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# MATERIALS RESEARCH FOR THE CLEAN UTILIZATION OF COAL

## Quarterly Progress Report

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## I. SUMMARY OF PROGRESS TO DATE

### Brief Summary

### 1. Materials Performance and Properties

Data Center personnel have received training in the use of the new Data Base Management System (DBMS). The Failure Information Data Base has been transferred to the new DBMS and is now operative on it. The normal activities of the Data Center have proceeded, especially with regard to production of the book of data for materials for coal gasification.

### 2. Creep and Related Properties of Refractories

This report describes the twelve station creep facility and presents data obtained on fused-cast-alumina and hot-pressed silicon carbide.

## II. DETAILED DESCRIPTION OF TECHNICAL PROGRESS

### 1. Materials Performance and Properties (H. M. Ondik, B. W. Christ, A. Perloff, W. A. Willard)

Progress: Since the last quarterly report for this project the Data Center personnel have received training in the use of the vendor's computer. This training included:

1. Classes providing instruction in the capabilities of the vendor's installation and the services offered (including a tour of the facilities), the sign-on procedures to gain access to the computer, and the control language for use of the vendor's time-sharing system.
2. Classes teaching the user how to access and query the specific Data Base Management System (DBMS) to be utilized by NBS. This training included hands-on practice in on-line querying of a sample data base using computer terminals in the classroom.
3. Several consultation sessions with the systems analysts assigned to NBS in the design and establishment of the data base.

The Failure Information Data Base (to be designated in the future as the Materials and Components Plant Performance Data Base) has been completely transferred to the new DBMS. The vendor company did the programming needed to take the magnetic tape copy of the data base as it was structured in the old DBMS on another computer and read it into their computer, transforming the structure to fit the new DBMS. Pre-programmed statements, known as macros, for querying this data file were also prepared by the systems analysts. In spite of the large effort expended by the vendor, Data Center personnel, upon review of the entire new data base and the macro queries had to spend an appreciable amount of time and effort in correcting a fair number of data errors and in changing the macro queries to fit the desired NBS pattern. The Failure File containing Plant Performance information is now fully operative on the new system.

During the last six months (since the last quarterly report) the Data Center has received eleven reports of failures and failure analyses. These reports have been abstracted and entered into the current Data Base. The Center also received twenty-two requests for information and in response to these requests has provided a total of 3436 computer abstracts of failure events and analyses, sixty statistical tables, and fourteen hard copy reports. Summary statistics for the current file of failure events in pilot plants included in the Data Base are given in Tables 1 through 4.

The Materials Properties Data Base has gone through various design changes during the lengthy procurement period preceding the awarding of the computer services contract. These changes have been with regard to structure and definition within the computer, however, and not with respect to either the number or identify of the data items or the relationships between them. The currently planned design takes advantage of certain characteristics of the DBMS which reduce the need for storage of repetitive data but do not affect the ease of searching for data. Although the data will appear to the user to be contained in one data base there will, in reality, be several data bases, one for references, one for materials, and one for properties. The DBMS is known as a "user friendly" system and permits queries structured so that multiple data bases are searched as if they were actually one data base. At this time a fourth data base is under consideration, one for test conditions. The current arrangement of the data includes the test conditions in the properties data base but the use of the fourth base would reduce the amount of keyboarding of data required and also reduce the amount of computer storage needed. Tests of the data base structure are being conducted and a small amount of information has been entered for test purposes.

Work on the book "Construction Materials for Coal Gasification--Performance and Properties Data" has progressed well. Over 400 pages are complete to date of the 600 pages projected for the first issue of the looseleaf format book. A full outline of the book was given in an earlier quarterly report of this series and, although there have been a few revisions to the outline, the major divisions of the book are the same. These divisions are:

| | |
|---|---|
| Section A. | Materials Considerations and Performance Data |
| Section B. | Materials Tests Results |
| Section C. | Properties of Candidate Materials |
| Section D. | Properties of Experimental Materials |
| Section E. | References |
| Section F. | Index |

The first issue of the book will contain information in Sections A, B, E, and F. The materials in Section A is subdivided by component area: Coal Handling and Preparation Equipment which will include sections on conveying equipment, grinding and crushing equipment, drying equipment, fines control equipment such as cyclones, wet scrubbers, and bag houses, heating equipment; Vessels (including reactors, devolatilizers, lockhoppers, etc.) which will include sections on pressure-containing shells, refractory linings and components for both dry-bottom and slagging vessels, and metal internal components; Gas Clean Up Equipment including solids separation equipment (scrubbers and cyclones), cooling-down systems (quench systems and heat exchangers), and gas removal systems; Water-Gas Shift Equipment; Methanation Equipment; Compressors; Piping for gas, solids transfer, slurry, and liquids; Pumps for gas, liquids, and slurry; Valves for gas, liquids, slurry and solids.

The information in each component area section will be organized in the following way:

Operating Requirements (a brief discussion of the major problem(s) of that component area)

Performance Data

Plant Experience (summary tables and brief discussion of the pertinent data in the NBS/DOE Failure Data Base)

| | |
|---|---|
| Component Test and Development | (data obtained by testing prototypes or real components on test stands, or in constructing and testing portions of or models of components--such data are not available for all component areas) |
| Materials Evaluation | (summaries of pertinent data tables contained in Section B of the book with evaluations) |
| Candidate Materials | (discussion of possible materials for use in the component area based on the information included in the entire Performance Data section) |

Section B is divided into four main headings:

1. Corrosion Effects, Chemical Reactions, and Phase Changes
2. Erosion, Erosion/Corrosion, and Abrasion Effects
3. Mechanical Properties Testing
4. Physical Properties Testing

Each of these categories is divided into sections for alloys, refractories, and, where such data exist, one for coatings. The main source of the information in this section is the collection of DOE materials research contractor's reports. These tables and graphs summarize the data in as condensed a form as possible. The tests include laboratory experiments as well as the results of exposure of small test specimens in pilot plants. These data are organized in this separate section rather than in Section A to avoid the duplication resulting from the applicability of the same information to more than one component area of a gasification plant. Sections C and D will contain handbook-type data for those materials mentioned in Section A as showing some promise for possible use. Examples of pages from the book are attached in the appendix.

