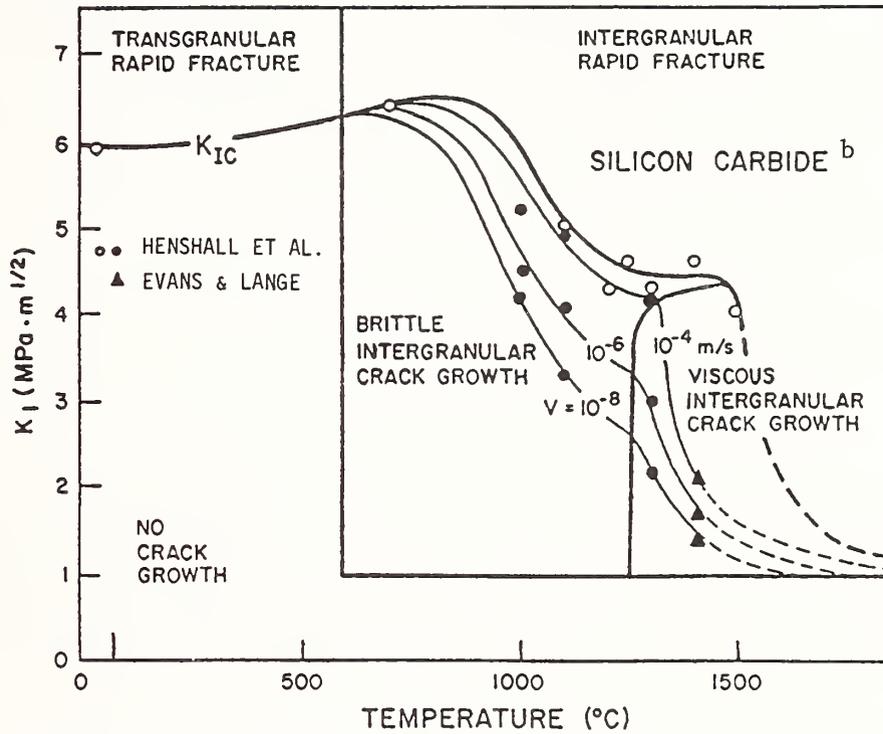
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(Data Continued)

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CRACK GROWTH DATA[a] FOR A SILICON CARBIDE[b] AND A SILICON NITRIDE[c] TESTED
IN AIR[107], Continued

SILICON CARBIDE[b] -

Temperature °C	Crack Velocity (m/s)	Stress Intensity Factor K_I (MPa $\sqrt{m}$)	Reference[d]
1400	10^{-2}	2.5	1
	10^{-4}	2.1	1
	10^{-6}	1.7	1
	10^{-8}	1.4	1
1300	10^{-4}	4.2	2
	10^{-6}	3.0	2
	10^{-8}	2.2	2
1100	10^{-4}	5.0	2
	10^{-6}	4.1	2
	10^{-8}	3.3	2
1000	10^{-4}	5.2	2
	10^{-6}	4.5	2
	10^{-8}	4.2	2
	10^{-10}	3.7	2

Temperature °C	Stress Intensity Factor K_{Ic} (MPa $\sqrt{m}$)	Reference[d]
1500	4	2
1400	4.6	2
1300	4.3	2
1250	4.6	2
1200	4.3	2
1100	5.0	2
700	6.4	2
25	6.0	2

SILICON NITRIDE[c] -

Temperature °C	Crack Velocity (m/s)	Stress Intensity Factor K_I (MPa $\sqrt{m}$)	Reference[d]
1400	10^{-2}	7.8	3
	10^{-4}	5.0	3
	10^{-6}	3.1	3
1350	10^{-2}	9.5	3
	10^{-4}	6.0	3
	10^{-6}	3.7	3
1300	10^{-2}	9.1	3
	10^{-4}	7.4	3
	10^{-6}	4.7	3
1250	10^{-2}	6.8	3
	10^{-4}	6.4	3
	10^{-6}	5.6	3

- -

(Data Continued)

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CRACK GROWTH DATA[a] FOR A SILICON CARBIDE[b] AND A SILICON NITRIDE[c] TESTED
IN AIR[107], Continued

SILICON NITRIDE[c], continued -

Temperature °C	Crack Velocity (m/s)	Stress Intensity Factor K_I (MPa $\sqrt{m}$)	Reference[d]
1200	10^{-2}	5.7	3
	10^{-4}	5.5	3
	10^{-6}	5.1	3

Temperature °C	Stress Intensity Factor K_{Ic} (MPa $\sqrt{m}$)	Reference[d]
1350	12.0	3
1300	9.5	3
1200	6.0	3
1000	5.1	3

[a] Stress intensity factors and crack velocity data at various temperatures were taken from the literature (see footnote d). Crack growth mechanism maps were constructed from the published data. A computer program was used which maps K-v (stress intensity factor versus crack velocity) data with contours of constant temperature onto a map of K-T (stress intensity factor versus temperature) data with contours of constant crack velocity.

[b] NC 203 (Norton), a hot-pressed SiC.

[c] HS 130, a commercial hot-pressed Si_3N_4.

[d] References:
1. Evans, A.G. and Lange, F.F., Journal of Materials Science, 10, 1659 (1975).
2. Henshall, J.L., Rowcliffe, D.J., and Edington, J.W., Journal of the American Ceramic Society, 62, 36 (1979).
3. Evans, A.G. and Wiederhorn, S.M., Journal of Materials Science, 9, 270 (1974).

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FRACTURE TOUGHNESS[a] OF VARIOUS SILICON-BASED CERAMICS[107]

Figure A

Figure B

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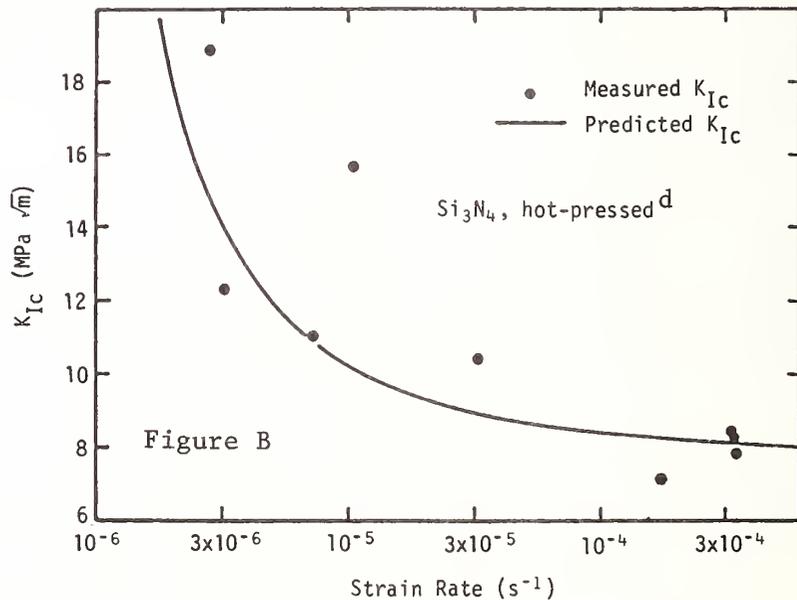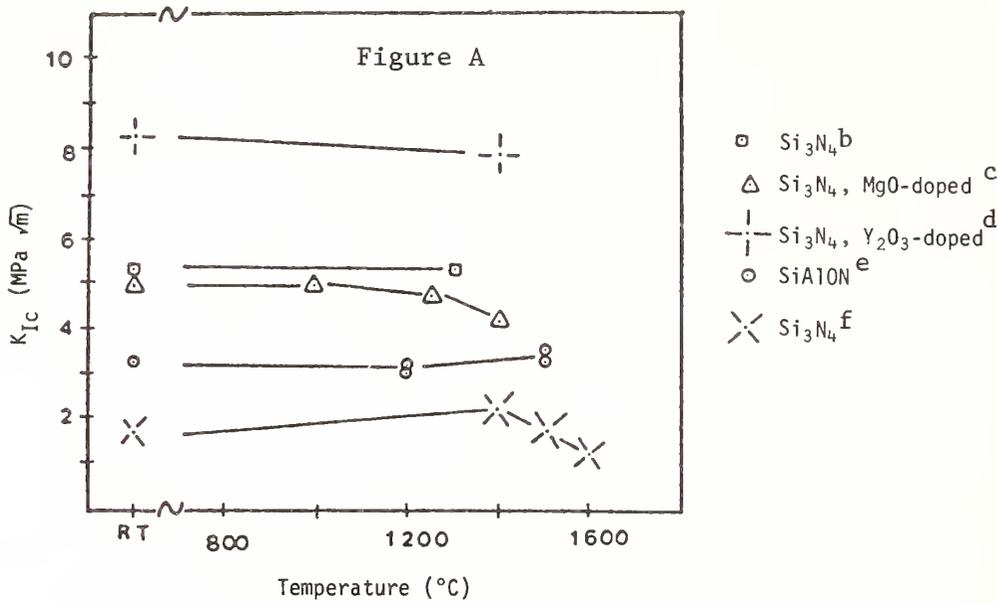
[a]Notched bars, 10 mm x 5 mm x 50 mm with a 3-mm deep blunt notch machined in the 10-mm direction, were tested in 4-point bending under constant displacement rate. Sharp stable cracks were grown from the blunt notch by bending at elevated temperature, annealing at the pre-cracking temperature one hour to relax the stresses produced, and then loading to fracture at various displacement rates and temperatures in air.

Figure A shows the critical stress intensity factors for various materials as a function of temperature.

- -

(Data Continued)

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FRACTURE TOUGHNESS[a] OF VARIOUS SILICON-BASED CERAMICS[107], Continued

Footnotes continued

    Figure B shows the critical stress intensity factor for Si_3N_4 at 1400 °C as a function of strain rate and compares the results with predicted values.

[b]Hot-pressed silicon nitride (HS-130, Norton).

[c]Hot-pressed, MgO-doped silicon nitride (NC 132, Norton).

[d]Hot-pressed, Y_2O_3-doped silicon nitride (NCX 34, Norton).

[e]Si_5AlON_7 obtained from the National Industrial Research Institute of Kyushu, Japan.

[f]Reaction-bonded silicon nitride (NC 350, Norton).

FRACTURE TOUGHNESS[a] DATA FOR SOME SILICON NITRIDES[b][107]

Wt % Garnet[b]	Fabrication[c]	Dimensions (thickness x width, mm)	Crack Length mm	Crosshead Speed in/min	Peak Load kg	Fracture Load kg	Critical Stress Intensity K_{Ic} (MPa√m)[d]
20 + Pt	HP 1 h 1800, PHT 24 h 1300	3.03 x 3.44	2.86	0.02	8.4	5.2	18.0
10	S in BN 1/2 h 1700, HP 1/2 h 1750	3.23 x 4.46	2.63	0.02	27.8	25.4	14.6
10 + Pt	HP 1 h 1800, PHT 24 h 1300	3.22 x 3.63	2.95	0.02	10.2	5.6	14.3
10[e]	HP 1 h 1800	3.27 x 4.37	2.50	0.10	25.7	25.7	14.1
10 + Pt	HP 1/2 h 1750	3.21 x 4.88	2.86	0.02	27.0	24.8	12.4
10 + Pt	HP 1 h 1800	2.81 x 3.94	2.94	0.002	12.1	6.8	11.2
20	HP 1 h 1800, PHT 24 h 1300	3.19 x 4.54	2.95	0.02	17.0	15.0	10.8
10 + Pt	HP 1 h 1750, PHT 24 h 1300	3.06 x 3.75	2.15	0.02	14.3	13.2	9.8
10	HP 1/2 h 1850, PHT 24 h 1300	3.16 x 4.71	1.65	0.05	37.2	37.2	9.7
10 + Pt	S 1/2 h 1750, HP 1 h 1750, PHT 24 h 1300	3.04 x 3.63	1.23	0.05	20.0	20.0	7.8
10	HP 1/2 h 1750	3.32 x 4.41	1.27	0.05	27.7	27.7	6.5
20	HP 1 h 1800	-- not given --	-- --	0.02	13.8	none	---
10	HP 1/2 h 1750, PHT 24 h 1300	-- not given --	-- --	0.005	8.8	none	---
10	HP 1 h 1800, PHT 24 h 1300	-- not given --	-- --	0.02	6.6	none	---
10	S 1/2 h 1600, HP 1/2 h 1750	-- not given --	-- --	0.05	4.1	none	---

(Table Continued)

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FRACTURE TOUGHNESS[a] DATA FOR SOME SILICON NITRIDES[b][107], Continued

[a]Notched bars were tested in air in 4-point bending in air at 1300 °C. Specimens tested at 1400 °C were badly oxidized.

[b]Si_3N_4 was hot-pressed with yttrium aluminum garnet plus minute amounts of Pt or $PtCl_2$ (prepared at the University of Michigan).

[c]S = sintered, HP = hot-pressed, PHT = post heat treated; treatment time is given in hours, temperatures are in °C.

[d]These values are high due to creep effects.

[e]The yttrium alumina garnet added as crystalline garnet.

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EFFECT OF CREEP[a] ON THE FRACTURE[b] OF SILICON-BASED CERAMICS[107]

Figure A

Figure B

Figure C

- -
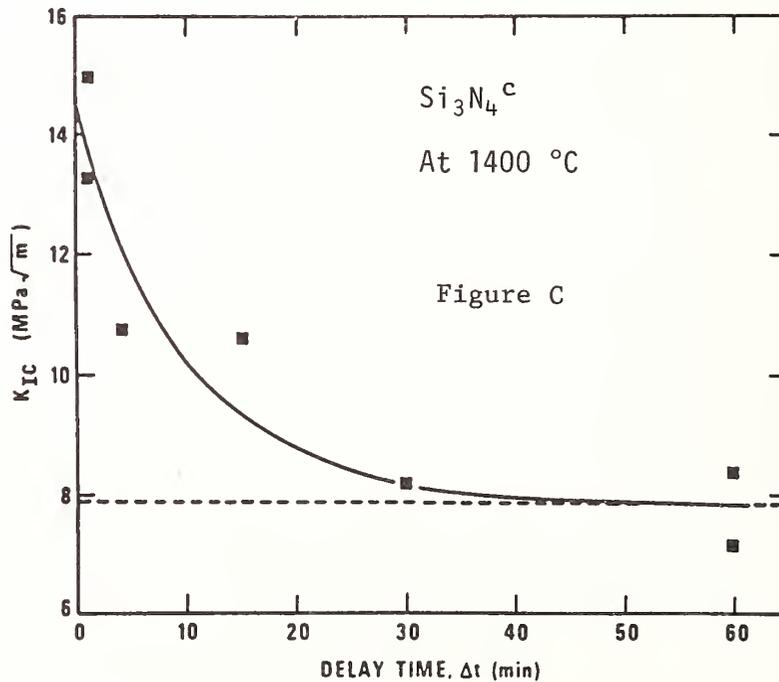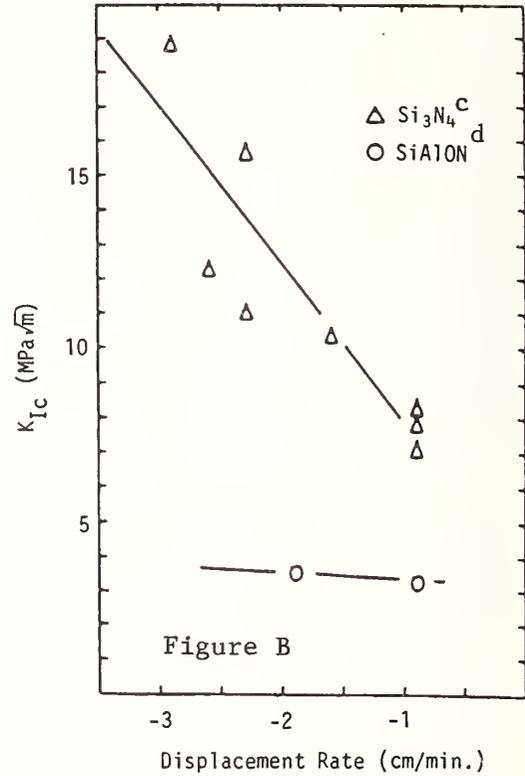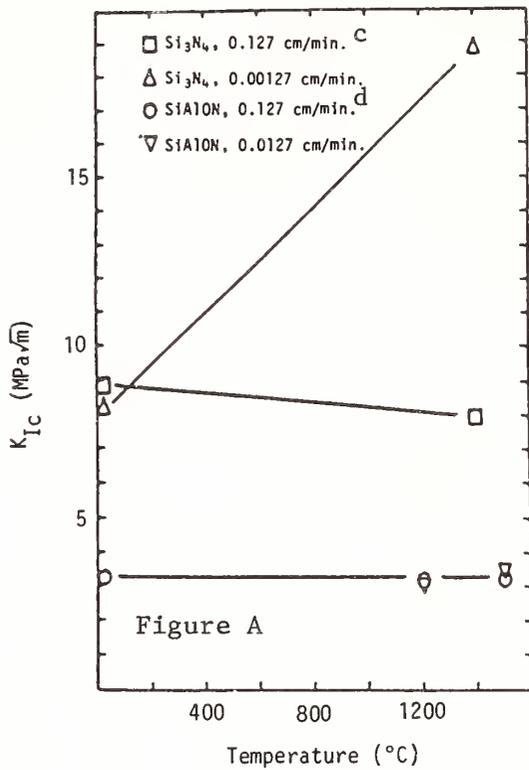
(Data Continued)

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EFFECT OF CREEP[a] ON THE FRACTURE[b] OF SILICON-BASED CERAMICS[107], Continued

Footnotes

[a]See Section B.3.2.161 for creep data for these materials. The data in this section, B.3.2.160, show the dependence of the critical stress intensity factor for fracture on deformation at the crack tip.

[b]Specimen bars 5 x 10 x 50 mm were notched with a thin (0.5 mm) cutting wheel to a depth of 2-5 mm in the 10 mm direction. The bars were tested in 4-point bending at crosshead speeds of 0.00127 to 0.127 cm/min. (0.0005 to 0.05 in./min. on a universal testing machine in air in a high-temperature furnace. The major span was 40 mm, the minor span 10 mm. The specimens were loaded slowly at constant displacement rate to fracture.

Figure A show the critical stress intensity factor, K_{Ic}, versus temperature for a silicon nitride and a SiAlON. The K_{Ic} was determined from the crack length at the onset of rapid fracture and the load at which rapid fracture took place.

Figure B shows the K_{Ic} versus displacement rate at 1400 °C for the silicon nitride and at 1500 °C for the SiAlON.

Figure C shows the effect of holding at temperature before rapid fracture providing stress relaxation. The bars were slowly loaded at 0.00127 cm/min. until crack growth was observed as a distinct non-linearity in the load dis-placement curve. At this point the bars were unloaded rapidly. After holding at temperature for a time, Δt, the bars were reloaded at 0.127 cm/min. to fracture.

[c]Commercial silicon nitride, hot-pressed with 8% Y_2O_3(NCX 34, Norton).

[d]Nominal composition Si_5AlON_7, prepared at the National Industrial Research Institute of Kyushu.

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CREEP DATA[a] FOR TWO SILICON-BASED CERAMICS[107]
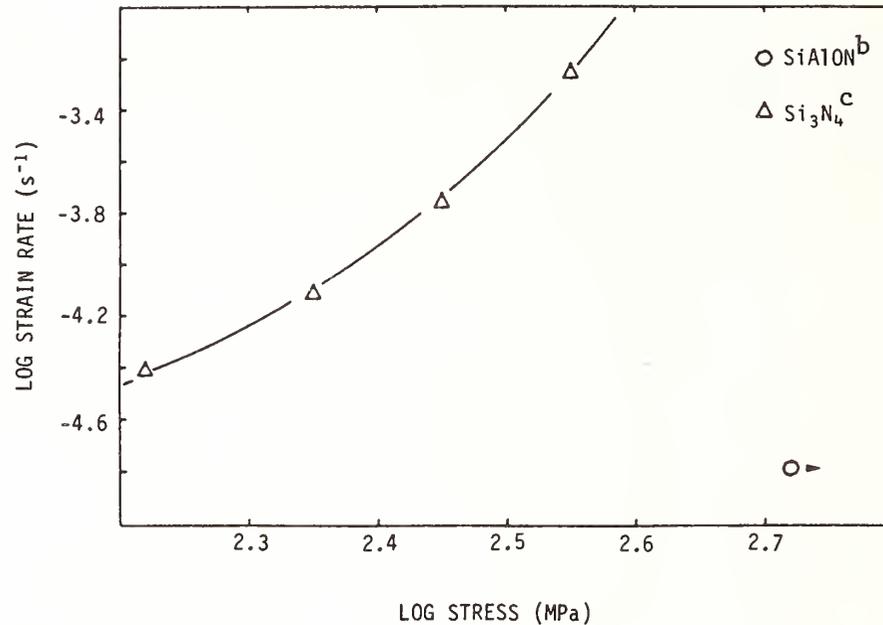
^{a}The total strain, ε_T, in a body is equal to the sum of an elastic component, ε_e, plus a creep component, ε_c. The elastic component is independent of time at constant load and the creep component increases with time. In this figure, the stress for which $\varepsilon_c = \varepsilon_e$ is plotted as a function of strain rate. Compression tests were performed on specimens 5 x 5 x 10 mm at crosshead displacement rates between 0.002 and 0.00002 in./min. The stress at which $\varepsilon_c = \varepsilon_e$ was determined from load-displacement curves and specimen dimensions.

^{b}Nominal composition Si_5AlON_7, prepared at the National Industrial Research Institute of Kyushu. Tested at 1500 °C.

^{c}Commercial silicon nitride, hot-pressed with 8% Y_2O_3 (NCX 34, Norton). Tested at 1400 °C.

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CREEP CRACK GROWTH DATA[a] FOR A SIALON MATERIAL[107]
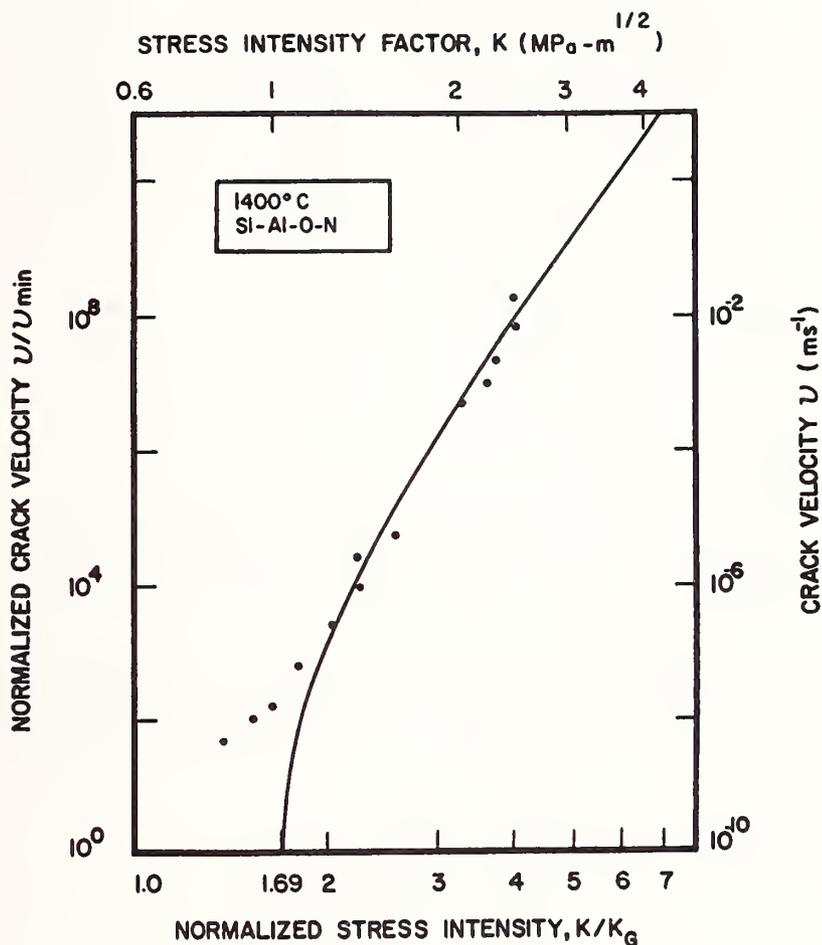
[a]Data obtained from the literature and plotted versus a theoretical prediction
(solid line). The theory assumes the existence of a grain boundary crack
growing in steady state due to externally sustained loads by stress-assisted
surface and grain-boundary self-diffusion. K_G is the critical K_I predicted
by theory and υ_{min} is the "threshold" velocity below which no crack growth
is possible. The plot of υ/υ_{min} versus K/K_G produces a single curve regard-
less of material or temperature.

==

FRACTURE TOUGHNESS DATA[a] FOR SILICON CARBIDES AND SILICON NITRIDE[107]

Specimen Type	Temperature °C	Critical Stress Intensity Factor K_{Ic} (MPa$\sqrt{m}$)	Evidence of Slow Crack Growth
SINTERED SILICON CARBIDE[b] –			
Through-notched	1500	4.06	None ⎫ ⎧but areas near
Chevron	1500	9.3[c]	None ⎬ ⎨notch were heavi-
Chevron	1600	10.9[c]	None ⎭ ⎩ly cavitated.
REACTION-BONDED SILICON CARBIDE[d] –			
Chevron	1050	12.1[c]	---
Chevron	1200	24.4[c]	Yes
Chevron	1350	23.6[c]	---
Chevron	1400	15.8[c]	Yes
HOT-PRESSED SILICON NITRIDE[e] –			
Indentation	ambient	6.0	---
Through-notched[f]	ambient	9.0	---

[a]Determined in 4-point bending under constant displacement rate.

[b]Sintered α-SiC (Carborundum).

[c]Data are marked as preliminary. Although this type of specimen forms a
sharp crack during loading, the computation of K_{Ic} is more difficult than it
is for blunt-notched specimens which do not always provide sharp cracks.

[d]KT-SiC (Carborundum).

[e]Hot-pressed Si_3N_4 with 8% Y_2O_3 (NCX 34, Norton).

[f]Specimens precracked at 1400 °C by slow bending.

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PRELIMINARY FRACTURE TOUGHNESS DATA[a] FOR A SILICON CARBIDE[b][107]

Width mm	Height mm	Temperature °C	Loading Rate in./min.	Peak Load kg	Apparent K_{Ic} MPa$\sqrt{m}$
2.023	4.971	ambient	0.02	4.1	4.66
2.024	4.972	ambient	0.02	3.60	4.76
2.021	4.963	ambient	0.02	3.85	4.51
2.018	4.971	ambient	0.02	3.95	4.39
1.991	4.972	1050	0.0002	4.87	5.41
2.009	4.974	1050	0.02	4.63	5.16
2.019	4.972	1050	0.02	4.10	4.66
1.971	4.974	1050	0.0002	4.75	5.17
1.973	4.970	1200	0.0002	6.33	6.74
1.992	4.970	1200	0.0002	4.83	5.02
2.003	4.969	1200	0.02	5.41	6.17
2.003	4.971	1200	0.02	5.62	6.03
1.989	4.974	1350	0.0002	5.68	6.13
1.983	4.972	1350	0.0002	6.74	7.32
1.995	4.974	1350	0.002	5.47	6.01
1.990	4.970	1350	0.02	4.97	5.37
2.006	4.970	1350	0.02	4.15	4.72

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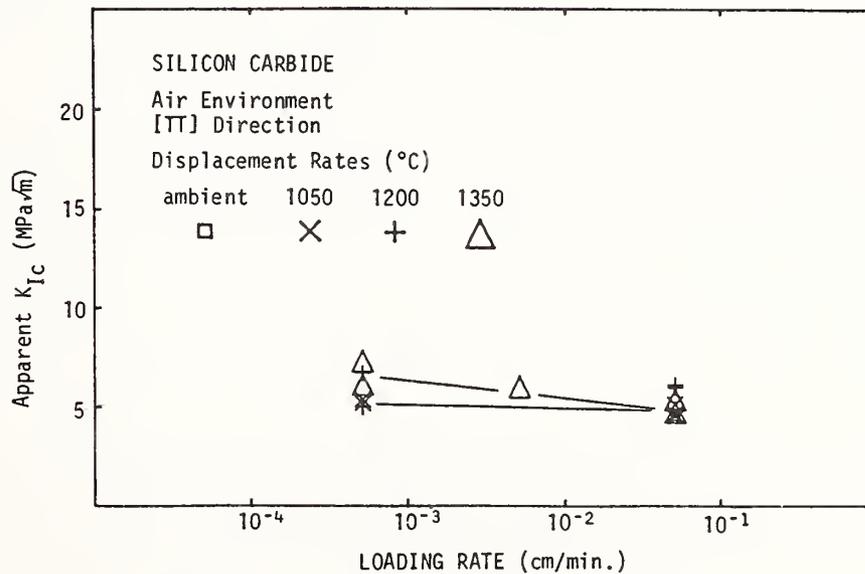
(Data Continued)

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PRELIMINARY FRACTURE TOUGHNESS DATA[a] FOR A SILICON CARBIDE[b][107], Continued

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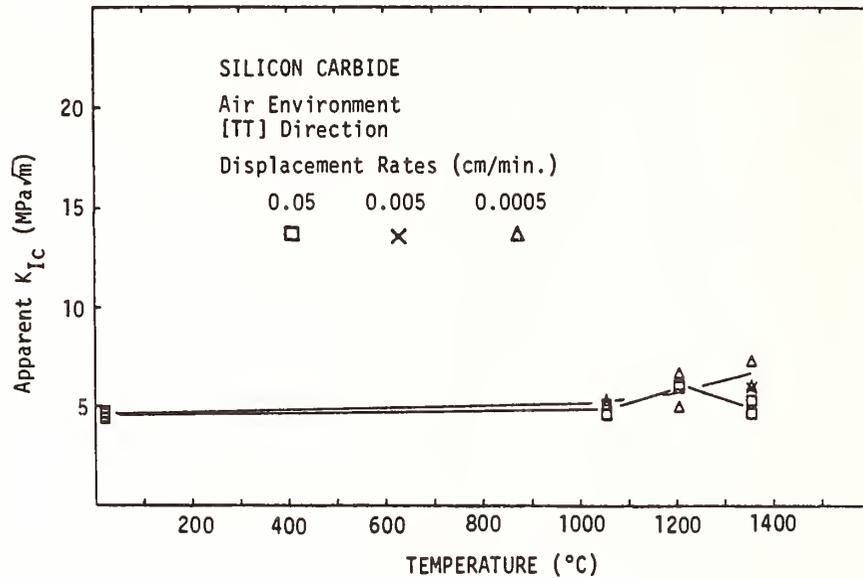
[a]Chevron-notched beam specimens were fractured under 4-point bending for
loading rates in the range 0.0002 in./min. to 0.02 in./min. and temperatures
from ambient to 1350 °C. Most specimens failed in the transgranular mode.
The apparent K_{Ic} values were determined from peak load and minimum Y, a
parameter dependent on specimens and crack geometry. True K_{Ic} values require
measurement of actual crack length at which fast fracture occurs. The figures
show apparent K_{Ic} versus temperature for given loading rates and K_{Ic} versus
loading rate for given temperatures.

[b]Commercial sintered siliconized SiC (NC-430, Norton).

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FRACTURE TOUGHNESS DATA[a] FOR SILICON CARBIDES[b] IN A SIMULATED COAL
COMBUSTION GAS[b][107]

Environment[c]	Temperature °C	Crack Orientation[d]	Loading Rate mm/min.	Critical Stress Intensity Factor, K_{Ic}[e] MPa$\sqrt{m}$
SINTERED SILICON CARBIDE[b]				
Air	25	TP	0.5	7.8
		TT	0.5	6.1
		TT	0.005	6.3
Mixed Gas	1200	TP	0.5	9.1
		TP	0.5	10.6
		TP	0.005	9.3
		TP	0.005	8.7
		TT	0.5	9.0
		TT	0.005	7.2
Mixed Gas	1500	TT	0.5	6.1
		TT	0.005	5.7
		TT	0.001	5.3
SILICONIZED SILICON CARBIDE[b]				
Mixed Gas	1050	TT	0.5	6.1
		TT	0.005	15.7
Mixed Gas	1200	TT	0.5	12.7
		TT	0.005	22.8
Mixed Gas	1350	TT	0.5	12.7
		TT	0.005	18.5

[a] Fracture testing was conducted in a furnace designed to provide controlled gaseous environments at elevated temperatures. Chevron-notched specimens were tested in 4-point bending. Before testing the specimens were equilibrated at temperature and exposed to the flowing gas environment for 4 hours. Tests were conducted at a constant displacement rate to failure. The rates were 0.5 and 0.005 mm/min. The lower rate indicates the presence of subcritical crack growth and/or creep shielding at the crack tip.

[b] Sintered α-SiC (Hexoloy SA, Carborundum; sintered siliconized SiC (Hexoloy KT, Carborundum).

[c] The combustion gas environment was a mixture of 75% N_2, 7% O_2, 11% CO_2, 7% H_2O, and 0.1% SO_2.

[d] These materials were supplied as plate billets, 12 x 50 x 50 mm. Two test orientations were used for the specimens, TP and TT where T denotes a transverse orientation, P a plate direction. For both cases, the crack propagated on a transverse plane (T), but in the TP orientation the propagation direction was perpendicular to the plate, and in the TT orientation it was parallel to the plate.

[e] Determined from the maximum fracture load assuming the crack went catastrophic at the minimum of the K_I versus crack length curve, valid only when subcritical crack growth is minimal.

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EFFECT OF A SIMULATED COAL COMBUSTION GAS[a] ON THE FRACTURE TOUGHNESS[b] OF A SILICON CARBIDE[c][107]

REACTION-BONDED SILICON CARBIDE
Displacement Rates, mm/min.

	0.5	0.005	
Air	◇	△	
Mixed Gas	◆	▲	
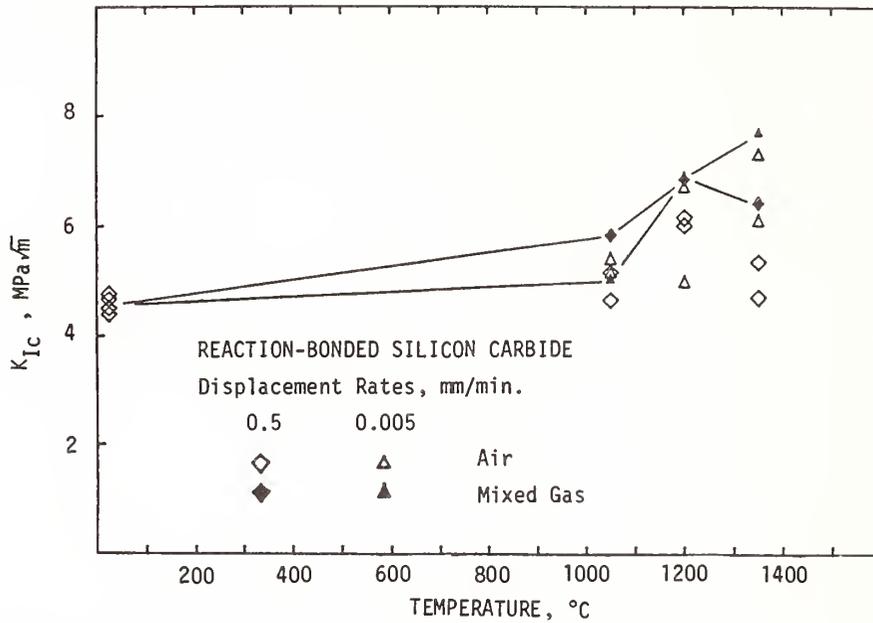
Displacement Rate, mm/min.	K_{Ic} , MPa$\sqrt{m}$		
	1050 °C	1200 °C	1350 °C
0.5	5.96	7.01	6.57
	5.54	7.75	7.75
0.005	5.17	6.98	7.89
	6.89	7.19	7.27
Mean and Standard Deviation	5.9±0.7	7.2±0.4	7.4±0.6

[a]Fracture testing was conducted in a furnace designed to provide controlled gaseous environments at elevated temperatures. Before testing the specimens were equilibrated at temperature and exposed to the flowing gas environment for 4 hours. The combustion gas environment was a mixture of 75% N_2, 7% O_2, 11% CO_2, 7% H_2O and 0.1% SO_2.

[b]Chevron-notched specimens were tested in 4-point bending. See Section B.3.2.165, footnotes a and e, for other test information.

[c]Mixed-grain size reaction-bonded SiC (NC 430, Norton).

FLEXURE STRENGTH[a] OF SILICON CARBIDE[b] UNDER STATIC LOAD AS A FUNCTION OF TIME [107]
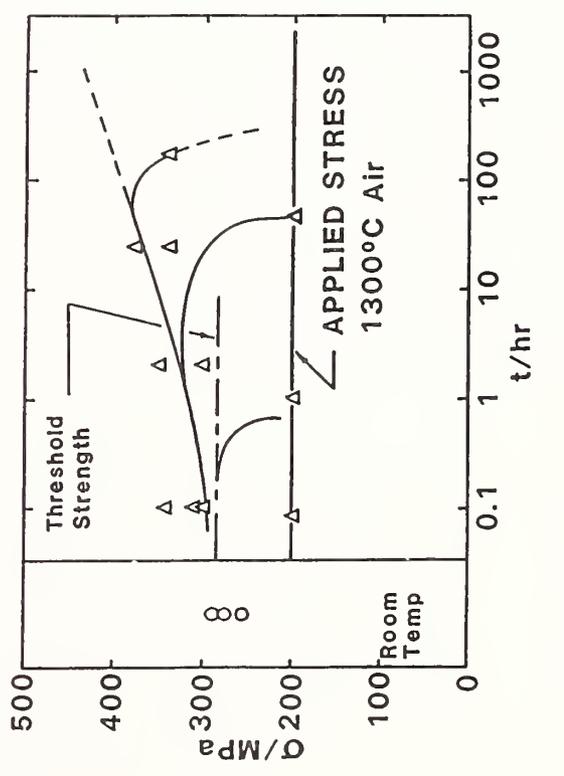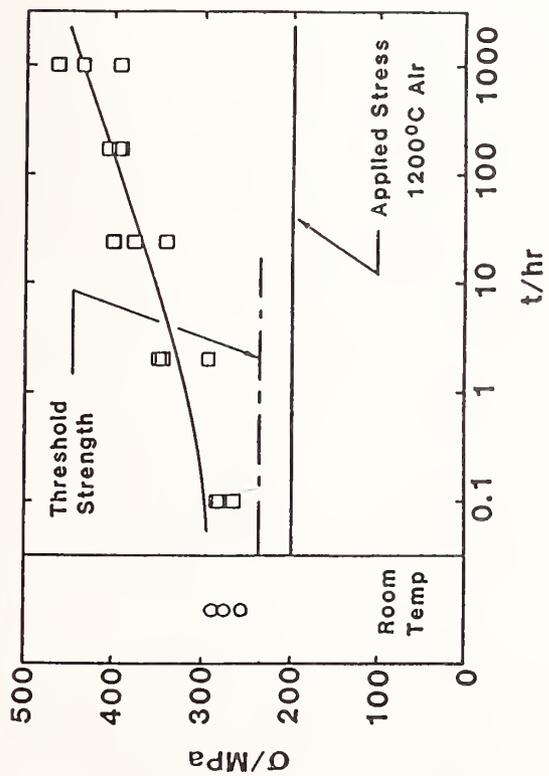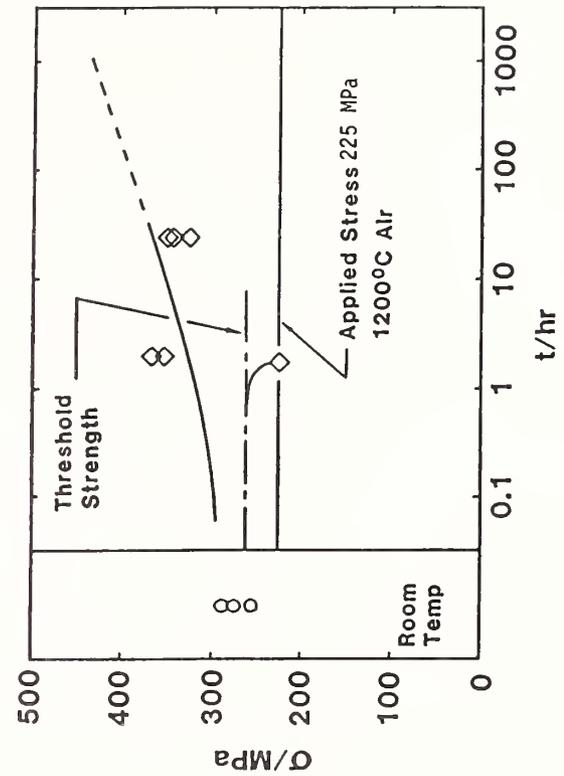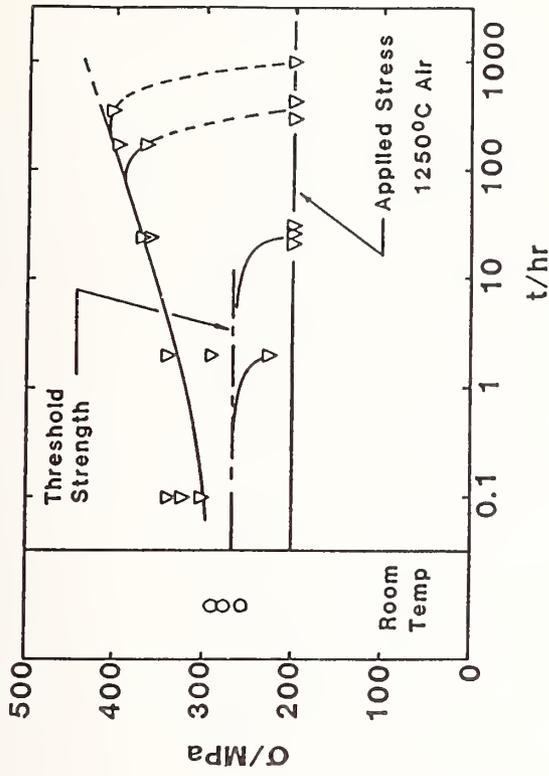
(Data Continued)

FLEXURE STRENGTH[a] OF SILICON CARBIDE[b] UNDER STATIC LOAD AS A FUNCTION OF TIME[107], Continued
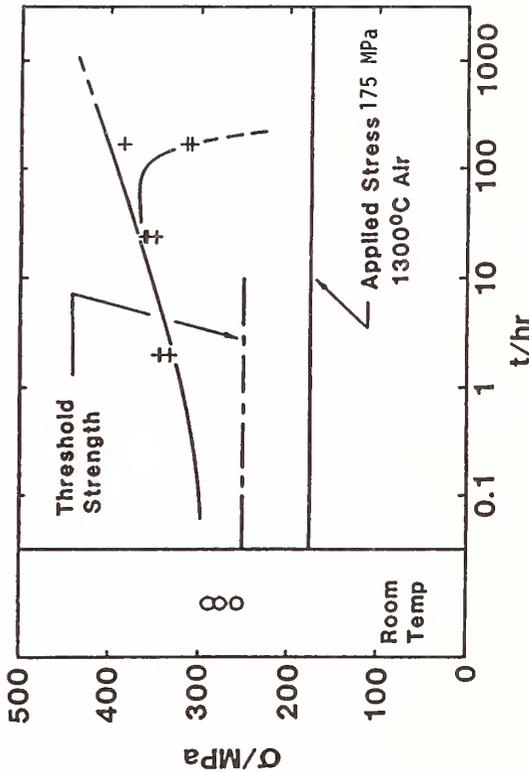
[a] As-machined specimens were tested at ambient temperature in 4-point bending and a medium strength of 274 MPa was determined from 4 specimens. An applied stress of 200 MPa was chosen and temperature testing performed to find a suitable temperature regime for further testing. The applied stress of 200 MPa (and later 225 and 175 MPa) was used at 1200 °C, 1250 °C, and 1300 °C for predetermined periods of time. If specimen failure did not occur in the allotted time period, specimens were broken and residual strength recorded. Delayed failures are plotted on the applied stress line in the figures. At short times there is an apparent strength threshold below which failure is delayed, and above which subsequent strength generally increases with time. At very long times, a rapid strength degradation occurs. The degradation moves to shorter times with increasing temperature or increasing applied stress.

[b] A sintered α-SiC (Hexoloy SA, Carborundum).