Plans: The first issue of the book on materials for coal gasification will be completed. Keyboarding of data for the Materials Properties Data Base will proceed. The regular activities of the Data Center will be maintained.

## TABLE 1

## FAILURES IN COAL CONVERSION INDUSTRY

| PROCESS | NO. OF INCIDENTS | PERCENT |
|---|---|---|
| BIGAS | 18 | 4.00 |
| BMI | 35 | 7.78 |
| CARBONATE | 5 | 1.11 |
| CLEAN COKE | 27 | 6.00 |
| $CO_2$ | 52 | 11.56 |
| CPC | 1 | 0.22 |
| CRESAP | 20 | 4.44 |
| EXXON | 3 | 0.67 |
| GFETC | 3 | 0.67 |
| H-COAL | 1 | 0.22 |
| HYGAS | 48 | 10.67 |
| LETC | 3 | 0.67 |
| LIGNITE | 18 | 4.00 |
| METC | 14 | 3.11 |
| MISC. | 15 | 3.33 |
| PETC | 2 | 0.44 |
| SRC | 22 | 4.89 |
| SRC-W | 17 | 3.78 |
| SYNTHANE | 114 | 25.33 |
| SYNTHOIL | 2 | 0.44 |
| WESTINGHOUSE | 30 | 6.67 |

TOTAL NUMBER OF INCIDENTS = 450

## TABLE 2

## FAILURE MODE ANALYSIS

| PROCESS | FAILURE MODE | | | | | |
|---|---|---|---|---|---|---|
| | CORROSION | EROSION/ WEAR | MANUFACT DEFECT(1) | PROCESS CONTROL(2) | SCC | STRESS/ TEMP(3) |
| BIGAS | 0 | 3 | 2 | 9 | 4 | 0 |
| BMI | 3 | 4 | 6 | 12 | 0 | 2 |
| CARBONATE | 1 | 1 | 1 | 0 | 2 | 0 |
| CLEAN COKE | 3 | 7 | 6 | 4 | 0 | 5 |
| $CO_2$ | 20 | 8 | 4 | 7 | 8 | 2 |
| CPC | 0 | 0 | 0 | 0 | 0 | 1 |
| CRESAP | 3 | 9 | 5 | 2 | 1 | 0 |
| EXXON | 2 | 1 | 0 | 0 | 0 | 0 |
| GFETC | 1 | 0 | 1 | 0 | 1 | 0 |
| H-COAL | 0 | 0 | 0 | 0 | 1 | 0 |
| HYGAS | 13 | 10 | 3 | 6 | 9 | 5 |
| LETC | 0 | 2 | 0 | 1 | 0 | 0 |
| LIGNITE | 6 | 1 | 0 | 2 | 6 | 1 |
| METC | 3 | 6 | 2 | 1 | 0 | 2 |
| MISC. | 7 | 0 | 0 | 1 | 1 | 5 |
| PETC | 1 | 0 | 0 | 1 | 0 | 0 |
| SRC | 6 | 4 | 2 | 4 | 4 | 1 |
| SRC-W | 2 | 7 | 4 | 0 | 3 | 1 |
| SYNTHANE | 24 | 33 | 11 | 18 | 7 | 8 |
| SYNTHOIL | 0 | 1 | 0 | 1 | 0 | 0 |
| WESTINGHOUSE | 5 | 6 | 4 | 3 | 9 | 3 |
| | 100 | 103 | 51 | 72 | 56 | 36 |

(1) INCLUDES FAILURES BY DESIGN, FABRICATION, AND QUALITY CONTROL
(2) INCLUDES FAILURES BY OVERHEATING AND OVERSTRESSING
(3) INCLUDES FAILURES BY CREEP, FATIGUE, AND THERMAL STRESS

# TABLE 3

## COMPONENT FAILURES IN COAL CONVERSION INDUSTRY

| PROCESS | COMPONENT | | | | | |
|---|---|---|---|---|---|---|
| | AUX EQUIP(1) | COAL HAND(2) | PIPING | PUMPS | VALVES | VESSELS (3) |
| BIGAS | 8 | 0 | 7 | 1 | 1 | 1 |
| BMI | 14 | 8 | 4 | 4 | 5 | 0 |
| CARBONATE | 1 | 0 | 1 | 0 | 2 | 1 |
| CLEAN COKE | 5 | 0 | 10 | 6 | 3 | 3 |
| $CO_2$ | 2 | 1 | 42 | 0 | 2 | 5 |
| CPC | 0 | 0 | 0 | 0 | 0 | 1 |
| CRESAP | 5 | 0 | 4 | 8 | 1 | 2 |
| EXXON | 0 | 0 | 3 | 0 | 0 | 0 |
| GFETC | 0 | 0 | 1 | 0 | 1 | 1 |
| H-COAL | 0 | 0 | 1 | 0 | 0 | 0 |
| HYGAS | 7 | 0 | 33 | 5 | 2 | 1 |
| LETC | 0 | 0 | 2 | 0 | 1 | 0 |
| LIGNITE | 1 | 1 | 10 | 4 | 1 | 1 |
| METC | 0 | 0 | 3 | 0 | 9 | 2 |
| MISC. | 2 | 0 | 10 | 0 | 0 | 3 |
| PETC | 0 | 0 | 2 | 0 | 0 | 0 |
| SRC | 2 | 0 | 4 | 4 | 2 | 10 |
| SRC-W | 2 | 0 | 0 | 6 | 3 | 6 |
| SYNTHANE | 12 | 4 | 34 | 24 | 24 | 16 |
| SYNTHOIL | 0 | 0 | 0 | 0 | 2 | 0 |
| WESTINGHOUSE | 8 | 0 | 19 | 0 | 3 | 0 |
| | 69 | 14 | 190 | 62 | 62 | 53 |

(1) INCLUDES COMPRESSORS, CYCLONES, THERMOCOUPLES, AND OTHERS
(2) INCLUDES BAGHOUSE, FILTERS, CONVEYORS, ELEVATORS, AND OTHERS
(3) INCLUDES GASIFIERS, REGENERATORS, DISSOLVERS, SEPARATORS, AND OTHERS

## TABLE 4

## COMPONENT VS. FAILURE MODE

| COMPONENT | FAILURE MODE | | | | | |
|---|---|---|---|---|---|---|
| | CORROSION | EROSION/ WEAR | MANUFACT DEFECT(1) | PROCESS CONTROL(2) | SCC | STRESS TEMP(3) |
| AUXILIARY EQUIPMENT | 5 | 4 | 7 | 12 | 0 | 4 |
| COAL HANDLING | 2 | 1 | 1 | 6 | 0 | 2 |
| COMPRESSORS | 2 | 3 | 6 | 5 | 0 | 1 |
| CYCLONES | 0 | 5 | 0 | 0 | 0 | 0 |
| PIPING | 58 | 32 | 10 | 22 | 46 | 12 |
| PUMPS | 4 | 29 | 9 | 9 | 0 | 4 |
| THERMOCOUPLES | 7 | 0 | 0 | 1 | 0 | 0 |
| VALVES | 8 | 25 | 11 | 10 | 2 | 1 |
| VESSELS | 14 | 4 | 7 | 7 | 8 | 12 |
| | 100 | 103 | 51 | 72 | 56 | 36 |

(1) INCLUDES FAILURES BY DESIGN, FABRICATION, AND QUALITY CONTROL
(2) INCLUDES FAILURES BY OVERHEATING AND OVERSTRESSING
(3) INCLUDES FAILURES BY CREEP, FATIGUE, AND THERMAL STRESS

# APPENDIX

Samples of data from the book "Construction Materials for Coal Gasification--Performance and Properties Data"

## COMPRESSOR IN-SERVICE PERFORMANCE(5)

| Material | Compressor/ Components | Plant/ Process | Service Life | Atmosphere | Temp. °F | Press. PSIG | Failure Mode |
|---|---|---|---|---|---|---|---|
| Aluminum | Process air compressor K-701 A/B (wheels and bearings) | Battelle, Columbus | 5000 hr | Air | Ambient | N.A. | Corrosion and/or erosion of wheels caused an imbalance which damaged the bearing. |
| Cast Iron | Recycle make gas booster compressor | Battelle, Columbus | N.A. | Process gas/ cooling water | <32 | N.A. | Crack occurred between the water jacket and cylinder bore possibly caused by freezing of residual cooling water. |
| Cast Iron | Duplex recycle compressor (oil injection pump plungers) | Clean Coke | N.A. | Oil | N.A. | N.A. | Plungers on oil pumps failed on startup after oil injection pumps were extensively rebuilt |
| Cast Iron | Inert gas compressor (valve disc) | Battelle, Columbus | 3000 hr | Inert gas | Ambient | N.A. | Broken valve disc. Cause unknown. |
| 304 S.S.<br>Inconel X-750 | Hydrogen compressor (helical coil springs from a two-way pressure valve) | SRC (Wilsonville) | 21 hr | Hydrogen/ nitrogen | 77-190 | N.A. | Improper material substitution (304 S.S. for Inconel X-750) led to eventual failure of valve springs and a total plant shutdown. |
| 416 S.S. | Three stage reciprocating (valve assembly leaf springs) | Hygas | ∿42 hr | $H_2/CO/CO_2/H_2O$ | 75-219 | 1050 | Several springs fractured, others suffered pitting corrosion. Possible causes are moisture in the gas, preload on springs too high, stress corrosion cracking. |
| Teflon Steel | Inert gas compressor (piston ring, shaft) | Synthane | 6 mos | Recycle $CO_2/H_2O$ | N.A. | N.A. | Teflon piston ring wear has accelerated possibly due in part to increased moisture in $CO_2$. Most of shaft wear of 0.008" max. occurred in initial operation and was due to internal rust and contamination. |

## SOLIDS PIPING IN-SERVICE PERFORMANCE[5]

| Material | Location | Description (Plant ID) | Plant/ Process | Service Life | Environment | Temp. °F | Press., PSIG | Failure Mode |
|---|---|---|---|---|---|---|---|---|
| C-Mo Steel | Pipe welded to a 1 in 1500 lb sock-o-let in char transfer line, 1 P2112 E2B | 1 in Schedule 80 | Synthane | N.A. | Char/steam/ coal gases | N.A. | 600 | A hole occurred at edge of weld due to erosion/ corrosion. |
| Incoloy 800 | Elbow on fines feed line, C102B | N.A. | Westing-house | 100 hr | Coke breeze/ recycle gas | 1200 | 240 | Hole eroded in elbow. |
| Incoloy 800 | Piping in spent acceptor lift line CD-208 | 4 in ID x 3/16 in wall thickness | $CO_2$ Acceptor | 630 hr | Dolomite/re-cycle gas | 1450 | 150 | A hole was eroded in the inner liner close to lower slip joint due to misalignment of the slip joint. |
| Incoloy 800 | Piping in spent acceptor lift line, CD-208 | 4 in ID x 3/16 in wall thickness | $CO_2$ Acceptor | N.A. | Dolomite/re-cycle gas | 1350 | 150 | Line exploded during attempt to clear a plug using forced air. |
| Incoloy 800 | Piping in char recycle gas line, Dolomite diverter | 1 in Schedule 40 | Westing-house | 30 hr | Dolomite/re-cycle gas | 1200 | 240 | Hole eroded in "Y" branc caused by impact of dolomite |
| Incoloy 800 | Pipe below 4 in x 1 in reducer on line C-103B | 1in pipe | Westing-house | 120 hr | Coke breeze/ recycle gas | 400 | 220 | Hole eroded in wall by fines impingement. |
| Incoloy 800 | Spool piece be-low C-103B | N.A. | Westing-house | 2 yrs | Char/recycle gas | 400 | 220 | Pinhole leak and hairlin cracks - possibly stress corrosion cracking. |
| Incoloy 800 | Tee on char feed line near C-105B | 4 in Tee | Westing-house | 200 hr | Coke breeze/ recycle gas | 500 | 200 | Hole eroded in tee by fines impact. |
| Incoloy 800 coated with nickel aluminide | Transition cones of slip joint | 4 in ID x 3/16 in wall thickness | $CO_2$ Acceptor | 184 hr | Dolomite/re-cycle gas | 1450 | 150 | Erosion damage to transition cones. |
| Incoloy 800 coated with Stellite 12 | Transition cones of slip joint | 4 in ID x 3/16 in wall thickness | $CO_2$ Acceptor | 835 hr | Dolomite/re-cycle gas | 1450 | 150 | Erosion damage to transition cones. |
| Incoloy 800 coated with 75% chromium carbide/25% nichrome | Transition cones of slip joint ' | 4 in ID x 3/16 in wall thickness | $CO_2$ Acceptor | 110 hr | Dolomite/re-cycle gas | 1450 | 150 | Erosion damage to transition cones. |
| Tabular $Al_2O_3$ castable | Refractory lined spool piece at bottom of riser leg | 5 in-thick liner within 14 in ID pipe | Battelle, Columbus | ∿200 hr | Mulcoa (cal-cined bauxite)/ lift gas | Ambient | N.A. | Spool piece refractory erosion caused by excessive lift gas flow. |
| 304 S.S. | Tee in char transfer line P2112 | 1 in Pipe | Synthane | 3 mos | Coal char/ steam/coal gas/ $CO_2/N_2$ | 400 | 600 | Coal char erosion of line tee. |
| 310 S.S. | Inner liner of outlet from re-generator line | 6 in pipe with 0.250 wall thick-ness | $CO_2$ Acceptor | 850 hr | Dolomite/flue gas | 1830 | 150 | Inner lining broke off and fell into vessel. Failed by sulfur corrosion and thermal cycling. |
| 316 S.S. | Elbow from coal feed line, above nozzle 118 | 2 in/45° pipe elbow | Synthane | 9 mos | Coal/oxygen/ steam/coal gas | N.A. | 600 | Hole eroded in elbow from coal particle impingement. |
| 446 S.S. | Pipe in solids transfer line of hydrogasifier | N.A. | Hygas | N.A. | Process solids/gas | 1500 | 1500 | Circumferential cracking and complete fractures in welds and parent metal, possibly caused by stress corrosion cracking. |