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CRACK GROWTH DATA[a] FOR A SILICON CARBIDE[b][107]

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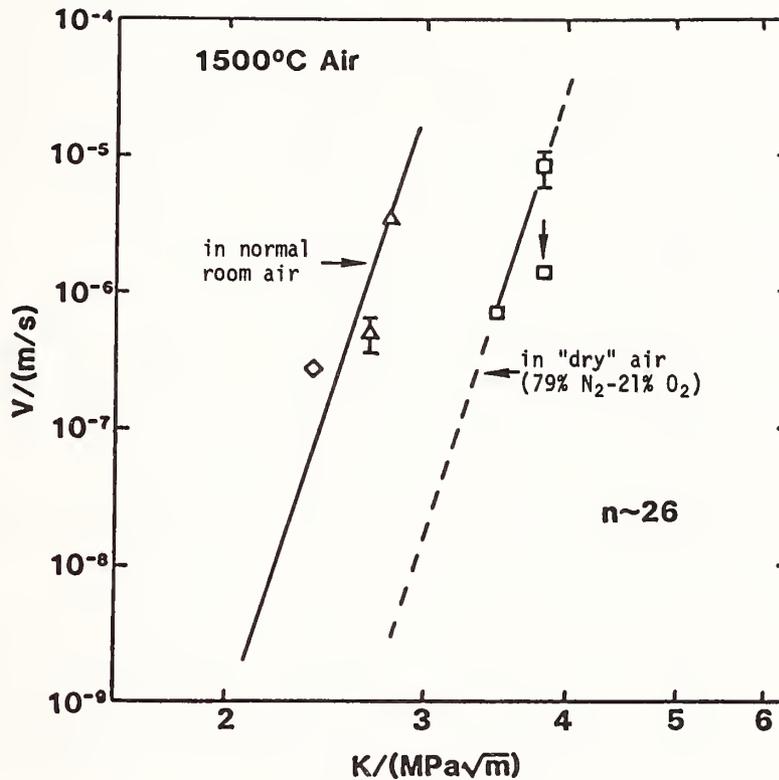
[a] Double-torsion fracture specimens were static loaded for a fixed time at 1500 °C. Crack advance was monitored with optical microscopy at ambient temperature following the high-temperature exposure. A sharp crack was introduced initially by extending a sawn-in notch at ambient temperature via several microhardness indentation cracks ahead of the notch. Such a crack will propagate at elevated temperature at an average velocity of about 10^{-6} m/s. When the crack is loaded a second time at elevated temperature without resharpening the crack, the average velocity is slowed and finally stops. This deceleration behavior is indicated in the figure by the data point with the arrow on the right-hand curve of crack velocity versus stress intensity factor.

[b] A sintered α-SiC (Hexoloy SA, Carborundum).

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FRACTURE TOUGHNESS[a] OF A SILICON CARBIDE[b] IN AIR AND IN A SIMULATED COAL COMBUSTION GAS[c][107]

Temperature °C	Loading Method	a_o/W [d]	K_{Ic} (MPa$\sqrt{m}$) Air 0.005 mm/min.	Air 0.5 mm/min.	Combustion Gas[c] 0.005 mm/min.	Combustion Gas[c] 0.5 mm/min.
25	4-point	0	4.79	3.96		
	4-point	0.2	3.44	3.61		
	4-point	0.2	3.33	3.49		
	4-point	0.2	3.43	3.74		
	3-point	0.2	3.36			
1050	4-point	0	13.2	7.03		
	4-point	0.2	9.74	8.01	15.70	6.13
	4-point	0.2	11.02			
	4-point	0.2	13.89			
	3-point	0.2	13.86	6.18		
1200	4-point	0.2			22.8	12.72
	3-point	0.2	20.6	8.34		
1350	4-point	0	23.8			
	4-point	0.2	15.7	9.72		
	4-point	0.2			18.46	12.74
	3-point	0.2	14.05	15.27		

[a] Chevron-notched flexure specimens were tested in 4- and 3-point bending at two displacement rates, 0.5 and 0.005 mm/min.

[b] A fine-grain, reaction-bonded, siliconized SiC (Hexoloy KX-01, Carborundum).

[c] The tests were performed in a furnace designed to provide controlled gaseous environments at elevated temperatures. Before testing the specimens were equilibrated at temperature and exposed to the flowing gas environments for 4 hours. The combustion gas environment was a mixture of 75% N_2, 7% O_2, 11% CO_2, 7% H_2O and 0.1% SO_2.

[d] a_o is the position of the chevron apex with respect to the tensile side of the specimen.

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FLEXURAL STRENGTH[a] AS A FUNCTION OF PROBABILITY OF FAILURE[b] FOR TWO
SILICON-BASED CERAMICS[15]

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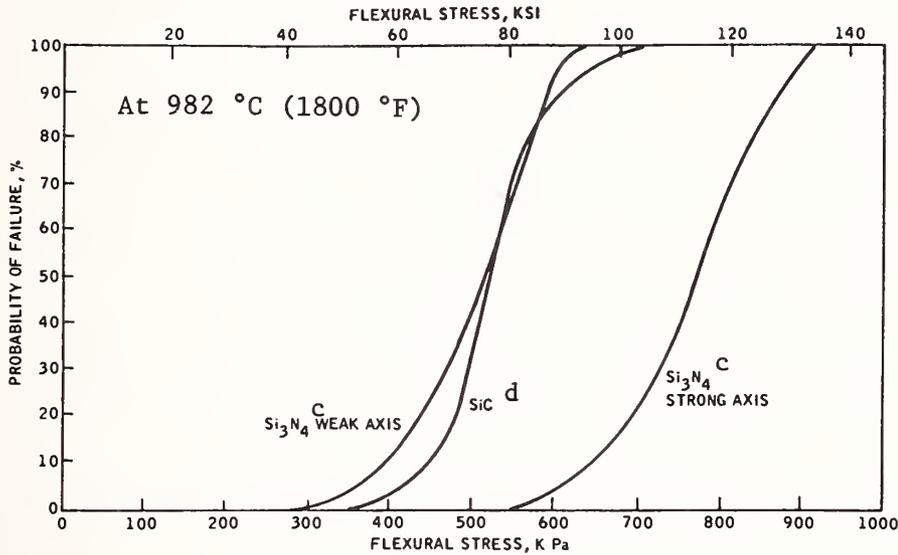
[a] Data from the literature.

[b] Constructed from the Weibull statistics (distribution of percent cumulative
failures versus stress).

[c] Hot-pressed silicon nitride (HS-130, Norton).

[d] Hot-pressed silicon carbide (NC-203, Norton).

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            a Coal-Oil Fuel Combustor

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COMPARISON OF THERMAL CONDUCTIVITY[a] FOR HIGH-ALUMINA REFRACTORIES[9]
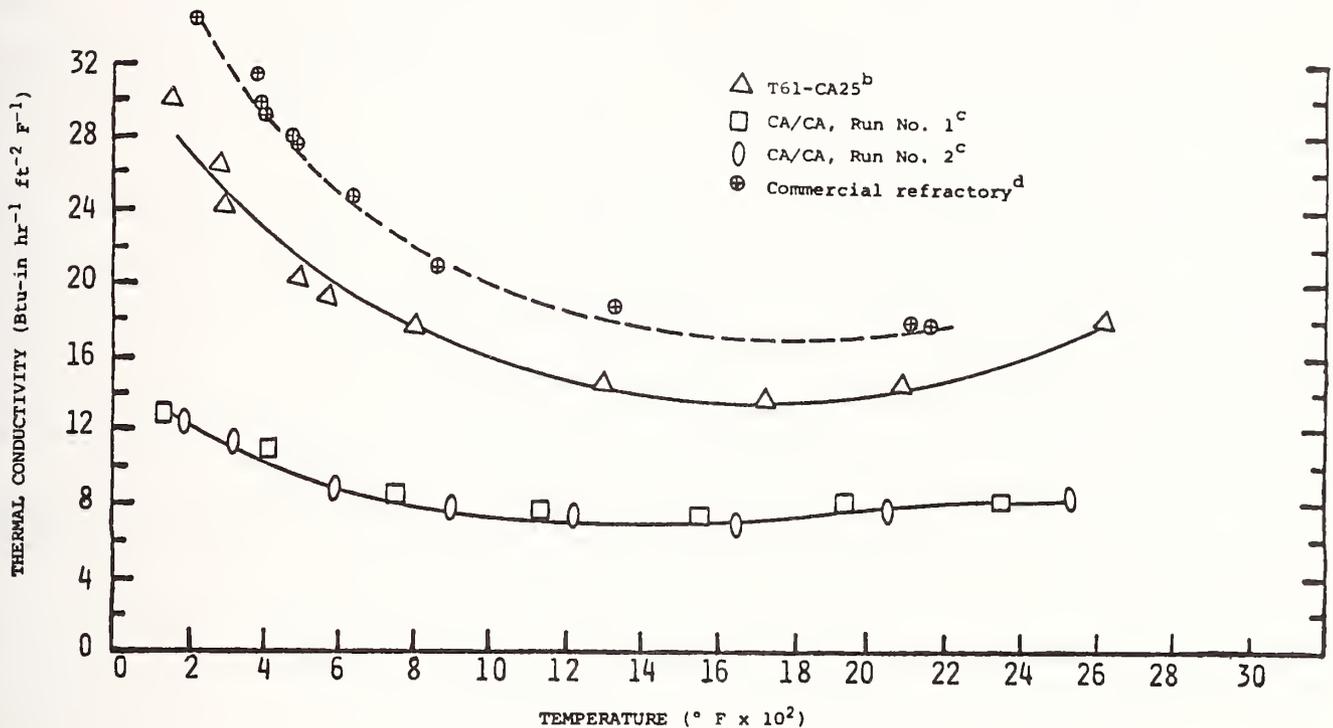
a Duplicate determinations were made of each composition at each temperature.

b T61-CA25 is a 95% alumina refractory concrete, a generic preparation; bulk density 157-163 lb/ft^3.

c CA/CA is a 78% alumina refractory concrete, prepared by Tuscaloosa Metallurgy Research Center; bulk density 132 lb/ft^3; aggregate is made from sintered calcium aluminate cement.

d The commercial refractory is 95% alumina refractory concrete; bulk density 157-163 lb/ft^3. All of the above refractories had about the same cement/aggregate ratio and the same aggregate size distributions.

===

PHYSICAL PROPERTIES[a] OF REFRACTORIES[b] EXPOSED TO THE CONDITIONS[c] IN COAL GASIFICATION PILOT PLANTS[12,90]

Refractory (Brand Name)	Exposure[d]	Weight Change %	Apparent Porosity[a] %	% Change	Apparent Specific Gravity[a]	% Change	Bulk Density[a] g/cm³	% Change
Two exposures								

Two exposures in gasifier off-gas (SYNTHANE): reducing gas, H_2O, CO_2, CO, H_2, H_2S, ash and char (small amounts); 800-1500 °F (average 1290 °F), 600 psi, operational time 781.8 hours (first exposure), 181 hours (second exposure).

Refractory (Brand Name)	Exposure[d]	Weight Change %	Apparent Porosity[a] %	% Change	Apparent Specific Gravity[a]	% Change	Bulk Density[a] g/cm³	% Change
90+% Al_2O_3 dense castable, CA bond[e] (Castolast G)	U		27.5		3.63		2.62	
	E1	+0.4	27.7	+ 0.7	3.59	− 1.1	2.59	− 1.1
	U		29.9		3.69		2.59	
	E2	−5.0	28.7	− 4.0	3.66	− 0.8	2.61	+ 0.8
60% Al_2O_3 dense castable, CA bond (Mizzou Castable)	U		25.1		3.00		2.25	
	E1	−0.4	27.7	+10.4	3.11	+ 3.7	2.27	+ 0.9
	U		25.1		3.04		2.28	
	E2	−1.5	24.4	− 2.8	3.07	+ 1.0	2.32	+ 1.8
90+% Al_2O_3 light castable, CA bond (Purolite 30)	U		46.6		2.65		1.42	
	E1	−3.3	Disintegrated, could not be tested					
90+% Al_2O_3 light castable, CA bond (Greencast-97L)	U		49.9		2.82		1.41	
	E1	+2.0	54.9	+10.0	3.17	+12.4	1.43	+ 1.4
54% Al_2O_3 dense castable, CA bond (Super Brikcast A)	U		24.7		2.88		2.17	
	E2	−1.8	25.34	+ 2.6	2.94	+ 2.1	2.19	+ 0.9
54% Al_2O_3 light insulating castable, CA bond (Litecast 75-28)	U		37.9		2.36		1.51	
	E1	−1.4	55.0	+45.1	3.18	+34.8	1.43	− 5.3
	U		40.7(24.7)[f]		1.98(2.99)[f]		1.17(2.17)[f]	
	E2	−5.3	61.3	+50.6(+148)[f]	3.12	+57.6(+8.3)[f]	1.21	+ 3.4(−44.2)[f]
70% Al_2O_3 (mullite), vitreous bond high-fired brick (Mul-8)	U		17.2		3.05		2.53	
	E1	−0.2	19.1	+11.0	3.08	+ 1.0	2.49	− 1.6
	U		18.9		3.07		2.49	
	E2	−1.1	19.9	+ 5.8[j]	3.10	+ 1.0	2.48	− 0.4
85% Al_2O_3 phosphate bond, dense fired brick (Chemal 85 B)	U		15.7		3.36		2.83	
	E1	0	16.8	+ 7.0	3.51	+ 4.5	2.91	+ 2.8
	U		16.6		3.43		2.83	
	E2	−0.9	19.7	+18.7	3.49	+ 1.8	2.80	− 1.1
90+% Al_2O_3 self-bonded dense fired brick (Kricor)	U		16.7		3.54		2.95	
	E1	−0.2	18.7	+12.0	3.58	+ 1.1	2.91	− 1.4
	U		17.5		3.55		2.93	
	E2	−0.7	19.2	+ 9.7	3.58	+ 2.3[j]	2.88	− 1.7
99+% Al_2O_3 self-bonded dense fired brick (H-W Corundum)	U		19.0		3.74		3.03	
	E1	+0.2	20.2	+ 6.3	3.80	+ 1.6	3.03	0
	U		20.3		3.77		3.02	
	E2	−0.6	21.7	+ 6.9	3.83	+ 1.6	2.94	− 1.0[j]
77% Al_2O_3 self-bonded light insulating fired brick (B&W Insalcor)	U		46.5		2.81		1.53	
	E1	+0.4(+0.04)[f]	61.4	+32.0	1.31	−53.4	1.29	−15.7
98+% Al_2O_3 self-bonded light insulating fired brick (Alfrax B 101)	U		60.7		3.74		1.47	
	E1	+1.3	59.4	− 2.1	2.84	−24.1	1.56	+ 6.1
99+% Al_2O_3 self-bonded dense fused-cast brick (Monofrax A)	U		0.8		3.86		3.83	
	E1	−0.3	4.7	+488[g]	3.89	+ 0.8	3.71	− 3.1
	U		0.09		3.82		3.79	
	E2	−0.2	3.14	+3388[g]	3.87	+ 1.3	3.75	− 1.1
SiC, silicon nitride bonded, dense fired brick (Refrax 20)[h]	U		15.1		3.12		2.64	
	E1	+3.1	19.2	+27.2	2.88	− 7.7(−8.3)[f]	2.07	−21.6
	U		14.9		3.18		2.67	
	E2	−1.3	18.16	+21.9	2.88	− 9.4	2.36	−11.6
Alumina-zirconia-silica fused-cast brick (AZS)	U		0.09		3.88		3.85	
	E2	−1.1	11.58	+12,767[g]	3.95	+ 1.8	3.49	− 9.4
60% Al_2O_3 tar-impregnated vitreous fired brick (Ufala TI)	U		14.2[k]		2.70		2.69	
	E2	−0.5	15.84	+11.6	2.98	+10.4	2.50	− 7.1
refired Ufala TI[i]	U		14.5[k]		2.96		2.53	
	E2	−0.6	16.6	+14.5	2.98	+ 0.7	2.49	− 1.6
45-50% Al_2O_3 clay vitreous bond, high-fired super-duty brick (KX-99)	U		12.3		2.52		2.29	
	E2	−0.9	16.5	+34.2	2.70	+ 7.1	2.26	− 1.3
refired KX-99[i]	U		11.5		2.62		2.32	
	E2	−0.7	15.0	+30.4	2.70(2.80)[f]	+ 3.1	2.30	− 0.9
88% Al_2O_3 phosphate-bonded mortar (Chemal 111)	U		--		--		--	
	E1	+0.2	--		--		--	

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(Table Continued)

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PHYSICAL PROPERTIES[a] OF REFRACTORIES[b] EXPOSED TO THE CONDITIONS[c] IN COAL GASIFICATION PILOT PLANTS[12,90], Continued

Refractory (Brand Name)	Expo-sure[d]	Weight Change %	Apparent Porosity[a] %	% Change	Apparent Specific Gravity[a]	% Change	Bulk Density[a] g/cm^3	% Change
Two exposures in gasifier off-gas (SYNTHANE), continued								
60% Al$_2$O$_3$ phosphate-bonded ramming mix (Wasp #60)	U		14.6		3.08		2.63	
	E1	−1.4	21.2	+45.2	2.84	− 7.8	2.24	−14.8
90% Al$_2$O$_3$ phosphate-bonded ramming mix (Brikram 90)	U		16.3		3.52		2.95	
	E1	−1.9	15.6	− 4.3	3.44	− 2.3	2.90	− 1.7
	U		14.6		3.50		2.99	
	E2	−0.8	17.2	+17.8	3.56	+ 1.7	2.95	− 1.3
52% Al$_2$O$_3$ light castable, CA bond (Litecast 80)	U		34.6		2.57		1.68	
	E1	−0.1	48.7	+40.8	3.24	+26.1	1.66	− 1.2
70+% Al$_2$O$_3$ vitreous fired brick (Hi Lumite 70 D)	U		24.3		3.14		2.34	
	E1	−2.0	26.8	+10.3	3.27	+ 4.1	2.39	+ 2.1
85% Al$_2$O$_3$ phosphate-bonded fired brick (Altex 85-B)	U		12.0		3.36		2.96	
	E1	−0.2	17.1	+42.5	3.51	+ 4.5	2.91	− 1.7
	U		12.0		3.36		2.96	
	E2	−0.7	21.65	+80.4	3.53	+ 5.1	2.76	− 6.8
80+% Al$_2$O$_3$ phosphate-bonded fired brick (Alumex P-8)	U		13.8		3.32		2.86	
	E1	−0.1	21.8	+58.0	3.51	+ 5.7	2.74	− 4.2
	U		13.8		3.32		2.86	
	E2	−0.8	18.25	+32.3	3.37	+ 1.5	2.76	− 3.5
90+% Al$_2$O$_3$ phosphate-bonded castable (Resco Cast AA-22)	U		23.9		3.53		2.69	
	E1	−−	26.6	+11.3	3.09	−12.5	2.27	−15.6
	U		23.6		3.47		2.66	
	E2	−2.2	27.3	+15.7	3.65	+ 5.2	2.65	− 0.4
SiC, oxynitride bond, coupons/ bricks (Crystolon 63)	U		15.6		3.04		2.55	
	E2	−1.4	19.8	+26.9	2.98	− 2.0	2.39	− 6.3
73% Al$_2$O$_3$ chemical bond ramming mix (Blu Ram)	U		18.2		3.07		2.52	
	E2	−1.3	20.68	+13.6	3.16	+ 3.0	2.51	− 0.4
73% Al$_2$O$_3$ chemical bond ramming mix (Blu Ram H.S.)	U		21.9		3.12		2.44	
	E2	−1.8	21.92(31.92)[f]	+0.1	3.11	− 0.3	2.43	− 0.4
One exposure in the fluidized bed of the gasifier (SYNTHANE): reducing gas as in above plus coal fines, ash and char; 800-1650 °F(average 1434 °F), 600 psi, operational time 181 hours.								
90+% Al$_2$O$_3$ dense castable, CA bond (Castolast G)	U		30.0		3.73		2.61	
	E	−5.2	27.6	− 8.0	3.65	− 2.1	2.70	+ 3.5
60% Al$_2$O$_3$ dense castable, CA bond (Mizzou Castable)	U		25.3		3.08		2.30	
	E	−1.2	25.7	+ 1.6	3.07	− 0.3	2.28	− 0.9
90+% Al$_2$O$_3$ light castable, CA bond (Greencast-97L)	U		49.6		2.78		1.40	
	E	−3.8	61.0	+23.1	3.74	+34.5	1.46	+ 4.3
54% Al$_2$O$_3$ dense castable, CA bond (Super Brikcast A)	U		24.9		2.87		2.16	
	E	−0.8	25.4	+ 2.0	2.94	+ 2.43	2.20	+ 1.9
54% Al$_2$O$_3$ light insulating castable, CA bond (Litecast 75-28)	U		44.7		−−		1.24	
	E	−3.2	58.7	+31.3	3.14	+40.0	1.30	+ 4.8
70% Al$_2$O$_3$ (mullite), vitreous bond high-fired brick (Mul-8)	U		18.0		3.06		2.51	
	E	−0.6	19.8	+ 6.1[j]	3.09	+ 1.0	2.48	− 1.2
85% Al$_2$O$_3$ phosphate bond, dense fired brick (Chemal 85 B)	U		16.2		3.43		2.87	
	E	−0.2	19.2	+18.5	3.45	+ 0.6	2.78	− 3.1
90+% Al$_2$O$_3$ self-bonded dense fired brick (Kricor)	U		17.1		3.53		3.03	
	E	−0.7	17.8	+ 4.1	3.59	+ 1.7	2.95	− 2.6
99+% Al$_2$O$_3$ self-bonded dense fired brick (H-W Corundum)	U		19.9		3.77		3.02	
	E	−0.3	20.9	+ 5.0	3.79	+ 0.5	3.00	− 0.7
77% Al$_2$O$_3$ self-bonded light insu- lating fired brick (B&W Insalcor)	U		47.5		2.81		1.47	
	E	−1.8	54.5	+14.5	3.22	+14.6	1.47	0
98+% Al$_2$O$_3$ self-bonded light insu- lating fired brick (Alfrax B 101)	U		60.4		3.75		1.48	
	E	−0.5	59.0	− 2.3	3.88	+ 3.5	1.56	+ 5.4
99+% Al$_2$O$_3$ self-bonded dense fused- cast brick (Monofrax A)	U		0.9(0.09)[f]		3.84		3.80	
	E	−0.03	3.8	+322	3.96	+ 3.1	3.81	+ 0.3
SiC, silicon nitride bonded, dense fired brick (Refrax 20)[h]	U		15.6		3.14		2.65	
	E	−1.1	30.8	+33.3[j]	2.78	−11.5	1.92	−27.6
Zirconium silicate, vitreous bond coupon (Zircon)	U		16.2		4.59		3.85	
	E	−0.1	16.9	+ 4.3	4.61	+ 0.4	3.83	− 0.5

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(Table Continued)

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PHYSICAL PROPERTIES[a] OF REFRACTORIES[b] EXPOSED TO THE CONDITIONS[c] IN COAL GASIFICATION PILOT PLANTS[12,90], Continued

Refractory (Brand Name)	Exposure [d]	Weight Change %	Apparent Porosity[a] %	% Change	Apparent Specific Gravity[a]	% Change	Bulk Density[a] g/cm³	% Change
One exposure in the fluidized bed of the gasifier (SYNTHANE), continued								
60% Al_2O_3 tar-impregnated vitreous fired brick (Ufala TI), refired[l]	U		14.2[k]		2.70		2.69	
	E	-0.2	15.65	+10.2	2.97	+10.0	2.58	- 4.1
45-50% Al_2O_3 high-fired super-duty brick (KX-99)	U		11.6		2.61		2.31	
	E	-0.5	14.9	+28.5	2.70	+ 3.5	2.30	- 0.4
60% Al_2O_3 phosphate-bonded ramming mix (Wasp # 60)	U		18.0		2.91		2.38	
	E	-1.4	21.5	+23.9[j]	2.81	- 3.4	2.24	- 5.9
90% Al_2O_3 phosphate-bonded ramming mix (Brikram 90)	U		15.4		3.55		2.92	
	E	-0.3	13.3	-13.6	3.50	- 1.4	3.03	+ 3.8
70+% Al_2O_3 vitreous fired brick (Hi Lumite 70 D)	U		21.5		2.77		2.17	
	E	-1.3	26.2	+26.5[j]	2.75	- 0.7	2.03	- 6.5
85% Al_2O_3 phosphate-bonded fired brick (Altex 85-B)	U		16.3		3.45		2.89	
	E	-0.2	21.3	+30.8	3.51	+ 2.0[j]	2.76	- 4.5
80+% Al_2O_3 phosphate-bonded fired brick (Alumex P-8)	U		15.8		3.34		2.81	
	E	-1.5	17.9	+13.3	3.35	+ 0.3	2.94(2.76)[f]	+ 4.6(-1.8)[f]
90+% Al_2O_3 phosphate-bonded castable (Resco Cast AA-22)	U		27.2		3.54		2.58	
	E	-0.5	23.4	-14.0	3.65	+ 3.1	2.80	+ 8.5
SiC, oxynitride bond, coupons/bricks (Crystolon 63)	U		14.7		3.05		2.60	
	E	-0.4	31.1	+111.7	2.86	- 6.2	2.19	-15.8
Three exposures in gasifier off-gas (Conoco, CO_2 Acceptor): 48 H_2, 23 H_2O, 12 CH_4, 8.5 CO, 6 CO_2, 2.5 N_2(all vol. %); 1500 °F, 150 psig, ∿800 hours (exposure 1), 1000-2400 hours (exposure 2), 740-1450 hours (exposure 3).								
90+% Al_2O_3 dense castable, CA bond (Castolast G)	U		27.5		3.63		2.62	
	E1	-1.0	27.5	0	3.64	+ 0.3	2.64	+ 0.8
	U		27.5		3.63		2.62	
	E2	-0.33	31.7	+15.	3.83	+ 5.5	2.61	- 0.4
	U		31.2		3.82		2.64	
	E3	-0.32	25.6	-18.	3.49	- 8.6	2.60	- 1.5
60% Al_2O_3 dense castable, CA bond (Mizzou Castable)	U		25.1		3.00		2.25	
	E1	-1.2(-1.6)[f]	22.3	-11.	2.98	- 0.7	2.31	+ 2.7
	U		25.1		3.00		2.35	
	E2	+0.02	23.7	- 5.5	2.98	- 0.7	2.28	+ 2.7[j]
	U		24.6		2.98		2.25	
	E3	-0.84	26.5	+ 8.	3.06	+ 2.7	2.25	0
90+% Al_2O_3 light castable, CA bond (Purolite 30)	U		46.6		2.65		1.42	
	E1	-4.5	52.9	+14.	3.05	+15.	1.42(1.43)[f]	0(+0.7)[f]
	U		44.6		2.65		1.42	
	E2	-2.1	55.6	+24.	3.04	+15.	1.35	- 5.0
90+% Al_2O_3 light castable, CA bond (Greencast-97L)	U		49.9		2.82		1.41	
	E1	-0.9	30.1	-40.	2.04	-27.	1.43(1.94)[f]	+ 0.7[j](+37.6)[f]
	U		49.9		2.82		1.41	
	E2	+1.15	51.5	+ 3.2	2.99	+ 6.0	1.45	+ 2.8
	U		50.8		2.69		1.26	
	E3	-4.1	61.1	+20.	3.74	+39.	1.41	+12.(+1.2)[f]
54% Al_2O_3 dense castable, CA bond (Super Brikcast A)	U		25.5		2.85		2.18	
	E3	-0.74	28.0	+10.	2.91	+ 2.1	2.13	- 2.3
54% Al_2O_3 light insulating castable, CA bond (Litecast 75-28)	U		41.3		2.33		1.37	
	E1	-2.1	52.0	+25.9	3.04(3.10)[f]	+30.5(+33.0)[f]	1.46	+ 6.6
	U		37.9		2.36		1.51	
	E2	-0.60	51.6	+36.	3.00	+27.	1.45	- 3.9
	U		48.9		2.34		1.20	
	E3	-1.6	60.5	+24.	3.01	+29.	1.19	- 0.8
70% Al_2O_3 (mullite), vitreous bond high-fired brick (Mul-8)	U		17.2(18.2)[f]		3.05(3.06)[f]		2.53(2.50)	
	E1	-0.2	19.2	+12.(+5.5)	3.09	+ 1.3(+1.0)	2.50	- 1.2(0)
	U		17.2		3.05		2.53	
	E2	+0.05	17.1	- 0.6	3.00	- 1.6	2.53	0
	U		19.0		3.07		2.49	
	E3	-0.16	19.6	+ 3.2	3.08	+ 0.3	2.48	- 0.4

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(Table Continued)

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PHYSICAL PROPERTIES[a] OF REFRACTORIES[b] EXPOSED TO THE CONDITIONS[c] IN COAL GASIFICATION PILOT PLANTS[12,90], Continued

Refractory (Brand Name)	Exposure[d]	Weight Change %	Apparent Porosity[a] %	% Change	Apparent Specific Gravity[a]	% Change	Bulk Density[a] g/cm³	% Change
Three exposures in gasifier off-gas (Conoco, CO$_2$ Acceptor), continued								
85% Al$_2$O$_3$ phosphate bond, dense fired brick (Chemal 85 B)	U		15.7(17.6)[f]		3.36(3.44)[f]		2.83(2.84)[f]	
	E1	-0.3(-0.2)[f]	16.0	+ 1.9(-9.1)	3.50	+ 4.2(+1.7)	2.94	+ 3.9(+3.5)
	U		15.7		3.36		2.83	
	E2	-0.07	14.5	- 7.6	3.48	+ 3.6	2.64	- 6.7
	U		15.0		3.45		2.94	
	E3	-0.21	16.2	+ 8.0	3.49	+ 1.2	2.92	- 0.7
90+% Al$_2$O$_3$ self-bonded dense fired brick (Kricor)	U		16.7(15.1)[f]		3.54(3.51)[f]		2.95(2.99)[f]	
	E1	0(-0.3)[f]	17.6(16.7)	+ 5.4(+10.6)	3.55	+ 0.3(+1.1)	2.96(2.95)	+ 0.3(-1.3)
	U		16.7		3.54		2.95	
	E2	-0.5	17.8	+ 6.6	3.53	- 0.3	2.91	- 1.3
99+% Al$_2$O$_3$ self-bonded dense fired brick (H-W Corundum)	U		19.0		3.74(3.64)[f]		3.03(2.95)[f]	
	E1	-0.1(-0.3)[f]	20.3	+ 6.8	3.77	+ 0.8(+3.6)	3.00	- 1.0(+1.7)
	U		19.0		3.74		3.03	
	E2	+0.11	20.6	+ 8.4	3.78	+ 1.1	3.00	- 1.0
	U		24.3		3.76		3.00	
	E3	-0.12	21.0	-13.	3.77	+ 0.3	2.98	- 0.7
77% Al$_2$O$_3$ self-bonded light insulating fired brick (B&W Insalcor)	U		46.5(50.5)[f]		2.81(2.77)[f]		1.53(1.35)[f]	
	E1	-10.9(-1.0) -1.24[f]	54.6(56.1)	+17.4(+11.1)	2.82(2.94)	+ 0.4(+5.1)[j]	1.28(1.29)	-16.3(-4.4)
	U		46.5		2.81		1.53	
	E2	+0.85	55.2	+19.	2.82	+ 0.4	1.23	-20.
	U		48.7		2.81		1.44	
	E3	-1.34	55.7	+14.	3.28	+17.	1.45	+ 0.7
98+% Al$_2$O$_3$ self-bonded light insulating fired brick (Alfrax B 101)	U		60.7(57.7)[f]		3.74(3.70)[f]		1.47(1.57)[f]	
	E1	-0.1(-1.2)[f]	57.4(52.5)	- 5.4(-9.0)	3.75(3.02)	+ 0.3(-18.4)	1.59(1.43)	+ 5.4[j](-8.9)
	U		60.7		3.74		1.47	
	E2	+1.1	59.2	- 2.5	3.76	+ 0.6	1.53	+ 4.1
	U		59.9		3.68		1.47	
	E3	+0.17	60.0	+ 0.2	3.68	0	1.52	+ 3.4
99+% Al$_2$O$_3$ self-bonded dense fused-cast brick (Monofrax A)	U		0.8		3.86		3.83	
	E1	-0.10	0.4	-50.	3.90	+ 1.0	3.89	+ 1.6
	U		0.8		3.86		3.83	
	E2	-0.06	1.33	+66.	3.82	- 1.0	3.77	- 1.6
	U		0.9		3.85		3.80	
	E3	+0.05	1.85	+106.	3.93	+ 2.1	3.78	- 0.5
SiC, silicon nitride bonded, dense fired brick (Refrax 20)[h]	U		15.1(14.6)[f]		3.12(3.10)[f]		2.64(2.65)[f]	
	E1	+0.29	16.1	+ 6.6(+10.3)	3.10	- 0.7(0)	2.60	- 0.9[j](-1.9)[j]
	U		15.1		3.12		2.64	
	E2	corroded	20.5	+36.	2.86	- 8.3	2.28	-14.
	U		17.3		3.16		2.61	
	E3	+0.11	20.9	+21.	2.92	-39.[j]	2.30	-12.
70% Al$_2$O$_3$ (mullite), vitreous dense fired brick (Alumex 70-HD)	U		20.7		3.25		2.58	
	E3	-0.46	20.6	- 0.4	3.25	0	2.57	- 0.4
Zirconium sulfate vitreous bond coupon (Zircon)	U		16.2		4.58		3.84	
	E3	-0.39	16.5	+ 1.8	4.60	+ 0.4	3.83	- 0.3
60% Al$_2$O$_3$ tar-impregnated vitreous fired brick (Ufala TI)	U		0.9[k]		2.70		2.68	
	E3	-6.8	15.3	+960.[j]	2.95	+ 9.3	2.50	- 6.7
45-50% Al$_2$O$_3$ high-fired super-duty brick (KX-99)	U		12.3		2.62		2.30	
	E3	-0.62	14.2	+15.	2.67	+ 1.9	2.29	- 0.4
60% Al$_2$O$_3$ phosphate-bonded ramming mix (Wasp #60)	U		14.6(20.1)[f]		3.08(2.82)[f]		2.63(2.25)[f]	
	E1	Specimen cracked						
	U		14.6		3.08		2.63	
	E2	fractured	20.8	+42.	2.86	- 7.1	2.26	-14.
	U		19.7		2.82		2.26	
	E3	-2.5	19.9	+ 1.0	2.82	0	2.26	0

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(Table Continued)

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PHYSICAL PROPERTIES[a] OF REFRACTORIES[b] EXPOSED TO THE CONDITIONS[c] IN COAL GASIFICATION PILOT PLANTS[12,90], Continued

Refractory (Brand Name)	Expo-sure[d]	Weight Change %	Apparent Porosity[a] %	% Change	Apparent Specific Gravity[a]	% Change	Bulk Density[a] g/cm³	% Change
Three exposures in gasifier off-gas (Conoco, CO_2 Acceptor), continued								
90% Al_2O_3 phosphate-bonded ramming mix (Brikram 90)	U		16.3		3.52		2.95	
	E1		Specimen cracked					
	U		16.3		3.52		2.95	
	E2	+0.07	16.2	− 0.6	3.49	− 0.9	2.93	− 0.7
	U		19.5		3.62		2.92	
	E3	−0.19	16.5	−15.	3.50	− 3.3	2.92	0
52% Al_2O_3 light castable, CA bond (Litecast 80)	U		37.3		2.66		1.67	
	E1	−3.5	46.3	+24.1	2.94	+10.5	1.57	− 6.0
	U[f]		34.6		2.57		1.68	
	E1[f]		Specimen cracked					
	U		34.6		2.57		1.68	
	E2	−2.8	46.7	+35.	3.13	+30.[j]	1.64	− 0.6[j]
90+% Al_2O_3 dense castable, CA bond (Greencast-97)	U		27.7(31.2)[f]		3.65(3.78)[f]		2.64(2.61)[f]	
	E1	−0.86	30.5	+10.(−2.2)	3.82	+ 4.9[j](+1.1)	2.65(2.66)	+ 0.4(+1.9)
70+% Al_2O_3 vitreous fired brick (Hi Lumite 70 D)	U		24.3(21.2)[f]		3.14(2.68)[f]		2.34(2.11)[f]	
	E1	−6.6	26.7	+ 9.9(+25.9)	3.25	+ 3.2[j](+21.3)	2.38	+ 1.7(+12.8)
	U		24.3		3.14		2.34	
	E2	−0.89	25.9	+ 6.6	3.25	+ 3.5	2.31	+ 3.0[j]
85% Al_2O_3 phosphate-bonded fired brick (Altex 85-B)	U		12.0(15.2)[f]		3.36(3.44)[f]		2.96(2.90)[f]	
	E1	−0.20	16.7	+39.(+9.9)	3.50	+ 3.9[j](+1.7)	2.92	− 1.3(+0.7)
80+% Al_2O_3 phosphate-bonded fired brick (Alumex P-8)	U		13.8(15.2)[f]		3.32(3.33)[f]		2.86(2.82)[f]	
	E1	−0.02(−0.2)[f]	15.2(15.1)	+10.(−0.7)	3.36(3.35)	+ 1.2(+0.6)	2.85	− 0.3(+1.1)
	U		13.8		3.36		2.86	
	E2	+0.07	15.4	+12.	3.33	− 0.9	2.82	− 1.4
90% Al_2O_3/10% Cr_2O_3 solid solution fired brick (CS 612)	U		17.4		3.82		3.15	
	E1	−1.0(−0.3)[f]	18.6(18.7)[f]	+ 6.9(+7.5)	3.84	+ 0.5	3.12	− 1.0
90+% Al_2O_3 phosphate-bonded castable (Resco Cast AA-22)	U		23.7		3.53		2.69	
	E3	−0.38	24.9	+ 5.0	3.53	0	2.68	− 0.4
SiC, oxynitride bond, coupons/ bricks (Crystolon 63)	U		14.2		3.08		2.64	
	E3	−18.1	20.0	+41.	2.97	− 3.6	2.38	− 9.8
Three exposures in dolomite regenerator off-gas (Conoco, CO_2 Acceptor): 70 N_2, 27 CO_2, 3 CO, H_2S (trace) (in vol. %); 1850 °F, 150 psi, ∿800 hours (exposure 1), 1000–2400 hours (exposure 2), 740–1450 hours (exposure 3).								
90+% Al_2O_3 dense castable, CA bond (Castolast G)	U		27.5		3.63		2.62	
	E1	−1.2	31.2	+13.5	3.72	+ 2.2[j]	2.56	− 2.3
	U		27.5		3.63		2.62	
	E2	0	31.8	+16.	3.74	+ 3.0	2.55	− 2.6
	U		32.3		3.80		2.58	
	E3	+0.45	27.0	−16.	3.50	− 7.9	2.55	− 1.2
60% Al_2O_3 dense castable, CA bond (Mizzou Castable)	U		25.1		3.00		2.25	
	E1	−2.1	23.2	− 4.7[j]	2.96	− 1.3	2.27	+ 0.9
	U		25.1		3.00		2.35(2.25)[f]	
	E2	−1.9	25.8	+ 2.8	3.02	+ 0.7	2.24	− 4.7(−0.4)
	U		27.8		3.13		2.26	
	E3	+1.3	25.5	− 8.	3.06	− 2.2	2.28	+ 0.9
90+% Al_2O_3 light castable, CA bond (Purolite 30)	U		46.6(44.6)[f]		2.65		1.42	
	E1	−9.0(−5.5)[f]	53.0	+14.(+19.)	3.03	+14.	1.43	+ 0.7
	U		44.6		2.65		1.42	
	E2	−34.	54.3	+22.	2.97	+12.	1.36	− 4.2
90+% Al_2O_3 light castable, CA bond (Greencast-97L)	U		49.9		2.82		1.41	
	E1	−3.3	44.5	−11.	2.56	− 9.2	1.42	+ 0.7
	U		49.9		2.82		1.41	
	E2	+1.8	54.5	+ 9.2	3.12	+11.	1.42	+ 0.6
	U		45.9		2.57		1.39	
	E3	+3.2	60.8	+33.	3.57	+39.	1.40	+ 0.7
54% Al_2O_3 dense castable, CA bond (Super Brikcast A)	U		26.4		2.94		2.13	
	E3	+2.3	26.0	− 1.5	2.92	− 0.7	2.16	+ 1.4

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(Table Continued)

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PHYSICAL PROPERTIES[a] OF REFRACTORIES[b] EXPOSED TO THE CONDITIONS[c] IN COAL GASIFICATION PILOT PLANTS[12,90], Continued

Refractory (Brand Name)	Expo-sure[d]	Weight Change %	Apparent Porosity[a] %	% Change	Apparent Specific Gravity[a]	% Change	Bulk Density[a] g/cm³	% Change
Three exposures in dolomite regenerator off-gas (Conoco, CO_2 Acceptor), continued								
54% Al_2O_3 light insulating castable, CA bond (Litecast 75-28)	U		41.3(37.9)[f]		2.33(2.36)[f]		1.37(1.51)[f]	
	E1	-1.3(-1.5)[f]	49.8	+20.6(+9.6)[j]	2.85(2.87)	+22.3(+21.6)	1.45	+ 5.8(-4.0)
	U		37.9		2.36		1.51	
	E2	+1.1	62.0	+64.	2.93	+24.	1.77	+17.
	U		48.4		2.34		1.19	
	E3	-0.34	57.5	+19.	2.91	+24.	1.24	+ 4.2
70% Al_2O_3 (mullite), vitreous bond high-fired brick (Mul-8)	U		17.2(18.2)[f]		3.05		2.53(2.50)[f]	
	E1	+0.3(+0.4)[f]	17.9(18.0)	+ 4.1(-1.1)	3.06	+ 0.3	2.51	- 0.8(+0.4)
	U		17.2		3.05		2.53	
	E2	+2.2	17.0	- 1.2	3.05	0	2.53	0
85% Al_2O_3 phosphate bond, dense fired brick (Chemal 85 B)	U		15.7(17.6)[f]		3.36(3.44)[f]		2.83(2.84)[f]	
	E1	+0.4	16.4(17.4)	+ 4.5(-1.1)	3.47	+ 3.3(+0.9)	2.90	+ 2.5(+2.1)
	U		15.7		3.36		2.83	
	E2	-0.8	20.5	+31.	3.49	+ 3.9	2.77	- 2.1
	U		18.1		3.46		2.84	
	E3	+0.73	22.6	+25.	3.48	+ 0.6	2.84	0
90+% Al_2O_3 self-bonded dense fired brick (Kricor)	U		15.1(16.7)[f]		3.51(3.54)[f]		2.99(2.95)[f]	
	E1	+0.4	16.7	+10.6(0)	3.53	+ 0.6(-0.3)	2.94	- 1.7(-0.3)
	U[f]		16.7		3.54		2.95	
	E1[f]	0	16.6	- 0.6	3.52	- 0.6	2.94	- 0.3
	U		16.7		3.54		2.95	
	E2	Specimen not recovered						
	U		18.2		3.60		2.94	
	E3	+1.05	17.9	- 1.6	3.55	- 1.4	2.96	+ 0.7
99+% Al_2O_3 self-bonded dense fired brick (H-W Corundum)	U		19.0		3.64(3.74)[f]		2.95(3.03)[f]	
	E1	-0.9(0)[f]	21.4(21.0)[f]	+12.6(+10.5)	3.77(3.76)	+ 3.6(+0.6)	2.96(2.97)	+ 0.3(-2.0)
	U		19.0		3.74		3.03	
	E2	+0.23	21.9	+15.	3.76	+ 0.6	2.84	- 6.3
	U		20.2		3.78		3.00	
	E3	+0.71	20.2	0	3.78	0	3.02	+ 0.7
77% Al_2O_3 self-bonded light insulating fired brick (B&W Insalcor)	U		46.5		2.81		1.53	
	E1	-1.2(+10.9)[f]	56.0	+20.4	2.94	+ 4.6	1.30	-13.1[j]
	U[f]		50.5		2.77		1.35	
	E1[f]	+1.0	54.8(54.6)[f]	+ 8.5(+8.1)	2.89(2.82)[f]	+ 4.3(+1.8)	1.28	- 5.2
	U		46.5		2.81		1.53	
	E2	+5.3	54.5	+17.	2.85	+ 1.4	1.30	-15.
	U		49.0		2.83		1.44	
	E3	+5.4	54.4	+11.	3.26	+15.	1.47	+ 2.1
98+% Al_2O_3 self-bonded light insulating fired brick (Alfrax B 101)	U		60.7(57.7)[f]		3.74(3.70)[f]		1.47(1.57)[f]	
	E1	-0.15(+2.0)[f]	57.2	- 5.8(-0.9)	3.67	- 1.9(-0.8)	1.59	+ 5.4[j](+1.3)
	U		60.7		3.74		1.47	
	E2	+2.2	57.9	- 4.6	3.77	+ 0.8	1.54	+ 4.8
	U		60.6		3.83		1.51	
	E3	+5.1	45.5	-25.	3.04	-21.	1.66	+10.
99+% Al_2O_3 self-bonded dense fused-cast brick (Monofrax A)	U		0.8		3.86		3.83	
	E1	-0.15(+0.2)[f]	0.7(0.6)[f]	-13.(-25.)	3.83(3.89)[f]	- 0.8(+0.8)	3.80(3.87)	- 0.8(+1.0)
	U		0.8		3.86		3.83	
	E2	0	1.1	+38.	3.82	- 1.0	3.78(3.82)	- 1.3(-0.3)
	U		1.05		3.82		3.78	
	E3	+0.56	1.41	+34.	3.82	0	3.76	- 0.5
SiC, silicon nitride bonded, dense fired brick (Refrax 20)[h]	U		15.1(14.6)		3.12(3.10)		2.64(2.65)	
	E1	+3.1	8.5(9.4)	-44.(-35.6)	2.97	- 4.8(-4.2)	2.72(2.71)	+ 3.0(+2.3)
	U		15.1		3.12		2.64	
	E2	+3.7	6.5	-57.	2.85	- 8.7	2.67	+ 1.1
	U		15.4		3.15		2.67	
	E3	+0.33	7.9	-48.	2.97	- 5.1	2.74	+ 2.6

(Table Continued)

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PHYSICAL PROPERTIES[a] OF REFRACTORIES[b] EXPOSED TO THE CONDITIONS[c] IN COAL GASIFICATION PILOT PLANTS[12,90], Continued

Refractory (Brand Name)	Expo-sure[d]	Weight Change %	Apparent Porosity[a] %	Apparent Porosity[a] % Change	Apparent Specific Gravity[a]	Apparent Specific Gravity[a] % Change	Bulk Density[a] g/cm³	Bulk Density[a] % Change
			Three exposures in dolomite regenerator off-gas (Conoco, CO_2 Acceptor), continued					
70% Al_2O_3 (mullite), vitreous dense fired brick (Alumex 70-HD)	U		20.1		3.26		2.60	
	E3	+1.9	20.4	+ 1.5	3.23	− 0.9	2.57	− 1.2
60% Al_2O_3 tar-impregnated vitreous fired brick (Ufala TI)	U		1.1[k]		2.79		2.76	
	E3	−4.2	12.1	+1000.[k]	2.96	+ 6.1	2.60	− 5.8
45-50% Al_2O_3 high-fired super-duty brick (KX-99)	U		13.3		2.63		2.28	
	E3	+1.4	13.2	− 0.7	2.67	+ 1.5	2.31	+ 1.3
60% Al_2O_3 phosphate-bonded ramming mix (Wasp #60)	U		14.6(20.1)		3.08(2.82)		2.63(2.25)	
	E1		Specimen cracked					
	U		14.6		3.08		2.63	
	E2	+1.7	19.2	+31.	2.78	− 9.7	2.24	−15.
	U		19.9		2.82		2.26	
	E3	+2.5	19.0	− 4.7	2.78	− 1.4	2.25	− 0.4
90% Al_2O_3 phosphate-bonded ramming mix (Brikram 90)	U		16.3		3.52		2.95	
	E1	−0.52(+0.4)	16.8(17.0)	+ 3.1(+4.3)	3.46	− 1.7	2.88	− 2.6[j]
	U		16.3		3.52		2.95	
	E2	−0.20	17.2	+ 5.5	3.44	− 2.3	2.98(2.85)	+ 1.0(−3.4)
	U		19.6		3.63		2.92	
	E3	+1.6	14.8	−25.	3.44	−12.[j]	2.93	+ 0.3
52% Al_2O_3 light castable, CA bond (Litecast 80)	U		37.3		2.66		1.67	
	E1	−1.3	46.0	+23.3	2.91	+ 9.4	1.56	− 6.6
	U[f]		34.6		2.57		1.68	
	E1[f]	−2.2	42.6	+23.	2.78	+ 8.2	1.60	− 4.8
	U		34.6		2.57		1.68	
	E2	+1.1	46.2	+34.	3.56(3.12)	+39.(+21.)	1.67	− 0.6
90+% Al_2O_3 dense castable, CA bond (Greencast-97)	U		27.7(31.2)		3.65(3.78)		2.64(2.61)	
	E1	−0.16	25.7	− 7.8[j](−18.)	3.56(3.55)	− 2.5(−6.1)	2.64	0(−1.1)
70+% Al_2O_3 vitreous fired brick (Hi Lumite 70 D)	U		21.2(24.3)		2.68(3.14)		2.11(2.34)	
	E1	−0.14(+0.5)	24.1(23.8)	+13.7(−2.1)	3.02(3.22)	+12.7(+2.3)	2.45(2.45)	+16.1(+4.7)
	U		24.3		3.14		2.34	
	E2	−0.18	16.9	−30.	2.58	−18.	2.15	− 8.1
85% Al_2O_3 phosphate-bonded fired brick (Altex 85-B)	U		12.0(15.2)		3.36(3.44)		2.96(2.90)	
	E1	+0.07(+0.7)	16.2(15.3)	+35.(+0.6)	3.48(3.48)	+ 3.6(+1.2)	2.91	− 1.7(+0.3)
80+% Al_2O_3 phosphate-bonded fired brick (Alumex P-8)	U		13.8(15.2)		3.32(3.33)		2.86(2.82)	
	E1	−0.04(+0.4)	17.2(16.4)	+25.(+7.9)	3.33	+ 0.3(0)	2.78	− 2.8(−1.4)
	U		13.8		3.36		2.86	
	E2	− 0.16	19.5	+41.	3.33	− 0.9	2.68	− 6.3
90% Al_2O_3/10% Cr_2O_3 solid solu-tion fired brick (CS 612)	U		17.4		3.82		3.15	
	E1	+0.05(+0.1)	18.4(18.8)	+ 5.8(+2.3)[j]	3.83(3.84)	+ 0.3(+0.5)	3.12	− 1.0
90+% Al_2O_3 phosphate-bonded castable (Resco Cast AA-22)	U		24.1		3.53		2.68	
	E3	+1.8	26.6	+14.[j]	3.59	+ 1.7	2.63	− 1.9
SiC, oxynitride bond, coupons/bricks (Crystolon 63)	U		14.9		3.07		2.61	
	E3	+1.2	9.4	−37.	2.92	− 4.9	2.65	+ 1.5
			Two exposures in gasifier off-gas (Bi-Gas): H_2O, CO_2, CO, H_2, N_2, CH_4, H_2S, ash and char (entrained); 1800 °F 750 psi, exposure time not given (exposure 1); 1525-1750 °F, 750 psig, 1100 hours in operating conditions (exposure 2).					
54% Al_2O_3 dense castable, CA bond (Super Brikcast A)	U		24.8[l]		2.87[l]		2.17[l]	
	E1	−8.4	26.1	+ 5.2	2.89	+ 0.7	2.13	− 1.8
	U		26.6		2.90		2.13	
	E2[m]	--	27.3	+ 3.	2.92	+ 1.	2.12	0(−0.5)
54% Al_2O_3 light insulating castable, CA bond (Litecast 75-28)	U		37.9[l]		2.36[l]		1.51[l]	
	E1	−10.4	62.2	+64.	3.06	+30.	1.15	−24.[m]
	U		41.1		2.12		1.13	
	E2[m]	--	63.3	+54.	3.04	+44.	1.11	− 2.
85% Al_2O_3 phosphate bond, dense fired brick (Chemal 85 B)	U		15.7[l]		3.36[l]		2.83[l]	
	E1	−2.55	15.9	+ 1.3	3.36	0	2.83	0
	U		18.5		3.49		2.82	
	E2	--	20.0	+ 8.	3.50	0	2.80	+ 1.