EROSION DATA[a] FOR THE GASIFIER REFRACTORY LINING[b] OF A GASIFICATION PILOT PLANT[c] [18]

| MATERIAL<br>Sample Location[d] | Erosion[a]<br>kg/kg abrasive | Density<br>kg/m$^3$ |
|---|---|---|
| ABRASION-RESISTANT CASTABLE[b] | | |
| Control specimen[e] | $1.03 \times 10^{-2}$ | 2100.8 |
| Control specimen[e] | $9.36 \times 10^{-3}$ | 2115 |
| Bottom-of-bed, east, cold end | $4.49 \times 10^{-3}$ | 2112 |
| hot end | $5.50 \times 10^{-3}$ | 2081.1 |
| Bottom-of-bed, south, hot end | $6.67 \times 10^{-3}$ | 1921.1 |
| Bottom-of-bed, north, hot end | $2.42 \times 10^{-3}$ | 2079 |
| Mid-bed, east, hot end | $5.88 \times 10^{-3}$ | 1957.9 |
| Mid-bed, north, cold end | $3.63 \times 10^{-3}$ | 1967.7 |
| hot end | $4.37 \times 10^{-3}$ | 2051 |
| DENSE CALCINED FLINT CLAY CASTABLE[b] | | |
| Control specimen[e] | $1.07 \times 10^{-2}$ | 2043 |
| Control specimen[e] | $1.02 \times 10^{-2}$ | 2035 |
| Mid-bed, south, cold end | $7.51 \times 10^{-3}$ | 1899 |
| hot end | $6.52 \times 10^{-3}$ | 1970.9 |
| Mid-bed, north, cold end | $4.87 \times 10^{-3}$ | 1980.5 |
| hot end | $6.18 \times 10^{-3}$ | 1925.5 |
| Mid-bed, north-east, hot end | $5.98 \times 10^{-3}$ | 1934 |
| Mid-bed, west, hot end | $6.71 \times 10^{-3}$ | 1934 |

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[a] Erosion samples were 0.5 in x 0.5 in x 0.5 in, cut by diamond sawing and diamond surface grinding; samples were subjected to erosion by 100 mesh SiC, at 30 psig, ambient temperature, 90° angle of impingement, 400 grams of abrasive per sample.

[b] Lining hot face consisted of a 6-in layer of an abrasion resistant castable ($\sim$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 ($\sim$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 inches in diameter; samples were from various locations within the portion of the vessel occupied by the char(lignite)-dolomite fluidized bed; the compass point designations were a convenient way to keep track of samples; core samples could not be taken from higher levels because of the extreme hardness of the material in the upper region; hot end designates the portion of a core close to the hot face surface; cold end designates the portion of a core adjacent to the underlying insulating refractory or, in the case of cores taken from vapor stops, adjacent to the metal shell of the vessel.

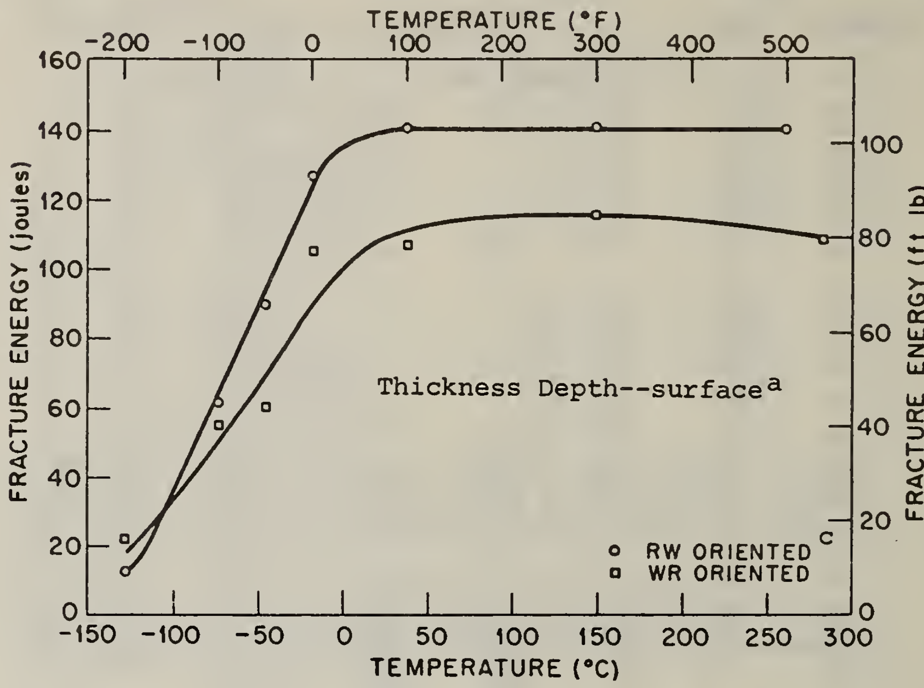
[e] Control specimens are laboratory-prepared and fired to 900 °C for 16 hr.

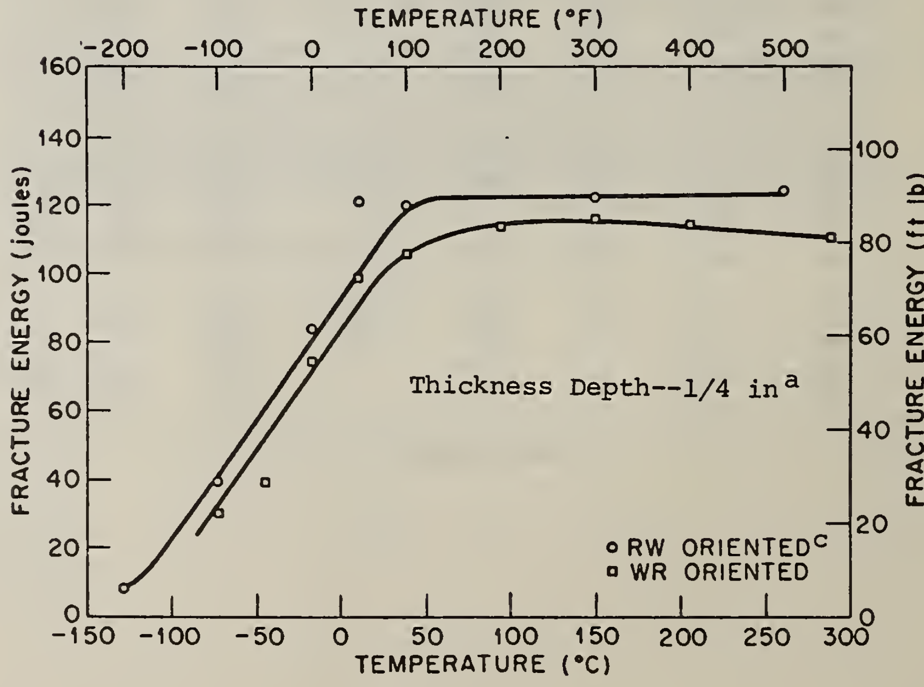
STRESS RUPTURE TESTS[a] OF SOME ALLOYS IN AIR AND IN COAL GASIFICATION ATMOSPHERE[b][10]

| Environment | Temperature °F | Stress ksi | Time (hours) to 0.1% El.[c] | Time (hours) to 0.5% El.[c] | Time (hours) to 1.0% El.[c] | Time (hours) to Rupture | Elong. % | RA % | Minimum Creep Rate %/hr |
|---|---|---|---|---|---|---|---|---|---|
| | | | | INCOLOY 800H[d] | | | | | |
| Air, 1 atm | 1200 | 31.0 | 0.1 | 2.7 | 26.7 | 174.9 | 36.4 | 43.5 | 0.030 |
| | 1200 | 31.0 | 0.1 | 2.0 | 21.4 | 184.4 | 34.9 | 41.5 | 0.018 |
| | 1200 | 31.0 | - | - | - | 176.2 | 38.4 | 54.2 | 0.070 |
| | 1200 | 31.0 | - | - | - | 77.0 | 36.9 | 46.9 | 0.082 |
| | 1200 | 31.0 | - | - | - | 95.0 | 33.6 | 38.5 | 0.165 |
| | 1200 | 31.0 | - | - | - | 87.0 | 37.6 | 45.6 | 0.152 |
| | 1200 | 27.5 | 0.2 | 4.9 | 51 | 555.9 | 30.8 | 36.3 | 0.018 |
| | 1200 | 23.5 | 0.9 | 12.7 | 105 | 1659.1 | 24.2 | 30.2 | 0.003 |
| CGA[b], 68 atm | 1253 | 35.5 | - | - | - | 6.9 | 18.7 | 46.8 | 1.178 |
| | 1253 | 31.0 | - | - | - | 2.7 | 35.1 | 53.5 | 8.0 |
| | 1253 | 17.5 | - | - | - | 173.5 | 26.1 | 63.6 | 0.180 |
| | 1200 | 15.0 | - | - | - | 3780.6 | 33.2 | 45.9 | - |
| | 1200 | 13.0 | - | - | - | 3777.5[e] | 32.4[e] | 35.7[e] | - |
| Argon, 68 atm | 1200 | 31.0 | - | - | - | 143.0 | 25.0 | 42.6 | 0.390 |
| Air, 1 atm | 1500 | 9.6 | 0.3 | 2.3 | 5.2 | 155.9 | 39.1 | 43.5 | 0.017 |
| | 1500 | 9.6 | - | - | - | 82.0 | 44.7 | 53.0 | 0.483 |
| | 1500 | 8.2 | 1.7 | 20.7 | 50.9 | 641.4 | 26.0 | 40.6 | 0.021 |
| | 1500 | 6.8 | 5.5 | 129 | 240 | 1739.8 | 24.9 | 35.9 | 0.005 |
| CGA, 68 atm | 1583 | 8.5 | - | - | - | 81.8 | 25.5 | 51.4 | 0.052 |
| | 1583 | 6.5 | - | - | - | 138.0 | 24.0 | 13.1 | 0.090 |
| | 1500 | 5.4 | - | - | - | 499.0 | 33.4 | 27.5 | 0.004 |
| Air, 1 atm | 1800 | 4.85 | 0.4 | 15.2 | 27.5 | 66.8 | 30.0 | 38.5 | 0.0215 |
| | 1800 | 3.5 | 19.5 | 260 | 306 | 432.9 | 11.0 | 21.2 | 0.001 |
| | 1800 | 2.75 | 269 | 633 | 738 | 1018.9 | 13.9 | 18.2 | 0.0007 |
| | 1800 | 2.31 | 210 | 1293 | 1630 | 2579.0 | 17.4 | 16.9 | 0.0002 |
| | 1800 | 1.9 | 370 | 5280 | 6820 | 9540.3[f] | - | - | 0.00006 |
| CGA, 68 atm | 1800 | 2.0 | - | 10 | 15 | 37.5 | 33.0 | 48.8 | 0.0938 |
| | 1800 | 1.8 | - | - | - | 78.1 | 50.8 | 50.6 | - |
| | 1800 | 1.5 | - | - | - | 457.9 | 21.6 | 32.5 | - |
| | | | | INCOLOY 800H, Al[g] | | | | | |
| Air, 1 atm | 1200 | 28.5 | 0.1 | 0.6 | 1.5 | 101.2 | 53.8 | 54.5 | 0.155 |
| | 1200 | 25.0 | 0.2 | 1.5 | 4.5 | 356.4 | 54.2 | 56.0 | 0.0358 |
| | 1200 | 23.0 | 0.3 | 2.7 | 6.9 | 533.7 | 49.7 | 59.0 | 0.0199 |
| CGA, 68 atm | 1200 | 29.4 | - | - | - | 63.0 | 43.0 | 44.2 | 0.111 |
| | 1200 | 22.5 | - | 4.0 | 7.0 | 178.0 | 46.8 | 46.7 | 0.0794 |
| | 1200 | 17.5 | - | - | - | 975.0 | 39.3 | 40.4 | 0.00529 |
| Air, 1 atm | 1500 | 8.6 | 0.1 | 0.6 | 1.3 | 102.9 | 73.0 | 71.6 | 0.363 |
| | 1500 | 7.6 | 0.2 | 2.0 | 9.5 | 248.8 | 74.3 | 61.6 | 0.144 |
| | 1500 | 6.4 | 3.1 | 20.6 | 41.2 | 1350.4 | 52.6 | 55.6 | 0.0192 |
| CGA, 68 atm | 1500 | 8.6 | - | - | - | 59.2 | 51.7 | 55.0 | - |
| | 1500 | 7.6 | - | - | - | 191.4 | 38.1 | 60.8 | 0.188 |
| | 1500 | 6.5 | - | - | - | 524.0 | 28.7 | 44.6 | - |
| Air, 1 atm | 1800 | 3.4 | 7.5 | 38 | 58 | 180.6 | 13.8 | 22.5 | 0.012 |
| | 1800 | 2.8 | 0.3 | 6.1 | 13.6 | 194.6 | 28.8 | 27.9 | 0.0688 |
| | 1800 | 2.5 | 175 | 585 | 726 | 847.9 | 5.5 | 7.6 | 0.00039 |
| CGA, 68 atm | 1800 | 2.5 | - | 1.75 | 3.5 | 23.5 | 55.2 | 62.0 | 0.0741 |
| | 1800 | 1.8 | - | - | - | 50.8 | 34.8 | 35.2 | - |
| | 1800 | 0.8 | - | - | - | 94.7 | 66.3 | 58.3 | - |