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(Table Continued)

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PHYSICAL PROPERTIES[a] OF REFRACTORIES[b] EXPOSED TO THE CONDITIONS[c] IN COAL GASIFICATION PILOT PLANTS[12,90], Continued

Refractory (Brand Name)	Exposure[d]	Weight Change %	Apparent Porosity[a] %	% Change	Apparent Specific Gravity[a]	% Change	Bulk Density[a] g/cm³	% Change
Two exposures in gasifier off-gas (Bi-Gas), continued								
70% Al_2O_3 (mullite), vitreous	U		19.2		3.23		2.61	
dense fired brick (Alumex 70-HD)	E1	-1.04	19.4	+ 1.0	3.29	+ 1.9	2.65	+ 1.5
60% Al_2O_3 tar-impregnated vitreous	U		1.1[k]		2.69		2.66	
fired brick (Ufala TI)	E1	-6.4	15.5	+1409.[j]	2.97	+10.4	2.51	- 5.6
90+% Al_2O_3 phosphate-bonded	U		23.9[l]		3.53[l]		2.69[l]	
castable (Resco Cast AA-22)	E1	+0.03	25.2	+ 5.4	3.60	+ 2.0	2.71	+ 0.7
	U		30.4		3.53		2.46	
	E2	--	31.6	+ 4.	3.57	+ 1.	2.44	- 1.
60% Al_2O_3 dense castable, CA bond	U		27.8		3.12		2.25	
(Mizzou Castable)	E2	--	31.7	+14.	3.19	+ 2.	2.18	- 3.
90+% Al_2O_3 self-bonded dense	U		18.5		2.58		2.93	
fired brick (Kricor)	E2	--	20.0	+ 8.	3.62	+ 1.[j]	2.82	- 4.
Alumina-zirconia-silica fused-	U		1.2		3.87		3.82	
cast brick (AZS)	E2		Specimen not recovered					
45-50% Al_2O_3 high-fired super-	U		12.7		2.62		2.30	
duty brick (KX-99)	E2[n]	--	14.6	+15.	2.65	+ 1.	2.26	- 2.
90% Al_2O_3 phosphate-bonded ramming	U		13.9		3.47		2.99	
mix (Brikram 90)	E2	--	15.3	+10.	3.46	0	2.93	- 2.
One exposure in gasifier off-gas (Battelle): 1800 °F, 100 psi, ∿50 hour exposure.								
60% Al_2O_3 dense castable, CA bond	U		28.2		3.14		2.25	
(Mizzou Castable)	E	-16.4	27.5	- 2.5	3.07	- 2.2	2.23	- 0.9
70% Al_2O_3 (mullite), vitreous bond	U		17.5		3.07		2.53	
high-fired brick (Mul-8)	E	-0.04	17.8	+ 1.8	3.08	+ 0.3	2.53	0
85% Al_2O_3 phosphate bond, dense	U		14.5		3.38		2.90	
fired brick (Chemal 85 B)	E	+0.01	14.9	+ 2.8	3.41	+ 0.9	2.90	0
Silicon carbide, clay bonded	U		16.6		3.06		2.55	
(Harbide)	E	+0.36	17.2	+ 3.6	3.08	+ 0.7	2.55	0
90% Al_2O_3 phosphate-bonded ramming	U		12.3		2.98		2.61	
mix (Brikram 90)	E	+0.04	12.3	0	2.98	0	2.62	+ 0.4
80+% Al_2O_3 phosphate-bonded ramming	U		14.7		3.33		2.84	
mix (Alumex P-8)	E	0.0	15.6	+ 6.1	3.37	+ 1.2	2.84	0
90+% Al_2O_3 phosphate-bonded	U		29.6		3.55		2.50	
castable (Resco Cast AA-22)	E	-0.19	28.2	- 4.7	3.56	+ 0.3	2.56	+ 2.4
One exposure in combustor off-gas (Battelle): 2000 °F, >100 psi, ∿50 hour exposure.								
60% Al_2O_3 dense castable, CA bond	U		30.6		3.20		2.21	
(Mizzou Castable)	E	-38.5	27.0	-12.	3.10	- 3.1	2.26	+ 2.3
Silicon carbide, clay bonded	U		16.7		3.05		2.54	
(Harbide)	E	+0.13	19.5	+17.	3.07	+ 0.7	2.48	- 2.4
60% Al_2O_3 tar-impregnated vitreous	U		0.2[k]		2.07[l]		2.69[l]	
fired brick (Ufala TI)	E	-5.8	15.1	+7450.	3.05	+13.	2.59	- 3.7
45-50% Al_2O_3 high-fired super-	U		11.35		2.62		2.32	
duty brick (KX-99)	E	0.0	11.5	+ 1.3	2.67	+ 2.7[j]	2.33	+ 0.4
90% Al_2O_3 phosphate-bonded ramming	U		13.2		3.46		3.00	
mix (Brikram 90)	E	+0.08	13.0	- 1.5	3.46	0	3.01	- 0.3
90+% Al_2O_3 phosphate-bonded	U		26.0		3.55		2.63	
castable (Resco Cast AA-22)	E	-3.3	25.7	- 1.2	3.58	+ 0.8	2.66	+ 1.1
Two exposures in gasifier off-gas (U-GAS): reducing gas, H_2O, CO_2, CO, H_2, CH_4, N_2, H_2S, ash and char (entrained); 1803-1930 °F, 5.5-43 psig, exposure times not given.								
90+% Al_2O_3 dense castable, CA bond	U		31.4		3.79		2.60	
(Castolast G)	E1	+0.02	26.4	-15.9	3.50	-7.65	2.57	-1.15
	U		26.1		3.57		2.64	
	E2	--	24.7	- 5.	3.48	- 3.	2.62	- 1.
60% Al_2O_3 dense castable, CA bond	U		25.3		3.04		2.27	
(Mizzou Castable)	E1	+0.05	26.1	+ 3.2	3.07	+ 0.99	2.27	0
	U		25.3		2.97		2.22	
	E2	--	25.7	+ 2.	3.01	+ 1.	2.24	+ 1.

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(Table Continued)

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PHYSICAL PROPERTIES[a] OF REFRACTORIES[b] EXPOSED TO THE CONDITIONS[c] IN COAL GASIFICATION PILOT PLANTS[12,90], Continued

Refractory (Brand Name)	Exposure[d]	Weight Change %	Apparent Porosity[a] %	% Change	Apparent Specific Gravity[a]	% Change	Bulk Density[a] g/cm³	% Change
Two exposures in gasifier off-gas (U-GAS), continued								
54% Al₂O₃ dense castable, CA bond (Super Brikcast A)	U		24.1		2.84		2.15	
	E1	+0.03	25.9	+ 7.5	2.92	+ 2.8	2.16	+ 0.47
	U		27.6		2.86		2.07	
	E2	--	27.3	0[j]	2.89	+ 1.	2.09	+ 1.
85% Al₂O₃ phosphate bond, dense fired brick (Chemal 85 B)	U		15.1		3.47		2.95	
	E1	+0.04	16.2	+ 7.3	3.49	+ 0.58	2.92	- 1.02
	U		19.8		3.40		2.74	
	E2	--	19.0	- 4.	3.38	- 1.	2.73	0
SiC, silicon nitride bonded, dense fired brick (Refrax 20)	U		14.6		3.11		2.66	
	E1	+0.01	9.2	-37.	2.97	-14.[j]	2.69	+ 1.13
	U		18.9		3.18		2.58	
	E2	--	11.1	-41.	2.96	- 7.	2.64	+ 2.
60% Al₂O₃ tar-impregnated vitreous fired brick (Ufala TI)	U		1.1[k]		2.69		2.66	
	E2	--	14.6	+1227.	2.93	+ 9.	2.51	- 5.
90% Al₂O₃ phosphate-bonded ramming mix (Brikram 90)	U		18.4		3.50		2.80	
	E1	+0.02	18.0	- 2.2	3.46	- 1.14	2.83	+ 1.07
	U		18.2		3.48		2.85	
	E2	--	17.1	- 6.	3.47	0	2.88	+ 1.
90% Al₂O₃ phosphate-bonded castable (Resco Cast AA-22)	U		23.3		3.47		2.67	
	E1	+0.03	25.2	+ 8.2	3.58	+ 3.17	2.68	+ 0.37
	U		25.5		3.58		2.67	
	E2	--	25.1	0[j]	3.60	+ 1.	2.70	+ 1.
70% Al₂O₃ (mullite), vitreous dense fired brick (Alumex 70-HD)	U		20.0		3.25		2.60	
	E1	+0.05	20.4	+ 2.0	3.25	0	2.59	- 0.38

Three exposures in gasifier off-gas of high-temperature reactor (HYGAS): H_2O, CO, CO_2, H_2, H_2S, CH_4, char; ∿1250 °F, 900-1100 psi, exposure times for 1 and 2 not given, exposure 3 >1355 hours.

Refractory(Brand Name)	Exposure[d]	Brick[o] Portion	Weight Change %	Apparent Porosity[a] %	% Change	Apparent Specific Gravity[a]	% Change	Bulk Density[a] g/cm³	% Change
90+% Al₂O₃ dense castable, CA bond (Castolast G)	U			27.5		3.63		2.62	
	E1	hot face }	-3.2	25.8	- 6.2	3.51	- 3.3	2.64	+ 0.8
		cold face }		27.8	+ 1.1	3.58	- 1.4	2.58	- 1.5
	U	whole[l]		27.5		3.63		2.62	
	E3	whole	+0.5						
		hot face		28.2	+ 2.5	3.63	0	2.61	- 0.4
		cold face		27.5	0	3.60	- 0.8	2.61	- 0.4
60% Al₂O₃ dense castable, CA bond (Mizzou Castable)	U	whole		25.1		3.00		2.25	
	E2	hot face }	+0.4	22.6	-10.	2.99	- 0.3	2.31	+ 2.7
		cold face }		23.0	- 8.4	2.99	- 0.3	2.30	+ 2.2
(two specimens in exposure 2)		hot face }	+0.7	22.2	-12.	2.97	- 1.0	2.31	+ 2.7
		cold face }		22.8	- 9.2	2.98	- 0.7	2.30	+ 2.2
	U	whole[l]		25.1		3.00		2.25	
	E3	whole	+0.7						
		hot face		23.2	- 7.6	3.01	+ 0.3	2.31	+ 2.7
		cold face		24.0	- 4.4	3.03	+ 1.0	2.30	+ 2.2
54% Al₂O₃ dense castable, CA bond (Super Brikcast A) (2 bricks)	U	whole[l]		24.8		2.87		2.17	
	E3	whole (1)	+1.1						
		whole (2)	+0.9						
		hot face (1)		20.5	-17.	2.62	- 8.7	2.22	+ 2.3
		hot face (2)		20.2	-19.	2.85	- 0.7	2.21	+ 1.8
		cold face (1)		21.9	-12.	2.83	- 1.4	2.21	+ 1.8
		cold face (2)		21.0	-15.	2.84	- 1.0	2.23	+ 2.2(+2.7)[f]
54% Al₂O₃ light insulating castable, CA bond (Litecast 75-28)	U	whole[l]		37.9		2.36		1.51	
	E3	whole	-0.3						
		hot face		63.8	+68.	3.03	+28.	1.10	-27.
		cold face		62.2	+64.	3.04	+29.	1.15	-24.

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(Table Continued)

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PHYSICAL PROPERTIES[a] OF REFRACTORIES[b] EXPOSED TO THE CONDITIONS[c] IN COAL GASIFICATION PILOT PLANTS[12,90], Continued

Refractory(Brand Name)	Expo-sure[d]	Brick[o] Portion	Weight Change %	Apparent Porosity[a]		Apparent Specific Gravity[a]		Bulk Density[a]	
				%	% Change		% Change	g/cm^3	% Change
Three exposures in gasifier off-gas of high-temperature reactor (HYGAS), continued									
70% Al$_2$O$_3$ (mullite), vitreous bond high-fired brick (Mul-8)	U	whole		17.2		3.05		2.53	
	E1	hot face }	+0.3	20.2	+17.	3.09	+ 1.3	2.47	− 2.4
		cold face		19.1	+11.	3.08	+ 1.0	2.49	− 1.6
	U	whole		17.2		3.05		2.53	
	E2	hot face }	+0.5	22.0	+28.	3.12	+ 2.3	2.43	− 4.0
		cold face		20.8	+21.	3.11	+ 2.0	2.46	− 2.8
	U	whole[1]		17.2		3.05		2.53	
	E3	whole	+0.3						
		hot face		20.5	+19.	3.09	+ 1.3	2.40	− 5.1
		cold face		18.2	+ 6.	3.09	+ 1.3	2.51	− 0.8
85% Al$_2$O$_3$ phosphate bond, dense fired brick (Chemal 85 B)	U	whole		15.7		3.36		2.83	
	E1	hot face }	0.0	17.6	+12.	3.46	+ 3.0	2.88	+ 1.8
		cold face		18.8	+20.	3.48	+ 3.6	2.83	0
	U	whole		15.7		3.36		2.83	
	E2	hot face }	+0.8	18.2	+16.	3.45	+ 2.7	2.82	− 0.4
		cold face		16.4	+ 4.5	3.43	+ 2.1	2.87	+ 1.4
	U	whole[1]		15.7		3.36		2.83	
	E3	whole	+1.1						
		hot face		19.9	+27.	3.43	+ 2.1	2.74	− 3.2
		cold face		18.6	+18.	3.42	+ 1.8	2.70	− 4.6
90+% Al$_2$O$_3$ self-bonded dense fired brick (Kricor)	U	whole		16.7		3.54		2.95	
	E1	hot face }	−0.7	17.5	+ 4.8	3.54	0	2.92	− 1.0
		cold face		15.1	− 9.6	3.50	− 1.1	2.97	+ 0.7
	U	whole		16.7		3.54		2.95	
	E2	hot face }	−0.1	16.6	− 0.6	3.57	+ 0.8	2.97	+ 0.7
		cold face		17.5	+ 4.8	3.54	0	2.92	− 1.0
	U	whole[1]		16.7		3.54		2.95	
	E3	whole	+2.0						
		hot face		17.8	+ 6.6	3.54	0	2.91	− 1.4
		cold face		15.2	− 9.0	2.51(3.51)[f]	− 0.8	2.98	+ 1.0
99+% Al$_2$O$_3$ self-bonded dense fired brick (H-W Corundum)	U	whole		19.0		3.74		3.03	
	E1	hot face }	+0.3	19.0	0	3.77	+ 0.8	3.06	+ 1.0
		cold face		19.5	+ 2.6	3.76	+ 0.5	3.03	0
	U	whole		19.0		3.74		3.03	
	E2	hot face }	+0.96	19.7	+ 3.7	3.78	+ 1.1	3.03	0
		cold face		20.9	+10.	3.81	+ 1.9	3.01	− 0.7
	U	whole[1]		19.0		3.74		3.03	
	E3	whole	− 0.02						
		hot face		20.5	+ 7.9	3.77	+ 0.8	3.00	− 1.0
		cold face		21.4	+13.	3.78	+ 1.1	2.98	− 1.7
77% Al$_2$O$_3$ self-bonded light insulating fired brick (B&W Insalcor)	U	whole		46.5		2.81		1.53	
	E1	hot face }	−1.3	56.4	+21.	2.87	+ 2.1	1.26	−18.
		cold face		57.6	+24.	2.98	+ 6.0	1.27	−17.
70% Al$_2$O$_3$ (mullite) vitreous dense fired brick (Alumex 70-HD)	U	whole[1]		19.2		3.23		2.61	
	E3	whole	+0.7						
		hot face		19.1	− 0.5	3.24	+ 0.3	2.62	+ 0.4
		cold face		19.6	+ 2.1	3.27	+ 1.2	2.63	+ 0.8
98+% Al$_2$O$_3$ self-bonded light insulating fired brick (Alfrax B 101)	U	whole		60.7		3.74		1.47	
	E1	hot face }	−0.15	57.7	− 4.9	3.72	− 0.5	1.57	+ 6.8
		cold face		56.7	− 6.6	3.61	− 3.5	1.56	+ 6.1
60% Al$_2$O$_3$ tar-impreg-nated vitreous fired brick (Ufala TI)	U	whole[1]		0.2[k]		2.70		2.69	
	E3	whole	−5.1						
		hot face		12.7	+6250.	2.95	+ 9.3	2.58	− 4.1
		cold face		11.5	+5650.	2.95	+ 9.3	2.61	− 3.0
45-50% Al$_2$O$_3$ clay vitreous bond, high-fired super-duty brick (KX-99)	U	whole[1]		12.3		2.62		2.29	
	E3	whole	−0.9						
		hot face		21.1	+72.	2.72	+ 3.8	2.15	− 6.1
		cold face		14.4	+17.	2.66	+ 1.5	2.28	− 0.4

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(Table Continued)

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PHYSICAL PROPERTIES[a] OF REFRACTORIES[b] EXPOSED TO THE CONDITIONS[c] IN COAL GASIFICATION PILOT PLANTS[12,90], Continued

Refractory(Brand Name)	Expo- sure[d]	Brick[o] Portion	Weight Change %	Apparent Porosity[a]		Apparent Specific Gravity[a]		Bulk Density[a]	
				%	% Change		% Change	g/cm³	% Change
Three exposures in gasifier off-gas of high-temperature reactor (HYGAS), continued									
90% Al₂O₃ phosphate- bonded ramming mix (Brikram 90)	U	whole		16.3		3.52		2.95	
	E2	hot face	+0.6	17.3	+ 6.1	3.54	+ 0.6	2.93	- 0.7
		cold face		17.2	+ 5.5	3.53	+ 0.3	2.93	- 0.7
(two specimens in exposure 2)		hot face	+0.01	16.6	+ 1.8	3.56	+ 1.1	2.97	+ 0.7
		cold face		16.0	- 1.8	3.54	+ 0.6	2.98	+ 1.0
	U	whole[1]		16.3		3.52		2.95	
	E3	whole	+0.6						
		hot face		14.7	- 9.8	3.47	- 1.4	2.91	- 1.4
		cold face		21.9	+34.	3.64	+ 3.4	2.84	- 3.7
90+% Al₂O₃ dense cast- able, CA bond (Greencast-97)	U	whole		27.7		3.65		2.64	
	E2	hot face	-1.46	27.9	+ 0.7	3.66	+ 0.3	2.64	0
		cold face		27.4	- 1.1	3.67	+ 0.5	2.67	+ 1.1
70+% Al₂O₃ vitreous fired brick (Hi Lumite 70 D)	U	whole		24.3		3.14		2.34	
	E1	hot face	-0.03	27.1	+12.	3.27	+ 4.1	2.39	+ 2.1
		cold face		24.7	+ 1.6	3.24	+ 3.2	2.44	+ 4.3
	U	whole		24.3		3.14		2.34	
	E2	hot face	-0.67	22.6	- 7.0	3.12	- 0.6	2.43	+ 3.8
		cold face		20.8	-14.	3.11	- 1.0	2.46	+ 5.1
85% Al₂O₃ phosphate- bonded fired brick (Altex 85-B)	U	whole		12.0		3.36		2.96	
	E1	hot face	+0.09	15.3	+28.	3.48	+ 3.6	2.94	- 0.7
		cold face		15.8	+32.	3.47	+ 3.3	2.93	- 1.0
	U	whole		12.0		3.36		2.96	
	E2	hot face	+0.40	16.2	+35.	3.49	+ 3.9	2.92	- 1.4
		cold face		15.1	+26.	3.49	+ 3.9	2.97	+ 0.3
80+% Al₂O₃ phosphate- bonded fired brick (Alumex P-8)	U	whole		13.8		3.32		2.86	
	E1	hot face	--	14.4	+ 4.3	3.23	- 2.7	2.76	- 3.5
		cold face		16.3	+18.	3.33	+ 0.3	2.79	- 2.4
	U	whole		13.8		3.32		2.86	
	E2	hot face	+0.18	18.3	+33.	3.38	+ 1.8	2.76	- 3.5
		cold face		17.7	+28.	3.40	+ 2.4	2.72	- 4.9

[a] Apparent porosity, specific gravity, and bulk density measured by liquid immersion method according to ASTM C20-46, using kerosine as the displacing medium; specimens were first evacuated 2 h, kerosine introduced, and evacuation continued for 3 to 4 hours. Values are for one specimen.

[b] Brick specimens were prepared by diamond saw sectioning of commercial refractories supplied by manufacturers. Castables were mixed with amount of distilled water recommended by manufacturers, poured into 9 in. straights in wooden molds, cured in 100% humidity for 48 h, then saw cut, fired to 1500 °F and cooled. All refractories were fired to 1500 °F for 4 h in air before all testing both before and after exposure in the plants. Sample sizes varied for different pilot plants: Synthane and Conoco, 4 1/2 x 2 x 3/4 in.; HYGAS, 2 1/2 x 4 1/2 x 9 in.; Bi-Gas, 4 1/2 x 2 1/2 x 3/4 in.; Battelle 1.6 x 1.6 x 1 in.: U-GAS, 4 1/2 x 2 1/2 x 3/4 in.

[c] Full conditions to which the refractories were subjected were not available in direct relation to the exposure times. Conditions fluctuated during the exposures. Time periods, when given, are only approximate.

[d] U = unexposed, E1 = first exposure, E2 = second exposure, etc.

[e] CA bond = calcium aluminate bond.

[f] Values in parentheses or values given on separate lines for the same exposure are conflicting values for the same data reported in different tables of the set of reports.

[g] Large increases in values seen for fused-cast materials are due to development of a network of hairline cracks caused by thermal shock.

[h] Material partially disintegrated, fragments were weighed together.

[i] Specimens were refired to 2640 °F for 4 hours before exposure.

[j] Value reported is not consistent with the unexposed and exposed values but it is not possible to determine which of the three values, unexposed, exposed, or percent change, is incorrect.

[k] Values may be in error; note that in parts of this table the unexposed apparent porosity value is ∿14, in other parts ∿1.

[l] Average properties of all unexposed specimens evaluated in this exposure.

[m] An incorrect direction for this percent change is given in the project final report.

[n] Specimen broke.

[o] HYGAS samples, 9 x 2 1/2 x 4 1/2 in. bricks, were placed so that the 2 1/2 x 4 1/2 in. face was exposed to the gasifier conditions. Slices were cut from each end, one inch thick, for tests.

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EFFECT OF HEAT TREATMENT AND GASEOUS ENVIRONMENT[a] ON THE WEIGHT AND DIMENSIONS OF SAMPLES OF ALUMINA REFRACTORIES[b][39]

Treatment Conditions

DENSE HIGH-ALUMINA COMMERCIAL CASTABLES

Atmosphere[a]	Temperature °C / °F	Pressure MPa/psi	Time days	94% Al_2O_3[d]	93% Al_2O_3[e]	90-95% Al_2O_3[f]	90-95% Al_2O_3[g]
				Weight Change, % / Dimensional Change, %[c]			
Air (fired)	260/ 500	ambient	3/4	-3.1/-0.1	-2.9/-0.3	-2.3/nil	-3.0/-0.2
Air (fired)	538/1000	ambient	3/4	-4.5/-0.1	-4.7/-0.4	-3.9/-0.1	-4.1/nil
CGA exposure	260/ 500	3.4/500	10	-0.4/nil	-0.8/+0.2	+0.2/nil	+0.4/+0.2
			20	-1.5/-0.2	-2.1/-0.4	-0.7/nil	-0.6/-0.3
			30	-0.2/-0.2	-0.6/-0.4	nil/-0.3	+0.6/-0.3
CGA exposure	538/1000	6.9/1000	10	-2.6/+0.2	-4.3/-0.4	-1.1/-0.3	+0.4/-0.1
			20	-1.9/-0.3	-3.2/-0.6	-1.0/nil	-1.4/-0.1
			30	-1.9/-0.4	-3.7/-1.1	-0.9/-0.2	-1.4/-0.3
CGA with H_2S	538/1000	6.9/1000	10	-2.3/-0.1	-2.5/-0.3	-1.7/-0.1	-1.8/-0.1
			20	-2.3/-0.1	-3.9/-0.2	-2.0/-0.1	1.9[h]/+0.03
			30	-2.2/-0.02	-3.8/-0.2	-1.8/+0.03	-1.8/nil
CGA, H_2O saturated[i]	231/ 447	6.9/1000	30	+0.1/+0.4	-1.6/-0.1	not tested	+2.0/-0.2
CGA with H_2S, H_2O satd.[i]	231/ 447	6.9/1000	30	-0.9/+0.2	-2.3/+0.5	+0.1/+0.2	-0.5/+0.3
CO/H_2O, H_2O saturated	199/ 390	3.2/465	10	-0.5/-0.1	-0.1/nil	+0.1/-0.2	-0.4/-0.1
CO/H_2O	260/ 500	3.2/465	10	+0.3/-0.1	+1.5/-0.2	+0.3/-0.1	+0.6/nil
CO/H_2O	538/1000	3.2/465	10	-2.5/-0.2	-3.8/-0.4	-1.9/-0.1	-2.0/-0.1

DENSE HIGH-ALUMINA CASTABLES (GENERIC PREPARATION)

Atmosphere[a]	Temperature °C / °F	Pressure MPa/psi	Time days	93% Al_2O_3[j]	91% Al_2O_3[k]	91% Al_2O_3[l]	88% Al_2O_3[m]	84% Al_2O_3[n]
Air (fired)	260/ 500	ambient	3/4	-3.2/-0.6	-4.3/-0.2	-3.0/-1.2	-4.0/nil	-3.7/nil
Air (fired)	538/1000	ambient	3/4	-5.0/-0.3	-5.0/-0.1	-4.0/-0.3	-4.4/nil	-4.1/nil
CGA exposure	260/ 500	3.4/500	10	-0.4/+0.1	nil/nil	+1.0/-0.2		
			20	-1.6/-0.6	-1.8/nil	-1.4/-0.5		
			30	-0.4/-0.3	-0.1/-0.2	+0.2/-0.4		
CGA exposure	538/1000	6.9/1000	10	-2.5/-0.1	-3.7/-0.1	-5.0/-0.5		
			20	-1.8/-0.4	-3.3/-0.4	-3.5/-1.0		
			30	-2.0/-0.2	-3.6/-0.5	-3.8/-0.9	NOT TESTED	NOT TESTED
CGA with H_2S	538/1000	6.9/1000	10	-2.0/-0.2	-4.2/-0.2	-2.5/-0.2		
			20	-2.2/-0.2	-4.0/-0.2	-2.5/-0.3		
			30	-1.2/-0.2	-4.0/-0.2	-2.8/-0.1		
CGA, H_2O saturated[i]	231/ 447	6.9/1000	30	+0.2/+0.1	--/--	-1.7/+0.3		
CGA with H_2S, H_2O satd.[i]	231/ 447	6.9/1000	30	+0.5/+0.3	-2.4/+0.3	+0.5/+0.7		
CO/H_2O, H_2O saturated	199/ 390	3.2/465	10	-0.2/-0.2	-1.4/-0.1	+0.7/-0.1		
CO/H_2O	260/ 500	3.2/465	10	-0.2/-0.1	+0.7/-0.1	+2.5/-0.2		
CO/H_2O	538/1000	3.2/465	10	-2.6/-0.2	-4.8/-0.2	-4.8/-0.3		

INTERMEDIATE-ALUMINA DENSE AND INSULATING CASTABLES

Atmosphere[a]	Temperature °C / °F	Pressure MPa/psi	Time days	54-57% Al_2O_3[o]	57% Al_2O_3[p]	59% Al_2O_3[q]	54% Al_2O_3[r]
Air (fired)	260/ 500	ambient	3/4	-3.0/nil	-3.2/-0.1	-3.4/nil	-2.1/-0.3
Air (fired)	538/1000	ambient	3/4	-4.7/-0.2	-5.0/-0.2	-5.0/+0.1	-3.5/nil
CGA exposure	260/ 500	3.4/500	10	-0.1/nil	-0.8/-0.1	-1.5/-0.1	-1.0/+0.3
			20	-1.8/nil	-2.2/nil	-2.0/-0.4	-1.5/+0.1
			30	-0.8/-0.3	-1.1/-0.3	-1.7/-0.1	-1.1/+0.2
CGA exposure	538/1000	6.9/1000	10	-4.2/nil	-4.0/-0.1	-4.2/-0.2	-2.9/+0.1
			20	-3.8/nil	-4.2/nil	-4.7/-0.1	-3.1/nil
			30	-4.2/nil	-4.4/-0.1	-5.0/-0.2	-3.2/+0.1
CGA with H_2S	538/1000	6.9/1000	10	-4.0/-0.1	-4.5/-0.2	-4.7/-0.2	-2.9/-0.1
			20	-3.7/-0.05	-4.0/-0.2	-4.5/-0.2	-2.7/nil
			30	-4.1/+0.05	-4.1/+0.1	-4.7/-0.1	-2.9/nil
CGA, H_2O saturated[i]	231/ 447	6.9/1000	30	-4.2/+0.1	-5.2/nil	-5.0/nil	-1.9/-0.1
CGA with H_2S, H_2O satd.[i]	231/447	6.9/1000	30	-1.9/+0.3	-2.7/+0.2	+0.3/+0.1	+1.4/+0.6
CO/H_2O, H_2O saturated	199/ 390	3.2/465	10	-1.5/nil	-1.6/+0.1	-2.7/nil	-2.3/+0.3
CO/H_2O	260/ 500	3.2/465	10	-1.6/nil	-0.1/+0.1	-2.1/nil	-1.3/+0.5
CO/H_2O	538/1000	3.2/465	10	-4.5/-0.2	-4.5/-0.1	-4.1/-0.2	-4.7/-0.2

(Table Continued)

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EFFECT OF HEAT TREATMENT AND GASEOUS ENVIRONMENT[a] ON THE WEIGHT AND DIMENSIONS OF SAMPLES OF ALUMINA REFRACTORIES[b][39], Continued

Atmosphere[a]	Treatment Conditions Temperature °C / °F	Pressure MPa/psi	Time days	Weight Change, % / Dimensional Change, %[c] 96% Al_2O_3[s]	96% Al_2O_3[t]	90% Al_2O_3[u]
- PHOSPHATE-BONDED RAMMING MIXES -						
CGA exposure	260/ 500	3.4/500	10	+0.1/-0.3	+0.2/-0.2	-1.1/nil
			20	+0.1/-0.1	nil/nil	-0.1/-0.1
			30	nil/nil	+0.1/nil	-0.1/nil
CGA exposure	538/1000	6.9/1000	10	-0.1/nil	+0.4/nil	-0.7/-0.1
			20	-0.2/nil	-0.1/nil	-0.3/-0.1
			30	-0.1/nil	+0.2/nil	-0.1/nil
CGA with H_2S	538/1000	6.9/1000	10	-0.3/-0.1	-0.2/nil	-0.4/nil
			20	-0.3/+0.2	-0.2/nil	-0.6/+0.1
			30	-0.3/+0.2	-0.1/+0.1	-0.7/nil
CGA, H_2O saturated[i]	231/ 447	6.9/1000	30	+0.2/+0.2	+1.0/+0.1	-3.3/nil
CGA with H_2S, H_2O satd.[i]	231/ 447	6.9/1000	30	+2.0/+0.3	+2.9/+0.3	-0.3/+0.1
CO/H_2O, H_2O saturated	199/ 390	3.2/465	10	-1.0/-0.1	-0.4/-0.1	-2.5/-0.1
CO/H_2O	260/ 500	3.2/465	10	+0.1/nil	+0.1/-0.1	+0.1/-0.2
	538/1000	3.2/465	10	-0.3/-0.1	nil/-0.1	-0.6/nil

[a]Environments included a simulated coal gasification atmosphere (CGA) with an overall composition (in vol %) of 18 CO, 12 CO_2, 24 H_2, 5 CH_4, and 41 H_2O; another CGA which has 1% H_2S added; and a $CO-H_2O$ atmosphere which is 52.5 CO and 46.5 H_2O (vol %). Samples were exposed in two pressure vessels. One vessel was a steam generator and tests specifying saturated conditions occurred in this vessel. After placing samples, the vessel was heated to the desired steam pressure and the other gases were then added so as to obtain the concentrations specified above. The second vessel was connected to the steam generator and after samples were placed and the vessel heated to the test temperature, the vessel was pressurized with the steam/gas mixture from the steam generator. A gaseous flow rate for both vessels was maintained so as to provide one complete change in atmosphere every 24 hours.

[b]All cement-bonded castables were cast in molds either 12 x 3 x 1/2 in. or 3 x 3/4 x 3/4 in., cured 24 h in air at 100% humidity at ambient temperature, and dried at 230 °F for 24 h. The larger slabs were then cut to 3 x 1/2 x 1/2 in. bars. Phosphate-bonded specimens were prepared by hand ramming in 3 x 3/4 x 3/4 in. molds, drying immediately for 24 h at 230 °F, and then firing at 1000 °F for 18 h. All specimens were stored at 230 °F until tested.

[c]Data are the averages for seven specimens except where noted. Change in weight or dimension is with as-dried value as initial value (as-fired for the phosphate-bonded materials); + indicates increase, - indicates decrease.

[d]94% alumina dense castable, calcium aluminate (79% alumina, 18% calcia) bonded (Greencast-94, A.P. Green; CA-25 Calcium Aluminate Cement, Alcoa).

[e]93% alumina dense castable, calcium aluminate (72% alumina, 25% calcia) bonded (B&W Kao-Tab 93, Babcock & Wilcox; C-3 cement, Babcock & Wilcox).

[f]90-95% alumina dense castable, calcium aluminate (79% alumina, 18% calcia) bonded (Castolast G, Harbison-Walker; CA-25 Calcium Aluminate Cement, Alcoa).

[g]90-95% alumina dense castable, calcium aluminate (79% alumina, 18% calcia) bonded (Purotab, Kaiser Refractories; CA-25 Calcium Aluminate Cement, Alcoa).

[h]Original reports do not indicate direction of the change.

[i]Data are for two specimens only.

[j]93% alumina dense castable, UMR-1 generic preparation (70% tabular alumina + 30% calcium aluminate cement) (T-61 alumina, Alcoa; CA-25 Calcium Aluminate Cement, Alcoa).

[k]91% alumina dense castable, UMR-2 generic preparation (70% tabular alumina + 30% calcium aluminate cement) (T-61 alumina, Alcoa; Secar 71(250), a 72% alumina-26% calcia cement, Lone Star Lafarge).

[l]91% alumina dense castable, UMR-3 generic preparation (70% tabular alumina + 30% calcium aluminate cement) (T-61 alumina, Alcoa; C-3, a 72% alumina-25% calcia cement, Babcock & Wilcox).

[m]88% alumina dense castable, UMR-6 generic preparation (UMR-1 preparation with 5% SiO_2 added as 99.9% pure bone dry Wedron silica flour).

[n]84% alumina dense castable, UMR-7 generic preparation (UMR-1 preparation with 10% SiO_2 added as 99.9% pure bone dry Wedron silica flour).

[o]54-57% alumina, 34-37% silica castable, calcium aluminate (79% alumina, 18% calcia) bonded (Lo-Abrade, A.P. Green; CA-25 Calcium Aluminate Cement, Alcoa).

[p]57% alumina, 34% silica castable, calcium aluminate (79% alumina, 18% calcia) bonded (RC-3, General Refractories; CA-25 Calcium Aluminate Cement, Alcoa).

[q]59% alumina, 33% silica insulating castable, calcium aluminate bonded (Kast-O-Lite 30, A.P. Green; CA-25 cement, Alcoa).

[r]54% alumina, 40% silica insulating castable, calcium aluminate bonded (Cer-Lite #75, C-E Refractories; CA-25 cement, Alcoa).

[s]96% alumina dense phosphate-bonded ramming mix (Greenpak-90P, A.P. Green).

[t]96% alumina dense phosphate-bonded ramming mix (90 Ram H.S., C-E Refractories).

[u]90% alumina dense phosphate-bonded ramming mix (Brikram 90R, General Refractories).

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EFFECT OF HEAT TREATMENT AND GASEOUS ENVIRONMENT[a] ON THE DENSITY AND POROSITY OF VARIOUS ALUMINA REFRACTORIES[b][39]

DENSE HIGH-ALUMINA COMMERCIAL CASTABLES

Atmosphere[a]	Temperature °C / °F	Pressure MPa/psi	Time days	94% Al_2O_3[d]	93% Al_2O_3[e]	90-95% Al_2O_3[f]	90-95% Al_2O_3[g]
Air (dried)	110/ 230	ambient	1	2.77/14	2.62/19	2.85/14	2.88/14
Air (fired)	260/ 500	ambient	3/4	2.75/20	2.56/25	2.81/17	2.84/17
Air (fired)	538/1000	ambient	3/4	2.67/25	2.49/28	2.74/20	2.78/22
CGA exposure	260/ 500	3.4/500	10	2.81/19	2.59/25	2.87/19	2.87/19
			20	2.78/21	2.59/26	2.85/19	2.88/20
			30	2.81/20	2.63/24	2.88/19	2.90/18
CGA exposure	538/1000	6.9/1000	10	2.77/24	2.57/28	2.85/21	2.87/21
			20	2.75/24	2.58/28	2.85/21	2.85/22
			30	2.75/24	2.57/28	2.85/21	2.87/21
CGA with H_2S	538/1000	6.9/1000	10	2.76/24	2.54/28	2.77/23	2.86/21
			20	2.77/24	2.54/28	2.77/24	2.87/21
			30	-- /24	2.55/28	2.77/24	2.89/21
CGA, H_2O saturated[h]	231/ 447	6.9/1000	30	2.79/13	2.53/29	not tested	2.94/ 6
CGA with H_2S, H_2O satd.[h]	231/ 447	6.9/1000	30	2.77/16	2.51/29	2.79/16	2.87/13
CO/H_2O, H_2O saturated	199/ 390	3.2/465	10	2.78/ 5	2.54/27	2.87/ 4	2.89/ 4
CO/H_2O	260/ 500	3.2/465	10	2.83/20	2.67/23	2.89/17	2.93/18
	538/1000	3.2/465	10	2.75/24	2.56/27	2.84/21	2.85/21

DENSE HIGH-ALUMINA CASTABLES (GENERIC PREPARATION)

Atmosphere[a]	Temperature °C / °F	Pressure MPa/psi	Time days	93% Al_2O_3[i]	91% Al_2O_3[j]	91% Al_2O_3[k]	88% Al_2O_3[l]	84% Al_2O_3[m]
Air (dried)	110/ 230	ambient	1	2.75/16	2.66/16	2.47/23	-- /-	-- /-
Air (fired)	260/ 500	ambient	3/4	2.71/20	2.58/24	2.45/25	2.62/22	2.58/24
Air (fired)	538/1000	ambient	3/4	2.68/23	2.52/28	2.35/31	2.59/27	2.54/27
CGA exposure	260/ 500	3.4/500	10	2.74/23	2.70/22	2.50/27		
			20	2.76/22	2.62/23	2.47/29		
			30	2.75/21	2.68/22	2.52/27		
CGA exposure	538/1000	6.9/1000	10	2.73/24	2.61/26	2.46/31		
			20	2.74/24	2.60/26	2.44/31		
			30	2.73/26	2.59/27	2.44/32		
CGA with H_2S	538/1000	6.9/1000	10	2.70/26	2.59/27	2.44/31	NOT TESTED	NOT TESTED
			20	2.76/25	2.61/26	2.44/31		
			30	2.78/24	2.60/27	2.45/31		
CGA, H_2O saturated[h]	231/ 447	6.9/1000	30	2.75/11	not tested	2.44/28		
CGA with H_2S, H_2O satd.[h]	231/ 447	6.9/1000	30	2.73/15	2.58/18	2.44/28		
CO/H_2O, H_2O saturated	199/ 390	3.2/465	10	2.72/ 5	2.61/ 7	-- /-		
CO/H_2O	260/ 500	3.2/465	10	2.74/22	2.70/21	-- /-		
	538/1000	3.2/465	10	2.73/24	2.58/26	-- /-		

INTERMEDIATE-ALUMINA DENSE AND INSULATING CASTABLES

Atmosphere[a]	Temperature °C / °F	Pressure MPa/psi	Time days	54-57% Al_2O_3[n]	57% Al_2O_3[o]	59% Al_2O_3[p]	54% Al_2O_3[q]
Air (dried)	110/ 230	ambient	1	2.33/13	2.31/16	1.53/43	1.58/39
Air (fired)	260/ 500	ambient	3/4	2.29/17	2.27/19	1.52/46	1.55/41
Air (fired)	538/1000	ambient	3/4	2.26/20	2.21/23	1.46/49	1.54/42
CGA exposure	260/ 500	3.4/500	10	2.32/18	2.30/19	1.50/48	1.55/45
			20	2.30/19	2.28/21	1.50/49	1.51/44
			30	2.32/18	2.30/20	1.50/48	1.52/44
CGA exposure	538/1000	6.9/1000	10	2.26/21	2.24/22	1.48/52	1.53/50
			20	2.27/20	2.25/22	1.48/52	1.53/51
			30	2.25/21	2.25/22	1.49/52	1.53/52
CGA with H_2S	538/1000	6.9/1000	10	2.23/22	2.24/23	1.52/51	1.54/51
			20	2.23/21	2.25/22	1.53/50	1.54/51
			30	2.23/22	2.24/22	1.53/51	1.54/51
CGA, H_2O saturated[h]	231/ 447	6.9/1000	30	2.24/12	2.15/22	1.45/54	1.54/52
CGA with H_2S, H_2O satd.[h]	231/ 447	6.9/1000	30	2.26/16	2.26/17	1.59/45	1.59/45
CO/H_2O, H_2O saturated	199/ 390	3.2/465	10	2.24/18	2.31/11	1.47/51	1.52/48
CO/H_2O	260/ 500	3.2/465	10	2.32/19	2.34/17	1.51/49	1.55/45
	538/1000	3.2/465	10	2.27/20	2.24/23	1.49/51	1.54/47

Density (g/cm^3) / Apparent Porosity (%)[c]

(Table Continued)

EFFECT OF HEAT TREATMENT AND GASEOUS ENVIRONMENT[a] ON THE DENSITY AND POROSITY OF VARIOUS ALUMINA REFRACTORIES[b][39], Continued

Atmosphere[a]	Treatment Conditions Temperature °C / °F	Pressure MPa/psi	Time days	Density (g/cm^3) / Apparent Porosity (%)[c] 96% Al_2O_3[r]	96% Al_2O_3[s]	90% Al_2O_3[t]
				PHOSPHATE-BONDED RAMMING MIXES		
Air (fired)	538/1000	ambient	3/4	2.95/17	2.92/20	2.96/17
CGA exposure	260/ 500	3.4/500	10	2.91/19	2.90/20	2.90/20
			20	2.97/18	2.94/20	2.95/18
			30	2.97/18	2.94/20	2.94/18
CGA exposure	538/1000	6.9/1000	10	2.90/18	2.91/18	2.94/16
			20	2.94/18	2.94/19	3.00/15
			30	2.95/18	2.96/19	3.00/16
CGA with H_2S	538/1000	6.9/1000	10	2.87/20	2.92/20	2.97/17
			20	2.88/20	2.94/19	3.00/16
			30	2.87/20	2.91/19	2.95/18
CGA, H_2O saturated[h]	231/ 447	6.9/1000	30	2.96/21	2.94/21	2.93/21
CGA with H_2S, H_2O satd.[h]	231/ 447	6.9/1000	30	2.90/21	2.99/19	2.97/19
CO/H_2O, H_2O saturated	199/ 390	3.2/465	10	2.87/21	2.89/21	3.02/17
CO/H_2O	260/ 500	3.2/465	10	2.87/26	2.94/20	3.08/14
	538/1000	3.2/465	10	2.90/19	2.90/20	3.04/15

[a]Environments included a simulated coal gasification atmosphere (CGA) with an overall composition (in vol %) of 18 CO, 12 CO_2, 24 H_2, 5 CH_4, and 41 H_2O; another CGA which has 1% H_2S added; and a $CO-H_2O$ atmosphere which is 52.5 CO and 46.5 H_2O (vol %). Samples were exposed in two pressure vessels. One vessel was a steam generator and tests specifying saturated conditions occurred in this vessel. After placing samples, the vessel was heated to the desired steam pressure and the other gases were then added so as to obtain the concentrations specified above. The second vessel was connected to the steam generator and after samples were placed and the vessel heated to the test temperature, the vessel was pressurized with the steam/gas mixture from the steam generator. A gaseous flow rate for both vessels was maintained so as to provide one complete change in atmosphere every 24 hours.

[b]All cement-bonded castables were cast in molds either 12 x 3 x 1/2 in. or 3 x 3/4 x 3/4 in., cured 24 h in air at 100% humidity at ambient temperature, and dried at 230 °F for 24 h. The larger slabs were then cut to 3 x 1/2 x 1/2 in. bars. Phosphate-bonded specimens were prepared by hand ramming in 3 x 3/4 x 3/4 in. molds, drying immediately for 24 h at 230 °F, and then firing at 1000 °F for 18 h. All specimens were stored at 230 °F until tested.

[c]Data are the averages for seven specimens except where noted. ASTM C20-74 procedure followed except that kerosene was used.

[d]94% alumina dense castable, calcium aluminate (79% alumina, 18% calcia) bonded (Greencast-94, A.P. Green; CA-25 Calcium Aluminate Cement, Alcoa).

[e]93% alumina dense castable, calcium aluminate (72% alumina, 25% calcia) bonded (B&W Kao-Tab 93, Babcock & Wilcox; C-3 cement, Babcock & Wilcox).

[f]90-95% alumina dense castable, calcium aluminate (79% alumina, 18% calcia) bonded (Castolast G, Harbison-Walker; CA-25 Calcium Aluminate Cement, Alcoa).

[g]90-95% alumina dense castable, calcium aluminate (79% alumina, 18% calcia) bonded (Purotab, Kaiser Refractories; CA-25 Calcium Aluminate Cement, Alcoa).

[h]Data are for two specimens only.

[i]93% alumina dense castable, UMR-1 generic preparation (70% tabular alumina + 30% calcium aluminate cement) (T-61 alumina, Alcoa; CA-25 Calcium Aluminate Cement, Alcoa).

[j]91% alumina dense castable, UMR-2 generic preparation (70% tabular alumina + 30% calcium aluminate cement) (T-61 alumina, Alcoa; Secar 71(250), a 72% alumina-26% calcia cement, Lone Star Lafarge).

[k]91% alumina dense castable, UMR-3 generic preparation (70% tabular alumina + 30% calcium aluminate cement) (T-61 alumina, Alcoa; C-3, a 72% alumina-25% calcia cement, Babcock & Wilcox).

[l]88% alumina dense castable, UMR-6 generic preparation (UMR-1 preparation with 5% SiO_2 added as 99.9% pure bone dry Wedron silica flour).

[m]84% alumina dense castable, UMR-7 generic preparation (UMR-1 preparation with 10% SiO_2 added as 99.9% pure bone dry Wedron silica flour).

[n]54-57% alumina, 34-37% silica castable, calcium aluminate (79% alumina, 18% calcia) bonded (Lo-Abrade, A.P. Green; CA-25 Calcium Aluminate Cement, Alcoa).

[o]57% alumina, 34% silica castable, calcium aluminate (79% alumina, 18% calcia) bonded (RC-3, General Refractories; CA-25 Calcium Aluminate Cement, Alcoa).

[p]59% alumina, 33% silica insulating castable, calcium aluminate bonded (Kast-O-Lite 30, A.P. Green; CA-25 cement, Alcoa).

[q]54% alumina, 40% silica insulating castable, calcium aluminate bonded (Cer-Lite #75, C-E Refractories; CA-25 cement, Alcoa).

[r]96% alumina dense phosphate-bonded ramming mix (Greenpak-90P, A.P. Green).

[s]96% alumina dense phosphate-bonded ramming mix (90 Ram H.S., C-E Refractories).

[t]90% alumina dense phosphate-bonded ramming mix (Brikram 90R, General Refractories).

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EFFECT OF HEAT TREATMENT, GASEOUS ENVIRONMENT, AND SATURATED CONDITIONS[a] ON THE WEIGHT AND DIMENSIONS OF SAMPLES OF CALCIUM ALUMINATE CEMENTS[b][39]

Atmosphere[a]	Treatment Conditions Temperature °C / °F	Pressure MPa/psi	Time days	Initially Dry Cement[b] % Weight Change[c]	Hydrated Cement[b] % Weight Change[c]	Hydrated Cement[b] % Dimensional Change[c]
- - - - - - - - - - - - - - -	79% ALUMINA, 18% CALCIA CEMENT[d]	- - - -	- -	- - - - -	- - - - -	- - - - -
CGA exposure	260/ 500	3.4/500	10	+16.5		
			20	+ 8.8	nil	- 4.3
			30	+16.0		
CGA exposure	538/1000	6.9/1000	10	+12.5	- 2.2	+ 0.6
			20	+11.3	- 1.1	- 1.3
			30	+13.0	- 1.6	- 0.3
CGA with H_2S	538/1000	6.9/1000	10	+12.6	- 4.0	- 1.2
			20	+12.4	- 3.0	- 0.6
			30	+14.8	- 4.7	- 0.2
CGA, H_2O saturated[e]	231/ 447	6.9/1000	30	+18.3	+ 2.0	+ 0.7
CGA with H_2S, H_2O satd.[e]	231/ 447	6.9/1000	30	+ 7.3	- 2.6	+ 1.0
CO/H_2O, H_2O saturated	199/ 390	3.2/465	10	+ 9.5	+ 5.4	- 0.6
CO/H_2O	260/ 500	3.2/465	10	+20.3	+ 7.3	+ 1.1
	538/1000	3.2/465	10	+ 5.9	- 2.3	- 0.9
Saturated steam { vapor	231/ 447	2.8/410	10	+20.6	+ 5.4	+ 0.8
{ liquid	231/ 447	2.8/410	10		+ 3.8	+ 1.2
Saturated steam { vapor	285/ 545	6.9/1000	10	+ 9.7	+ 5.66	+ 0.38
{ liquid	285/ 545	6.9/1000	10		+ 3.82	+ 0.77
CGA with H_2S, { vapor	231/ 447	6.9/1000	10		+11.60	+ 0.72
H_2O saturated { liquid	231/ 447	6.9/1000	10		+11.83	+ 0.73
- - - - - - - - - - - - - -	72% ALUMINA, 26% CALCIA CEMENT[f]	- - -	- -	- - - - -	- - - - -	- - - - -
CGA exposure	260/ 500	3.4/500	10	+16.3		
			20	+15.2	+ 3.7	- 0.6
			30	+16.2		
CGA exposure	538/1000	6.9/1000	10	+16.9	+ 2.4	+ 0.7
			20	+19.9	- 1.8	- 1.2
			30	+18.0	+ 5.4	- 0.3
CGA with H_2S	538/1000	6.9/1000	10	+ 8.4	- 5.3	- 1.5
			20		- 0.3	- 1.0
			30		- 3.3	- 0.6
CGA, H_2O saturated[e]	231/ 447	6.9/1000	30		+ 5.5	+ 0.7
CGA with H_2S, H_2O satd.[e]	231/ 447	6.9/1000	30		+ 0.6	+ 0.8
CO/H_2O, H_2O saturated	199/ 390	3.2/465	10	+25.7	+ 6.7	+ 0.7
CO/H_2O	260/ 500	3.2/465	10	+ 6.4	+13.5	+ 0.6
	538/1000	3.2/465	10	+ 4.3	- 6.6	- 1.7
Saturated steam { vapor	231/ 447	2.8/410	10	+22.5	nil	+ 0.7
{ liquid	231/ 447	2.8/410	10		- 1.0	+ 1.3
Saturated steam { vapor	285/ 545	6.9/1000	10	+21.3	+7.27	+ 0.28
{ liquid	285/ 545	6.9/1000	10		+ 4.91	+ 0.49
CGA with H_2O, { vapor	231/ 447	6.9/1000	10		+10.88	+ 0.72
H_2O saturated { liquid	231/ 447	6.9/1000	10		+10.55	+ 0.72

(Table Continued)

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EFFECT OF HEAT TREATMENT, GASEOUS ENVIRONMENT, AND SATURATED CONDITIONS[a] ON THE WEIGHT AND
DIMENSIONS OF SAMPLES OF CALCIUM ALUMINATE CEMENTS[b][39], Continued

| | Treatment Conditions | | | Initially Dry Cement[b] | Hydrated Cement[b] | |
Atmosphere[a]	Temperature °C / °F	Pressure MPa/psi	Time days	% Weight Change[c]	% Weight Change[c]	% Dimensional Change[c]
— — — — — — — — — — — 72% ALUMINA, 25% CALCIA CEMENT[g] — — — — — — — — — — — — — —						
CGA exposure	260/ 500	3.4/500	10	+19.3		
			20	+ 6.4	+ 4.8	− 3.1
			30	+19.5		
CGA exposure	538/1000	6.9/1000	10	+11.7	− 6.3	+ 0.6
			20	+13.8	− 2.3	− 1.7
			30	+11.8	− 2.9	− 0.2
CGA with H_2S	538/1000	6.9/1000	10	+11.5	− 6.1	− 1.5
			20	+ 3.3	− 4.4	− 1.0
			30	+14.2	− 3.7	− 0.9
CGA, H_2O saturated[e]	231/ 447	6.9/1000	30	+18.7	− 4.2	+ 2.0
CGA with H_2S, H_2O satd.[e]	231/ 447	6.9/1000	30		+ 1.9	+ 1.1
CO/H_2O, H_2O saturated	199/ 390	3.2/465	10	+ 2.3	+10.3	+ 2.4
CO/H_2O	260/ 500	3.2/465	10	+31.1	+16.5	nil
	538/1000	3.2/465	10	− 0.7	− 5.5	− 1.0
— — — — — — — — — — — — 58% ALUMINA, 33% CALCIA CEMENT[h] — — — — — — — — — — — — — —						
CGA exposure	260/ 500	3.4/500	20	+ 7.6	+ 1.0	− 0.6
CGA exposure	538/1000	6.9/1000	10	+12.8	− 5.0	− 0.2
			20	+17.0	− 4.7	− 1.0
			30	+12.1	+ 0.7	nil
CGA with H_2S	538/1000	6.9/1000	10	+ 3.7	− 6.3	− 1.2
			20	+15.7	− 2.2	− 0.9
			30	+ 9.6	− 3.7	− 0.7
CGA, H_2O saturated[e]	231/ 447	6.9/1000	30		+ 4.3	+ 1.0
CGA with H_2S, H_2O satd.[e]	231/ 447	6.9/1000	30		+ 0.1	+ 0.6
CO/H_2O, H_2O saturated	199/ 390	3.2/465	10	+15.1	+ 2.4	+ 0.8
CO/H_2O	260/ 500	3.2/465	10	+32.2	+10.6	+ 4.6
	538/1000	3.2/465	10	− 1.9	− 7.1	− 1.1
Saturated steam { vapor	231/ 447	2.8/410	10	+15.7	+ 4.7	+ 0.9
{ liquid	231/ 447	2.8/410	10		+ 2.4	+ 1.1
Saturated steam { vapor	285/ 545	6.9/1000	10		+ 2.92	+ 0.24
{ liquid	285/ 545	6.9/1000	10		+ 2.73	+ 0.77
CGA with H_2S, { vapor	231/ 447	6.9/1000	10		+10.64	+ 0.71
H_2O saturated { liquid	231/ 447	6.9/1000	10		+10.71	+ 0.71

(Table Continued)

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EFFECT OF HEAT TREATMENT, GASEOUS ENVIRONMENT, AND SATURATED CONDITIONS[a] ON THE WEIGHT AND DIMENSIONS OF SAMPLES OF CALCIUM ALUMINATE CEMENTS[b][39], Continued

Treatment Conditions				Initially Dry Cement[b]	Hydrated Cement[b]	
Atmosphere[a]	Temperature °C / °F	Pressure MPa/psi	Time days	% Weight Change[c]	% Weight Change[c]	% Dimensional Change[c]
- - - - - - - - - - - - - - - 44% ALUMINA, 36% CALCIA CEMENT[i] - - - - - - - - - - - - - - -						
CGA exposure	260/ 500	3.4/500	20	+ 7.1	+ 1.5	- 0.8
CGA exposure	538/1000	6.9/1000	10		- 2.3	- 0.7
			20	+13.8	- 4.2	- 1.3
			30		- 1.5	+ 0.5
CGA with H_2S	538/1000	6.9/1000	10	+ 0.14	- 6.1	- 1.5
			20	+ 6.84	- 3.9	- 1.3
			30	+ 0.5	- 4.9	- 0.8
CGA, H_2O saturated[e]	231/ 447	6.9/1000	30		+ 1.8	+ 1.4
CGA with H_2S, H_2O satd.[e]	231/ 447	6.9/1000	30		- 8.7	nil
CO/H_2O, H_2O saturated	199/ 390	3.2/465	10	+14.4	- 3.0	+ 1.0
CO/H_2O	260/ 500	3.2/465	10	+24.8	+ 4.9	- 0.1
	538/1000	3.2/465	10	- 0.5	- 8.0	- 2.0

[a] Environments included a simulated coal gasification atmosphere (CGA) with an overall composition (in vol %) of 18 CO, 12 CO_2, 24 H_2, 5 CH_4, and 41 H_2O; another CGA which has 1% H_2S added; and a CO-H_2O atmosphere which is 52.5 CO and 46.5 H_2O (vol %). Samples were exposed in two pressure vessels. One vessel was a steam generator and tests specifying saturated conditions occurred in this vessel. For some of the tests samples were placed both in the vapor and in the liquid in the bottom of the vessel. After placing samples, the vessel was heated to the desired steam pressure and the other gases were then added so as to obtain the concentrations specified above. The second vessel was connected to the steam generator and after samples were placed and the vessel heated to the test temperature, the vessel was pressurized with the steam/gas mixture from the steam generator. A gaseous flow rate for both vessels was maintained so as to provide one complete change in atmosphere every 24 hours.

[b] Cements were tested as initially dry powders and as neat cement specimens. Hydrated neat cement samples (water/cement ratio of $\sim$0.3) were cast as 1/2-inch cubes, cured in air at 100% humidity for 24 hours, and dried at 230 °F for 24 hours. Samples were stored at 230 °F until tested.

[c] Data are for seven samples except where noted. Change in weight or dimension is with as-dried value as the initial value; + indicates increase, - indicates decrease. No data are given for the fired condition.

[d] 79% Alumina, 18% calcia cement (CA-25 Calcium Aluminate Cement, Alcoa).

[e] Data are for two samples only.

[f] 72% Alumina, 26% calcia cement (Secar 71(250), Lone Star Lafarge).

[g] 71.5-72.5% Alumina, 24-25% calcia cement (C-3, Babcock & Wilcox).

[h] 58% Alumina, 33% calcia cement (Refcon, Universal Atlas Cement).

[i] 44% Alumina, 36% calcia cement (Lumnite, Universal Atlas Cement).

EFFECT OF SATURATED VAPOR AND LIQUID AND EXPOSURE[a] TO PRESSURE-TEMPERATURE CYCLING CAUSING BOEHMITE FORMATION[b] AND DECOMPOSITION ON THE WEIGHT AND DIMENSIONS OF CASTABLE ALUMINA REFRACTORY SPECIMENS[c][39]

Atmosphere[a]	Treatment Conditions Temperature °C / °F	Pressure MPa/psi	Time days	Weight Change, % / Dimensional Change, %[d]			
				— — — — — — — — DENSE CASTABLES — — — — — — — —			
				93% Al_2O_3[e]	87% Al_2O_3[f]	64% Al_2O_3[g]	58% Al_2O_3[h]
Air (fired)	260/ 500	ambient	3/4	-3.2/-0.6	-4.6/nil	-3.0/-0.1	-4.2/nil
Air (fired)	538/1000	ambient	3/4	-5.0/-0.3	-5.8/-0.1	-3.8/-0.1	-4.7/nil
Saturated steam { vapor	231/ 447	2.8/410	10	-0.2/+0.1	-0.7/+0.1	-1.8/+0.1	-0.8/+0.2
{ liquid	231/ 447	2.8/410	10	+0.5/+0.2	-0.5/+0.3	-1.5/+0.4	-1.2/+0.3
Saturated steam { vapor	285/ 545	6.9/1000	10	-0.8/+0.3	-1.0/+0.2	-1.8/+0.1	-1.6/+0.2
{ liquid	285/ 545	6.9/1000	10	+0.8/+0.1	nil/+0.1	-2.2/+0.1	-1.1/+0.2
CGA with H_2S, { vapor	231/ 447	6.9/1000	10	+1.2/+0.1	+3.7/+0.1	+0.1/+0.03	+2.1/-0.3
H_2O saturated { liquid	231/ 447	6.9/1000	10	+0.8/+0.1	+1.8/+0.04	-0.7/0	+2.0/-0.1
CGA with H_2S, { vapor	231/ 447	6.9/1000	44	+0.7/+0.5	+1.7/+0.5	-1.6/nil	nil/nil
H_2O saturated { liquid	231/ 447	6.9/1000	44	+1.3/+0.1	not tested	+0.1/nil	not tested
CGA with H_2S, saturated[i]	240/ 465	6.9/1000	5	+0.4/+0.3	+2.3/nil	+0.5/+0.1	+2.1/nil
followed by firing in air[j]	538/1000	ambient	3/4	-2.7/+0.4	-1.3/nil	-2.1/-0.2	-1.1/-0.1
Cycling tests--repeat of the preceding exposure followed by the same air firing							
			1 cycle	-2.4/-0.1	-1.0/nil	-2.0/-0.3	-1.1/-0.1
			2 cycles	-2.2/-0.1	-0.7/-0.1	-2.0/-0.2	-0.8/-0.1
			3 cycles	-2.2/nil	-1.3/-0.2	-2.3/-0.1	-1.4/-0.1
			4 cycles	-2.3/nil	-0.9/-0.2	-2.4/-0.1	-2.3/-0.2
			5 cycles	-1.8/+0.2	-0.5/-0.1	-2.1/nil	-1.2/-0.1
				— — — — — — — — INSULATING CASTABLES — — — — — — — —			
				54% Al_2O_3[k]	47% Al_2O_3[l]	35% Al_2O_3[m]	
Air (fired)	260/ 500	ambient	3/4	-2.1/-0.3	-5.4/nil	-4.5/nil	
Air (fired)	538/1000	ambient	3/4	-3.6/nil	-6.4/nil	-5.6/-0.4	
Saturated steam { vapor	231/ 447	2.8/410	10	-1.6/+0.3	-1.6/+0.7	-4.6/+0.5	
{ liquid	231/ 447	2.8/410	10	-2.3/+0.5	-3.6/+0.2	-5.3/+0.7	
Saturated steam { vapor	285/ 545	6.9/1000	10	-1.9/+0.7	-3.9/+0.6	-9.1/+0.5	
{ liquid	285/ 545	6.9/1000	10	-3.7/+1.2	-3.2/+0.2	-9.3/+0.5	
CGA with H_2S, { vapor	231/ 447	6.9/1000	10	+0.4/+0.5	+1.3/-0.04	-0.2/+0.2	
H_2O saturated { liquid	231/ 447	6.9/1000	10	+0.7/+0.6	+2.5/+0.1	+2.4/+0.9	
CGA with H_2S, { vapor	231/ 447	6.9/1000	44	+0.8/+0.4	-3.5/+0.2	-2.3/+0.7	
H_2O saturated { liquid	231/ 447	6.9/1000	44	+9.1/-0.3	not tested	+9.1/+0.1	
CGA with H_2S, saturated[i]	240/ 447	6.9/1000	5	-0.2/+0.4	+1.2/+0.2	+0.3/no test	
followed by firing in air[j]	538/1000	ambient	3/4	-1.6/no test	-3.4/no test	-4.8/no test	
Cycling tests--repeat of the preceding exposure followed by the same air firing							
			1 cycle	-2.2/+0.4	-3.9/+0.1	-4.7/+1.0	
			2 cycles	-2.0/+0.4	-3.9/+0.2	-4.3/+1.0	
			3 cycles	-2.2/+0.1	-2.7/nil	-4.2/+0.5	
			4 cycles	-2.5/+0.1	-4.2/-0.1	-5.6/+0.4	
			5 cycles	-2.7/+0.3	-3.8/nil	-4.6/+0.1	

[a] For testing the effect of water-saturated vapor and of liquid, samples were exposed in a steam generator, both in the vapor and in the liquid in the bottom of the vessel. After placement of samples, the vessel was heated to the desired steam pressure and where tests included other gases, these were then added so as to obtain the desired overall concentrations. The overall composition of the coal gasification atmosphere (CGA) is (in vol %) 18 CO, 12 CO_2, 24 H_2, 40 H_2O, 5 CH_4, and 1 H_2S. A gaseous flow rate was maintained so as to provide one complete change in atmosphere every 24 hours.

[b] Other tests showed that boehmite formation occurred in steam-saturated atmospheres and resulted in very large increases in the flexural strength. These cycling tests were designed to investigate the effect of repetitive formation and decomposition of boehmite on the properties of the refractories.

[c] After casting, specimens were cured 24 hours in air at 100% humidity at ambient temperature, and dried at

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(Table Continued)

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EFFECT OF SATURATED VAPOR AND LIQUID AND EXPOSURE[a] TO PRESSURE-TEMPERATURE CYCLING CAUSING BOEHMITE FORMATION[b] AND DECOMPOSITION ON THE WEIGHT AND DIMENSIONS OF CASTABLE ALUMINA REFRACTORY SPECIMENS[c][39], Continued

Footnotes continued

230 °F for 24 hours. Specimens were stored at 230 °F until tested.

[d]Values are averages for seven samples. Change in weight or dimension is with the as-dried value as the initial value; + indicates increase, - indicates decrease.

[e]93% alumina dense castable, UMR-1 generic preparation (70% tabular alumina + 30% calcium aluminate cement) (T-61 alumina, Alcoa; CA-25 Calcium Aluminate Cement, Alcoa).

[f]87.4% alumina dense castable, UMR-4 generic preparation (70% tabular alumina + 30% calcium aluminate cement) (T-61 alumina, Alcoa; Refcon, a 58% alumina-33% calcia cement, Universal Atlas).

[g]63.8% alumina, 28.3% silica dense castable, UMR-8 generic preparation (75% calcined kaolin aggregate + 25% calcium aluminate cement) (Mulcoa M-60 and Mulcoa M-47, C-E Minerals; CA-25 Calcium Aluminate Cement, a 79% alumina-18% calcia cement, Alcoa).

[h]58.6% alumina, 29.6% silica dense castable, UMR-5 generic preparation (75% calcined kaolin aggregate + 25% calcium aluminate cement) (Mulcoa M-60 and Mulcoa M-47, C-E Minerals; Refcon, a 58% alumina-33% calcia cement, Universal Atlas).

[i]85% steam saturated; this exposure is the one forming the first step in the cycling tests, forming boehmite.

[j]The firing step, following the saturated CGA exposure, completed one cycle, decomposing the boehmite.

[k]54% alumina, 40% silica insulating castable, calcium aluminate (79% alumina, 18% calcia) bonded (Cer-Lite #75, C-E Refractories; CA-25 Calcium Aluminate Cement, Alcoa).

[l]46.7% alumina, 40.2% silica insulating castable (Litecast 60-25, General Refractories).

[m]34.5% alumina, 52.5% silica insulating castable (VSL-50, A.P. Green).

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EFFECT OF SATURATED VAPOR AND LIQUID AND EXPOSURE[a] TO PRESSURE-TEMPERATURE CYCLING CAUSING BOEHMITE FORMATION[b]
AND DECOMPOSITION ON THE DENSITY AND POROSITY OF CASTABLE ALUMINA REFRACTORIES[c][39]

| Atmosphere[a] | Treatment Conditions | | | Density (g/cm^3) / Porosity (%)[d] | | | |
| | Temperature °C / °F | Pressure MPa/psi | Time days | DENSE CASTABLES | | | |
				93% Al_2O_3[e]	87% Al_2O_3[f]	64% Al_2O_3[g]	58% Al_2O_3[h]
Air (dried)	110/ 230	ambient	1	2.74/17.1	2.62/18.6	2.47/12.0	2.41/13.5
Air (fired)	260/ 500	ambient	3/4	2.69/22.5	2.58/23.4	2.38/16.5	2.44/16.3
Air (fired)	538/1000	ambient	3/4	2.66/26.2	2.54/27.0	2.38/19.9	2.37/18.9
Saturated steam { vapor	231/ 447	2.8/410	10	2.68/17.9	2.53/25.5	2.33/13.4	2.32/17.0
{ liquid	231/ 447	2.8/410	10	2.67/18.8	2.52/25.5	2.35/15.2	2.32/16.3
Saturated steam { vapor	285/ 545	6.9/1000	10	2.70/16.5	2.57/21.8	2.37/12.4	2.39/16.0
{ liquid	285/ 545	6.9/1000	10	2.67/10.2	2.54/14.6	2.36/13.2	2.36/15.0
CGA with H_2S, { vapor	231/ 447	6.9/1000	10	2.78/12.9	2.62/19.9	2.44/12.4	2.46/10.3
H_2O saturated { liquid	231/ 447	6.9/1000	10	2.75/13.2	2.63/20.2	2.41/12.0	2.45/10.5
CGA with H_2S, { vapor	231/ 447	6.9/1000	44	2.69/10.3	2.64/ 3.8	2.41/10.2	2.33/12.4
H_2O saturated { liquid	231/ 447	6.9/1000	44	2.73/ 8.3	not tested	2.41/10.3	not tested
CGA with H_2S, saturated[i]	240/ 465	6.9/1000	5	2.68/20.3	2.67/13.1	2.44/11.7	2.45/12.4
followed by firing in air[j]	538/1000	ambient	3/4	2.60/24.7	2.57/20.3	2.40/16.1	2.39/16.1
Cycling tests--repeat of the preceding exposure followed by the same air firing							
			1 cycle	2.69/25.7	2.59/24.8	2.42/18.3	2.38/17.9
			2 cycles	2.69/25.3	2.62/24.5	2.41/19.0	2.38/17.9
			3 cycles	2.70/25.7	2.62/24.5	2.42/17.6	2.37/17.7
			4 cycles	2.68/26.2	2.63/24.4	2.42/18.7	2.38/17.9
			5 cycles	2.69/25.1	2.62/24.6	2.43/19.4	2.39/17.7

| Atmosphere[a] | Temperature °C / °F | Pressure MPa/psi | Time days | INSULATING CASTABLES | | |
				54% Al_2O_3[k]	47% Al_2O_3[l]	35% Al_2O_3[m]
Air (dried)	110/ 230	ambient	1	1.52/43.0	1.22/48.2	0.93/56.0
Air (fired)	260/ 500	ambient	3/4	1.53/44.8	1.16/52.8	0.90/61.8
Air (fired)	538/1000	ambient	3/4	1.53/45.9	1.13/54.4	0.90/62.9
Saturated steam { vapor	231/ 447	2.8/410	10	1.45/52.5	1.13/57.8	0.86/67.1
{ liquid	231/ 447	2.8/410	10	1.47/52.6	1.11/59.8	0.95/63.9
Saturated steam { vapor	285/ 545	6.9/1000	10	1.49/51.3	1.16/56.1	0.88/65.3
{ liquid	285/ 545	6.9/1000	10	1.46/53.1	1.18/55.7	0.95/64.0
CGA with H_2S, { vapor	231/ 447	6.9/1000	10	1.54/48.7	1.25/51.4	1.04/60.3
H_2O saturated { liquid	231/ 447	6.9/1000	10	1.54/48.2	1.25/51.8	0.91/65.1
CGA with H_2S, { vapor	231/ 447	6.9/1000	44	1.57/30.9	1.20/56.5	0.97/63.7
H_2O saturated { liquid	231/ 447	6.9/1000	44	1.68/24.7	not tested	1.13/57.7
CGA with H_2S, saturated[i]	240/ 465	6.9/1000	5	1.58/44.3	1.28/51.9	1.04/59.5
followed by firing in air[j]	538/1000	ambient	3/4	1.55/46.7	1.24/53.9	0.95/64.5
Cycling tests--repeat of the preceding exposure followed by the same air firing						
			1 cycle	1.56/48.5	1.21/56.4	0.97/64.3
			2 cycles	1.55/48.0	1.21/55.7	0.96/64.5
			3 cycles	1.56/48.1	1.22/54.1	0.98/62.7
			4 cycles	1.56/49.3	1.24/56.0	0.98/63.8
			5 cycles	1.52/51.0	1.23/56.9	0.97/63.7

[a] For testing the effect of water-saturated vapor and of liquid, samples were exposed in a steam generator, both in the vapor and in the liquid in the bottom of the vessel. After placement of samples, the vessel was heated to the desired steam pressure and where tests included other gases, these were then added so as to obtain the desired overall concentrations. The overall composition of the coal gasification atmosphere (CGA) is (in vol %) 18 CO, 12 CO_2, 24 H_2, 40 H_2O, 5 CH_4, and 1 H_2S. A gaseous flow rate was maintained so as to provide one complete change in atmosphere every 24 hours.

[b] Other tests showed that boehmite formation occurred in steam-saturated atmospheres and resulted in very large increases in the flexural strength. These cycling tests were designed to investigate the effect of repetitive

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(Table Continued)

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EFFECT OF SATURATED VAPOR AND LIQUID AND EXPOSURE[a] TO PRESSURE-TEMPERATURE CYCLING CAUSING BOEHMITE FORMATION[b]
AND DECOMPOSITION ON THE DENSITY AND POROSITY OF CASTABLE ALUMINA REFRACTORIES[c][39], Continued

Footnotes continued
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formation and decomposition of boehmite on the properties of the refractories.

[c]After casting, specimens were cured 24 hours in air at 100% humidity at ambient temperature, and dried at 230 °F for 24 hours. Specimens were stored at 230 °F until tested.

[d]Values are averages for seven samples. ASTM C20-74 procedure followed except that kerosene was used.

[e]93% alumina dense castable, UMR-1 generic preparation (70% tabular alumina + 30% calcium aluminate cement) (T-61 alumina, Alcoa; CA-25 Calcium Aluminate Cement, Alcoa).

[f]87.4% alumina dense castable, UMR-4 generic preparation (70% tabular alumina + 30% calcium aluminate cement) (T-61 alumina, Alcoa; Refcon, a 58% alumina-33% calcia cement, Universal Atlas).

[g]63.8% alumina, 28.3% silica dense castable, UMR-8 generic preparation (75% calcined kaolin aggregate + 25% calcium aluminate cement) (Mulcoa M-60 and Mulcoa M-47, C-E Minerals; CA-25 Calcium Aluminate Cement, a 79% alumina-18% calcia cement, Alcoa).

[h]58.6% alumina, 29.6% silica dense castable, UMR-5 generic preparation (75% calcined kaolin aggregate + 25% calcium aluminate cement) (Mulcoa M-60 and Mulcoa M-47, C-E Minerals; Refcon, a 58% alumina-33% calcia cement, Universal Atlas).

[i]85% steam saturated; this exposure is the one forming the first step in the cycling tests, forming boehmite.

[j]The firing step, following the saturated CGA exposure, completed one cycle, decomposing the boehmite.

[k]54% alumina, 40% silica insulating castable, calcium aluminate (79% alumina, 18% calcia) bonded (Cer-Lite #75, C-E Refractories; CA-25 Calcium Aluminate Cement, Alcoa).

[l]46.7% alumina, 40.2% silica insulating castable (Litecast 60-25, General Refractories).

[m]34.5% alumina, 52.5% silica insulating castable (VSL-50, A.P. Green).

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EFFECT OF CARBON MONOXIDE/STEAM/HYDROGEN SULFIDE EXPOSURE[a] ON THE DENSITY AND POROSITY
OF VARIOUS ALUMINA REFRACTORIES[b][39]

Refractory[b]	$CO/H_2O = 0.1 + 1\% H_2S$		$CO/H_2O = 1.0 + 1\% H_2S$		$CO/H_2O = 3.0 + 1\% H_2S$	
	1000 °F Unsaturated	532 °F Saturated[c]	1000 °F Unsaturated	466 °F Saturated[c]	1000 °F Unsaturated	400 °F Saturated[c]
— — — — — — — — — — — — DENSITY,[d] g/cm^3 — — — — — — — — — — — — —						
DENSE HIGH-ALUMINA CASTABLES						
93% Alumina[e]	2.71	2.76 (2.77)	2.72	2.64 (2.67)	2.71	2.69 (2.53)
91% Alumina[f]	2.59	2.65 (2.63)	2.56	2.51 (2.55)	2.62	2.61 (2.38)
87% Alumina[g]	2.61	2.60 (2.66)	2.60	2.52 (2.59)	2.58	2.58 (2.46)
88% Alumina[h]	not tested		2.60	2.60 (2.53)	2.62	2.65 (2.56)
84% Alumina[i]	not tested		2.53	2.53 (2.62)	2.56	2.54 (2.51)
DENSE INTERMEDIATE-ALUMINA CASTABLES						
57% Alumina, 34% silica[j]	2.27	2.26 (2.29)	2.29	2.32 (2.26)	2.24	2.29 (2.15)
59% Alumina, 30% silica[k]	2.34	2.39 (2.41)	2.37	2.38 (2.38)	2.38	2.46 (2.33)
INSULATING CASTABLES						
54% Alumina, 40% silica[l]	1.52	1.51 (1.51)	1.55	1.57 (1.68)	1.52	1.53 (1.51)
46% Alumina, 40% silica[m]	1.17	1.19 (1.31)	1.18	1.24 (1.33)	1.16	1.22 (1.14)
35% Alumina, 53% silica[n]	0.91	0.86 (0.93)	0.89	0.84 (0.90)	0.90	0.89 (0.87)
PHOSPHATE-BONDED RAMMING MIX						
96% Alumina[o]	2.88	2.84 (2.84)	2.91	2.87 (2.97)	2.87	2.84 (2.88)
— — — — — — — — — — — POROSITY,[d] g/cm^3 — — — — — — — — — — — — —						
DENSE HIGH-ALUMINA CASTABLES						
93% Alumina[e]	26	13 (13)	25	26 (20)	25	22 (26)
91% Alumina[f]	28	15 (14)	27	22 (22)	25	22 (33)
87% Alumina[g]	24	19 (17)	25	26 (22)	26	23 (28)
88% Alumina[h]	not tested		25	23 (18)	25	8 (24)
84% Alumina[i]	not tested		26	18 (20)	25	6 (22)
DENSE INTERMEDIATE-ALUMINA CASTABLES						
57% Alumina, 34% silica[j]	22	17 (16)	20	18 (20)	22	9 (22)
59% Alumina, 30% silica[k]	22	16 (14)	19	17 (16)	18	13 (18)
INSULATING CASTABLES						
54% Alumina, 40% silica[l]	52	52 (52)	49	51 (35)	47	51 (52)
46% Alumina, 40% silica[m]	61	56 (51)	58	57 (45)	55	56 (60)
35% Alumina, 53% silica[n]	68	68 (64)	67	70 (67)	65	67 (69)
PHOSPHATE-BONDED RAMMING MIX						
96% Alumina[o]	20	23 (22)	19	21 (19)	20	22 (22)

[a] Samples were exposed in two pressure vessels. One vessel was a steam generator and tests which specify saturated conditions occurred in this vessel. After placing samples, the vessel was heated to the desired steam pressure and the other gases were then added so as to obtain the concentrations specified in the table. The second vessel was connected to the steam generator and after samples were placed and the vessel heated to the test temperature, the vessel was pressurized with the steam/gas mixture from the steam generator. Tests specified as unsaturated occurred in this vessel. A gaseous flow rate for both vessels was maintained so as to provide one complete change in atmosphere every 24 hours. The specimens were exposed at the temperatures specified in the table at 1000 psia for 20 days. For $CO/H_2O = 0.1$: CO 90 psia, H_2O 900 psia, H_2S 10 psia. For $CO/H_2O = 1.0$: CO 495 psia, H_2O 495 psia, H_2S 10 psia. For $CO/H_2O = 3.0$: CO 742.5 psia, H_2O 247.5 psia, H_2S 10 psia.

[b] All cement-bonded castables were cast in molds either 12 x 3 x 1/2 in. or 3 x 3/4 x 3/4 in., cured 24 hours in air at 100% humidity at ambient temperature, and dried at 230 °F for 24 hours. The 12 x 3 x 1/2 inch slabs were then cut to 3 x 1/2 x 1/2 in. bars. Phosphate-bonded specimens were prepared by hand ramming in 3 x 3/4 x 3/4 in. molds, immediately drying for 24 hours at 230 °F and then firing at 1000 °F for 18 hours. All specimens were stored at 230 °F until tested.

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(Table Continued)

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EFFECT OF CARBON MONOXIDE/STEAM/HYDROGEN SULFIDE EXPOSURE[a] ON THE DENSITY AND POROSITY
OF VARIOUS ALUMINA REFRACTORIES[b][39], Continued

Footnotes continued

[c]Numbers without parentheses are for samples exposed to saturated vapor. Adjacent numbers in parentheses
are for samples immersed in liquid (water) in the bottom of the steam generator under the same exposure
conditions.

[d]All values for samples exposed in vapor are the averages of seven samples. The values for the samples
exposed in liquid are the averages for two samples. ASTM C20-74 followed using kerosene.

[e]93% Alumina dense castable, UMR-1 generic preparation (70% tabular alumina + 30% calcium aluminate ce-
ment) (T-61 alumina, Alcoa; CA-25 Calcium Aluminate Cement, 79% alumina-18% calcia, Alcoa).

[f]91% Alumina dense castable, UMR-2 generic preparation (70% tabular alumina + 30% calcium aluminate ce-
ment) (T-61 alumina, Alcoa; Secar 71(250), 72% alumina-26% calcia cement, Lone Star Lafarge).

[g]87.4% Alumina dense castable, UMR-4 generic preparation (70% tabular alumina + 30% calcium aluminate ce-
ment) (T-61 alumina, Alcoa; Refcon, 58% alumina-33% calcia cement, Universal Atlas).

[h]88% Alumina dense castable, UMR-6 generic preparation (UMR-1 generic preparation with 5% SiO_2 added as
99.9% pure bone dry Wedron silica flour).

[i]84% Alumina dense castable, UMR-7 generic preparation (UMR-1 generic preparation with 10% SiO_2 added as
99.9% pure bone drey Wedron silica flour).

[j]57% Alumina, 34% silica castable, calcium aluminate (79% alumina, 18% calcia) bonded (RC-3, General Re-
fractories; CA-25 Calcium Aluminate Cement, Alcoa).

[k]58.6% Alumina, 29.6% silica castable, UMR-5 generic preparation (75% Mulcoa + Mulgrain M-60, C-E Minerals;
25% Refcon, 58% alumina-33% calcia cement, Universal Atlas).

[l]54% Alumina, 40% silica insulating castable, calcium aluminate (79% alumina-18% calcia) bonded (Cer-Lite
#75, C-E Refractories; CA-25 Calcium Aluminate Cement, Alcoa).

[m]46.7% Alumina, 40.2% silica insulating castable (Litecast 60-25, General Refractories).

[n]34.5% Alumina, 52.5% silica insulating castable (VSL-50, A.P. Green).

[o]96% Alumina dense phosphate-bonded ramming mix (Greenpak-90P, A.P. Green).