(Table Continued)

## VARIATION OF CHARPY-V IMPACT ENERGY WITH TEMPERATURE AND THICKNESS DEPTH[a] FOR ASTM A543 CLASS 1 STEEL PLATE[b][35]





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(Data Continued)

## 2. Creep and Related Properties of Refractories (N. J. Tighe, C. L. McDaniel, S. M. Wiederhorn)

Progress: During this quarter the twelve station creep reep facility was put into service and used to obtain creep data. Measurements were made on a fused-cast sodium-doped alumina and on a hot-pressed silicon carbide refractory. The materials evaluation included creep measurements, flexural strength and x-ray analysis of surface scales. Tests were conducted at 1400 and 1500 °C for one to six week periods.

Experimental Procedure: Creep measurements on the sodium-doped alumina designated "Monofrax A" were made using cores obtained from Montana State University. The cores were machined to have a 1/2 in diameter by 1 in long reduced cross-section in a 2 in long by 3/4 in cylinder. The silicon carbide samples were made from a hot-pressed material from Norton Co. designated NC 203. Test bars were 4 x 5 x 50 mm and were cut from billet 4294621198. This billet was 6 x 6 x 1 in and was cut into 390 specimens. The specimens were numbered according to their position in the billet; the numbers were randomized and specimens were tested in random order.

The test facility was designed and built in order to carry-out long term tests under static loads at temperatures up to 1600 °C in air. The completed facility is shown in figure 1. This facility has 4 furnaces and each furnace has 3 specimen stations. The furnaces have $MoSi_2$ heating elements and alumina-zirconia-silicate insulation, and are capable of holding temperatures of 1600 °C for extended periods. The heat loss produced when using all three stations restricts the optimum temperature somewhat as will be discussed later.

Loads are applied to the specimens through 3/4 in diameter rams attached to pneumatic cylinders. These details are seen in the photograph in figure 2. The figure shows the cylinders above the furnace and load cells below the furnace. The rams, shown in figure 3, are connected to the load cells and to the load cylinders through water-cooled jackets. The rams, made from sintered silicon carbide and from sintered alumina, are installed now and rams of other materials, such as fused-cast spinel, are being developed. The ram material must be compatible with both the test specimen material and with the temperature and load conditions.

Loads are applied manually to the specimens by varying the air pressure to the cylinder and reading the load from the load cell. Controls for applying the load are mounted to the frame of each furnace. Timers to indicate elapsed time and time of failure, are also mounted on the furnace frame. Load cells are connected to a 20 station scanner which has a digital load indicator and a printed read-out. The load cells can be switched to a continuous recorder when such reading is required for breaking the specimen or for determining yield information. The facility is capable, therefore, of recording both the static load applied over the 1000 hour periods and of recording the dynamic load applied over short time periods.

Furnace temperature control is achieved with the microprocessor-based system shown in detail in figure 4. With this system, each furnace is controlled on an independent time base and the rate of rise, soak time and rate of cooling can be programmed to suit the individual experiment. Temperatures can be cycled within the rise and fall capability of the furnace. For example, a cycle of 1400 °C to 1300 °C to 1400 °C can be achieved with a rise and fall time of 2 minutes. Various alarm control devices can be connected to the microprocessor board to further change or monitor the furnace during the test cycle. For example, the furnaces can be shut off when the creep reaches a fixed amount or after all specimens in a furnace have broken.

For the tests described in the following results section, the furnaces were brought to the test temperature in one hour, and held for 1/2 hour to allow for equilibration before applying the load. At the end of the test period, the specimens were either broken at temperature or cooled to room temperature under the appropriate cooling rate for the specimen material.

Experimental Results: The tests carried out during this quarter provided both significant deformation data and an assessment of the usefulness of the facility. The two specimen configurations, cylindrical compression and 4-pt bend bars, permit compressive creep, flexural creep and stress rupture data to be obtained.

Fused-cast Na doped $Al_2O_3$, "Monofrax A". This material is being used in the air-preheater experiment at Montana State University. This material changes composition during high temperature exposure and fractures readily under thermal shock conditions. The as-received material is two phase $\alpha Al_2O_3$ and $\beta Al_2O_3$ ($Na_2O \cdot 11Al_2O_3$). The $Na_2O$ volatizes after heating above 1200 °C and the "β phase" is not detected in x-ray patterns taken after cooling to room temperature.

Compressive yield of the alumina was obtained using a constant displacement rate of 2 x $10^{-4}$ in/min. The cylindrical specimens were used with SiC rams and alumina pads. The results are shown below in Table 1.

Table 1. Na-doped $Al_2O_3$, Compressive Yield

| Temperature, °C | Yield, ksi | Deformation % |
|---|---|---|
| 1400 | 13.0 | 2.9 |
| 1500 | 4.1 | 1.9 |
| 1600 | 1.8 | 1.5 |

The static load applied to the cylindrical specimens in the creep facility was 250 psi. After 1000 hrs at 1400 °C the average deformation for the 3 specimens was 0.13%. Cycling the temperature from 1400 °C to 1300 °C at 1/2 hr intervals accelerated the deformation and produced the average deformation of 0.13% in three specimens after 168 hrs with a stress of 250 psi. These results are significant and further cycling tests will be run to substantiate the data.

Silicon Carbide, NC203. Silicon carbide specimens were tested in the 4-pt. flexure configuration using SiC rams and SiC holders. Test temperatures of 1200, 1400 and 1500 °C were used. Specimens were placed with a holding load of approximately 1 lb during the exposure period and broken at temperature without removing them from the furnace.

The exposure times and flexure strength are presented in Table 2. Silicon carbide oxidizes readily above 1200 °C forming an amorphous and crystalline oxide scale identified as tridymite. It is seen in the table that specimens exposed 1030 hrs are stronger than those exposed 168 hr. This finding is similar to observations on some of the silicon nitride materials and will be analyzed further to identify the strengthening mechanism. The table shows also the decrease in strength observed at 1500 °C. These strength measurements provide basic information required to determine appropriate static load for long term creep experiments.

The x-ray diffraction patterns were taken from test bars before and after the exposures at 1200 and 1400 °C.

Table 2. Silicon Carbide, NC203, Billet 4294621198

| Temperature °C | Exposure, hr | Flexure Strength, MPa | X-ray Analysis |
|---|---|---|---|
| 25 | | 524 ±111 | αSiC, 6H+4H, WC |
| 1200 | 168 | 419 ±137 | |
| 1200 | 1030 | 605 ±43 | αSiC, 6H+4H, WC<br>$SiO_2$-tridymite SS |
| 1400 | 168 | 413 ±23 | αSiC, 6H+4H, WC<br>$SiO_2$-tridymite |
| 1500 | 168 | 339 ±14 | |

Plans: Thermal cycling data will be obtained using larger temperature differences. Both materials will be subjected to thermal cycle tests. Changes in microstructure will be monitored using x-ray diffraction analysis, optical and scanning electron microscopy. The deflection monitoring system for creep experiments will be installed at each specimen station.

FIGURE CAPTIONS

Figure 1. High temperature test facility showing (A) the four furnace stations, (B) the furnace programmer-controller, and (C) the scanner-recorder system.

Figure 2. Single furnace station showing (A) the furnace, (B) air cylinders, (C) controls for cylinders, (D) load cells.

Figure 3. Interior of furnace showing: (A) alumina creep specimen, (B) silicon carbide rams.

Figure 4. Facility control panels (A) scanner and recorder for load cells, (B) process control system for the furnace showing keypad and data input pad and furance power indicators.