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EFFECT OF CARBON MONOXIDE/STEAM/HYDROGEN SULFIDE EXPOSURE[a] ON THE WEIGHT AND DIMENSIONS
OF VARIOUS ALUMINA REFRACTORY SAMPLES[b][39]

Refractory[b]	CO/H_2O = 0.1 + 1% H_2S		CO/H_2O = 1.0 + 1% H_2S		CO/H_2O = 3.0 + 1% H_2S	
	1000 °F Unsaturated	532 °F Saturated[d]	1000 °F Unsaturated	466 °F Saturated[d]	1000 °F Unsaturated	400 °F Saturated[d]
— — — — — — — — — — WEIGHT CHANGE,[c] % — — — — — — — — — —						
DENSE HIGH-ALUMINA CASTABLES						
93% Alumina[e]	−2.1	+1.9 (+4.1)	−2.3	−2.9 (−2.2)	−2.0	−1.0 (−5.5)
91% Alumina[f]	−3.6	+2.4 (+1.4)	−3.8	+0.7 (−3.9)	−4.0	+1.7 (−9.5)
87% Alumina[g]	−2.4	+3.0 (+2.0)	−3.5	−0.3 (−1.4)	−3.3	+3.8 (−5.2)
88% Alumina[h]	not tested		−4.2	−3.2 (+0.7)	−3.4	−0.6 (−3.1)
84% Alumina[i]	not tested		−4.1	−0.9 (+0.6)	−3.6	−0.5 (−3.8)
DENSE INTERMEDIATE-ALUMINA CASTABLES						
57% Alumina, 35% silica[j]	−5.1	−2.8 (+1.6)	−3.8	−1.3 (−4.7)	−3.5	−0.3 (−5.3)
59% Alumina, 30% silica[k]	−5.0	−1.1 (−0.7)	−4.0	−0.4 (−3.1)	−2.7	+0.8 (−3.0)
INSULATING CASTABLES						
54% Alumina, 40% silica[l]	−3.2	−0.6 (+3.1)	−2.7	+0.5 (+5.5)	−2.6	−0.1 (−1.1)
46% Alumina, 40% silica[m]	−6.7	−1.6 (+5.5)	−6.2	+0.4 (+7.3)	−6.2	+1.4 (−4.6)
35% Alumina, 53% silica[n]	−7.0	−7.7 (−5.2)	−6.1	−6.2 (−4.6)	−6.5	−6.6 (−4.6)
PHOSPHATE-BONDED RAMMING MIX						
96% Alumina[o]	−0.2	−0.7 (+0.6)	−0.2	−0.4 (+3.2)	−0.2	−0.4 (+2.0)
— — — — — — — — — — DIMENSIONAL CHANGE,[c] % — — — — — — — — — —						
DENSE HIGH-ALUMINA CASTABLES						
93% Alumina[e]	−0.1	nil (+0.5)	−0.1	−0.1 (+0.6)	−0.2	+0.5 (+0.2)
91% Alumina[f]	−0.2	+0.2 (+0.6)	−0.4	+1.5 (+0.5)	−0.3	+1.4 (+0.4)
87% Alumina[g]	−0.2	+0.7 (+0.2)	−0.2	+1.4 (+0.3)	−0.3	+2.4 (nil)
88% Alumina[h]	not tested		−0.2	−0.1 (−0.2)	−0.1	+0.4 (+0.3)
84% Alumina[i]	not tested		−0.3	+0.2 (−0.3)	−0.1	+0.3 (nil)
DENSE INTERMEDIATE-ALUMINA CASTABLES						
57% Alumina, 35% silica[j]	−0.1	nil (+0.5)	nil	+0.1 (+0.4)	−0.2	+0.3 (+0.3)
50% Alumina, 38% silica[k]	−0.2	+0.1 (+0.6)	−0.3	+0.5 (+0.4)	−0.3	+0.3 (−0.1)
INSULATING CASTABLES						
54% Alumina, 40% silica[l]	−0.1	+0.6 (+1.1)	−0.1	+0.2 (+0.8)	nil	+0.3 (+0.5)
46% Alumina, 40% silica[m]	−0.1	+0.4 (+0.7)	+0.3	+0.2 (+0.2)	−0.2	+0.1 (+0.2)
35% Alumina, 53% silica[n]	−0.6	+0.8 (+9.8)[p]	−0.3	+0.5 (+0.6)	−0.8	nil (+1.3)
PHOSPHATE-BONDED RAMMING MIX						
96% Alumina[o]	−0.1	nil (+0.3)	nil	+0.3 (+0.3)	−0.1	+0.2 (+0.3)

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[a] Samples were exposed in two pressure vessels. One vessel was a steam generator and tests which specify saturated conditions occurred in this vessel. After placing samples, the vessel was heated to the desired steam pressure and the other gases were then added so as to obtain the concentrations specified in the table. The second vessel was connected to the steam generator and after samples were placed and the vessel heated to the test temperature, the vessel was pressurized with the steam/gas mixture from the steam generator. Tests specified as unsaturated occurred in this vessel. A gaseous flow rate for both vessels was maintained so as to provide one complete change in atmosphere every 24 hours. The specimens were exposed at the temperatures specified in the table at 1000 psia for 20 days. For CO/H_2O = 0.1: CO 90 psia, H_2O 900 psia, H_2S 10 psia. For CO/H_2O = 1.0: CO 495 psia, H_2O 495 psia, H_2S 10 psia. For CO/H_2O = 3.0: CO 742.5 psia, H_2O 247.5 psia, H_2S 10 psia.

[b] All cement-bonded castables were cast in molds either 12 x 3 x 1/2 in. or 3 x 3/4 x 3/4 in., cured 24 hours in air at 100% humidity at ambient temperature, and dried at 230 °F for 24 hours. The 12 x 3 x 1/2 inch slabs were then cut to 3 x 1/2 x 1/2 in. bars. Phosphate-bonded specimens were prepared by hand ramming in 3 x 3/4 x 3/4 in. molds, immediately drying for 24 hours at 230 °F and then firing at 1000 °F for 18 hours. All specimens were stored at 230 °F until tested.

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(Table Continued)

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EFFECT OF CARBON MONOXIDE/STEAM/HYDROGEN SULFIDE EXPOSURE[a] ON THE WEIGHT AND DIMENSIONS
OF VARIOUS ALUMINA REFRACTORY SAMPLES[b][39], Continued

Footnotes continued
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[c] Referenced to as-cast and dried (230 °F) weight; + indicates increase, - indicates decrease. Data are
average values for seven samples except for values in parentheses (see footnote d).

[d] Numbers without parentheses are for samples exposed to saturated vapor, average of seven samples. Adjacent numbers in parentheses are for samples immersed in liquid (water) under the same exposure conditions, average of two samples.

[e] 93% Alumina dense castable, UMR-1 generic preparation (70% tabular alumina + 30% calcium aluminate cement) (T-61 alumina, Alcoa; CA-25 Calcium Aluminate Cement, 79% alumina-18% calcia, Alcoa).

[f] 91% Alumina dense castable, UMR-2 generic preparation (70% tabular alumina + 30% calcium aluminate cement) (T-61 alumina, Alcoa; Secar 71(250), 72% alumina-26% calcia cement, Lone Star Lafarge).

[g] 87.4% Alumina dense castable, UMR-4 generic preparation (70% tabular alumina + 30% calcium aluminate cement) (T-61 alumina, Alcoa; Refcon, 58% alumina-33% calcia cement, Universal Atlas).

[h] 88% Alumina dense castable, UMR-6 generic preparation (UMR-1 generic preparation with 5% SiO_2 added as 99.9% pure bone dry Wedron silica flour).

[i] 84% Alumina dense castable, UMR-7 generic preparation (UMR-1 generic preparation with 10% SiO_2 added as 99.9% pure bone dry Wedron silica flour).

[j] 57% Alumina, 34% silica castable, calcium aluminate (79% alumina, 18% calcia) bonded (RC-3, General Refractories; CA-25 Calcium Aluminate Cement, Alcoa).

[k] 58.6% Alumina, 29.6% silica castable, UMR-5 generic preparation (75% Mulcoa + Mulgrain M-60, C-E Minerals; 25% Refcon, 58% alumina-33% calcia cement, Universal Atlas).

[l] 54% Alumina, 40% silica insulating castable, calcium aluminate (79% alumina, 18% calcia) bonded (Cer-Lite #75, C-E Refractories; CA-25 Calcium Aluminate Cement, Alcoa).

[m] 46.7% Alumina, 40.2% silica insulating castable (Litecast 60-25, General Refractories).

[n] 34.5% Alumina, 52.5% silica insulating castable (VSL-50, A.P. Green).

[o] 95% Alumina dense phosphate-bonded ramming mix (Greenpak-90P, A.P. Green).

[p] [9.8 value is most likely a misprint for 0.8.]

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EFFECT OF EXPOSURE TO AN UNSATURATED AND STEAM-SATURATED SIMULATED COAL GASIFICATION ENVIRONMENT[a] ON
THE WEIGHT, DENSITY, AND POROSITY OF VARIOUS ALUMINA REFRACTORIES[b][39]

Refractory[b]	Unsaturated (700 °F)			Saturated (447 °F)[c]		
	Wt. Change[d] %	Density gm/cc	Porosity %	Wt. Change[d] %	Density gm/cc	Porosity %
DENSE HIGH-ALUMINA CASTABLES						
94% Alumina[e]	-1.9	2.72	26	-1.4 (+4.5)	2.69 (2.72)	23 (18)
93% Alumina[f]	-1.9	2.71	27	+1.0 (+7.3)	2.72 (2.78)	21 (6)
91% Alumina[g]	-2.5	2.58	29	-1.5 (+4.4)	2.54 (2.60)	23 (24)
91% Alumina[h]	-0.6	2.44	31	-2.0 (+10.2)	2.35 (2.53)	32 (24)
88% Alumina[i]	nt[k]	nt[k]	nt[k]	nt[k] (+4.2)	nt[k] (2.77)	nt[k] (16)
84% Alumina[j]	nt	nt	nt	nt (+6.0)	nt (2.72)	nt (14)
DENSE INTERMEDIATE-ALUMINA CASTABLES						
57% Alumina, 34% silica[l]	-2.3	2.32	20	-1.2 (+5.2)	2.28 (2.38)	19 (13)
59% Alumina, 30% silica[m]	nt	nt	nt	-2.2 (nt)	2.40 (nt)	17 (nt)
INSULATING CASTABLES						
54% Alumina, 40% silica[n]	-1.4	1.51	52	+1.2 (+19.6)	1.54 (1.78)	50 (37)
46% Alumina, 40% silica[o]	nt	nt	nt	-2.1 (nt)	1.19 (nt)	57 (nt)
PHOSPHATE-BONDED RAMMING MIXES						
96% Alumina[p]	nt	nt	nt	+1.3 (nt)	2.89 (nt)	21 (nt)
90% Alumina[q]	nt	nt	nt	-0.8 (nt)	2.84 (nt)	22 (nt)

[a] Samples were exposed in two pressure vessels. One vessel was a steam generator and tests which specify saturated conditions occurred in this vessel. After placing samples, the vessel was heated to the desired steam pressure and other gases were then added so as to obtain the concentrations specified. The second vessel was connected to the steam generator and after samples were placed and the vessel heated to the test temperature, the vessel was pressurized with the steam/gas mixture from the steam generator. A gaseous flow rate for both vessels was maintained so as to provide one complete change in atmosphere every 24 hours. The simulated coal gasification atmosphere composition is (in vol%) 18 CO, 12 CO_2, 24 H_2, 40 H_2O and 1 H_2S. The samples were exposed at the temperatures indicated in the table at 1000 psia for 60 days.

[b] All cement bonded castables were cast in molds either 12x3x1/2 in or 3x3/4x3/4 in, cured 24 hr in air at 100% humidity at ambient temperature, and dried at 230 °F for 24 hr. The 12x3x0.5 in slabs were then cut to 3x1/2x1/2 in bars. Phosphate-bonded specimens were prepared by hand ramming in 3x3/4x3/4 in molds, immediately drying for 24 hr at 230 °F and then firing at 1000 °F for 18 hr. All specimens were stored at 230 °F until tested.

[c] Numbers without parentheses are for samples exposed to saturated vapor, average of seven samples. Adjacent numbers in parentheses are for samples immersed in liquid (water) under the same exposure conditions, average of two samples.

[d] Referenced to as-cast and dried (230 °F) weight.

[e] 94% Alumina dense castable, calcium aluminate (79% alumina, 18% calcia) bonded (Greencast-94, A.P. Green; CA-25 Calcium Aluminate Cement, Alcoa).

[f] 93% Alumina dense castable, UMR-1 generic preparation (70% tabular alumina + 30% calcium aluminate cement) (Tabular Alumina T-61, Alcoa; CA-25 Calcium Aluminate Cement, Alcoa).

[g] 91% Alumina dense castable, UMR-2 generic preparation (70% tabular alumina + 30% calcium aluminate cement) (Tabular Alumina T-61, Alcoa; Secar 71(250), a 72% alumina-26% calcia cement, Lone Star Lafarge).

[h] 91% Alumina dense castable, UMR-3 generic prepration (70% tabular alumina + 30% calcium aluminate cement) (Tabular Alumina T-61, Alcoa; C-3, a 72% alumina-25% calcia cement, Babcock & Wilcox).

[i] 88% Alumina dense castable, UMR-6 generic preparation (generic preparation UMR-1 with 5% SiO_2 added as 99.9% pure, bone dry Wedron silica flour).

[j] 84% Alumina dense castable, UMR-7 generic preparation (generic preparation UMR-1 with 10% SiO_2 added as 99.9% pure, bone dry Wedron silica flour).

[k] nt represents samples not tested in that exposure condition.

[l] 57% Alumina, 34% silica castable, calcium aluminate (79% alumina, 18% calcia) bonded (RC-3, General Refractories; CA-25 Calcium Aluminate Cement, Alcoa).

[m] 58.6% Alumina, 29.6% silica castable, UMR-5 generic preparation (75% Mulcoa + Mulgrain M-60, C-E Minerals; 25% Refcon, a 58% alumina-33% calcia cement, Universal Atlas).

[n] 54% Alumina, 40% silica insulating castable, calcium aluminate (79% alumina, 18% calcia) bonded (Cer-Lite #75; C-E Refractories; CA-25 Calcium Aluminate Cement, Alcoa).

[o] 46.7% Alumina, 40.2% silica insulating castable (Litecast 60-25, General Refractories).

[p] 96% Alumina dense phosphate-bonded ramming mix (Greenpak-90P, A.P. Green).

[q] 90% Alumina dense phosphate-bonded ramming mix (Brikram 90R, General Refractories).

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THERMAL EXPANSION[a] AND THERMAL CONDUCTIVITY[b] DATA FOR VARIOUS
REFRACTORIES[26]

Refractories	Coefficient of Thermal Expansion[a] x 10^{-6} in/in/°F			Thermal Conductivity[b] Btu/in/h/ft^2/°F
	RT-1875	700-1875	RT-1875	
Alumina castables				
50% generic[c]	2.06	2.90	--	7.0
50% commercial[d]	2.61	4.04	4.1	2.8
42% commercial[e]	-0.12	1.19	3.30	1.6
50% commercial[f]	2.71	3.15	--	10.0
+ 4 wt% 310 SS[g]	5.00	5.73[h]	4.51	--
+ 2 wt% 446 SS[i]	3.32	3.65[h]	--	--
+ 4 wt% 446 SS[i]	3.81	4.02[h]	--	--
90+% generic[j]	3.22	4.62	--	--
90+% generic[k]	3.94	5.90	4.88	13.1
90+% modified[l]	3.50	5.36	--	13.1
90+% commercial[m]	3.84	5.30	--	--
Phosphate-bonded alumina ramming mixes				
45% generic[n]	3.24	3.83	--	7.2
90% generic[o]	4.84	5.45	--	15.5
90% commercial[p]	--	--	--	15.5

[a] Thermal expansion was determined using a fused silica dilatometer. Tests were run from room temperature (RT) to 1875 °F at rates from 100 °F/h to 400 °F/h. Specimens were 2 in. long x 0.5 in. square. The coefficient was determined in 2 different temperature ranges, from ambient to the top temperature and from 700 to 1875 °F (the linear portion of the curve). Some materials were run through a second complete heating cycle and that coefficient was determined over the entire temperature range.

[b] Determined using ASTM C-417 procedure.

[c] Generic preparation: 75% calcined kaolin (70% to -20 mesh, 5% ball milled fines with 50% less than 325 mesh), 25% calcium aluminate cement (Casting Grade CA-25, Alcoa); 11% water added.

[d] Commercial lightweight insulating castable, calcium aluminate bonded (Litecast 75-28, General Refractories); 21% water added.

[e] Commercial lightweight insulating castable, 42% alumina (B&W Kaolite 2300 LI, Babcock & Wilcox); 59% water added.

[f] Coarse grain size, high-strength 50% alumina prototype castable (B&W Kaocrete XD50, Mix 36C, Babcock & Wilcox); 7.5% water added.

[g] 1 in. long fibers of 310 SS (Ribtec 310) were added to the Kaocrete XD50.

[h] Temperature range for these values is 680-1875 °F.

[i] 1 in. long fibers of 446 SS (Ribtec 446) were added to the Kaocrete XD50.

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THERMAL EXPANSION[a] AND THERMAL CONDUCTIVITY[b] DATA FOR VARIOUS
REFRACTORIES[26], Continued

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[j]DOE 90 generic preparation: 65% tabular alumina (6 to -20 mesh), 10% calcined alumina (-325 mesh), 25% calcium aluminate cement (Secar 250, Lone Star Lafarge); 10% water added.

[k]DOE 90 generic preparation: as in footnote j but with Casting Grade CA-25 (Alcoa) cement; 9.3% water added.

[l]Modified DOE 90 preparation: 70% tabular alumina (-48 mesh), 5% calcined alumina (-325 mesh), 25% calcium aluminate cement (Casting Grade CA-25, Alcoa); 8.5% water added.

[m]Commercial castable (B&W Kao-Tab 95, Babcock & Wilcox).

[n]Generic preparation of phosphate-bonded ramming mix: 15% calcined alumina, 65% calcined kaolin, 17% ball mill fines, 3% Bentonite (Wyoming), 1% hydrated alumina; bonded with 6% phosphoric acid (85% strength).

[o]Generic preparation of phosphate-bonded ramming mix: 82% tabular alumina, 15% calcined alumina, 3% Bentonite (Wyoming), 1% hydrated alumina; bonded with 6% phosphoric acid (85% strength).

[p]Commercial phosphate-bonded ramming mix: 90 Ram H.S. from C-E Refractories.

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THERMAL EXPANSION DATA[a] FOR SEVERAL REFRACTORIES[26]

	Coef. Therm. Exp. (700 - 1875) in/in/F x 10^{-6}
90+% Al_2O_3 dense castable, cement 1[b]	5.5
90+% Al_2O_3 dense castable, cement 2[c]	4.7
50% Al_2O_3 dense castable, cement 1[d]	2.9
50% Al_2O_3 dense castable, cement 3[e]	2.5
50% Al_2O_3 dense castable, cement 2[f]	2.5

Air Stored 18 Days (0.1% Linear Shrinkage to 1200 F)
Air Stored 5 Days (0.2 Linear Shrinkage to 1200 F)
High Humidity Stored 18 Days (0.2 Linear Shrinkage to 1875 F)
High Humidity Stored 5 Days (0.2 Linear Shrinkage to 1875 F)

Air Stored

Humidity Stored

90+% Al_2O_3 dense castable[b]

- -

(Data Continued)

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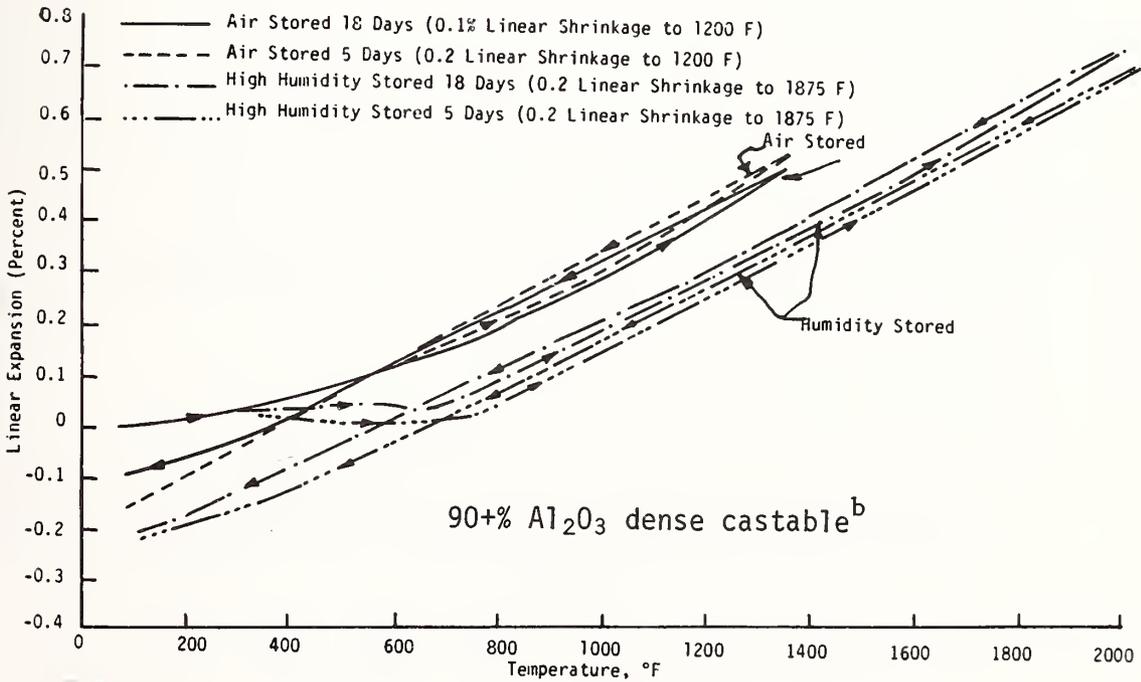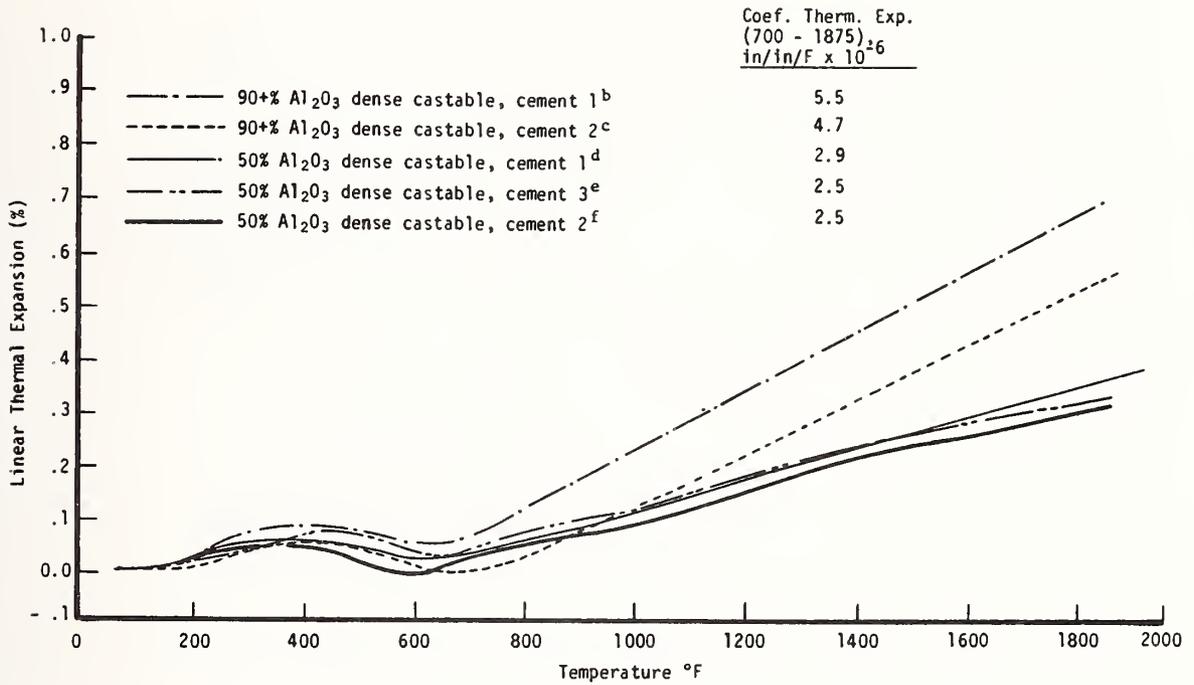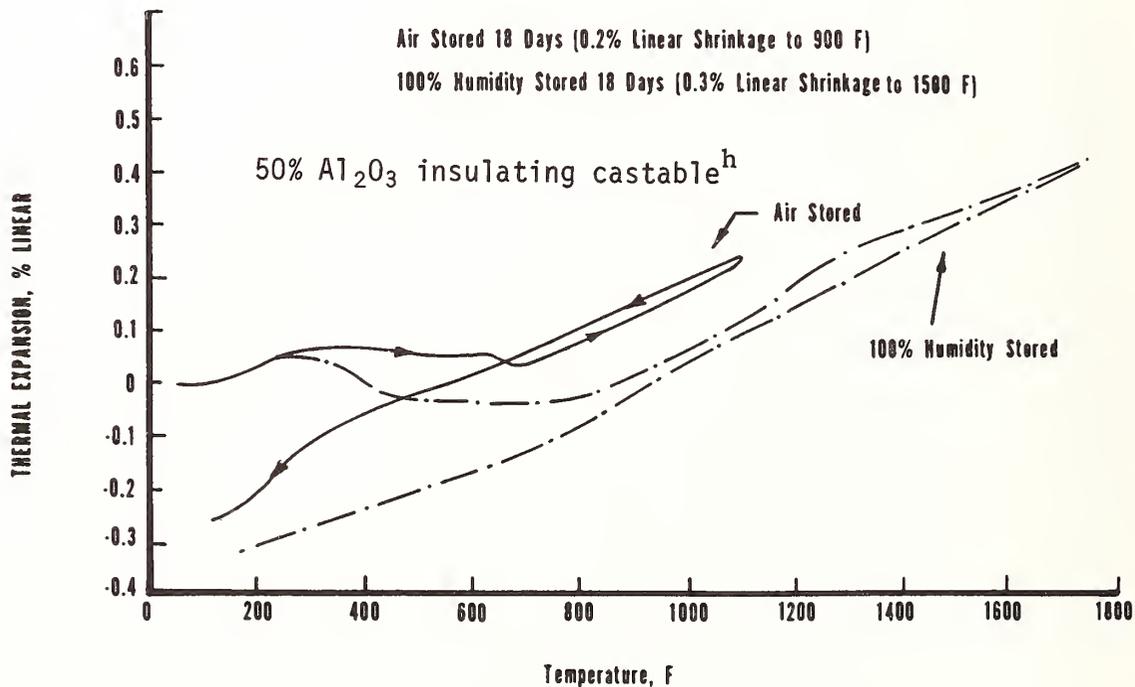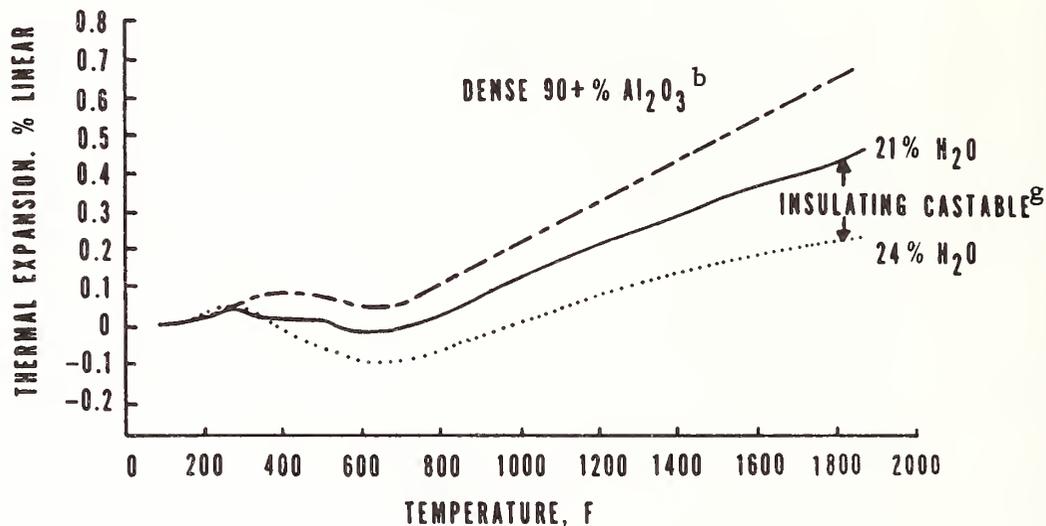
THERMAL EXPANSION DATA[a] FOR SEVERAL REFRACTORIES[26], Continued

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(Data Continued)

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THERMAL EXPANSION DATA[a] FOR SEVERAL REFRACTORIES[26], Continued

90% Al_2O_3 $\frac{i}{j}$ (0.1% Expansion After 1875 F)
45% Al_2O_3 $\frac{i}{j}$ (0.2% Expansion After 1875 F Test)

Phosphate-bonded Ramming Mixes

$\alpha = 4.84 \times 10^{-6}$ in/in/F

$\alpha = 3.24 \times 10^{-6}$ in/in/F

Percent Linear Expansion

Temperature, F

0.1% Shrinkage After 1875F Test

50% Al_2O_3 prototype dense castable[k]

$\alpha = 3.16 \times 10^{-6}$ in/in/F
(570 - 1975°F range)

PERCENT LINEAR EXPANSION

TEMPERATURE °F

(Data Continued)

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THERMAL EXPANSION DATA[a] FOR SEVERAL REFRACTORIES[26], Continued

Footnotes

[a]Thermal expansion was determined using a fused silica dilatometer. Tests
were run from room temperature (RT) to 1875 °F at a rate of 400 °F/h.
Specimens were 2 in. long x 0.5 in. square. Tests were run to compare the
effect of different storage conditions and different water contents as well
as to compare different refractories.

[b]Generic preparation: 65% tabular alumina (6 to -20 mesh), 10% calcined
alumina (-325 mesh), 25% calcium aluminate cement (Casting Grade CA-25,
Alcoa; cement 1); 9.3% water added.

[c]Same generic preparation as in footnote b except that the cement used was
regular grade CA-25 (Alcoa; cement 2); same water content.

[d]Generic preparation: 75% calcined kaolin (70% to -20 mesh, 5% ball milled
fines with 50% less than 325 mesh), 25% calcium aluminate cement (Casting
Grade CA-25, Alcoa; cement 1); 10% water added.

[e]Same generic preparation as in footnote d except a lower-purity calcium
aluminate cement was used (Refcon, Universal Atlas Cement; cement 3);
10.2% water added.

[f]Same generic preparation as in footnote d except regular grade CA-25
(Alcoa; cement 2) cement was used; 10.8% water added.

[g]Commercial lightweight insulating castable, 50% alumina, calcium aluminate
bonded (Litecast 75-28, General Refractories); 21 or 24% water added.

[h]Same commercial castable as in footnote g, 21% water added.

[i]Generic preparation of phosphate-bonded ramming mix: 82% tabular alumina,
15% calcined alumina, 3% Bentonite (Wyoming), 1% hydrated alumina; bonded
with 6% phosphoric acid (85% strength).

[j]Generic preparation of phosphate-bonded ramming mix: 15% calcined alumina,
65% calcined kaolin, 17% ball mill fines, 3% Bentonite (Wyoming), 1% hy-
drated alumina; bonded with 6% phosphoric acid (85% strength).

[k]Coarse grain size, high-strength 50% alumina prototype castable (B&W
Kaocrete XD50, Mix 36C, Babcock & Wilcox); 7.5% water added.

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THERMAL CONDUCTIVITY DATA[a] FOR SEVERAL REFRACTORIES[26]

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THERMAL CONDUCTIVITY DATA[a] FOR SEVERAL REFRACTORIES[26], Continued

50% Al_2O_3 Dense Castable[e]

Mean Temperature (°F X 100)

42% Al_2O_3 Commercial
Insulating Castable[f]

Mean Temperature (°F X 100)

- -

(Data Continued)

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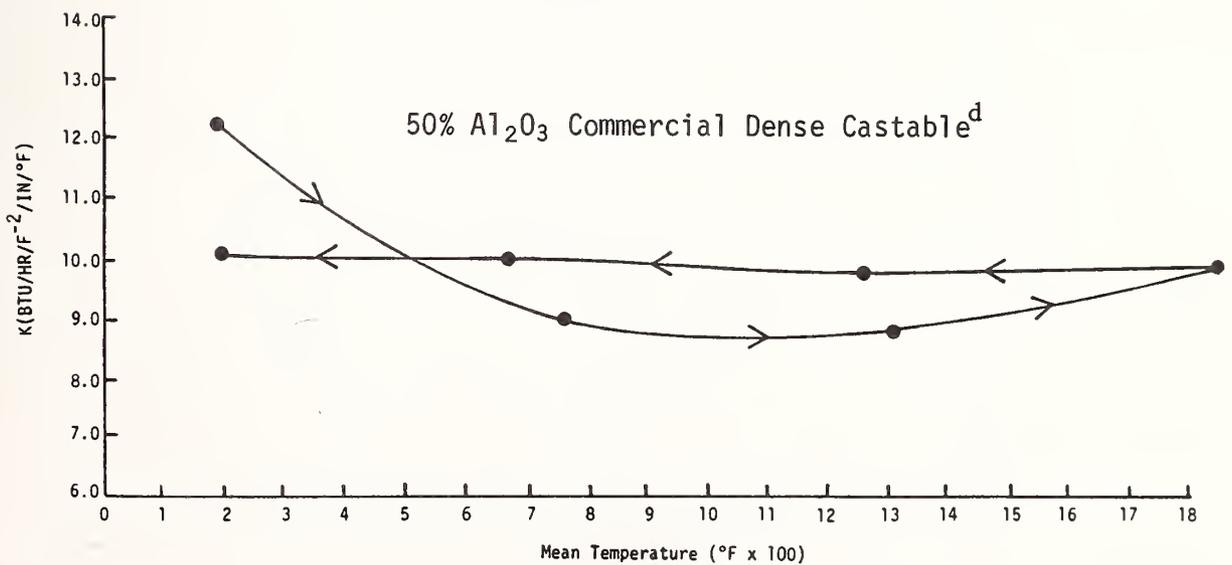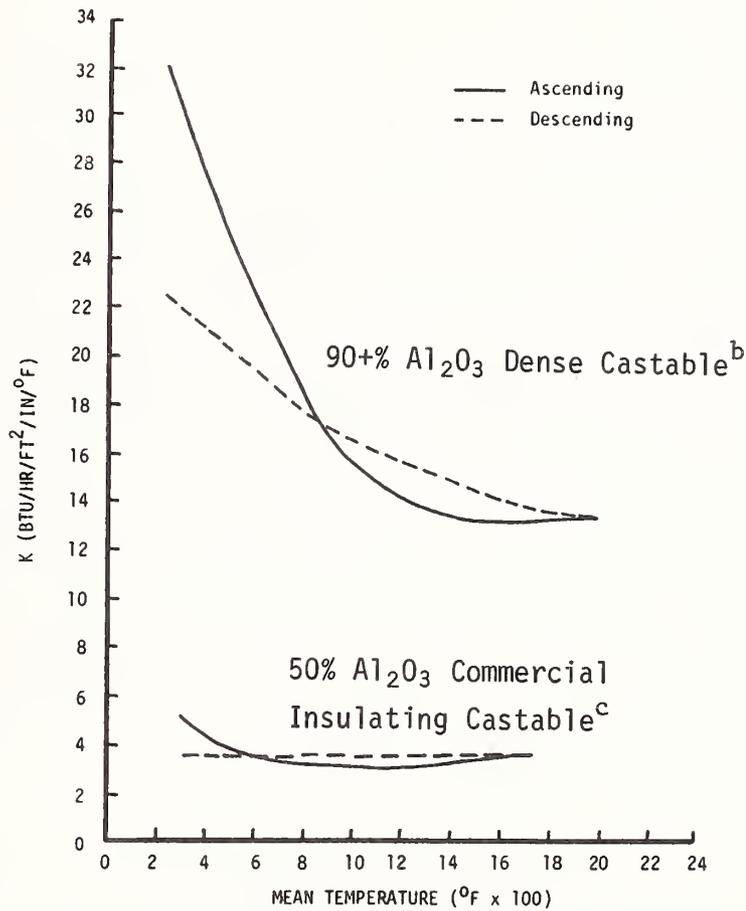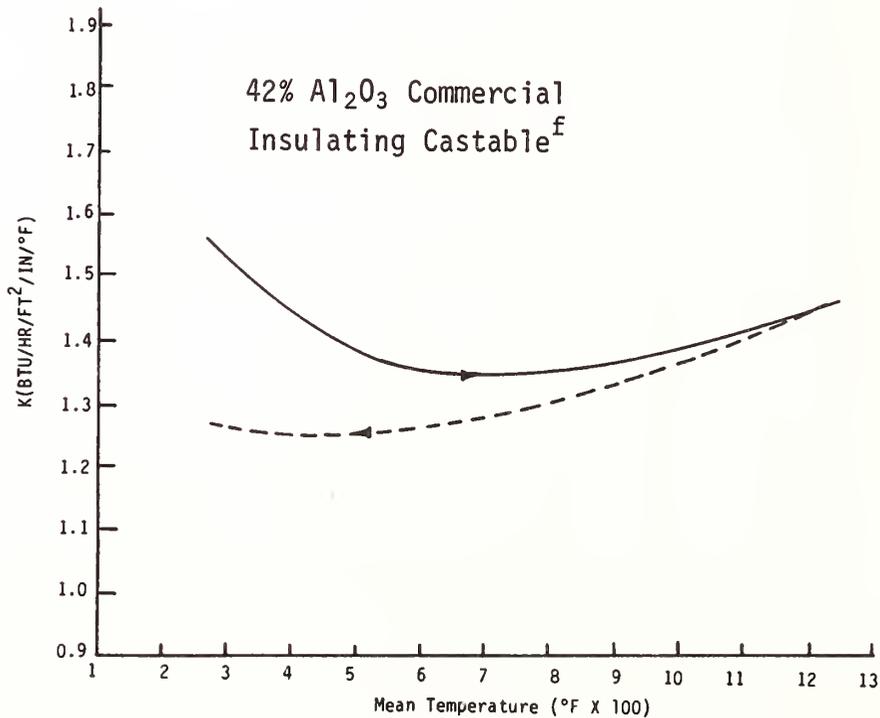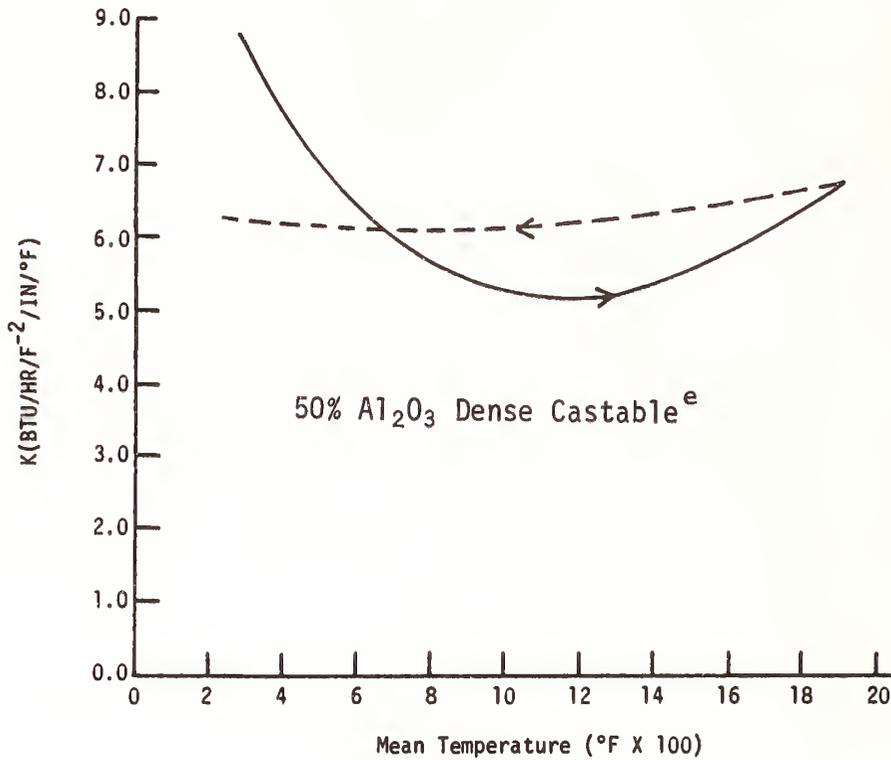
THERMAL CONDUCTIVITY DATA[a] FOR SEVERAL REFRACTORIES[26], Continued

Footnotes

[a]Determined using ASTM C-417 procedures.

[b]Generic preparation: 65% tabular alumina (6 to -20 mesh), 10% calcined alumina (-325 mesh), 25% calcium aluminate cement (Casting Grade CA-25, Alcoa).

[c]Commercial lightweight insulating castable, calcium aluminate bonded (Litecast 75-28, General Refractories); 21% water added.

[d]Coarse grain size, high-strength 50% alumina prototype castable (B&W Kaocrete XD50, Mix 36C, Babcock & Wilcox); 7.5% water added.

[e]Generic preparation: 75% calcined kaolin (70% to -20 mesh, 5% ball milled fines with 50% less than 325 mesh), 25% calcium aluminate cement (Casting Grade CA-25, Alcoa); 11% water added.

[f]Commercial lightweight insulating castable, 42% alumina (B&W Kaolite 2300 LI, Babcock & Wilcox); 59% water added.

BULK DENSITY[a] AND SHRINKAGE[b] OF SEVERAL REFRACTORIES[26]

90+% Alumina Refractories

	Standard 90+% Alumina Generic[c] with Cement 1[d]		with Cement 2[e]		Standard 90+% Alumina Generic[c] with Cement 3[f]				Modified 90+% Alumina Generic[g]	Commercial 90+% Alumina[h]	
Water Added, %	9.3	10.2	10.5	10.0	8.8	9.3	9.8	10.3	8.5	8.0	10.0
Bulk Density, lb/ft³ (brick)											
Ambient temperature cured	174(175)	173(173)	172(170)	171(172)	176(174)	175(174)	174(172)	172	177	177(178)	176(175)
After 24 h at 250 °F	169(169)	166(166)	169(168)	169(170)	174(172)	173(171)	171(169)	169	175	172(173)	169(167)
After 2 h at 1000 °F	--	--	--	--	165	167	--	--	--	--	--
After 2 h at 1500 °F	--	--	--	--	166	166	--	--	--	--	--
Shrinkage, % Linear (% Volume)											
Green to 250 °F for 24 h	0.1(0.4)	0.0(0.2)	0.0(0.3)	0.0(0.4)	0.1(1.0)	0.08(0.9)	0.1(0.5)	0.0(0.6)	0.05	0.1(0.4)	0.0(0.1)
Green to 450 °F for 24 h	--	--	--	--	--	--	--	--	--	0.14	--
Green to 1000 °F for 2 h	--	--	--	--	0.2(1.2)	0.2(1.2)	--	--	0.1	--	--
Green to 1500 °F for 2 h	--	--	--	--	0.1(0.5)	0.3(0.9)	--	--	0.2	--	--
Green to 1875 °F (no hold)	0.17	--	0.25	--	--	--	--	--	0.2	0.1	--

50% Alumina Refractories

	Standard 50% Alumina Generic[i] with Cement 1[d]		Cement 4[j]	Modified 50% Alumina Generic[k]		Commercial 50% Alumina[l]			Commercial 42% Alumina[m]	
Water Added, %	10.2	10.8	10.2	10	11	21	24	26	29	59
Bulk Density, lb/ft³ (brick)										
Ambient temperature cured	139(140)	140(142)	141(140)	143(142)	141(141)	85,220 °F cured --	--	--	--	62,220 °F cured
After 24 h at 250 °F	134(134)	133(133)	138(137)	140(139)	137(136)	--	--	--	--	--
After 2 h at 1000 °F	--	--	--	133	133	--	--	--	--	--
After 2 h at 1500 °F	--	--	--	133	132	--	--	--	--	--
Shrinkage, % Linear (% Volume)										
Green to 250 °F for 24 h	0.0(0.4)	--	0.0(0.6)	0.0(0.9)	0.1(0.6)	0.1(0.6)	0.4(1.1)	0.1(0.8)	0.1(1.0)	0.09
Green to 1000 °F for 2 h	--	--	--	0.2(1.2)	0.2(1.2)	0.3(1.5)	0.2(1.1)	0.3(1.1)	--	0.4
Green to 1500 °F for 2 h	--	--	--	0.1(0.6)	0.1(0.5)	0.3(1.2)	0.3(1.3)	0.4(1.4)	--	0.6
Green to 1875 °F (no hold)	0.22	0.2	0.19	0.25	0.2	0.3	0.4	--	--	--

Dense Castable and Ramming Mixes

	Commercial 50% Dense Castable[n] plain	+310 SS[o]	+2% 446[p]	+4% 446[p]	Phosphate-Bonded Alumina Ramming Mixes 45%[q]	90%[r]	90%[s]
Water Added, %	7.5	7.5	7.5	7.5	6	5	--
Bulk Density, lb/ft³							
Cured at 220 °F	140	143	142	143	--	--	--
After 24 h at 250 °F	--	--	--	--	150	195	182
After 2 h at 1000 °F	--	--	--	--	142.5	185	172
Shrinkage, % Linear							
After 220 °F for 24 h	0.1	0.1	--	--			
After 1000 °F for 2 h	0.1	0.1	--	--			
After 1500 °F for 2 h	0.1	0.1	--	0.04			
After 1875 °F (no time given)	0.05	0.05	0.04	0.04			
After 2000 °F (no time given)	0.0	0.0	--	--			

[a] Standard ASTM test procedures were followed; determined at ambient temperature on as-cured, dried, and fired samples.

[b] Linear and volume shrinkage determined by two methods: one method involved determining the change in length of the thermal expansion bars after testing. The second followed the ASTM C269-70 procedure.

[c] DOE 90 generic preparation: 65% tabular alumina (6 to -20 mesh), 10% calcined alumina (-325 mesh), 25% calcium aluminate cement.

[d] Cement 1 is regular CA-25 calcium aluminate cement (Alcoa).

[e] Cement 2 is calcium aluminate cement, Secar 250 (Lone Star Lafarge).

(Table Continued)

BULK DENSITY[a] AND SHRINKAGE[b] OF SEVERAL REFRACTORIES[26], Continued

Footnotes continued

[f] Cement 3 is high-purity calcium aluminate cement (Casting Grade CA-25, Alcoa).

[g] Modified DOE 90 preparation: 70% tabular alumina (-48 mesh), 5% calcined alumina (-325 mesh), 25% calcium aluminate cement (Casting Grade CA-25, Alcoa).

[h] Commercial castable (B&W Kao-Tab 95, Babcock & Wilcox).

[i] Generic preparation: 75% calcined kaolin (60% to -20 mesh, 15% ball milled fines with 50% less than 325 mesh), 25% calcium aluminate cement.

[j] Cement 4 is calcium aluminate cement (Refcon, Universal Atlas Cement).

[k] Modified generic preparation: 75% calcined kaolin (70% 6 to -20 mesh, 5% ball milled fines with 50% less than 325 mesh), 25% calcium aluminate cement (Casting Grade CA-25, Alcoa).

[l] Commercial lightweight insulating castable, calcium aluminate bonded (Litecast 75-28, General Refractories).

[m] Commercial lightweight insulating castable, 42% alumina (B&W Kaolite 2300 LI, Babcock & Wilcox).

[n] Coarse grain size, high-strength 50% alumina prototype castable (B&W Kaocrete XD50, Mix 36C, Babcock & Wilcox).

[o] 4 wt% of 1-in. long fibers of 310 SS (Ribtec 310) were added to the Kaocrete.

[p] 2 and 4 wt% of 1-in. long fibers of 446 SS (Ribtec 446) were added to the Kaocrete.

[q] Generic preparation of phosphate-bonded ramming mix: 15% calcined alumina, 65% calcined kaolin, 17% ball mill fines, 3% Bentonite (Wyoming), 1% hydrated alumina; bonded with 6% phosphoric acid (85% strength).

[r] Generic preparation of phosphate-bonded ramming mix: 82% tabular alumina, 15% calcined alumina, 3% Bentonite (Wyoming), 1% hydrated alumina; bonded with 6% phosphoric acid (85% strength).

[s] Commercial phosphate-bonded ramming mix: 90 Ram H.S. from C-E Refractories.

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THERMAL EXPANSION DATA[a] FOR SEVERAL ALUMINA-BASED REFRACTORIES[88]

90-95% Al_2O_3 dense castable [b]

α (100-900 °C) = 8.1 x 10^{-6}/°C
(4.5 x 10^{-6}/°F)

α (300-900 °C) = 4.0 x 10^{-6}/°C
(2.2 x 10^{-6}/°F)

(a)

(b)

54% Al_2O_3 lightweight insulating castable[c]

(a) 240 °F drying shrinkage =
0.1% (as-formed basis)

(b) 500 °F firing shrinkage =
0.2% (dried length basis)

This cooling curve shows anomously high shrinkage
compared to 0.35% expected on basis of other reheat
tests; cooling curve should also follow and have same
slope as heating curve.

Temperature, °C

Expansion, percent

59% Al_2O_3 brick [d]
α (100-900 °C) = 5.0 x 10^{-6}/°C
(2.8 x 10^{-6}/°F)

50-60% Al_2O_3 low-iron castable[e]

α (100-900 °C) = 4.3 x 10^{-6}/°C
(2.6 x 10^{-6}/°F)

Temperature, °C

Expansion, percent

- -

(Data Continued)

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THERMAL EXPANSION DATA[a] FOR SEVERAL ALUMINA-BASED REFRACTORIES[88], Continued

90% Al₂O₃ phosphate-bonded ramming mix [f]

α (100-900 °C) = 8.0 x 10⁻⁶/°C (4.4 x 10⁻⁶/°F)

(a) Measured drying shrinkage of only 0.2%
     for 5/8 x 5/8 x 2 inch specimen
     believed due to conatraint from bond-
     rich specimen surface.

(b) Dsshed curve shows estimated expansion
     on heating 2 x 2 x 7 inch bars which
     gave drying shrinkages of 1.0 to 1.5%
     typical for this type of material.

[a]Expansion specimens 5/8 x 5/8 x 2 inches were placed in a quartz tube
dilatometer immediately after an overnight moist cure period to obtain
drying shrinkage as well as expansion effects. The heating rate was
2.5 °C/minute (270 °F/h). Cooling rate was the natural furnace cool.
Expansion coefficients, α, were calculated from cooling curves (disre-
garding low temperature shrinkage effects) except where noted otherwise.

[b]B&W Kao-Tab 95 (Babcock & Wilcox).

[c]Litecast 75-28 (General Refractories). Expansion coefficient was calcu-
lated from heating curve with temperatures selected to disregard the low
temperature shrinkage effects.

[d]Ufala (Harbison-Walker Refractories).

[e]Steelkon (A.P. Green).

[f]90 Ram H.S. (C-E Refractories).

THERMAL CONDUCTIVITY[a] OF ALUMINA-BASED AND PERLITE-CONTAINING REFRACTORIES[b][88]

| Refractory[b] | Water Content[c] % | Bulk Density[d] lb/ft³ | Thermal Conductivity,[a] BTU/in/h/ft²/°F | | | | |
| | | | On Heating | | | | Cooling |
			500 °F	1000 °F	1500 °F	2000 °F	500 °F
90-95% Al₂O₃ dense castable (B&W Kao-Tab 95)	9.25	170.7	11.5 (24.7)	9.00 (15.8)	8.03 (13.2)	8.45 (13.2)	12.3 (20.2)
54% Al₂O₃ lightweight insulating castable (Litecast 75-28)	25.0	79.0	1.92 (2.1)	2.03 (2.4)	2.22 (2.6)	2.56 (3.0)	2.11 (--)
50-60% Al₂O₃ low-iron castable (Steelkon)	10.75	141.6	7.00 (--)	5.46 (--)	5.35 (--)	6.62 (5.3)	7.25 (4.4)
59% Al₂O₃ brick (Ufala)	---	155.9	10.9 (13.1)	9.20 (13.0)	8.80 (13.0)	9.35 (13.6)	12.2 (--)
90% Al₂O₃ phosphate-bonded ramming mix (90 Ram H.S.)	as received[e]	173.5	13.6 (--)	13.7 (17.6)	13.2 (14.6)	13.1 (13.2)	19.3 (--)
50 lb/ft³ perlite-containing insulating castable (Kaolite 2300 LI)	74.0	52.4	0.83 (1.38)	0.85 (1.37)	0.99 (1.57)	-- --	0.74 (1.26)

[a] Data obtained by ASTM C-201 by the Refractories Research Center at Ohio State University. The specimens used were 2.5 x 4.5 x 9 inches. Values given in parentheses are manufacturer's values.

[b] Castable specimens were fabricated by "spade casting", moist-curing overnight at ambient temperature, drying overnight at ambient temperature, then drying 8 hours at 140 °F and overnight at 240 °F. Ramming mix specimens were formed by air-ramming, air-drying overnight at ambient temperature, heating at ∿10 °F/h to 140 °F, holding overnight, heating at 10 °F/h to "∿180 °F" [200 °F was intended but in reality that temperature was not reached], holding overnight, heating to 240 °F, and holding overnight. [Delamination cracks formed in these ramming mix specimens. Whether these or others were used for these tests or what drying schedule was used for other specimens is not given. See B.4.2.21 for data for drying tests of the ramming mix.]

[c] Dry basis.

[d] Initial value after drying at 240 °F.

[e] Approximately 6.5% based on weight loss measurements.

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DRYING TESTS[a] TO PREVENT DELAMINATION CRACKING OF PHOSPHATE-
BONDED RAMMING MIX SLABS[b][88]

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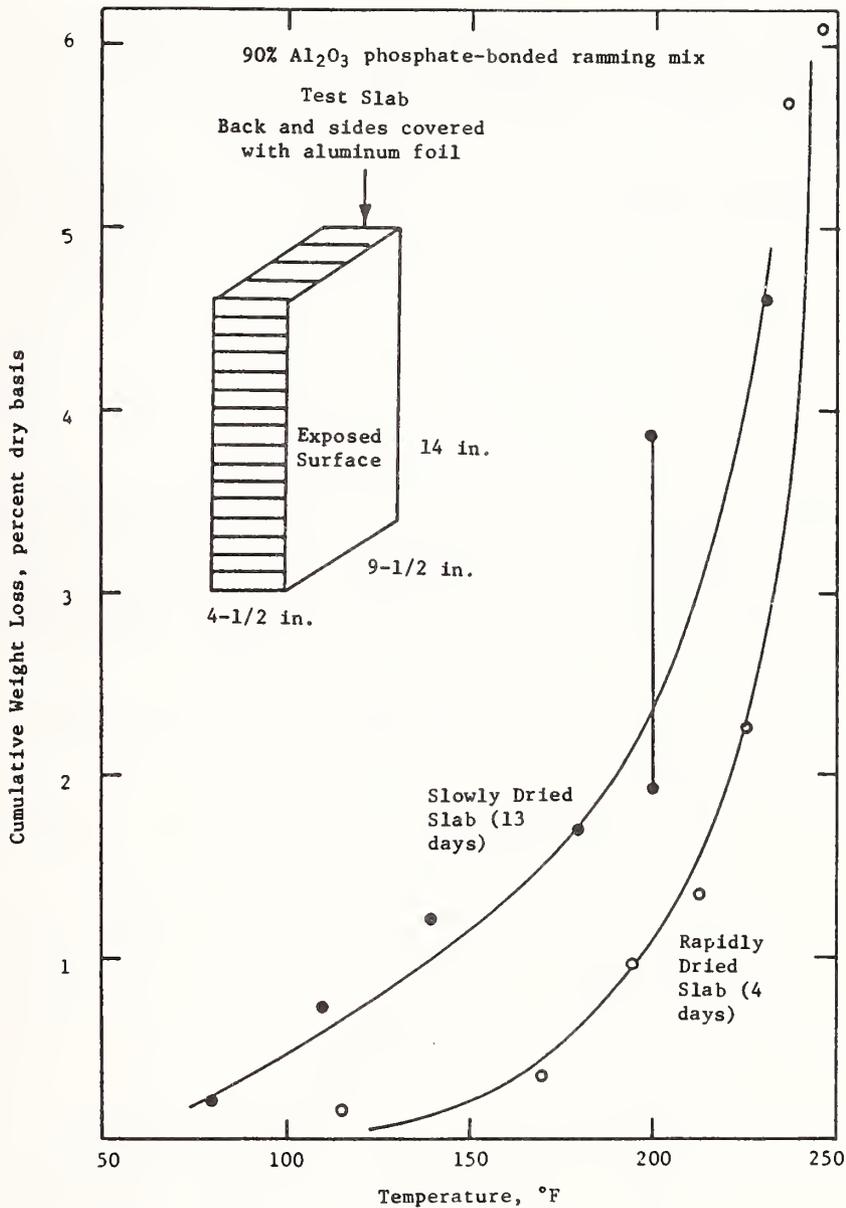
[a]To simulate the one-sided drying of refractory linings air-rammed slabs
of the mix were tightly covered on all but the top surface with aluminum
foil (see figure below for slab dimensions). The slabs were dried iso-
thermally (rather than under a temperature gradient). One slab was
dried slowly over a two-week period with one-day holds at various tem-
peratures. Note that only one-half of the moisture was removed after

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(Data Continued)

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DRYING TESTS[a] TO PREVENT DELAMINATION CRACKING OF PHOSPHATE-
BONDED RAMMING MIX SLABS[b][88], Continued

Footnotes continued

one day at 200 °F. A second slab was dried more rapidly but the heating
schedule was controlled so that the water vapor pressure was raised only
0.3 atmospheres with each temperature increase according to the follow-
ing schedule:

Saturated H_2O Vapor Pressure atmospheres	Temperature °F	Time at Temperature hours (approximate)
0.1	115	16
0.4	170	8
0.7	195	16
1.0	212	8
1.3	226	16
1.6	237	8
1.9	246	16

Delamination cracks did not form in this second slab. Water vapor
pressure within the specimen not only affects cracking but governs the
drying rate. The following Arrhenius plot is of data points for the
drying rates calculated from periodic weighings of the two test slabs
and a solid line representing the saturated water vapor pressure at
each temperature.
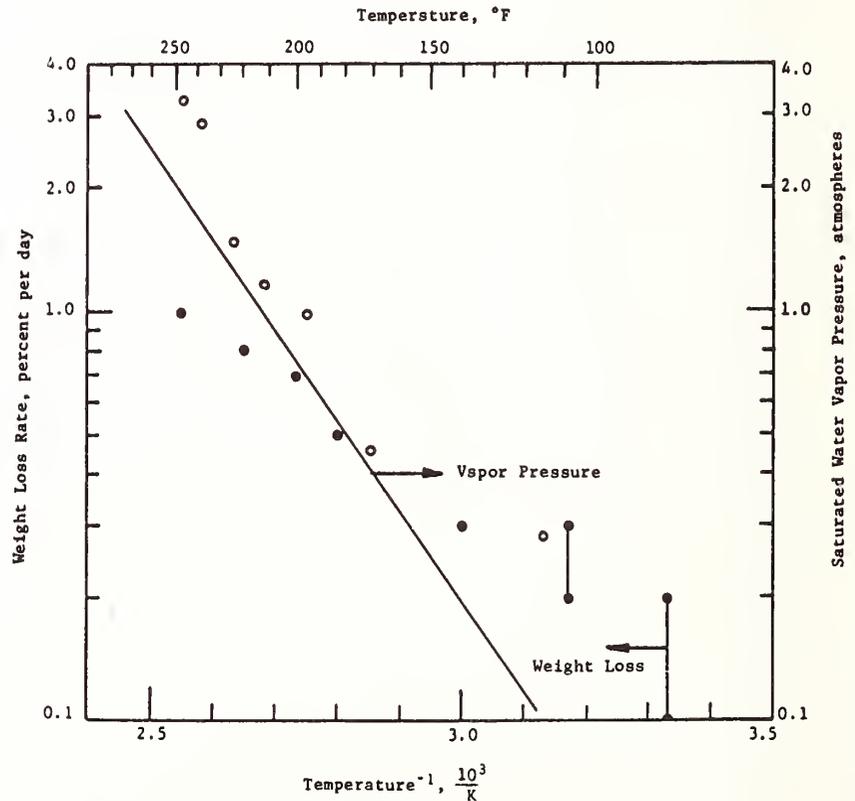
[b]90% Alumina phosphate-bonded ramming mix (90 Ram H.S.).

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EFFECT OF FIRING TEMPERATURE[a] ON LENGTH CHANGE OF MONOLITHIC
REFRACTORIES[88]

Refractory[c]	Water Content %	Length Change,[b] %				
		Based on As-Formed Length[d]				
		240 °F	500 °F	1000 °F	1500 °F	2000 °F
90-95% Al_2O_3 dense castable (B&W Kao-Tab 95)	9.25	-0.05	-0.15	-0.15	-0.17	nil
54% Al_2O_3 lightweight insulating castable (Litecast 75-28)	25.0	-0.10	-0.27	-0.35	-0.35	-0.45
50-60% Al_2O_3 low-iron castable (Steelkon)	10.75	-0.05	-0.15	-0.20	-0.20	-0.25
90% Al_2O_3 phosphate-bonded ramming mix (90 Ram H.S.)	as received[e]	-1.0	-1.10	-1.10	-1.0	-1.10
50 lb/ft^3 perlite-containing insulating castable (Kaolite 2300 LI)	74.0	-0.12	ND[f]	-0.68	ND[f]	-1.28

		Based on 240 °F Dried Length[g]			
		500 °F	1000 °F	1500 °F	2000 °F
90-95% Al_2O_3 dense castable (B&W Kao-Tab 95)	9.25	-0.10	-0.10	-0.12	+0.05
54% Al_2O_3 lightweight insulating castable (Litecast 75-28)	25.0	-0.17	-0.25	-0.25	-0.35
50-60% Al_2O_3 low-iron castable (Steelkon)	10.75	-0.10	-0.15	-0.15	-0.20
90% Al_2O_3 phosphate-bonded ramming mix (90 Ram H.S.)	as received[e]	-0.10	-0.10	±0.05	-0.10
50 lb/ft^3 perlite-containing insulating castable (Kaolite 2300 LI)	74.0	ND[f]	-0.56	ND[f]	-1.16

[a]Specimens were heated and cooled at 100 °F/h with a 5-hour soak at the maximum temperature.

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(Table Continued)

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EFFECT OF FIRING TEMPERATURE[a] ON LENGTH CHANGE OF MONOLITHIC
REFRACTORIES[88], Continued

Footnotes continued

[b] Change which occurred in bar specimens, 2 x 2 x 7 in., after five hours
at the indicated temperatures.

[c] Castable specimens were fabricated by "spade casting", moist-curing over-
night at ambient temperature, drying overnight at ambient temperature,
drying 8 hours at 140 °F and overnight at 240 °F. Ramming mix specimens
were formed by air-ramming, air-drying overnight at ambient temperature,
heating at ∿10 °F/h to 140 °F, holding overnight, heating at 10 °F/h to
200 °F , holding overnight, heating to 240 °F, and holding overnight.

[d] Specified in ASTM test method C-179 for plastic refractories; data
given include drying shrinkage shown in thermal expansion curves.

[e] Approximately 6.5% based on weight loss measurements.

[f] ND = not determined.

[g] Specified in ASTM test method C-269 for castable refractories; data do
not include drying shrinkage shown in thermal expansion curves.

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EFFECT OF FIRING TEMPERATURE[a] ON THE BULK DENSITY[b] OF VARIOUS REFRACTORIES[88]

Refractory[c]	Water Content %	Method	Bulk Density,[b] lb/ft^3, after heating[a]				
			240°F	500°F	1000 °F	1500°F	2000°F
90-95% Al_2O_3 dense castable (B&W Kao-Tab 95)	9.25	C-134	170.3	168.1	166.4	165.0	164.0
		C-830	176.0	173.0	172.8	172.9	168.7
	9.5	C-134	171.4	168.2	166.7	165.9	164.6
		C-830	173.7[d]	ND[e]	ND[e]	ND[e]	165.5[d]
54% Al_2O_3 lightweight insulating castable (Litecast 75-28)	25.0	C-134	79.2	76.7	75.3	75.1	74.9
		C-830	80.8	79.1	79.2	78.7	76.8
	24.0	C-134	81.2	78.3	77.3	77.0	76.4
		C-830	83.9[d]	ND	ND	ND	78.3[d]
50-60% Al_2O_3 low-iron castable (Steelkon)	10.75	C-134	140.6	137.3	135.1	134.4	134.2
		C-830	143.9	142.4	142.2	140.5	139.0
90% Al_2O_3 phosphate-bonded ramming mix (90 Ram H.S.)	as re-ceived[f]	C-134	181.6	180.4	180.3	179.7	180.6
		C-830	184.6	184.3	183.7	183.8	183.6
45-60% Al_2O_3 pitch-impregnated brick (Ufala TI)	--	C-134	153.7	ND	ND	ND	ND
		C-830	156.7	ND	ND	ND	ND
50 lb/ft^3 perlite-containing insulating castable (Kaolite 2300 LI)	74.0	C-134	51.4	ND	48.7	ND	48.3
		C-830	52.7	ND	50.0	ND	49.7

[a] Specimens were heated and cooled at 100 °F/h with a 5-hour soak at the maximum temperature.

[b] Bulk densities were determined by two different methods. ASTM method C-134, based on specimen weight and dimensions, and method C-830, using kerosene as the impregnating liquid. Above values are either the average of 5 determinations using C-134 or 2 determinations using C-830 unless otherwise noted.

[c] Specimens were 2-inch cubes cut from 2 x 2 x 7 in. bars. Castable specimens were fabricated by "spade casting", moist-curing overnight at ambient temperature, drying overnight at ambient temperature, drying 8 hours at 140 °F and overnight at 240 °F. Ramming mix specimens were formed by air-ramming, air-drying overnight at ambient temperature, heating at ∿10 °F/h to 140 °F, holding overnight, heating at 10 °F/h to 200 °F, holding overnight, heating to 240 °F, and holding overnight.

[d] Single value, see footnote b.

[e] ND = not determined.

[f] Approximately 6.5% based on weight loss measurements.

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EFFECT OF FIRING TEMPERATURE[a] ON APPARENT POROSITY[b] OF VARIOUS
REFRACTORIES[88]

Refractory[c]	Water Content %	Apparent Porosity,[b] %, after heating[a]				
		240°F	500°F	1000°F	1500°F	2000°F
90-95% Al_2O_3 dense castable (B&W Kao-Tab 95)	9.25	19.0	22.2	22.6	22.6	23.6
	9.5	20.6[d]	ND[e]	ND[e]	ND[e]	25.6[d]
54% Al_2O_3 lightweight insulating castable (Litecast 75-28)	25.0	47.1	49.8	50.3	51.1	54.9
	24.0	35.7[d]	ND	ND	ND	54.5[d]
50-60% Al_2O_3 low-iron castable (Steelkon)	10.75	18.8	19.6	20.7	21.5	23.3
90% Al_2O_3 phosphate-bonded ramming mix (90 Ram H.S.)	as received[f]	18.2	18.6	18.8	18.4	18.5
45-60% Al_2O_3 pitch-impregnated brick (Ufala TI)	--	15.9	ND	ND	ND	ND
50 lb/ft^3 perlite-containing insulating castable (Kaolite 2300 LI)	74.0	60.5	ND	64.9	ND	71.1

[a]Specimens were heated and cooled at 100 °F/h with a 5-hour soak at the maximum temperature.

[b]Apparent porosities were determined using ASTM method C-830. Values are the average of two determinations, unless otherwise noted.

[c]Specimens were 2-inch cubes cut from 2 x 2 x 7 in. bars. Castable specimens were fabricated by "spade casting", moist-curing overnight at ambient temperature, drying overnight at ambient temperature, drying 8 hours at 140 °F and overnight at 240 °F. Ramming mix specimens were formed by air-ramming, air-drying overnight at ambient temperature, heating at ∿10 °F/h to 140 °F, holding overnight, heating at 10 °F/h to 200 °F, holding overnight, heating to 240 °F and holding overnight.

[d]Single value, see footnote b.

[e]ND = not determined.

[f]Approximately 6.5% based on weight loss measurements.

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EFFECT OF FIRING ON PORE SIZE DISTRIBUTION[a] IN TWO CASTABLE
REFRACTORIES[b][88]

Volume Percent Pores

Mean Pore Diameters
0.3, 0.7, 1.4, & 1.5 microns

Dried
240 °F

Fired
2000 °F

———— 90-95% Al_2O_3 dense castable[c]

– – – 54% Al_2O_3 lightweight [d]
insulating castable

Pore Radius, microns
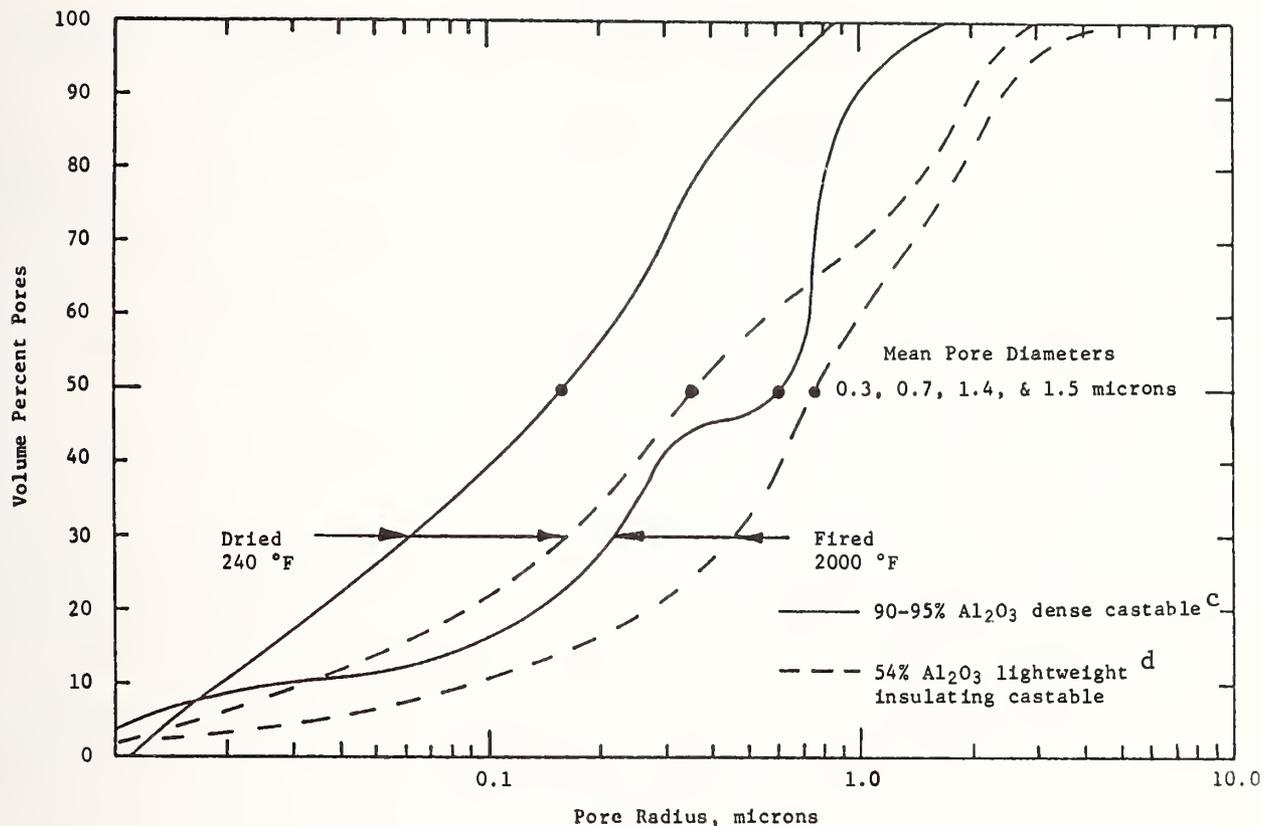
[a]Determined by mercury porosimetry. Residual mercury in the specimens cal-
culated from weight measurements indicated that 1/2 to 2/3 of the measured
porosity consisted of "ink-well" pores, large internal pores connected to
the surface by fine matrix pore channels. Measured pore sizes, therefore,
only reflect the apparent size of connecting channels, not the actual size
of the internal pores. Microscopic examination revealed that internal
pores, 0.1 inch (2500 microns) in diameter, were common.

[b]Castable specimens were fabricated by "spade casting", moist-curing over-
night at ambient temperature, drying overnight at ambient temperature,
drying 8 hours at 140 °F and overnight at 240 °F.

[c]B&W Kao-Tab 95, 9.25% water content.

[d]Litecast 75-28, 25.0% water content.

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CLOSED AND TOTAL POROSITY OF TWO CASTABLE REFRACTORIES[88]

Property	Dense Castable[a]		Lightweight Insulating Castable[b]	
	Dried 240°F	Fired 2000°F	Dried 240°F	Fired 2000°F
Apparent Porosity,[c] %				
Hg porosimetry	16.3	21.6	49.3	52.7
ASTM C-830 (kerosene)	19.0	23.6	47.1	54.9
True Density (assumed)				
lb/ft^3	237.2	237.2	187.3	187.3
g/cm^3	3.8	3.8	3.0	3.0
Bulk Density,[d]				
lb/ft^3	176.0	168.6	80.5	76.8
g/cm^3	2.82	2.70	1.29	1.23
Total Porosity,[e] %				
(assumed)	26.0	29.0	57.0	59.0
Closed Porosity, %				
(by difference)	7.0	5.0	10.0	4.0

[a] 90-95% Alumina dense castable (B&W Kao-Tab 95).

[b] 54% Alumina lightweight insulating castable (Litecast 75-28).

[c] Apparent porosity determined using both mercury porosimetry and ASTM method C-830 with kerosene as the impregnating liquid.

[d] Bulk density was determined by ASTM method C-830 with kerosene as the impregnating liquid.

[e] Total porosity = $(1 - \dfrac{\text{bulk density}}{\text{true density}})$ x 100.

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APPARENT POROSITY DATA[a] OF REFRACTORY TEST PANEL[b] MATERIALS SUBJECTED
TO VARIOUS HEAT TREATMENTS[c][88]

Specimen Location	Panel→	4 1/2-in. dense & 7 1/2 in. insulating castables[d]					12-in. low-iron castable[e]	4 1/2-in. ramming mix[f]	12 1/2-in. perlite-containing castable[g]
		1	2	3	4	5	6	7	8
Hot Face		22.9	--	20.2	22.9	24.7	22.1	--	73.0
	--	20.8	22.4	25.1	25.9	25.7	22.2	--	68.1
Interface		55.0	56.1	--	54.1	54.2	23.2	--	65.6
	--	52.8	55.4	--	52.4	52.9	22.4	--	65.6
		52.8	54.7	51.7	51.9	--	22.3	19.1	65.3
Base	--	52.2	51.7	49.5	49.5	54.0	21.7	25.9[h]	60.3

[a] Apparent porosity determined using ASTM method C-830 using nominally 2-inch cubes removed from the central portion of each panel at several depths in the refractory. See Section 4.2.24 for apparent porosity values for these materials after heating at different temperatures.

[b] Test panels for tests at atmospheric pressure were 38-inch squares cast directly onto a 1 inch thick steel plate. For panels 12 inches thick, 10 1/2-inch 304 SS V anchors were used, for panels 4 1/2 inches thick 3-inch 304 SS V anchors were used. The panel for the high-pressure test was an 18-inch square. Castables were placed by spading and rodding. Several batches were required. Two or three batches were cast back-to-back and moist cured overnight and for panels 1 through 5 where two refractories constituted the panel, the second refractory was then also prepared in batches, cast back-to-back, and moist cured overnight. Single material panels were fully cast at one time. The panels were then air dried at ambient temperature several days, installed in the test furnace, the panel surface heated to 220 °F overnight with the furnace lid propped open, and then heated to 1000 °F at 50 °F/h. At 450 °F the furnace lid was closed and a dry-air purge begun. The temperature was held at 1000 °F overnight, then heated to 1500 °F at 50 or 100 °F/h. Ramming mix was installed by air-ramming 2 x 2 x 7 in. bars in molds, placing the bars around the anchors and thermocouple instrumentation, and then air-ramming the mix to complete the balance of the panel. The ramming mix was predried more slowly than the castables by using a heating plate with cone heaters held 12 inches above the surface and heating to 140 °F overnight. Surface temperature was raised to 170 °F and was to stay overnight but rose to 208 °F (some surface and delamination cracks formed). The temperature was decreased to 150 °F and held about 1 week, then in the furnace the surface temperature was raised to 400 °F over three days (cold face 190 °F), and then heated to 750 °F at 50 °F/h.

[c] Test panels were actually prepared for performing heat transfer studies. The

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(Table Continued)

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APPARENT POROSITY DATA[a] OF REFRACTORY TEST PANEL[b] MATERIALS SUBJECTED
TO VARIOUS HEAT TREATMENTS[c][88], Continued

Footnotes continued

conditions for the studies for each panel follow:

Test Panel	Gas*	Temperature °F	Time at Nominal Hot Face Temperature, h	Transition Time Between Steady-State Conditions, h
1	Air	1000		
	Air	1500	Not	Not
	Air	1750	given	given
	Air	2000		
2	Air	1500	8	
	(Top 1-1/2 inches of material were removed.)			
	Air	1500	96	
3	Air	1500	55	
	Hydrogen	1500	48	72
4	Air	1500	32	
	Hydrogen	1500	79	152
5	Air(15 psia)	1500	63	
	Air(515 psia)	1000	7	26
	H₂(15 psia)	1100	68	84
	H₂(515 psia)	600	18	22
6	Air	1000	43	
	Air	1500	67	32
	CO₂	1500	70	24
	Hydrogen	1400	32	26
	Air	1700	50	46
7	Air	600	74	
	Hydrogen	550	24	70
8	Air	1800	135	
	Air	1500	55	24
	Hydrogen	1500	90	48

*Unless otherwise indicated, all gases are at atmospheric pressure.

[d]Panel consisted of 90-95% alumina dense castable (B&W Kao-Tab 95) and 54% alumina lightweight insulating castable (Litecast 75-28). Panel 1 was used in the shakedown testing of the equipment. Panel 2 testing was interrupted to remove the top 1-1/2 inches of the refractory for the heat transfer study. Panel 3 was fabricated with vertical cracks from the surface to both tips of the anchors. Panel 4 was fabricated with vertical cracks from the surface to the steel base plate.

[e]Panel material was 50-60% alumina low-iron castable (Steelkon).

[f]Panel material was 90% alumina phosphate-bonded ramming mix (90 Ram H.S.).

[g]Panel material was 50 lb/ft³ perlite-containing insulating castable (Kaolite 2300 LI).

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EFFECT OF CURING AND FIRING TEMPERATURES[a] ON THE BULK DENSITY[b] OF
SEVERAL ALUMINA REFRACTORIES[16]

Curing and Firing Temperature °C	Bulk Density, g/cm^3		
	Tabular alumina/ CA cement[c]	Tabular alumina/ CA cement[d]	Calcined bauxite/ CA cement[e]
Ambient	2.78	2.63	2.27
100	2.69	2.56	2.18
200	2.65	2.54	2.14
400	2.63	2.48	2.11
600	2.61	2.47	2.10
800	2.61	2.45	2.07
1000	2.61	2.39	2.04
1200	2.59	2.36	2.08
1350	2.64	2.36	2.12
	Calcined bauxite/ CA cement[f]	Calcined kaolin/ CA cement[g]	Calcined kaolin/ CA cement[h]
Ambient	2.26	2.25	2.20
100	2.20	2.18	2.17
200	2.15	2.17	2.14
400	2.12	2.11	2.11
600	2.12	2.10	2.10
800	2.10	2.07	2.07
1000	2.06	2.04	2.04
1200	2.04	2.08	2.01
1350	2.07	2.12	2.16
	Insulating Castables		
	57% Alumina[i]	50% Alumina[j]	
Ambient	1.45	1.35	
100	1.42	1.32	
200	1.41	1.30	
400	1.36	1.26	
600	1.37	1.26	
800	1.38	1.28	
1000	1.40	1.28	
1200	1.38	1.28	

[a] Samples were cast, vibrated one minute, cured sealed in plastic 24 hours at ambient temperature, cured in air 24 hours at ambient temperature, dried in air 24 hours at 100 °C, fired at 60 °C/h to 200, 400, 600, 800, 1000, 1200, and 1350 °C, soaked at temperature 5 hours, and furnace cooled to ambient temperature.

[b] Bulk densities were determined by the simple geometric method, calculating from sample weights and dimensions.

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(Table Continued)

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EFFECT OF CURING AND FIRING TEMPERATURES[a] ON THE BULK DENSITY[b] OF
SEVERAL ALUMINA REFRACTORIES[16], Continued

Footnotes continued

[c]75% T-61 alumina (Alcoa) aggregate, 25% Casting Grade CA-25 cement (Alcoa), water to ball-in-hand consistency 10.4% of aggregate and cement.

[d]75% T-61 alumina (Alcoa) aggregate, 25% Secar 71 cement (Lone Star Lafarge), water to ball-in-hand consistency 12.7% of aggregate and cement.

[e]75% Mulcoa M-60 (C-E Minerals) aggregate, 25% Casting Grade CA-25 (Alcoa) cement, water to ball-in-hand consistency 10.6% of aggregate and cement.

[f]75% Mulcoa M-60 (C-E Minerals) aggregate, 25% Secar 71 cement (Lone Star Lafarge), water to ball-in-hand consistency 14.6% of aggregate and cement.

[g]75% Mulcoa M-47 (C-E Minerals) aggregate, 25% Casting Grade CA-25 (Alcoa) cement, water to ball-in-hand consistency 11.3% of aggregate and cement.

[h]75% Mulcoa M-47 (C-E Minerals) aggregate, 25% Refcon cement (Universal Atlas), water to ball-in-hand consistency 13.5% of aggregate and cement.

[i]A commercial lightweight insulating castable (Cer-Lite #75, C-E Refractories), water to ball-in-hand consistency 22% of mix.

[j]A commercial lightweight insulating castable (Litecast 75-28, General Refractories), water to ball-in-hand consistency 24% of mix.

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BULK DENSITY[a] VERSUS CURING/FIRING TEMPERATURE[b] FOR A HIGH-ALUMINA CASTABLE[c]

WITH VARIOUS AGGREGATE PARTICLE SIZE DISTRIBUTIONS[d][16]

Curing/Firing Temperature °C	Bulk Density,[a] g/cm³			
	Generic Castable[d]	Continuous Aggregate Formulation[d]		
		20% Cement	25% Cement	30% Cement
Ambient	2.78±0.01	2.81±0.02	2.81±0.02	2.76±0.01
100	2.69±0.01	2.75±0.01	2.71±0.01	2.68±0.01
200	2.65±0.01	2.72±0.01	2.68±0.01	2.62±0.01
400	2.63±0.02	2.71±0.01	2.61±0.01	2.57±0.01
600	2.61±0.01	2.69±0.01	2.59±0.01	2.54±0.01
800	2.61±0.01	2.67±0.01	2.59±0.01	2.54±0.01
1000	2.61±0.01	2.66±0.01	2.55±0.01	2.49±0.01
1200	2.59±0.01	2.69±0.01	2.54±0.01	2.51±0.01
1350	2.64±0.01	--	--	--

	Gap-Sized Aggregate Formulation[d]		
	20% Cement	25% Cement	30% Cement
Ambient	2.98±0.02	2.91±0.01	2.85±0.01
100	2.92±0.01	2.88±0.01	2.83±0.01
200	2.09±0.01	2.75±0.01	2.72±0.01
400	2.86±0.01	2.74±0.02	2.67±0.01
600	2.86±0.02	2.79±0.01	2.65±0.01
800	2.86±0.01	2.77±0.02	2.64±0.01
1000	2.83±0.01	2.73±0.01	2.60±0.01
1200	2.83±0.01	2.72±0.01	2.62±0.01
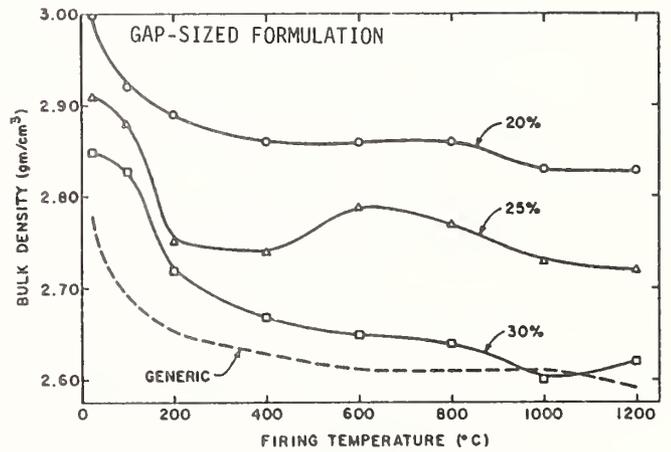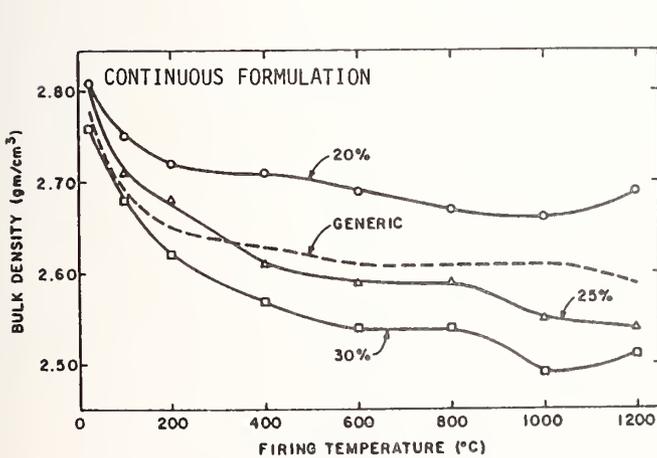
[a]Bulk densities were determined by the simple geometric method, calculating from sample weights and dimensions.

[b]Samples were cast, vibrated one minute, cured sealed in plastic 24 hours at ambient temperature, cured in air 24 hours at ambient temperature, dried in air 24 hours at 100 °C, fired at 60 °C/h to 200, 400, 600, 800, 1000,

(Data Continued)

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BULK DENSITY[a] VERSUS CURING/FIRING TEMPERATURE[b] FOR A HIGH-ALUMINA CASTABLE[c]
WITH VARIOUS AGGREGATE PARTICLE SIZE DISTRIBUTIONS[d][16], Continued

Footnotes continued

1200, and 1350 °C, soaked at temperature 5 hours, and furnace cooled to
ambient temperature.

[c]T-61 tabular alumina (Alcoa) aggregate, Casting Grade CA-25 calcium alumi-
nate cement (Alcoa). Generic formulation 75% aggregate, 25% cement, water
to ball-in-hand consistency 10.3% of aggregate and cement. Continuous for-
mulation: 20% cement has 9.9% water, 25% cement has 10.6% water, 30% cement
has 11.1% water. Gap-sized formulation: 20% cement has 8.0% water, 25%
cement has 9.1% water, 30% cement has 9.8% water.

[d]Various samples were prepared to study the effect of aggregate particle
size distribution on the properties of the castable. The generic castable
has a size distribution typical of a standard industrial product and is the
as-received material. The other two formulations are designed for maximum
density of the aggregate and the distributions were calculated according to
formulas for each case. The continuous formula contains aggregate continu-
ously graded so that the difference between successive particle sizes is a
factor of $\sqrt{2}$, with the largest particles sized at U.S. Sieve Number 4 x 6
and the smallest -325. Gap-sized packing provides sizes and proportions
so that the larger voids created between larger particles are filled by
smaller particles and new voids thus created filled by even smaller parti-
cles. The formulation used here is 61.5% of 4x6, 24.5% of 16x20, 9.8% of
70x100, and 4.2% of -325 mesh.

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LINEAR THERMAL EXPANSION COEFFICIENTS[a] FOR ALUMINA CASTABLES[b][16]

Refractory — Curing/Firing Temperature→	Linear Thermal Expansion Coefficient,[a] (x 10^{-6}/°C)								
	ambient	100	200	400	600	800	1000	1200	1350
Tabular alumina/CA cement[c]	5.8	5.3	6.5	6.6	6.6	6.7	6.6	6.8	7.2
Calcined bauxite/CA cement[d]	5.4	5.3	5.7	6.4	6.6	6.9	6.9	6.9	7.2
Calcined bauxite/CA cement[e]	4.7	4.5	4.4	4.8	5.2	5.3	5.3	5.3	5.8
Calcined kaolin/CA cement[f]	6.2	6.1	6.1	6.4	6.5	6.5	6.7	6.9	6.9
Calcined kaolin/CA cement[g]	6.7	6.3	6.3	6.5	6.9	6.9	6.7	6.7	6.8
57% Alumina insulating castable[h]	6.2	6.2	6.5	7.5	7.6	8.2	8.2	8.4	--
50% Alumina insulating castable[i]	5.9	5.9	6.1	6.8	6.9	7.4	7.6	7.6	--
Tabular alumina/CA cement[j]									
Generic formula, 25% cement[k]	7.0	6.0	5.0	6.0	7.0	7.4	8.1	8.3	--
Continuous formula, 20% cement[k]	6.3	5.0	4.8	5.9	6.8	7.1	7.4	7.9	--
Continuous formula, 25% cement	6.4	5.3	5.7	6.1	6.6	7.0	7.2	7.6	--
Continuous formula, 30% cement	6.5	5.3	5.7	6.4	6.8	7.2	7.4	7.7	--
Gap-sized formula, 20% cement[k]	6.5	5.3	5.1	5.9	6.8	7.4	7.8	8.3	--
Gap-sized formula, 25% cement	6.5	6.7	5.1	5.3	7.5	6.1	7.9	9.1	--
Gap-sized formula, 30% cement	6.8	5.3	6.8	7.4	7.8	7.8	8.0	8.2	--

[a] Thermal expansion was determined using an automatic recording dilatometer. Specimens were heated at a rate of 3 °C/minute to temperature in a horizontal tube furnace. The coefficients were calculated from plots of percent expansion versus temperature. Thermal expansion was measured on a specimen of each refractory for each curing and firing schedule.

[b] Samples were cast, vibrated one minute, cured sealed in plastic 24 hours at ambient temperature, cured in air 24 hours at ambient temperature, dried in air 24 hours at 100 °C, fired at 60 °C/h to 200, 400, 600, 800, 1000, 1200, and 1350 °C, soaked at temperature 5 hours, and furnace cooled to ambient temperature.

[c] 75% T-61 alumina (Alcoa) aggregate, 25% Secar 71 cement (Lone Star Lafarge), water to ball-in-hand consistency 12.7% of aggregate and cement.

(Data Continued)

LINEAR THERMAL EXPANSION COEFFICIENTS[a] FOR ALUMINA CASTABLES[b][16], Continued

Footnotes continued

[d] 75% Mulcoa M-60 (C-E Minerals) aggregate, 25% Casting Grade CA-25 (Alcoa) cement, water to ball-in-hand consistency 10.6% of aggregate and cement.

[e] 75% Mulcoa M-60 (C-E Minerals) aggregate, 25% Secar 71 cement (Lone Star Lafarge), water to ball-in-hand consistency 14.6% of aggregate and cement.

[f] 75% Mulcoa M-47 (C-E Minerals) aggregate, 25% Casting Grade CA-25 (Alcoa) cement, water to ball-in-hand consistency 11.3% of aggregate and cement.

[g] 75% Mulcoa M-47 (C-E Minerals) aggregate, 25% Refcon cement (Universal Atlas), water to ball-in-hand consistency 13.5% of aggregate and cement.

[h] A commercial lightweight insulating castable (Cer-Lite #75, C-E Refractories), water to ball-in-hand consistency 22% of mix.

[i] A commercial lightweight insulating castable (Litecast 75-28, General Refractories), water to ball-in-hand consistency 24% of mix.

[j] T-61 alumina (Alcoa) aggregate, Casting Grade CA-25 cement (Alcoa).

[k] Various samples were prepared to study the effect of aggregate particle size distribution on the properties of the castable. The generic castable has a size distribution typical of a standard industrial product and is the as-received material. The other two formulations are designed for maximum density of the aggregate and the distributions were calculated according to formulas for each case. The continuous formula contains aggregate continuously graded so that the difference between successive particle sizes is a factor of $\sqrt{2}$, with the largest particles sized at U.S. Sieve Number 4 x 6 and the smallest -325. Gap-sized packing provides sizes and proportions so that the larger voids created between larger particles are filled by smaller particles and new voids thus created filled by even smaller particles. The formulation used here is 61.5% of 4x6, 24.5% of 16x20, 9.8% of 70x100, and 4.2% of -325 mesh. The generic formulation: 75% aggregate, 25% cement, water to ball-in-hand consistency 10.3% of aggregate and cement. Continuous formulation: 20% cement has 9.9% water, 25% cement has 10.6% water, 30% cement has 11.1% water. Gap-sized formulation: 20% cement has 8.0% water, 25% cement has 9.1% water, 30% cement has 9.8% water.

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COEFFICIENTS OF LINEAR THERMAL EXPANSION[a] FOR SOME ALUMINA REFRACTORIES[2]

Composition	Brand Name[b]	in./in./°F
99.5% Al_2O_3	--[c]	4.50×10^{-6}
96% Al_2O_3	Plicast 40	4.2×10^{-6}
94.5% Al_2O_3-0.15% SiO_2	Greencast-94	4.56×10^{-6}
93.7% Al_2O_3	Taycor 414-FH	4.0×10^{-6}

[a]Thermal expansion procedure of ASTM C372-56 followed.

[b]See Section B.3.2.10 for manufacturers.

[c]Manufacturer Wesgo.

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EFFECT OF EXPOSURE[a] TO VARIOUS ATMOSPHERES[b] ON THE THERMAL EXPANSION[c]
OF ALUMINA CASTABLE REFRACTORIES[d][39]

94% Alumina dense castable[e]

1 = control (as-cast and dried)

2 = CO/H_2O = 1.1, 100% steam saturated,
199 °C, 3.2 MPa, 6 days

3 = same exposure as in 2 but
for 18 days

93% Alumina dense castable[f]

C = control (as-cast and dried)

S = CGA, 100% steam saturated, 30 days, 231 °C

US = CGA, unsaturated, 30 days, 538 °C

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(Data Continued)

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EFFECT OF EXPOSURE[a] TO VARIOUS ATMOSPHERES[b] ON THE THERMAL EXPANSION[c]
OF ALUMINA CASTABLE REFRACTORIES[d][39], Continued

93% Alumina dense caatable[f]

A = CGA, 100% steam saturated, 44 days, 231 °C

B = CGA, 85% steam aaturated, 5 daya, 240 °C

C = Exposed aa in B followed by firing at 1000 °F for 18 houra

87% Alumina dense caatable[g]

A = CGA, 100% steam aaturated, 44 daya, 231 °C

B = CGA, 85% steam aaturated, 5 days, 240 °C

C = Exposed aa in B followed by firing at
    1000 °F for 18 hours

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(Data Continued)

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EFFECT OF EXPOSURE[a] TO VARIOUS ATMOSPHERES[b] ON THE THERMAL EXPANSION[c]
OF ALUMINA CASTABLE REFRACTORIES[d][39], Continued

64% Alumina-28% Silica castable

A = CGA, 100% steam saturated, 44 days, 231 °C
B = CGA, 85% ateam saturated, 5 days, 240 °C
C = Expoaed as in B followed by firing at 1000 °F for 18 hours

59% Alumina, 30% Silica caatable

A = CGA, 100% steam saturated, 44 days, 231 °C
B = CGA, 85% ateam aaturated, 5 days, 240 °C
C = Exposed as in B followed by firing at 1000 °F for 18 hours

(Data Continued)

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EFFECT OF EXPOSURE[a] TO VARIOUS ATMOSPHERES[b] ON THE THERMAL EXPANSION[c]
OF ALUMINA CASTABLE REFRACTORIES[d][39], Continued

57% Alumina-34% Silica castable[j]

C = control (as-cast and dried)
S = CGA, 100% steam saturated, 30 days, 231 °C
US = CGA, unsaturated, 30 days, 538 °C

54% Alumina-40% Silica insulating castable[k]

C = control (as-cast and dried)
S = CGA, 100% steam saturated, 30 days, 231 °C
US = CGA, unsaturated, 30 days, 538 °C

(Data Continued)

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EFFECT OF EXPOSURE[a] TO VARIOUS ATMOSPHERES[b] ON THE THERMAL EXPANSION[c]
OF ALUMINA CASTABLE REFRACTORIES[d][39], Continued

54% Alumina-40% Silica inaulating castable [k]

A = CGA, 100% ateam ssturated, 44 dsya, 231 °C
B = CGA, 85% ateam asturated, 5 days, 240 °C
C = Exposed aa in B followed by firing st 1000 °C for 18 hours

47% Alumina-40% Silica Inaulating Caatable [l]

A = CGA, 100% steam saturated, 44 dsys, 231 °C
B = CGA, 85% ateam aaturated, 5 days, 240 °C
C = Expoaed as in B followed by firing at 1000 °F for 18 hours

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(Data Continued)

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EFFECT OF EXPOSURE[a] TO VARIOUS ATMOSPHERES[b] ON THE THERMAL EXPANSION[c]
OF ALUMINA CASTABLE REFRACTORIES[d][39], Continued

[a]Samples were exposed in two pressure vessels. One vessel was a steam gen-
erator and tests which specify saturated conditions occurred in this vessel.
After placing samples, the vessel was heated to the desired steam pressure
and the other gases were then added so as to obtain the test concentrations.
The second vessel was connected to the steam generator and after samples
were placed and the vessel heated to the test temperature, the vessel was
pressurized with the steam/gas mixture from the steam generator. Tests
specified as unsaturated occurred in this vessel. A gaseous flow rate for
both vessels was maintained so as to provide one complete change of atmo-
sphere every 24 hours. Except where otherwise noted the exposures were at
6.9 MPa (1000 psi).

[b]CGA = coal gasification atmosphere. Composition (in vol %): 18 CO, 12 CO_2,
24 H_2, 5 CH_4, and 41 H_2O. One exposure used a CO/H_2O atmosphere (in vol %):
52.5 CO and 46.5 H_2O.

[c]After exposures as indicated on the various figures, the thermal expansion
of 5 cm specimens was measured continuously during heating from 20 to 950 °C
at 250 °C/hour, and during cooling to ambient temperatures.

[d]After casting, the specimens were cured 24 hours in air at 100% humidity at
ambient temperature, and dried at 230 °F for 24 hours. All specimens were
stored at 230 °F until tested.

[e]94% alumina dense castable, calcium aluminate (79% alumina-18% calcia) bond-

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(Data Continued)

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EFFECT OF EXPOSURE[a] TO VARIOUS ATMOSPHERES[b] ON THE THERMAL EXPANSION[c]
OF ALUMINA CASTABLE REFRACTORIES[d][39], Continued

Footnotes continued

ed (Greencast-94, A.P. Green; CA-25 Calcium Aluminate Cement, Alcoa).

[f] 93% alumina dense castable, UMR-1 generic preparation (70% tabular alumina + 30% calcium aluminate cement) (T-61 alumina, Alcoa; CA-25 Calcium Aluminate Cement, Alcoa).

[g] 87.4% alumina dense castable, UMR-4 generic preparation (70% tabular alumina + 30% calcium aluminate cement) (T-61 alumina, Alcoa; Refcon, a 58% alumina-33% calcia cement, Universal Atlas).

[h] 63.8% alumina, 28.3% silica dense castable, UMR-8 generic preparation (75% calcined kaolin aggregate + 25% calcium aluminate cement) (Mulcoa M-60 and Mulcoa M-47, C-E Minerals; CA-25 Calcium Aluminate Cement, a 79% alumina-18% calcia cement, Alcoa).

[i] 58.6% alumina, 29.6% silica dense castable, UMR-5 generic preparation (75% calcined kaolin aggregate + 25% calcium aluminate cement) (Mulcoa M-60 and Mulcoa M-47, C-E Minerals; Refcon a 58% alumina-33% calcia cement, Universal Atlas).

[j] 57% alumina, 34% silica castable, calcium aluminate (79% alumina, 18% calcia) bonded (RC-3, General Refractories; CA-25 Calcium Aluminate Cement, Alcoa).

[k] 54% alumina, 40% silica insulating castable, calcium aluminate (79% alumina, 18% calcia) bonded (Cer-Lite #75, C-E Refractories; CA-25 Calcium Aluminate Cement, Alcoa).

[l] 46.7% alumina, 40.2% silica insulating castable (Litecast 60-25, General Refractories).

[m] 34.5% alumina, 52.5% silica insulating castable (VSL-50, A.P. Green).

THERMAL EXPANSION DATA[a] FOR ALUMINA CASTABLES[b] AS AFFECTED BY VARIOUS EXPOSURE CONDITIONS[c] COMPARED WITH BOEHMITE CONTENT[d][39]

Atmosphere[c]	Temperature °C / °F	Pressure MPa/psi	Time days	Average Thermal Expansion Coefficient 25-950 °C $\alpha \times 10^{-6}$/°C [e]	Fractional Thermal Expansion, 500-600 °C $\Delta L/L$, %	Final Fractional Thermal Expansion $(\Delta L/L)$final, %	% Boehmite
93% ALUMINA DENSE CASTABLE[e]							
Air (dried) — vapor	110/230	ambient	1	7.35	0	-0.2	0
Saturated steam — liquid	231/447	2.8/410	10	7.57	0.02	+0.1	20.2
Saturated steam — vapor	231/447	2.8/410	10	7.89	0.02	+0.01	22.
Saturated steam — liquid	285/545	6.9/1000	10	8.64	0.08	+0.07	22.5
Saturated steam — vapor	285/545	6.9/1000	10	7.89	0.01	+0.01	23.5
Saturated CGA[f] — liquid	231/447	6.9/1000	10	11.04	0.05	+0.2	22.3
Saturated CGA[f] — vapor	231/447	6.9/1000	10	not measured	not measured	not measured	21.8
Saturated CGA[f] — liquid	231/447	6.9/1000	60	8.86	0.1	+0.1	32.
Saturated CGA[f] — vapor	231/447	6.9/1000	60	8.	0.09	+0.02	29.
CO/H_2O = 0.1[f] — liquid	278/532	6.9/1000	20	9.3	0.07	0.13	30.
CO/H_2O = 0.1[f] — vapor	278/532	6.9/1000	20	8.86	0.03	0	25.3
CO/H_2O = 1.0[f] — liquid	241/466	6.9/1000	20	11.24	0.01	+0.11	18.6
CO/H_2O = 1.0[f] — vapor	241/466	6.9/1000	20	9.72	0.12	+0.15	26.1
CO/H_2O = 3.0[f] — liquid	204/400	6.9/1000	20	10.71	0.08	+0.25	23.3
CO/H_2O = 3.0[f] — vapor	204/400	6.9/1000	20	8.11	0.00	+0.03	21.6
87% ALUMINA DENSE CASTABLE[g]							
Saturated steam — vapor	285/545	6.9/1000	10	5.56	0.03	-0.02	20.0
Saturated steam — liquid	285/545	6.9/1000	10	7.56	0.02	-0.02	18.3
CO/H_2O = 1.0 — vapor	241/466	6.9/1000	20	not measured	not measured	not measured	9.9
CO/H_2O = 1.0 — liquid	241/466	6.9/1000	20	8.65	0.00	-0.1	10.7
64% ALUMINA-28% SILICA DENSE CASTABLE[h]							
Saturated steam — vapor	231/447	2.8/410	10	4.86		-0.06	9.5
Saturated steam — liquid	231/447	2.8/410	10	5.08		-0.04	11.5
Saturated steam — vapor	285/545	6.9/1000	10	4.97		-0.07	11.4
Saturated steam — liquid	285/545	6.9/1000	10	5.08		-0.05	10.8
Saturated steam — vapor	231/447	6.9/1000	10	5.0		-0.5	11.1
Saturated steam — liquid	231/447	6.9/1000	10	not measured		not measured	14.
59% ALUMINA-30% SILICA DENSE CASTABLE[i]							
Air (dried) — vapor	110/230	ambient	1	4.43		-0.2	0
Saturated steam — liquid	231/447	2.8/410	10	not measured		not measured	8.7
Saturated steam — vapor	231/447	2.8/410	10	4.11		-0.08	10.1
Saturated steam — liquid	285/545	6.9/1000	10	4.11		-0.14	11.1
Saturated steam — vapor	285/545	6.9/1000	10	5.08		-0.05	10.3
Saturated CGA — liquid	231/447	6.9/1000	10	5.56		-0.06	14.6
Saturated CGA — vapor	231/447	6.9/1000	10	not measured		not measured	13.2
CO/H_2O = 1.0 — liquid	241/466	6.9/1000	20	5.19		-0.17	10.
CO/H_2O = 1.0 — vapor	241/466	6.9/1000	20	not measured		not measured	23.1
CO/H_2O = 3.0 — liquid	204/400	6.9/1000	20	4.54		-0.08	13.3
CO/H_2O = 3.0 — vapor	204/400	6.9/1000	20	6.38		-0.04	10.3

(Table Continued)

THERMAL EXPANSION DATA[a] FOR ALUMINA CASTABLES[b] AS AFFECTED BY VARIOUS EXPOSURE CONDITIONS[c] COMPARED WITH BOEHMITE CONTENT[d][39], Continued

Atmosphere[c]		Treatment Conditions Temperature °C / °F	Pressure MPa/psi	Time days	Average Thermal Expansion Coefficient 25-950 °C $\alpha \times 10^{-6}/°C$	Fractional Thermal Expansion, 500-600 °C $\Delta L/L$, %[j]	Final Fractional Thermal Expansion $(\Delta L/L)$final, %	% Boehmite
54% ALUMINA-40% SILICA INSULATING CASTABLE[j]								
Air (dried)		110/ 230	ambient	1	6.65		-0.12	0
Saturated steam	vapor	285/ 545	6.9/1000	10	6.27		-0.16	3.6
	liquid	285/ 545	6.9/1000	10	5.4		-0.14	6.1
Saturated CGA	vapor	231/ 447	6.9/1000	10	not measured		not measured	6.4
	liquid	231/ 447	6.9/1000	10	2.27		-0.35	6.0
Saturated CGA	vapor	231/ 447	6.9/1000	60	7.03		-0.08	3.1
	liquid	231/ 447	6.9/1000	60	not measured		not measured	3.3
CO/H$_2$O = 1.0	vapor	241/ 466	6.9/1000	20	7.57		-0.23	6.7
	liquid	241/ 466	6.9/1000	20	not measured		not measured	6.8
CO/H$_2$O = 3.0	vapor	204/ 400	6.9/1000	20	5.4		-0.26	5.6
	liquid	204/ 400	6.9/1000	20	6.48		-0.13	8.2
47% ALUMINA-40% SILICA INSULATING CASTABLE[k]								
Air (dried)		110/ 230	ambient	1	4.43		-0.37	0
Saturated CGA	vapor	231/ 447	6.9/1000	10	3.32		-0.7	16.2
	liquid	231/ 447	6.9/1000	10	not measured		not measured	16.5
CO/H$_2$O = 1.0	vapor	241/ 466	6.9/1000	20	5.08		-0.38	14.9
	liquid	241/ 466	6.9/1000	20	5.4		-0.22	17.7
CO/H$_2$O = 3.0	vapor	204/ 400	6.9/1000	20	3.09		-0.42	17.6
	liquid	204/ 400	6.9/1000	20	2.22		-0.26	16.
35% ALUMINA-53% SILICA INSULATING CASTABLE[l]								
Air (dried)		110/ 230	ambient	1	0		-1.15	0
Saturated CGA	vapor	231/ 447	6.9/1000	10	2.27		-0.2	9.5
	liquid	231/ 447	6.9/1000	10	not measured		not measured	10.8

[a]After the indicated exposures, the thermal expansion of 5 cm samples was measured continuously during heating from 20 to 950 °C and during cooling to ambient temperatures. The average thermal expansion coefficient was calculated as $\alpha = \Delta L/L\Delta T$, where $\Delta L/L$ is the fractional length change between 25 and 950 °C and ΔT = 925 °C. The heating rate was 250 °C/hour.

[b]After casting, the specimens were cured 24 hours in air at 100% humidity at ambient temperature, and dried at 230 °F for 24 hours. All specimens were stored at 230 °F until tested.

[c]Samples were exposed in a steam generator. After placing samples, the vessel was heated to the desired steam pressure and the other gases were then added so as to obtain the test concentrations. Specimens were exposed both in the saturated vapor and in the liquid (water) in the bottom of the vessel. A gaseous flow rate was maintained so as to provide one complete change of atmosphere every 24 hours.

[d]Tests showed that boehmite formation occurred in steam-saturated atmospheres and resulted in very large increases in the flexural strength.

[e]93% alumina dense castable, UMR-1 generic preparation (70% tabular alumina + 30% calcium aluminate cement) (T-61 alumina,

(Table Continued)

THERMAL EXPANSION DATA[a] FOR ALUMINA CASTABLES[b] AS AFFECTED BY VARIOUS EXPOSURE CONDITIONS[c] COMPARED WITH BOEHMITE CONTENT[d][39], Continued

Footnotes continued

Alcoa; CA-25 Calcium Aluminate Cement (79% alumina-18% calcia), Alcoa).

[f]CGA = coal gasification atmosphere; composition (in vol %): 18 CO, 12 CO_2, 24 H_2, 5 CH_4, 40 H_2O, and 1 H_2S. The CO/H_2O exposures contained 1% H_2S.

[g]87.4% alumina dense castable, UMR-4 generic preparation (70% tabular alumina + 30% calcium aluminate cement) (T-61 alumina, Alcoa; Refcon, a 58% alumina-33% calcia cement, Universal Atlas).

[h]63.8% alumina, 28.3% silica dense castable, UMR-8 generic preparation (75% calcined kaolin aggregate + 25% calcium aluminate cement) (Mulcoa M-60 and Mulcoa M-47, C-E Minerals; CA-25 Calcium Aluminate Cement, a 79% alumina-18% calcia cement, Alcoa).

[i]58.6% alumina, 29.6% silica dense castable, UMR-5 generic preparation (75% calcined kaolin aggregate + 25% calcium aluminate cement) (Mulcoa M-60 and Mulcoa M-47, C-E Minerals; Refcon, a 58% alumina-33% calcia cement, Universal Atlas).

[j]54% alumina, 40% silica insulating castable, calcium aluminate (79% alumina-18% calcia) bonded (Cer-Lite #75, C-E Refractories; CA-25 Calcium Aluminate Cement, Alcoa).

[k]46.7% alumina, 40.2% silica insulating castable (Litecast 60-25, General Refractories).

[l]34.5% alumina, 52.5% silica insulating castable (VSL-50, A.P. Green).

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EFFECT OF A HIGH-BTU GAS EXPOSURE[a] ON THE BULK DENSITY[b] OF VARIOUS
ALUMINA CASTABLES[c] [95]

Castable Tested[c]	Bulk Density,[b] g/cm^3	
	Air-fired, 1100 °C, 6 h	Exposed, 500 °C, 1000 psi, 58 h
90% Al_2O_3	2.62	2.63
90% Al_2O_3 + 0.1% Fe	2.61	2.63
90% Al_2O_3 + 310 SS[d]	2.67	2.67
90% Al_2O_3 + alkali[e]	2.58	2.61
50% Al_2O_3	2.19	2.13
50% Al_2O_3 + 0.1% Fe	2.21	2.19
50% Al_2O_3 + 310 SS[d]	2.25	2.15
50% Al_2O_3 + alkali[e]	2.18	2.18

[a] Gas composition in mole percent: 38 H_2O, 24 H_2, 18 CO, 12 CO_2, 5 CH_4, 2 NH_3, 1 H_2S.

[b] Not explicitly stated but was probably determined following ASTM C20-73.

[c] No preparative information or other characterization was given in the reports and no commercial brand names were included. Both castables are dense, calcium aluminate bonded. No information was given about the manner of the Fe addition. See sections for reference [9] for earlier reports from the same organization.

[d] 310 stainless steel fibers (4 wt%) were added to the mix before casting.

[e] Alkali-containing specimens were prepared by soaking for 6 hours in a 50% Na_2CO_3-50% K_2CO_3 saturated solution prior to firing.

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EFFECT OF UNSATURATED ATMOSPHERES[a] OF STEAM AND CO/STEAM ON THE
DIMENSIONS OF ALUMINA REFRACTORIES[b][95]

Refractory[b]	Brand Name/ Manufacturer	Dimensional Change,[c] %		
		Air-Fired	Steam	CO/H_2O=3.5
High-Al_2O_3 dense castable	Plicast L-22-84/ Plibrico	-0.22	-0.10	-0.11
High-Al_2O_3 dense castable	Greenkon-33/ A.P. Green	-0.35	-0.20	-0.19
94% Al_2O_3 dense castable	Greencast-94/ A.P. Green	+0.15	+0.01	+0.26
High-Al_2O_3 insulating castable	B&W Kaolite 3300/ Babcock & Wilcox	-0.17	-0.06	-0.09
90+% Al_2O_3 brick	Kricor/Kaiser	-0.13	-0.05	-0.05
High-Al_2O_3 phosphate bonded ramming mix	Kritab/Kaiser	-0.16	-0.23	-0.13
High-Al_2O_3 phosphate bonded ramming mix	Lavalox X-8/Lava Crucible Refractories	-0.28	-0.56	-0.51
93% Al_2O_3 phosphate bonded castable	B&W KaoPhos 93/ Babcock & Wilcox	-0.51	-0.72	-0.64
90% Al_2O_3 phosphate bonded ramming mix	Brikram 90R/ General Refractories	+0.05	+0.40	+0.33
Intermediate-Al_2O_3 dense castable	Resco Cast RS-17E/ Resco Products	-0.18	-0.25	-0.28
Intermediate-Al_2O_3 dense castable	Blast Furnace Back-up Castable/C-E Refractories	-0.04	-0.82	-0.39
Intermediate-Al_2O_3 dense castable	Lo-Erode/Kaiser	-0.46	-0.63	-0.75
Intermediate-Al_2O_3 gunning mix	BF Castable 122-N/ General Refractories	-0.25	-0.20	-0.36
Fireclay dense castable	Plicast HyMOR 3000/ Plibrico	-0.36	-0.95	+0.65
Intermediate-Al_2O_3 insulating castable	Plicast LWI 206I/ Plibrico	-1.42	-0.74	NA[d]
59% Al_2O_3 insulating castable	Kast-O-Lite 30/ A.P. Green	-0.05	+0.12	+0.61
35% Al_2O_3 insulating castable	VSL-50/A.P. Green	-1.47	-1.65	-0.91
29% Al_2O_3 insulating castable	Greencast-29-L/ A.P. Green	-1.47	-1.03	NA[d]

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(Table Continued)

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EFFECT OF UNSATURATED ATMOSPHERES[a] OF STEAM AND CO/STEAM ON THE
DIMENSIONS OF ALUMINA REFRACTORIES[b][95], Continued

Refractory[c]	Brand Name/ Manufacturer	Dimensional Change,[c] %		
		Air-Fired	Steam	$CO/H_2O=3.5$
54% Al_2O_3 insulating castable	Litecast 75-28/ General Refractories	-0.25	-0.32	-0.33
Intermediate-Al_2O_3 gunning mix	72 Gunite Grade/ Sauereisen Cements	NA[d]	NA[d,e]	NA[d,e]

[a]Refractories were exposed to the indicated unsaturated atmospheres for 250 hours at 1000 °C. The firing took place at atmospheric pressure, the other exposures at 105 psig.

[b]Specimens were 2-inch cubes prepared and supplied by the manufacturers. Before testing the specimens were oven-dried at 110 °C for 24 hours.

[c]Negative values denote shrinkage, positive values denote expansion.

[d]NA = not available.

[e]This material fused during steam exposure.

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EFFECT OF UNSATURATED ATMOSPHERES[a] OF STEAM AND CO/STEAM ON THE BULK
DENSITY[b] OF ALUMINA REFRACTORIES[c][95]

Refractory[c]	Brand Name/ Manufacturer	Bulk Density,[b] lb/ft^3		
		Air-Fired	Steam	CO/H$_2$O=3.5
High-Al$_2$O$_3$ dense castable	Plicast L-22-84/ Plibrico	184	185	186
High-Al$_2$O$_3$ dense castable	Greenkon-33/ A.P. Green	187	188	190
94% Al$_2$O$_3$ dense castable	Greencast-94/ A.P. Green	163	167	166
High-Al$_2$O$_3$ insulating castable	B&W Kaolite 3300/ Babcock & Wilcox	64	67	NA[d]
90+% Al$_2$O$_3$ brick	Kricor/Kaiser	190	188	187
High-Al$_2$O$_3$ phosphate bonded ramming mix	Kritab/Kaiser	191	190	192
High-Al$_2$O$_3$ phosphate bonded ramming mix	Lavalox X-8/Lava Crucible Refractories	188	189	189
93% Al$_2$O$_3$ phosphate bonded castable	B&W KaoPhos 93/ Babcock & Wilcox	165	167	169
90% Al$_2$O$_3$ phosphate bonded ramming mix	Brikram 90R/ General Refractories	186	182	183
Intermediate-Al$_2$O$_3$ dense castable	Resco Cast RS-17E/ Resco Products	136	137	138
Intermediate-Al$_2$O$_3$ dense castable	Blast Furnace Back-up Castable/C-E Refractories	132	137	135
Intermediate-Al$_2$O$_3$ dense castable	Lo-Erode/Kaiser	127	128	129
Intermediate-Al$_2$O$_3$ gunning mix	BF Castable 122-N/ General Refractories	124	125	124
Fireclay dense castable	Plicast HyMOR 3000/ Plibrico	141	136	139
Intermediate-Al$_2$O$_3$ insulating castable	Plicast LWI 206I/ Plibrico	54	53	NA[d]
59% Al$_2$O$_3$ insulating castable	Kast-O-Lite 30/ A.P. Green	83	82	NA[d]
35% Al$_2$O$_3$ insulating castable	VSL-50/A.P. Green	43	43	NA[d]
29% Al$_2$O$_3$ insulating castable	Greencast-29-L/ A.P. Green	38	38	NA[d]

- -

(Table Continued)

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EFFECT OF UNSATURATED ATMOSPHERES[a] OF STEAM AND CO/STEAM ON THE BULK
DENSITY[b] OF ALUMINA REFRACTORIES[c][95], Continued

Refractory[c]	Brand Name/ Manufacturer	Bulk Density,[b] lb/ft^3		
		Air-Fired	Steam	CO/H$_2$O=3.5
54% Al$_2$O$_3$ insulating castable	Litecast 75-28/ General Refractories	74	75	75
Intermediate-Al$_2$O$_3$ gunning mix	72 Gunite Grade/ Sauereisen Cements	NA[d]	NA[d,e]	NA[d,e]

[a]Refractories were exposed to the indicated unsaturated atmospheres for 250 hours at 1000 °C. The firing took place at atmospheric pressure, the other exposures at 105 psig.

[b][Probably determined following ASTM C20-73.]

[c]Specimens were 2-inch cubes prepared and supplied by the manufacturers. Before testing the specimens were oven-dried at 110 °C for 24 hours.

[d]NA = not available.

[e]This material fused during steam exposure.

==

EFFECT OF UNSATURATED ATMOSPHERES[a] OF STEAM AND CO/STEAM ON THE APPARENT
POROSITY[b] OF ALUMINA REFRACTORIES[c][95]

Refractory[c]	Brand Name/ Manufacturer	Apparent Porosity,[b] %		
		Air-Fired	Steam	$CO/H_2O=3.5$
High-Al_2O_3 dense castable	Plicast L-22-84/ Plibrico	18	14	18
High-Al_2O_3 dense castable	Greenkon-33/ A.P. Green	16	14	16
94% Al_2O_3 dense castable	Greencast-94/ A.P. Green	30	21	33
High-Al_2O_3 insulating castable	B&W Kaolite 3300/ Babcock & Wilcox	65	31	NA[d]
90+% Al_2O_3 brick	Kricor/Kaiser	14	11	21
High-Al_2O_3 phosphate bonded ramming mix	Kritab/Kaiser	16	13	17
High-Al_2O_3 phosphate bonded ramming mix	Lavalox X-8/Lava Crucible Refractories	17	13	18
93% Al_2O_3 phosphate bonded castable	B&W KaoPhos 93/ Babcock & Wilcox	24	17	25
90% Al_2O_3 phosphate bonded ramming mix	Brikram 90R/ General Refractories	15	13	17
Intermediate-Al_2O_3 dense castable	Resco Cast RS-17E/ Resco Products	25	9	21
Intermediate-Al_2O_3 dense castable	Blast Furnace Back-up Castable/C-E Refractories	28	14	25
Intermediate-Al_2O_3 dense castable	Lo-Erode/Kaiser	29	24	28
Intermediate-Al_2O_3 gunning mix	BF Castable 122-N/ General Refractories	32	25	29
Fireclay dense castable	Plicast HyMOR 3000/ Plibrico	21	18	22
Intermediate-Al_2O_3 insulating castable	Plicast LWI 206I/ Plibrico	71	48	NA[d]
59% Al_2O_3 insulating castable	Kast-O-Lite 30/ A.P. Green	54	36	NA[d]
35% Al_2O_3 insulating castable	VSL-50/A.P. Green	76	52	NA[d]
29% Al_2O_3 insulating castable	Greencast-29-L/ A.P. Green	80	58	NA[d]

- -

(Table Continued)

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EFFECT OF UNSATURATED ATMOSPHERES[a] OF STEAM AND CO/STEAM ON THE APPARENT
POROSITY[b] OF ALUMINA REFRACTORIES[c][95], Continued

Refractory[c]	Brand Name/ Manufacturer	Apparent Porosity,[b] %		
		Air-Fired	Steam	CO/H_2O=3.5
54% Al_2O_3 insulating castable	Litecast 75-28/ General Refractories	56	39	58
Intermediate-Al_2O_3 gunning mix	72 Gunite Grade/ Sauereisen Cements	NA[d]	NA[d,e]	NA[d,e]

[a]Refractories were exposed to the indicated unsaturated atmospheres for 250 hours at 1000 °C. The firing took place at atmospheric pressure, the other exposures at 105 psig.

[b][Probably determined following ASTM C20-73.]

[c]Specimens were 2-inch cubes prepared and supplied by the manufacturers. Before testing the specimens were oven-dried at 110 °C for 24 hours.

[d]NA = not available.

[e]This material fused during steam exposure.

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EFFECT OF TIME OF EXPOSURE TO SIMULATED COAL GASIFIER ATMOSPHERE[a] ON THE
BULK DENSITY[b] OF ALUMINA CASTABLES[c] WITH VARIOUS CALCIUM ALUMINATE
CEMENTS[d][97]

(Data Continued)

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EFFECT OF TIME OF EXPOSURE TO SIMULATED COAL GASIFIER ATMOSPHERE[a] ON THE
BULK DENSITY[b] OF ALUMINA CASTABLES[c] WITH VARIOUS CALCIUM ALUMINATE
CEMENTS[d][97], Continued

BULK DENSITY VS EXPOSURE TIME

90+% Alumina castable
with 58% alumina cement

50+% Alumina castable
with 58% alumina cement

LOG TIME (HRS)

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[a]Specimen exposure to a coal gasifier atmosphere took place in the DoE IITRI
facility for periods of 10, 100, and 1000 hours. [Conditions were probably
982 °C (1800 °F), 1000 psi, gas composition (vol %) 24 H_2, 18 CO, 12 CO_2,
5 CH_4, 1 NH_3, 0–1.0 H_2S, balance H_2O.]

[b]Dashed line represents an extrapolation back to the value before exposure
(cured specimen value).

[c]Castables are generic materials, cast as brick in accordance with ASTM
C862–77. The 90+% alumina dense castable consisted of 70% tabular alumina
(–6 mesh + fines) (T–61, Alcoa), 5% calcined alumina (–325 mesh) (A–2, Alcoa),
and 25% calcium aluminate cement. The 50+% alumina dense castable consisted
of 75% calcined kaolin (27.5% –6 + 10 mesh, 22.5% –10 + 20 mesh, 20% –20
mesh, 5% ball milled fines 5% of which was <325 mesh) (Mulcoa 47, C–E Min-
erals) and 25% calcium aluminate cement.

[d]The 80% alumina cement: 79.7% Al_2O_3, 18.4% CaO (CA–25C, Alcoa). The
70% alumina cement: 70.6% Al_2O_3, 27.5% CaO (Secar 71, Lone Star Lafarge).
The 58% alumina cement: 58.0% Al_2O_3, 33.5% CaO, 5.6% SiO_2, 1.5% Fe_2O_3
(Refcon, Universal Atlas Cement).

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EFFECT OF TIME OF EXPOSURE TO SIMULATED COAL GASIFIER ATMOSPHERE[a] ON THE
APPARENT POROSITY[b] OF ALUMINA CASTABLES[c] WITH VARIOUS CALCIUM
ALUMINATE CEMENTS[d][97]

APPARENT POROSITY VS TIME EXPOSURE

50+% Alumina caatable
with 80% alumina cement

90+% Alumina caatable
with 80% alumina cement

90+% Alumina caatable
with 70% alumina cement

50+% Alumina caatable
with 70% alumina cement

APPARENT POROSITY VS EXPOSURE TIME

(Data Continued)

===

EFFECT OF TIME OF EXPOSURE TO SIMULATED COAL GASIFIER ATMOSPHERE[a] ON THE
APPARENT POROSITY[b] OF ALUMINA CASTABLES[c] WITH VARIOUS CALCIUM
ALUMINATE CEMENTS[d][97], Continued

[a]Specimen exposure to a coal gasifier atmosphere took place in the DoE IITRI facility for periods of 10, 100, and 1000 hours. [Conditions were probably 982 °C (1800 °F), 1000 psi, gas composition (vol %) 24 H_2, 18 CO, 12 CO_2, 5 CH_4, 1 NH_3, 0-1.0 H_2S, balance H_2O.]

[b]Dashed line represents an extrapolation back to the value before exposure (cured specimen value).

[c]Castables are generic materials, cast as brick in accordance with ASTM C862-77. The 90+% alumina dense castable consisted of 70% tabular alumina (-6 mesh + fines) (T-61, Alcoa), 5% calcined alumina (-325 mesh) (A-2, Alcoa), and 25% calcium aluminate cement. The 50+% alumina dense castable consisted of 75% calcined kaolin (27.5% -6 + 10 mesh, 22.5% -10 + 20 mesh, 20% -20 mesh, 5% ball milled fines 5% of which was <325 mesh) (Mulcoa 47, C-E Minerals) and 25% calcium aluminate cement.

[d]The 80% alumina cement: 79.7% Al_2O_3, 18.4% CaO (CA-25C, Alcoa). The 70% alumina cement: 70.6% Al_2O_3, 27.5% CaO (Secar 71, Lone Star Lafarge). The 58% alumina cement: 58.0% Al_2O_3, 33.5% CaO, 5.6% SiO_2, 1.5% Fe_2O_3 (Refcon, Universal Atlas Cement).

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DENSITY[a] AND POROSITY[a] FOR A HIGH-ALUMINA CASTABLE[b] AND CEMENT[c] BEFORE AND
AFTER CREEP TESTING[d][97]

Condition	Bulk Density g/cm^3	Apparent Density g/cm^3	Apparent Porosity %
- - - - - - - 90+% Alumina dense castable[b] - - - - - - - - - - - - - - - - -			
As-cured	2.62±0.03	3.34±0.04	22.82±0.53
Temperature treatment[e]	2.47±0.13	3.42±0.27	27.68±2.19
Crept[d]	2.45±0.08	3.57±0.18	31.41±2.92
- - - - - - - High-alumina calcium aluminate cement[c] - - - - - - - - - - - -			
As-cured	1.85±0.02	2.80±0.09	33.83±1.75
Temperature treatment	1.67±0.06	2.93±0.06	42.95±1.45
Crept	1.77±0.09	2.92±0.04	39.33±2.36

[a]Density and porosity were determined by the method of fluid displacement
using kerosene.

[b]A generic castable, cast as brick in accordance with ASTM C862-77. Test
specimens were core drilled from the brick. Composition: 70% tabular
alumina (-6 mesh + fines) (T-61, Alcoa), 5% calcined alumina (-325 mesh)
(A-2, Alcoa), and 25% calcium aluminate cement (CA-25C, Alcoa), with 9.0-
9.5% water.

[c]80% alumina-18% calcia cement (CA-25C, Alcoa) cast to specimen size, with
water/cement ratio = 0.3.

[d]Creep tests were performed in air in creep furnaces under compressive
loading. Creep strain was monitored with a linear voltage displacement
transducer until steady state creep was obtained. Experiments took place
over the range 538-1204 °C (1000-2200 °F) and 6.9-17.2 MPa (1000-2500 psi).
Specimens were heated to the first temperature, the load was applied, and
deformation was recorded. The load was then released, the specimen heated
to the next test temperature, the same load reapplied, and deformation
recorded, etc. After creep measurements were made at several temperatures,
the specimens were cooled to room temperature and the density and porosity
measured.

[e]Specimens were subjected to the same heat schedules as outlined in footnote
d in the creep testing, but loads were not applied to the specimens.

DENSITY AND POROSITY DATA[a] FOR CHROMIA-CONTAINING REFRACTORIES[97]

Specimen Designation	Composition,[b] wt %	Bulk[c] Volume cm^3	Open Pore Volume cm^3	Apparent Volume[d] cm^3	Bulk Density g/cm^3	Apparent Density g/cm^3	Apparent Porosity %	Absorption %
CR 10-1	90 Al$_2$O$_3$-10 Cr$_2$O$_3$	25.21	2.37	22.84	3.299	3.641	9.40	2.28
CR 10-2	90 Al$_2$O$_3$-10 Cr$_2$O$_3$	37.70	5.99	31.71	3.262	2.878	15.89	3.90
CR 10-3[e]	90 Al$_2$O$_3$-10 Cr$_2$O$_3$	37.70	7.24	30.46	3.132	3.876	19.20	4.91
CR 10-4	90 Al$_2$O$_3$-10 Cr$_2$O$_3$	25.46	2.29	23.16	3.24	3.56	9.00	2.20
CR 10-5	90 Al$_2$O$_3$-10 Cr$_2$O$_3$	25.88	2.52	23.35	3.21	3.56	9.75	2.40
CR 10-6	90 Al$_2$O$_3$-10 Cr$_2$O$_3$	25.04	1.74	23.30	3.20	3.44	6.96	1.72
CR 20-7	56.5 MgO-20 Cr$_2$O$_3$-8 Al$_2$O$_3$-10.5 FeO-2.5 SiO$_2$	33.70	2.37	31.33	3.592	3.864	7.03	1.56
CR 20-8	59 MgO-20 Cr$_2$O$_3$-6.5 Al$_2$O$_3$-9.5 Fe$_2$O$_3$-3 SiO$_2$	37.82	5.49	32.33	3.359	3.930	14.52	3.46
CR 20-9	56 MgO-20 Cr$_2$O$_3$-8 Al$_2$O$_3$-11 FeO-2.5 SiO$_2$	18.46	2.24	16.22	3.398	3.869	12.16	2.86
CR 73-10	73 Cr$_2$O$_3$-26 MgO	43.82	7.36	36.45	3.614	4.345	16.80	3.72
CR 76-11	76 Cr$_2$O$_3$-21 MgO-0.7 SiO$_2$-0.7 FeO	24.71	1.87	22.84	3.786	4.096	7.57	1.60
CR 78-12[f]	78 Cr$_2$O$_3$-18 MgO	37.32	5.99	31.33	3.729	4.442	16.05	3.44
CR 78-13	not given	31.42	3.98	27.43	3.96	4.53	12.67	2.62
CR 80-14	not given	24.96	0.87	24.09	4.413	4.573	3.50	0.63
CR 80-15	80 Cr$_2$O$_3$-8 Al$_2$O$_3$-4 Fe$_2$O$_3$-2.5 TiO$_2$-0.5 MgO	30.58	1.62	28.96	4.505	4.757	5.30	0.94
CR 80-16	80 Cr$_2$O$_3$-8 MgO-6 Fe$_2$O$_3$	25.35	1.41	23.93	4.29	4.54	5.58	1.03
CR 80-17[g]	not given	35.56	4.43	20.45	3.87	4.42	12.45	2.63
CR 93-18	93 Cr$_2$O$_3$-4 TiO$_2$	38.45	5.24	33.20	4.337	5.022	13.63	2.51
CR 93-19	93 Cr$_2$O$_3$-3.8 TiO$_2$-0.5 FeO	25.57	2.80	22.77	4.27	4.80	10.95	2.03

[a]Density and porosity measurements were by the fluid displacement method using kerosene as the fluid.

(Data Continued)

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DENSITY AND POROSITY DATA[a] FOR CHROMIA-CONTAINING REFRACTORIES[97], Continued

Footnotes continued

[b]Compositions as given. No brand names or other characterizations were given except for a few descriptions which are included under the footnotes for several specimen designations. Where no composition is given, the specimen designation does indicate the chromia content, i.e. CR 80. Different specimen designations may refer to materials of similar composition from different sources or to different samples of the same material.

[c]Exterior volume.

[d]Volume of the impervious portions.

[e]A coarse sintered tabular alumina enriched with chromia in the bond phase.

[f]A coarse rebonded magnesia-chromia spinel supplied by Argonne National Laboratory.

[g]A fine-grained high-purity spinel.

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THERMAL CONDUCTIVITIES OF AN INSULATING CASTABLE[a] AND COAL GASIFICATION
PROCESS GASES[99]
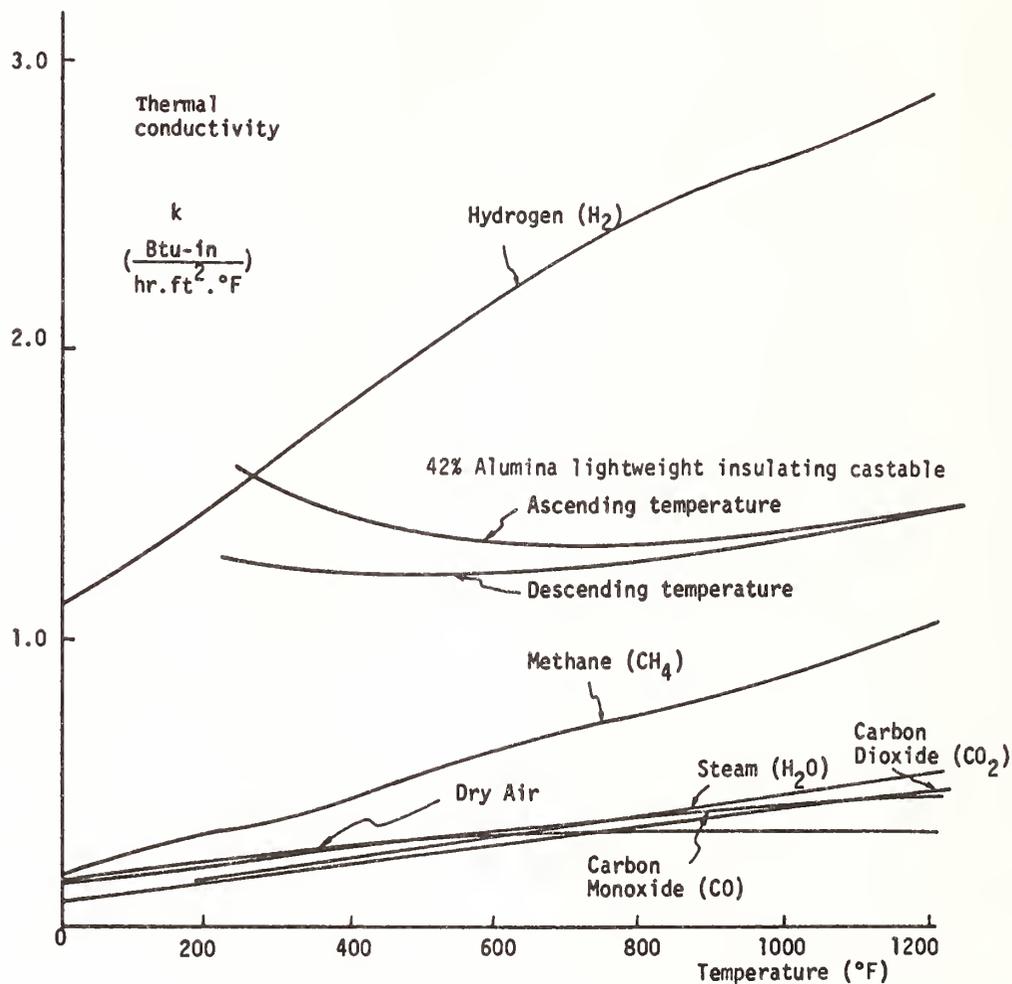
[a]Commercial refractory (B&W Kaolite 2300-LI, Babcock and Wilcox).

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THERMAL CONDUCTIVITIES OF SEVERAL REFRACTORIES[101]

Thermal Conductivity, $Wm^{-1}K^{-1}$ (Btu in. $h^{-1}ft^{-2}°F^{-1}$)

Temperature K (°F)	94% Alumina Dense Castable[a]			93% Alumina Lightweight Castable[b]			Lightweight Castable[c]
	In Air	In H₂, 20%[d]	In H₂, 35%[e]	In Air	In H₂, 55%[f]	In H₂, 70%[g]	In Air
366 (200)							0.12(0.80)
422 (300)							0.12(0.80)
473 (392)	1.92(13.35)	2.05(14.24)	2.08(14.42)	0.95(6.60)	1.13(7.85)	1.15(7.95)	0.12(0.80)
573 (572)	1.87(12.95)	2.02(13.99)	2.05(14.20)	0.88(6.10)	1.09(7.55)	1.11(7.67)	0.13(0.90)
752 (673)	1.80(12.50)	1.97(13.68)	2.00(13.91)	0.82(5.70)	1.06(7.35)	1.08(7.49)	0.14(0.95)
773 (932)	1.74(12.05)	1.92(13.33)	1.96(13.59)	0.79(5.45)	1.04(7.24)	1.07(7.39)	
873 (1112)	1.68(11.65)	1.88(13.06)	1.93(13.36)	0.76(5.30)	1.05(7.28)	1.07(7.45)	
973 (1292)	1.63(11.30)	1.85(12.82)	1.89(13.13)	0.74(5.15)	1.05(7.28)	1.08(7.46)	
1073 (1472)	1.58(10.95)	1.81(12.57)	1.86(12.90)	0.74(5.15)	1.07(7.42)	1.10(7.61)	
1173 (1652)	1.55(10.75)	1.80(12.48)	1.85(12.83)	0.74(5.10)	1.08(7.51)	1.11(7.72)	
1273 (1832)	1.53(10.60)	1.78(12.38)	1.84(12.75)	0.73(5.05)	1.09(7.55)	1.12(7.76)	

[a] 93.7% Al_2O_3, 0.5% Fe_2O_3, SiO_2 (Castolast G, Harbison-Walker Refractories).

[b] 92.6% Al_2O_3, 0.5% Fe_2O_3, SiO_2 (H-W Lightweight Castable 33, Harbison-Walker Refractories).

[c] [Composition given as 42.9% SiO_2, 8.7% SiO_2, 2.4% Fe_2O_3, 37.2% CaO; identified as H-W Lightweight Castable 10, Harbison-Walker Refractories.]

[d] Assumed porosity 20%, all filled with H_2.

[e] Assumed porosity 35%, all filled with H_2.

[f] Assumed porosity 55%, all filled with H_2.

[g] Assumed porosity 70%, all filled with H_2.

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THERMAL CONDUCTIVITIES[a] OF CASTABLE REFRACTORIES[101]

94% Alumina Dense Castable

93% Alumina Lightweight Castable

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(Data Continued)

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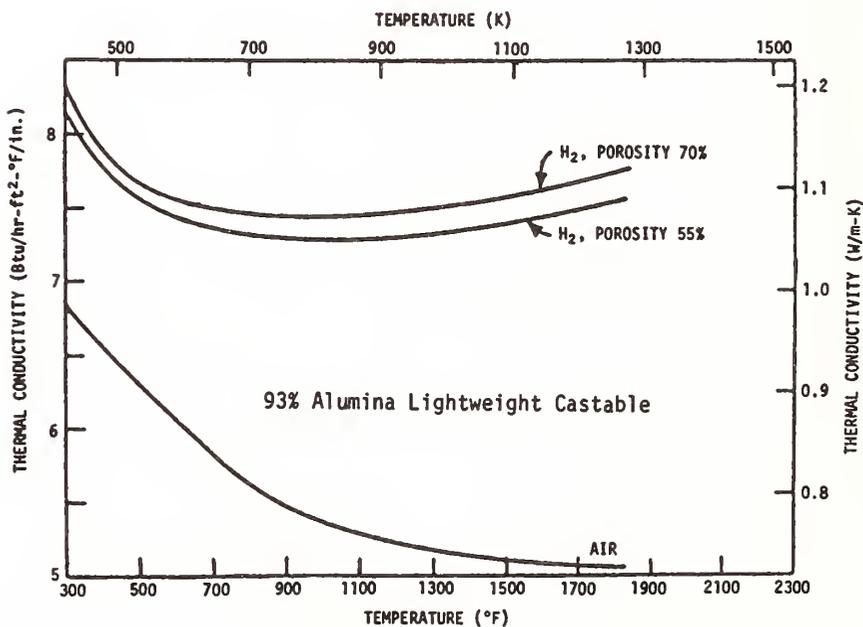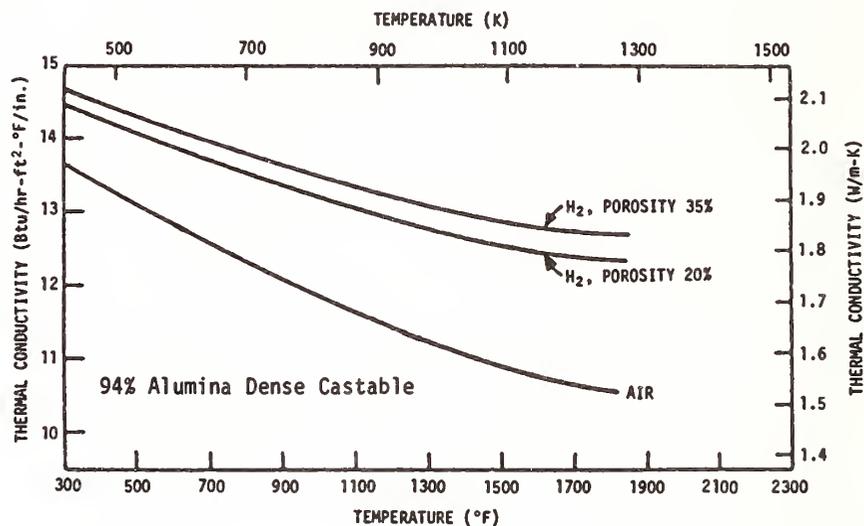
THERMAL CONDUCTIVITIES[a] OF CASTABLE REFRACTORIES[101], Continued

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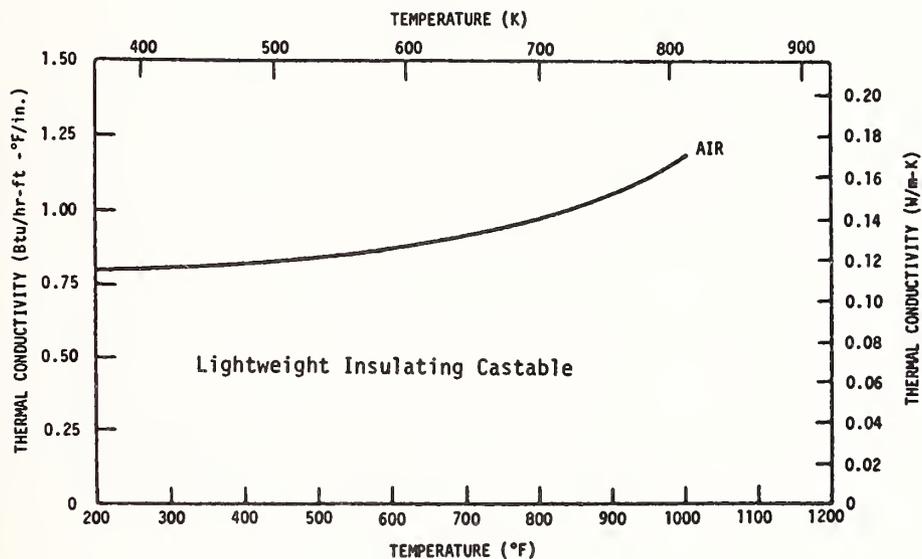
[a]See Section B.4.2.43 for the data plotted here as well as the identification
of the refractories.

GAS PERMEABILITY[a] OF CERAMIC TUBES SUBJECTED TO FUEL COMBUSTION ENVIRONMENTS[b][106]

Material	Test Exposure 1[b]	Permeability,[a] m^3/s (gas and ΔP given)		
		He, 7 kPa	He, 520 kPa	Air, 7 kPa
SiC, sintered-α (Carborundum)	pretest	$<1.0 \times 10^{-15}$	$<1.0 \times 10^{-15}$	not given
	posttest	9.7×10^{-14}	1.2×10^{-12}	not given
SiC, siliconized (KT-SiC, Carborundum)	pretest	$<1.0 \times 10^{-15}$	$<1.0 \times 10^{-15}$	not given
	posttest	6.5×10^{-15}	1.0×10^{-14}	not given
SiC, siliconized (NC 430, Norton)	pretest	$<1.0 \times 10^{-15}$	$<1.0 \times 10^{-15}$	not given
	posttest	2.7×10^{-14}	1.2×10^{-13}	not given
SiC, siliconized (Refel SiC, Pure Carbon Co.)	pretest	$<1.0 \times 10^{-15}$	$<1.0 \times 10^{-15}$	not given
	posttest	$<1.0 \times 10^{-15}$	8.1×10^{-15}	not given
Al_2O_3, high-purity (AD 998, Coors Porcelain)	pretest	$<1.0 \times 10^{-15}$	$<1.0 \times 10^{-15}$	not given
	posttest	2.3×10^{-11}	3.1×10^{-10}	not given
CVD SiC on SiC[c] (Deposits & Composites, Inc.)	pretest	$<1.1 \times 10^{-15}$	$<1.0 \times 10^{-15}$	not given
	posttest	1.1×10^{-7}	$>1.0 \times 10^{-9}$	not given
CVD SiC (Deposits & Composites, Inc.)	pretest	$<1.0 \times 10^{-15}$	$<1.0 \times 10^{-15}$	not given
	posttest	2.2×10^{-11}	$>1.0 \times 10^{-9}$	not given
SiC, clay-bonded (Carbofrax A, Carborundum)	pretest	4.6×10^{-5}	not given	1.3×10^{-5}
	posttest	1.8×10^{-5}	not given	4.0×10^{-6}
SiC, clay-bonded (Carbofrax M, Carborundum)	pretest	not given	not given	7.1×10^{-5}
	posttest	4.9×10^{-5}	not given	3.6×10^{-5}
SiC, Si_3N_4-bonded (Refrax 20, Carborundum)	pretest	not given	not given	1.2×10^{-6}
	posttest	4.9×10^{-5}	not given	0.8×10^{-6}
Aluminosilicate, high-purity (Mullite, Coors Porcelain)	pretest	not given d	$<1.0 \times 10^{-15}$ d	not given d
Magnesium aluminosilicate (MAS 8200, GTE Sylvania)	pretest	not given d	not given d	3.6×10^{-6} d
Magnesium aluminosilicate (MAS 8400, GTE Sylvania)	pretest	not given	not given	1.8×10^{-6}

(Table Continued)

GAS PERMEABILITY[a] OF CERAMIC TUBES SUBJECTED TO FUEL COMBUSTION ENVIRONMENTS[b][106], Continued

Material		Permeability,[a] m^3/s (gas and ΔP given)		
		He, 7 kPa	He, 520 kPa	Air, 7 kPa
Zirconia plus mullite (Zirmul, GTE Sylvania)	pretest	not given	not given	3.0×10^{-8}
	posttest	d	d	d

TEST EXPOSURE 2[b]

Material			He, 102 kPa	He, 623 kPa
SiC, siliconized (KT-SiC, Carborundum)	tube 1	pretest	e	3.0×10^{-15}
		posttest	1.6×10^{-15}	3.5×10^{-13}
	tube 2	pretest	1.4×10^{-14}	1.6×10^{-14}
		posttest	3.3×10^{-14}	1.5×10^{-12}
SiC, sintered-α (Carborundum)	tube 1	pretest	2.4×10^{-14}	2.8×10^{-14}
		posttest	3.7×10^{-15}	2.9×10^{-14}
	tube 2	pretest	e	2.7×10^{-14} f
		posttest	9.3×10^{-16}	7.5×10^{-14}
CVD SiC (Deposits & Composites)	tube 1	pretest	1.8×10^{-15}	3.6×10^{-15}
		posttest	5.7×10^{-16}	5.7×10^{-14}
	tube 2	pretest	e	5.0×10^{-15}
		posttest	e	3.0×10^{-14}
Al$_2$O$_3$, high-purity (AD 998, Coors Porcelain)	tube 1	pretest	8.0×10^{-15}	8.0×10^{-15}
		posttest	d	d
	tube 2	pretest	g	g
		posttest	d	d
SiAlON (GE 128, General Electric)	tube 1	pretest	5.0×10^{-16}	2.8×10^{-15}
		posttest	d	d
	tube 2	pretest	g	g
		posttest	d	d

TEST EXPOSURE 3[b]

Material			He, 101 kPa	He, 247 kPa	He, 446 kPa	He, 618 kPa
Al$_2$O$_3$, high-purity (AD 998, Coors Porcelain)	tube 1	pretest	e	e	4.6×10^{-15}	1.7×10^{-14}
		posttest	e	e	e	not given
	tube 2	pretest	g	g	g	g
		posttest	g	g	g	not given

(Table Continued)

GAS PERMEABILITY[a] OF CERAMIC TUBES SUBJECTED TO FUEL COMBUSTION ENVIRONMENTS[b][106], Continued

Material			Permeability,[a] m^3/s (gas and ΔP given)			
			He, 101 kPa	He, 247 kPa	He, 446 kPa	He, 618 kPa
SiC, sintered-α (Carborundum)	tube 1	pretest	e	5.6×10^{-16}	7.0×10^{-16}	4.5×10^{-15}
		posttest	e	2.6×10^{-15}	2.0×10^{-14}	not given
	tube 2	pretest	e	e	9.6×10^{-16}	g
		posttest	g	g	g	not given
SiC, siliconized (KT-SiC, Carborundum)	tube 1	pretest	e	e	e	4.0×10^{-15}
		posttest	e	e	$<2.9 \times 10^{-14}$	not given
	tube 2	pretest	e	3.3×10^{-15}	4.6×10^{-15}	4.8×10^{-15}
		posttest	5.2×10^{-15}	2.5×10^{-14}	1.2×10^{-13}	not given
SiC, siliconized (NC 430, Norton), with joint[h]		pretest	e	e	4.3×10^{-15}	8.5×10^{-15}
		posttest	3.3×10^{-14}	4.6×10^{-14}	7.1×10^{-14}	not given
SiC, siliconized (SC-2, Coors Porcelain)	tube 1	pretest	e	e	6.0×10^{-15}	g
		posttest	4.9×10^{-14}	1.1×10^{-13}	2.4×10^{-13}	not given
	tube 2	pretest	e	e	e	e
		posttest	d	d	d	not given
CVR SiC[i] (experimental material, Syntax)		pretest	e	e	e	e
		posttest	e	e	e	not given
SiAlON (GE 128, General Electric)		pretest	6.6×10^{-16}	6.6×10^{-16}	3.2×10^{-15}	g
		posttest	d	d	d	not given
SiAlON (GE 129, General Electric)		pretest	g	g	g	g
		posttest	d	d	d	d
SiAlON (GE 130, General Electric)		pretest	e	e	e	g
		posttest	5.6×10^{-14}	g	g	not given

TEST EXPOSURE 4[b]

Material			He, 101 kPa	He, 247 kPa	He, 446 kPa
SiC, sintered-α (Carborundum)	closed[j]	pretest	tube leaked through sealed end	--	- - -
		posttest	--	--	--
	open[j]	pretest	e	e	e
		posttest	e	e	e

(Table Continued)

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GAS PERMEABILITY[a] OF CERAMIC TUBES SUBJECTED TO FUEL COMBUSTION ENVIRONMENTS[b][106], Continued

Material			Permeability,[a] m³/s (gas and ΔP given)		
			He, 101 kPa	He, 247 kPa	He, 446 kPa
SiC, Si₃N₄-bonded (CS SiC, Norton)	open[j]	pretest	g	g	g
		posttest	d	d	d
Al₂O₃, high-purity (AD 998, Coors Porcelain)	closed[j]	pretest	e	e	e
		posttest	d	d	d

Siliconized SiC (KT-SiC, Carborundum; SC-2, Coors Porcelain; NC 430, Norton) materials all had as-received leak rates well below the limit of detection of the equipment; after exposure the leak rate was still below the limit of detection. These materials were all tested with both open and closed end tubes.

[a] Helium and air permeabilities measured. Measurements were made at ambient temperature. The pressure differentials between the inside and the outside of the tubes are given in the table. The detection limit of the apparatus used was 5 x 10^{-16} m³/s.

[b] Ceramic tubes were exposed in the flue gas of a combustor during 4 tests with 4 different fuels. The parameters for each test are given below. For some of the tests more than one tube of the material was exposed. In Test 4 some tubes were closed on one end and were pressurized to 0.68 MPa (100 psig).
TEST EXPOSURE 1--fired with No. 6 fuel oil with a high vanadium content (~83 weight ppm); tube wall thicknesses varied from 1.6 to 9.5 mm for the different materials; 7 heating-holding-cooling cycles occurred during the test.
TEST EXPOSURE 2--fired with Venezuelan crude-derived No. 6 fuel oil containing 10 wt% powdered bituminous coal, with over 98.8% of particle size <44 μm; ash content was acidic with base to acid ratio = 0.29; slag was viscous; 3 thermal cycles occurred during the test (1200→800→1200 °C).
TEST EXPOSURE 3--fired with Venezuelan crude-derived No. 6 fuel oil containing 20 wt% powdered coal from Hanna Seam, Wyoming, with 99.2% of particle size <44 μm; ash content was basic with base to acid ratio = 1.14; slag was fluid; tube wall thicknesses varied from 5.5 to 8.7 mm for the different materials. The values given in the table below for this exposure are given for the 222nd hour of the exposure.
TEST EXPOSURE 4--fired with Venezuelan crude-derived No. 6 fuel oil containing 20 wt% powdered coal with refractory ash (fusion temperature >1482 °C; ash was very acidic with base to acid ratio = 0.09; slag formed a sticky particulate buildup; see figures below for temperature excursions during test. Values given in the table for this exposure are for the 506th hour of the exposure.

All temperatures in the following table are in °C. The duration given for the test is the total time of the exposure.

(Table Continued)

GAS PERMEABILITY[a] OF CERAMIC TUBES SUBJECTED TO FUEL COMBUSTION ENVIRONMENTS[b][106], Continued

Footnotes continued

Test	Time h	Tube Temperature°C	Flue Gas Temperature°C Inlet	Flue Gas Temperature°C Outlet	Air Temperature°C Inlet	Air Temperature°C Outlet	Air Velocity m/s	Heat Transfer Rate, kW	Heat Flux kW/m^2
1	500	1100-1250	1410	1385	ambient	340	8.1	23.7	54
2	500	1200-1280	1390	1349	ambient	427	7.6	27.6	13.6
3	240	1220	1418	1378	47.0	353	10.9	23.7	49.4
4	350	see below	1302	1198	36	162	14.4	16.8	166

COMBUSTOR EXPERIENCED EIGHT MAJOR TEMPERATURE EXCURSIONS DURING TEST 4

TUBE TEMPERATURES EXPERIENCED MAJOR FLUCTUATIONS DURING TEST 4

c Chemically vapor deposited SiC on reaction-sintered SiC substrate.

d Tube cracked too much during exposure to be tested for permeability.

e The permeability was below the detection limit of the equipment, 5×10^{-16} m^3/s.

f [Value in the original reports was 10^{-16} but this then places the permeability below the limits of detection. It was assumed to be a misprint for 10^{-14}.]

g The leak rate was too high to be measured by the leak detector.

h This tube contained a proprietary butt-joint at tube midlength.

i Chemically vapor reacted SiC; silicon reacted with graphite mandrel at 1800 °C and a CVD SiC overlay applied.

j "Closed" refers to tubes which were sealed at one end and pressurized to 0.68 MPa with air during the combustion exposures. "Open" refers to tubes which were open at both ends and were subjected to the air passing through as shown above in the table in footnote b.

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EFFECT OF A FUEL COMBUSTION ENVIRONMENT[a] ON THE THERMAL EXPANSION[b] OF
CERAMIC TUBE MATERIALS[106]

Material	Linear Thermal Expansion Coefficient, 10^{-6}/°C		Change[c] %
	As-Received	Exposed	
SiC, sintered-α (Carborundum)	5.2	6.1	+ 17
SiC, siliconized (KT-SiC, Carborundum)	5.1[d]	5.2[d]	+ 2
SiC, siliconized (NC 430, Norton)	6.1[d]	5.9[d]	− 3
SiC, siliconized (Refel SiC, Pure Carbon Co.)	5.6	6.0	+ 7
CVD SiC[e] (Deposits & Composites Inc.)	5.6	5.6	0
CVD SiC on SiC[f] (Deposits & Composites)	5.7	6.7	+ 18
SiC, clay-bonded (Carbofrax A, Carborundum)	6.5	6.5	0
SiC, clay-bonded (Carbofrax M, Carborundum)	5.7	6.3	+ 11
SiC, Si_3N_4-bonded (Refrax 20, Carborundum)	5.7	5.6	− 2
Magnesium aluminosilicate (MAS 8200, GTE Sylvania)	4.0	7.1	+ 78
Magnesium aluminosilicate (MAS 8400, GTE Sylvania)	4.7	4.0	− 15
Zirconia plus mullite (Zirmul, GTE Sylvania)	7.3	8.6	+ 18
Al_2O_3, high-purity (AD 998, Coors Porcelain)	8.2	8.5	+ 4
Aluminosilicate, high-purity (Mullite, Coors Porcelain)	7.9	7.4	− 6

[a] Ceramic tubes were exposed to the flue gas of a combustor using a No. 6 fuel oil with a high vanadium content. For exposure details, see footnote b, Section B.4.2.45, for details of Test Exposure 1.

[b] Test specimens were oriented along the axial directions of the tubes. Specimens from the exposed tubes were taken from the upstream side of the tubes. Thermal expansion measurements were made from 25 °C to 1000 °C except for the one material noted.

[c] Change = (exposed − as-received)/as-received.

[d] The temperature range was 25–1100 °C.

[e] CVD = chemically vapor deposited.

[f] Chemically vapor deposited SiC on reaction-sintered SiC substrate.

THERMAL EXPANSION DATA FOR CERAMIC TUBE MATERIALS EXPOSED TO AN ACIDIC COAL
SLAG[a] IN A COAL-OIL FUEL COMBUSTOR[106]
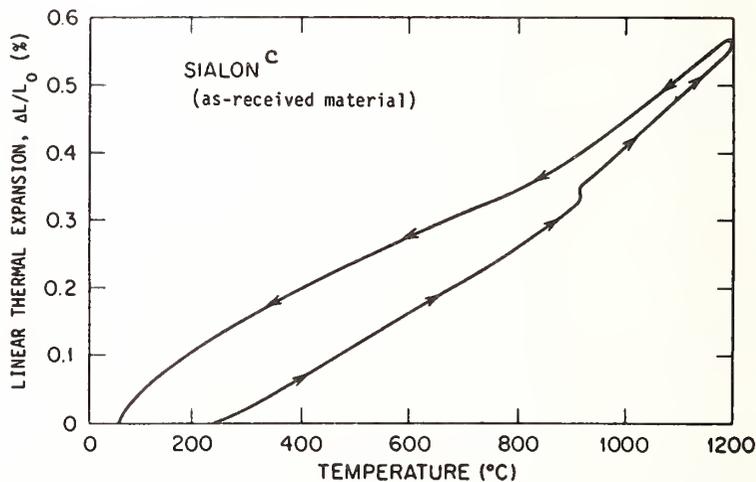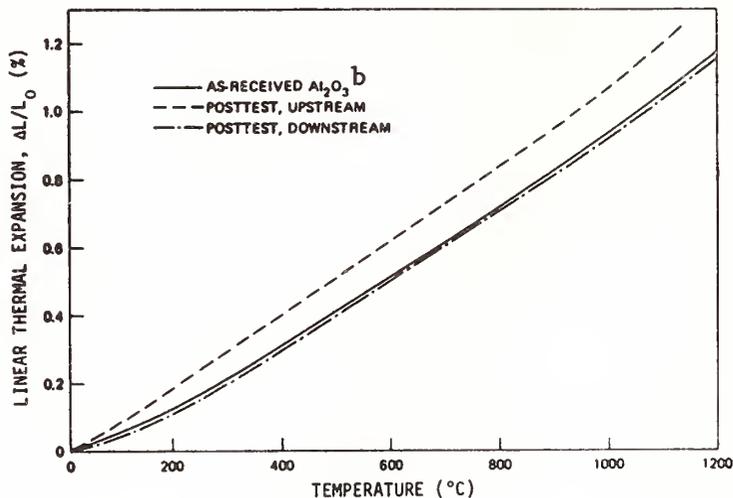
[a]See Section B.4.2.45, footnote b, for the conditions of Combustion Test Ex-
posure 2. The base to acid ratio of the slag was 0.29. See Section B.4.2.46
for thermal expansion data for materials exposed to fuel oil combustion prod-
ucts only. See Section B.3.2.150 for strength testing of materials after
combustion exposures.

[b]High-purity alumina (AD 998, Coors Porcelain). Each curve represents the
average percentage elongation obtained from the heating and cooling curves.

[c]GE 128, General Electric.

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CHANGE IN WALL THICKNESS[a] OF CERAMIC TUBES EXPOSED TO A BASIC SLAG[b] IN
A COAL-OIL FUEL COMBUSTOR[106]

| | Tube Wall Thickness | | | | |
| | As-Received | Downstream | | Upstream | |
Material	mm	mm	% Change	mm	% Change
Al$_2$O$_3$, high-purity (AD 998, Coors Porcelain)	2.770	2.920	+ 5	2.357	− 15
SiC, sintered-α[c] (Carborundum)	3.340	3.137	− 6	2.822	− 16
SiC, siliconized (NC 430, Norton)	3.925	3.566	− 9	2.634	− 33
SiC, siliconized (KT-SiC, Carborundum)	3.285	2.946	− 10	2.431	− 26
SiC, siliconized[d] (SC-2, Coors Porcelain)	3.125	2.885	− 7	2.131	− 32
	2.955	2.647	− 10	2.057	− 30
CVD SiC[e]	3.120	3.564	+ 14	2.695	− 13
SiAlON (GE-128, General Electric)	3.580	1.895	− 31	0.594	− 78
SiAlON (GE-129, General Electric)	4.250	3.045	− 28	1.877	− 56
SiAlON (GE-130, General Electric)	4.850	3.729	− 23	3.940	− 19

[a]Change in thickness = (exposed − as-received)/as-received thickness.

[b]See Section B.4.2.45, footnote b, for the conditions of Combustion Test Exposure
3. The base to acid ratio of the slag was 1.14. See Section B.3.2.150 for
strength testing of materials after combustion exposures.

[c]The figure shows the upstream and downstream thickness profiles for the α-SiC
after exposure.
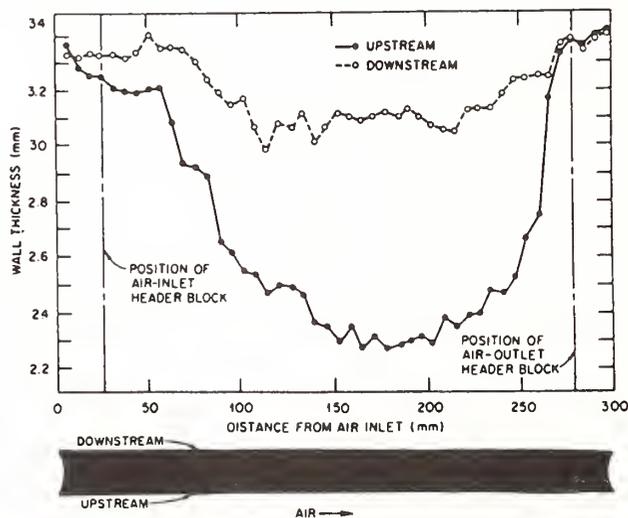
[d]Dimensions are reported for two different tubes for this siliconized SiC.

[e]Other data tables in the original reports show that CVD (chemically vapor de-
posited) SiC was not exposed in Test 3. The material in other sections is
given as CVR SiC (chemically vapor reacted SiC: silicon reacted with graphite
mandrel at 1800 °C and a CVD SiC overlay applied; see Section B.4.2.45).

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INTRODUCTION

    The references given here are the source material for the data reported in
this compilation. They are indicated in the text and in data sections by a
number in square brackets. Each reference usually lists a whole series of
reports for a given project sponsored by the Department of Energy and its pre-
decessor agencies. The order of the references is arbitrary depending on the
starting date of abstracting the data and has nothing to do with the relative
importance of the references or of their data content. For many projects both
the title and the investigators changed several times as the project continued
over a number of years. The references are, therefore, not restricted to a
uniform format but do vary in an attempt to minimize somewhat the repetition of
information within any one reference. The dates given for most reports repre-
sent the reporting period and not the publication date.

    Section D.5 provides a concordance of the references based on the reference
number ([1], [2], etc.), listing each section in this compilation which contains
information from each reference.

    Many of the projects reported here are still in progress. These projects and
a number of new projects sponsored by the Department of Energy are reported in the
<u>Oak Ridge National Laboratory Advanced Research and Technology Development Fossil
Energy Materials Program Quarterly Progress Reports</u> available from the National
Technical Information Service.

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[1]   -"Erosion Testing of Potential Valve Materials for Coal Gasification
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                Revision Y, September 1978, Revision Z, October 1978, Revision AA,
                December 1978, Revision AB, January 1979, Revision AC, March 1979,
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                sion AJ, December 1979, Revision AK, January 1980, Revision AL,

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sion B, June 1978-May 1979, Revision C, June 1979-May 1980;
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undated, Revision A, undated, Revision B, May 1980, Revision C, May
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[4]   "Update on Lockhopper Valve Development," J.F. Gardner and F.D. Freeburn,
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      presented at the American Institute of Chemical Engineers meeting,
      Houston, Texas, April 1979.

[5]   Compiled from the files of the Materials and Components Plant Performance
      Data Center, National Bureau of Standards.

[6]   "Valve Development for Coal Gasification Plants," reports of Fairchild
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             December 1983;
            -P.D. Shalek et al., ORNL/FMP-83/1, p. 275, December 1982.

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MATERIALS INDEXES

The indexes are ordered primarily on materials and secondarily on properties. If some property is of primary interest it is assumed that the reader will go to the appropriate A or B sections to locate that information.

The user is assumed to have some knowledge of the broad categories into which various materials are likely to be placed by virtue of their composition, form, or properties. For example, a specialty steel might be found in the Metals and Alloys index under Heat Resistant Alloys, High-Iron Alloys and Superalloys, or High Strength Steels depending on its normal industrial use. In the Refractories index it is necessary to know the form (Brick and Shapes, Castables, Cements and Mortars, or Plastics and Ramming Mixes) first and then the composition to locate a material of interest.

Many entries refer to A sections, B sections, and then A sections again. This is not due to a reorganization of the alphabet. The first A and B references are to data listings which include the specific material and property. Any A references which follow a B reference indicate discussion sections in which one or more of the B sections are explicitly mentioned. Please note that this does not mean a specific material is discussed in that section, but only that that material is included in a data set which is referred to in that section.

CONCORDANCE OF REFERENCES

Many of the reference reports contain data for more than one property or performance, i.e., corrosion, erosion, tensile strength, creep, thermal expansion, etc. The data from any one reference are, therefore, scattered in various sections of Parts A and B. It seemed useful to provide a listing by reference number showing all the sections containing information taken from each reference.

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[16]   B.1.2.10, B.1.2.11, B.3.2.32, B.3.2.33, B.3.2.34, B.3.2.35, B.3.2.36,
        B.3.2.81, B.3.2.82, B.3.2.83, B.3.2.84, B.3.2.85, B.3.2.86, B.3.2.87,
        B.3.2.88, B.3.2.89, B.3.2.90, B.3.2.91, B.3.2.92, B.3.2.93, B.3.2.94,
        B.3.2.95, B.3.2.96, B.3.2.97, B.3.2.98, B.3.2.99, B.3.2.100, B.3.2.101,
        B.3.2.102, B.3.2.103, B.3.2.104, B.3.2.148, B.3.2.149, B.4.2.28, B.4.2.29,
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[17]   B.1.1.7, B.1.1.11, B.1.1.13, B.1.1.15, B.3.1.28, B.3.1.29, B.3.1.30

[18]   A.1.1.2.2, A.1.2.2.2, A.2.2.2.1, A.2.2.2.1.2, A.2.2.2.1.3, A.2.2.2.1.4,
        A.2.2.2.1.5

[19]   B.1.1.8, B.1.1.9, B.1.1.10

[20]   B.1.1.14

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[21]   B.2.1.4, B.2.1.5, B.2.1.6, B.2.1.7, B.2.1.8, B.2.1.9, B.2.1.10, B.2.1.11,
       B.2.1.12, B.2.1.13, B.2.1.14, B.2.1.21, B.2.1.22

[22]   B.2.2.7, B.2.2.8, B.2.2.9, B.2.2.10, B.2.2.11, B.2.2.12, B.2.2.13

[23]   A.2.2.2.1.1, B.1.2.6, B.1.2.7, B.1.2.8, B.1.2.9, B.2.2.17, B.3.2.1,
       B.3.2.2, B.3.2.3, B.3.2.4, B.3.2.5, B.3.2.6, B.3.2.7, B.3.2.14, B.3.2.25,
       B.3.2.26, B.3.2.47, B.3.2.48

[24]   B.1.2.12

[25]   B.3.1.22, B.3.1.23, B.3.1.24, B.3.1.27, B.3.1.31

[26]   A.2.2.2.2.1, A.2.2.2.2.2, A.2.2.2.2.3, A.2.2.2.2.4, A.2.2.2.2.5,
       A.2.2.2.3.5, B.3.2.37, B.3.2.38, B.3.2.39, B.3.2.40, B.3.2.41, B.3.2.42,
       B.3.2.43, B.3.2.44, B.3.2.49, B.3.2.50, B.3.2.51, B.3.2.59, B.3.2.60,
       B.3.2.61, B.3.2.62, B.3.2.63, B.3.2.64, B.3.2.65, B.4.2.15, B.4.2.16,
       B.4.2.17, B.4.2.18

[27]   B.1.2.13, B.1.2.27, B.1.2.28, B.1.2.29, B.3.2.52, B.3.2.54, B.3.2.67,
       B.3.2.68, B.3.2.69, B.3.2.70, B.3.2.71, B.3.2.72, B.3.2.73, B.3.2.74,
       B.3.2.75, B.3.2.76

[28]   B.1.1.25, B.1.1.26, B.2.1.27, B.2.1.28, B.2.1.29, B.2.1.30, B.2.1.31,
       B.2.1.32, B.2.1.33, B.2.1.34, B.2.1.56, B.2.1.57, B.2.1.58, B.2.1.59

[29]   A.2.1.2.2, B.3.1.11, B.3.1.12

[30]   B.1.1.16, B.1.1.118, B.1.1.119, B.1.1.120, B.1.1.121, B.1.1.122, B.3.1.84,
       B.3.1.85, B.3.1.86, B.3.1.172, B.3.1.173

[31]   B.1.1.29, B.1.1.30, B.1.1.31, B.1.1.32, B.1.1.33, B.1.1.34, B.1.1.35,
       B.1.1.36, B.1.1.37, B.1.1.38, B.1.1.39, B.1.1.40, B.1.1.41, B.1.1.42,
       B.1.1.43, B.1.1.44, B.1.1.45, B.1.1.46, B.1.1.47, B.1.1.48, B.1.1.49,
       B.1.1.50, B.1.1.51, B.1.1.52, B.1.1.53, B.3.1.32, B.3.1.33, B.3.1.34

[32]   B.1.2.14, B.1.2.15, B.1.2.22, B.1.2.23, B.1.2.24, B.1.2.25, B.1.2.26

[33]   B.1.3.1, B.2.2.19, B.2.3.2, B.2.3.3, B.2.3.4, B.3.3.1

[34]   B.1.1.12, B.1.1.68, B.1.1.69, B.1.1.70, B.1.1.71, B.1.1.72, B.1.1.73,
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       B.1.1.81, B.1.1.82, B.1.1.83, B.1.1.92, B.1.1.93, B.1.1.94, B.1.1.95,
       B.1.1.96, B.1.1.98, B.1.1.99, B.3.1.35, B.3.1.36, B.3.1.37, B.3.1.38,
       B.3.1.39, B.3.1.40, B.3.1.52

[35]   A.2.1.2.2, B.3.1.41, B.3.1.42, B.3.1.43, B.3.1.44, B.3.1.45, B.3.1.46,
       B.3.1.47, B.3.1.48, B.3.1.49, B.3.1.50, B.3.1.127, B.3.1.128, B.3.1.129,
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       B.3.1.146, B.3.1.147, B.3.1.148, B.3.1.150, B.3.1.151

[36]   B.1.1.54, B.1.1.55, B.1.1.56, B.1.1.57, B.1.1.58, B.1.1.59, B.1.1.60,
       B.1.1.61, B.1.1.62, B.1.1.63, B.1.1.64, B.1.1.65, B.1.1.66, B.1.1.67

[37]   B.1.1.84, B.1.1.85, B.1.1.86, B.1.1.87, B.1.1.88, B.1.1.89, B.1.1.90,
       B.1.1.91, B.1.1.97, B.2.1.35, B.2.1.36, B.2.1.37, B.2.1.38, B.2.1.39,
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[38]   B.1.1.100, B.1.1.101, B.1.1.102, B.1.1.103, B.1.1.104, B.1.1.105,
        B.1.1.106, B.1.1.107, B.1.1.108, B.1.1.109, B.1.1.131, B.1.1.132,
        B.1.1.133, B.1.1.134, B.1.1.135

[39]   B.1.2.17, B.1.2.18, B.1.2.19, B.1.2.20, B.1.2.21, B.1.2.31, B.1.2.32,
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        B.4.2.8, B.4.2.9, B.4.2.10, B.4.2.11, B.4.2.12, B.4.2.13, B.4.2.14,
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[40]   A.2.1.2.2, B.1.1.192, B.3.1.64, B.3.1.65, B.3.1.66, B.3.1.67, B.3.1.68,
        B.3.1.69, B.3.1.70, B.3.1.71, B.3.1.72, B.3.1.73, B.3.1.74, B.3.1.75,
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[41]   B.2.1.40, B.2.1.41, B.2.1.42, B.2.1.43, B.2.1.60, B.2.1.61, B.3.1.53,
        B.3.1.54, B.3.1.55, B.3.1.56, B.3.1.57, B.3.1.58, B.3.1.59, B.3.1.60,
        B.3.1.61, B.3.1.62, B.3.1.63, B.3.1.111, B.3.1.112, B.3.1.113

[42]   A.2.1.2.2

[43]   B.2.1.23, B.2.1.24, B.2.1.26, B.2.1.44, B.2.1.45, B.3.1.87

[44]   B.1.1.27, B.1.1.28

[45]   B.1.1.136, B.1.1.137, B.1.1.138, B.1.1.139, B.1.1.140, B.1.1.141

[46]   B.1.1.136, B.1.1.137, B.1.1.138, B.1.1.139, B.1.1.140, B.1.1.141

[47]   A.2.1.2.2, B.1.1.143, B.1.1.144, B.1.1.145, B.1.1.146, B.3.1.88,
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[48]   B.1.1.142, B.2.1.46, B.2.1.47, B.2.1.48, B.2.1.49, B.2.1.50, B.2.1.51,
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[49]   A.9.3.2.3

[50]   A.2.1.2.2, B.1.1.145, B.1.1.146, B.3.1.90, B.3.1.93

[51]   B.3.1.94

[52]   B.2.1.54, B.2.1.55, B.2.1.62, B.2.1.63, B.2.1.64

[53]   B.2.1.65, B.2.1.66, B.2.1.67, B.2.1.68, B.2.1.69, B.2.1.70, B.2.1.71,
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[54]   B.3.1.26, B.3.1.95, B.3.1.96, B.3.1.97

[55]   A.2.1.2.2, B.1.1.147, B.1.1.148, B.1.1.149, B.1.1.150, B.1.1.151,
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[56]   A.2.1.2.2, B.3.1.101, B.3.1.102, B.3.1.103, B.3.1.104, B.3.1.105,
        B.3.1.106, B.3.1.107, B.3.1.108, B.3.1.109, B.3.1.110

[57]   A.2.1.2.2, B.1.1.154, B.1.1.155, B.1.1.156, B.3.1.114, B.3.1.115,
        B.3.1.116, B.3.1.117

[58]   A.2.1.2.2, B.3.1.118

[59]   A.2.1.2.2, B.3.1.119, B.3.1.120, B.3.1.121

[60]   A.2.1.2.2, B.3.1.122

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[61]   A.2.1.2.2, B.1.1.177, B.1.1.178, B.1.1.179, B.1.1.180, B.1.1.181,
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        B.3.1.126

[62]   A.2.1.2.2, B.1.1.178, B.1.1.179, B.1.1.180, B.1.1.181, B.1.1.182,
        B.1.1.183, B.1.1.184, B.1.1.185, B.1.1.186, B.3.1.124, B.3.1.125,
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[63]   A.2.1.2.2, B.3.1.123

[64]   B.1.1.157, B.1.1.158

[65]   B.1.1.159, B.1.1.160, B.1.1.161, B.1.1.162, B.1.1.163

[66]   B.1.1.159, B.1.1.160, B.1.1.161, B.1.1.162, B.1.1.163, B.1.1.164,
        B.1.1.166, B.1.1.169, B.1.1.170, B.1.1.173

[67]   B.1.1.160, B.1.1.163, B.1.1.164, B.1.1.165, B.1.1.166, B.1.1.173,
        B.1.1.174, B.1.1.175, B.1.1.176

[68]   B.1.1.164, B.1.1.166, B.1.1.169, B.1.1.170, B.1.1.171, B.1.1.172,
        B.1.1.173

[69]   B.1.1.167, B.1.1.168

[70]   A.2.4.2.1.1, A.2.4.2.1.2, A.2.4.2.1.3, A.7.1.2.1.1, A.7.1.2.1.2,
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[71]   A.2.1.2.2

[72]   A.2.1.2.2

[73]   A.2.1.2.2

[74]   A.2.1.2.2, B.3.1.130, B.3.1.131, B.3.1.132, B.3.1.133, B.3.1.134,
        B.3.1.135, B.3.1.136, B.3.1.145, B.3.1.146, B.3.1.147, B.3.1.148,
        B.3.1.150, B.3.1.151

[75]   A.2.1.2.2, B.3.1.137, B.3.1.138, B.3.1.139, B.3.1.144

[76]   A.2.1.2.2, B.3.1.149, B.3.1.151, B.3.1.152, B.3.1.153, B.3.1.154,
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[77]   A.2.1.2.2, B.3.1.149, B.3.1.151, B.3.1.152, B.3.1.153, B.3.1.154,
        B.3.1.155

[78]   A.2.1.2.2, B.3.1.156, B.3.1.159, B.3.1.160, B.3.1.161

[79]   A.2.1.2.2, B.1.1.187, B.3.1.156, B.3.1.157, B.3.1.158, B.3.1.159,
        B.3.1.160, B.3.1.161

[80]   A.2.1.2.2, B.3.1.174, B.3.1.175, B.3.1.176, B.3.1.182

[81]   A.2.1.2.2, B.3.1.177, B.3.1.178, B.3.1.179, B.3.1.180, B.3.1.181,
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[82]   A.2.1.2.2, B.3.1.171

[83]   A.2.1.2.2, B.3.1.171

[84]   B.1.1.188, B.1.1.189, B.1.1.190, B.1.1.191

[85]   A.2.1.2.2

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[86]   A.2.4.2.2.1

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[88]   A.2.2.2.3.5, B.3.2.66, B.4.2.19, B.4.2.20, B.4.2.21, B.4.2.22, B.4.2.23,
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[89]   B.1.2.26

[90]   B.1.2.16, B.4.2.6

[91]   A.2.2.2.1.6, B.1.2.30, B.2.2.20, B.2.2.21, B.2.2.22, B.3.2.77, B.3.2.78,
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[92]   A.9.3.2.2

[93]   A.9.3.2.3

[94]   A.2.3.2.2.2

[95]   B.1.2.35, B.1.2.36, B.2.2.25, B.2.2.26, B.3.2.109, B.3.2.110, B.3.2.111,
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[98]   A.2.3.2.2.1

[99]   A.2.2.2.3.5, B.3.2.136, B.3.2.137, B.3.2.138, B.3.2.139, B.3.2.140,
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[101] A.2.2.2.3.5, B.4.2.43, B.4.2.44

[102] A.9.3.2.1, A.9.3.2.1.2

[103] B.2.1.78, B.2.2.27, B.2.2.28, B.2.2.29, B.2.2.30, B.2.2.31, B.2.2.32,
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       B.2.3.9, B.3.2.141, B.3.2.142, B.3.2.143, B.3.2.144

[104] A.9.3.2.1, A.9.3.2.1.1

[105] A.9.3.2.3, B.2.2.43

[106] A.10.2.2, B.1.2.37, B.1.2.38, B.1.2.39, B.1.2.40, B.3.2.150, B.3.2.151,
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[107] A.10.2.2, B.1.2.41, B.1.2.42, B.3.2.157, B.3.2.158, B.3.2.159, B.3.2.160,
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This book expands the information provided in the original NBS/SP 642 and in NBS/SP 642 Supplement 1 publications, Construction Materials for Coal Conversion--Performance and Properties Data. These volumes are intended to provide a central source of materials information needed for the fossil fuel industry. Data have been collected and evaluated from Department of Energy-sponsored projects. The book is organized so that the information is given both with respect to the various component areas of coal gasification, liquefaction, and direct combustion plants and also with respect to the properties or possible failure mechanisms, e.g., corrosion, erosion, mechanical properties, and physical properties.

12. KEY WORDS (Six to twelve entries; alphabetical order; capitalize only proper names; and separate key words by semicolons)

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NOTE: The Journal of Physical and Chemical Reference Data (JPCRD) is published quarterly for NBS by the American Chemical Society (ACS) and the American Institute of Physics (AIP). Subscriptions, reprints, and supplements are available from ACS, 1155 Sixteenth St., NW, Washington, DC 20056.

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