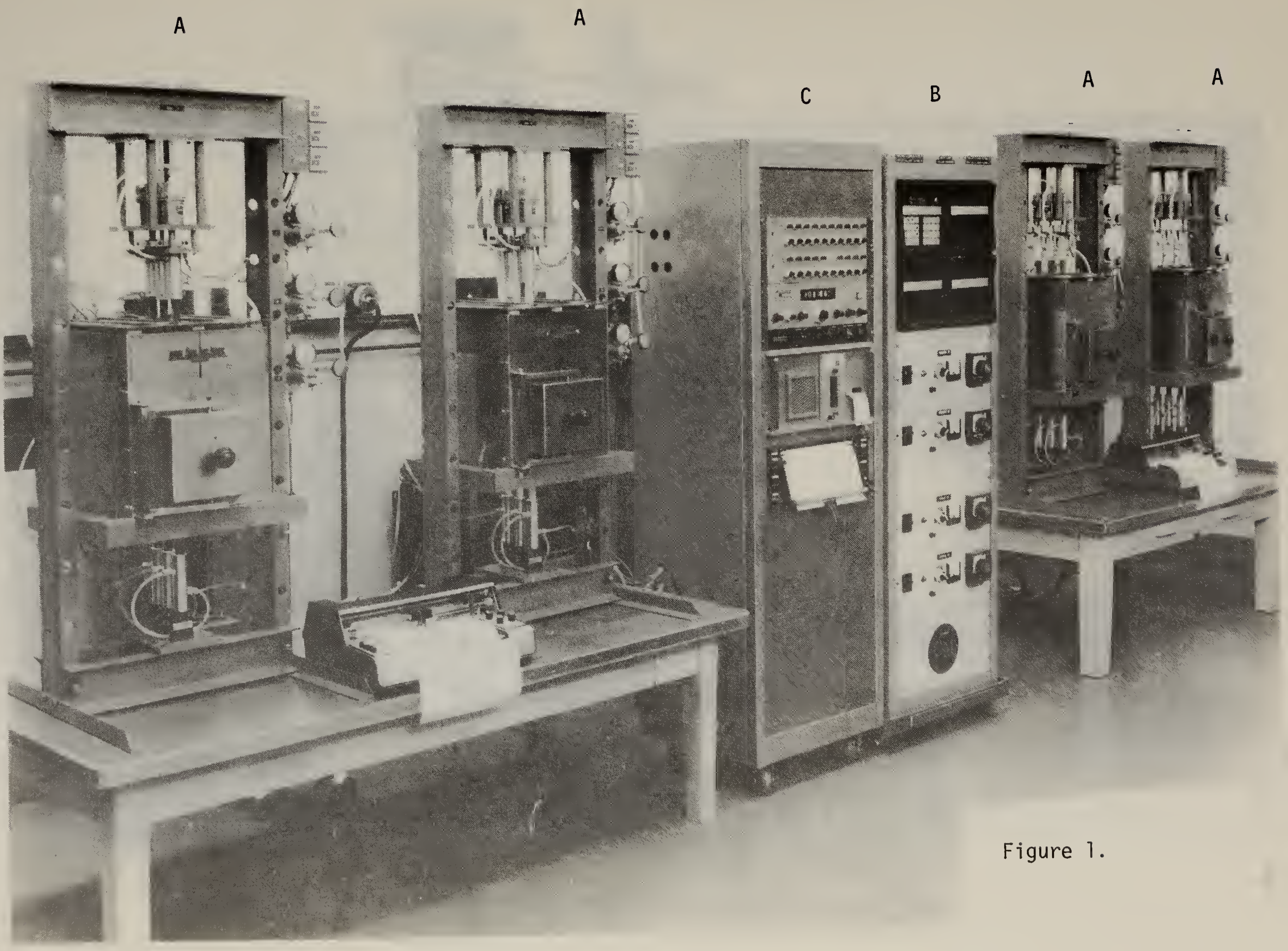
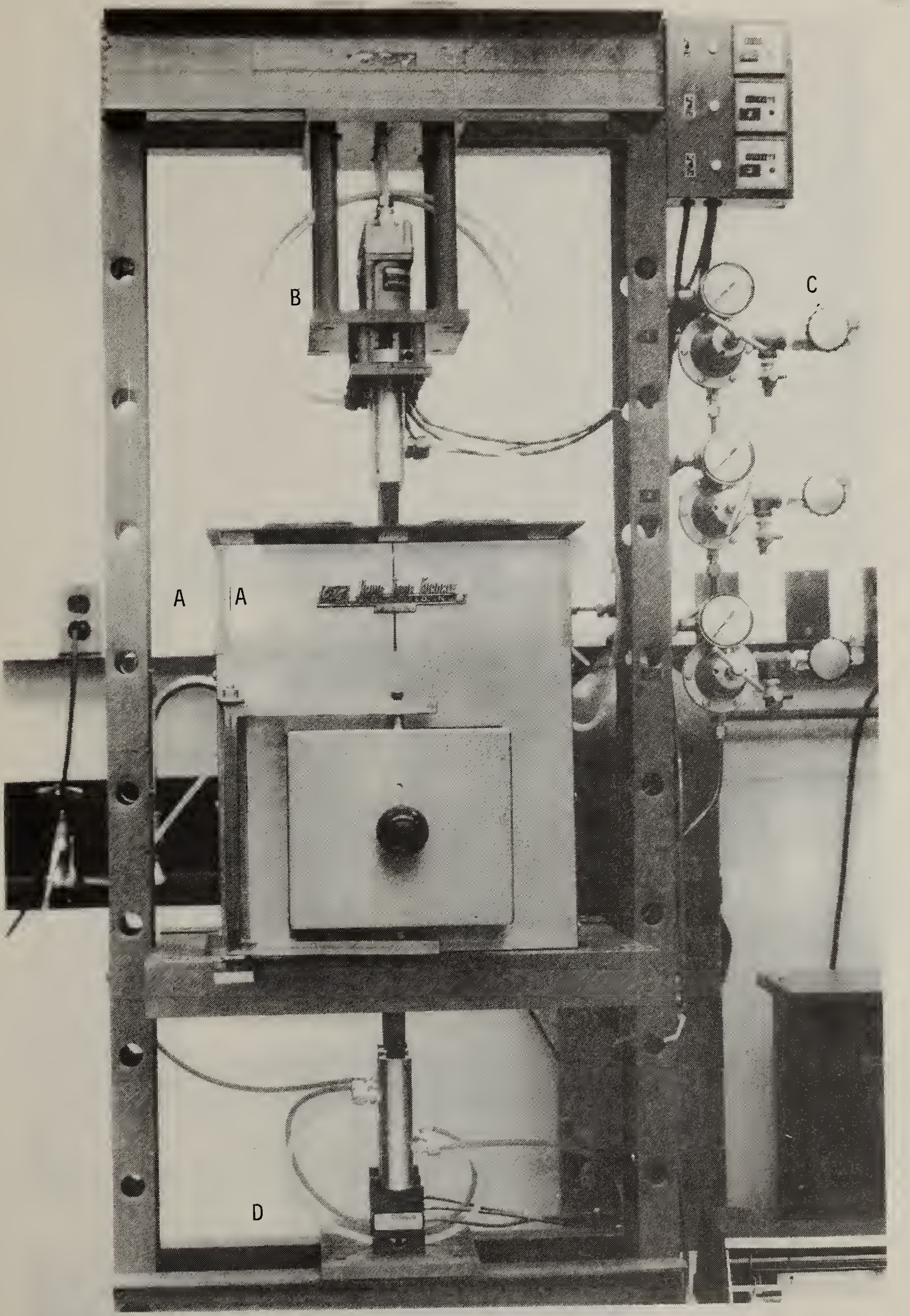


Figure 1.

Figure 2



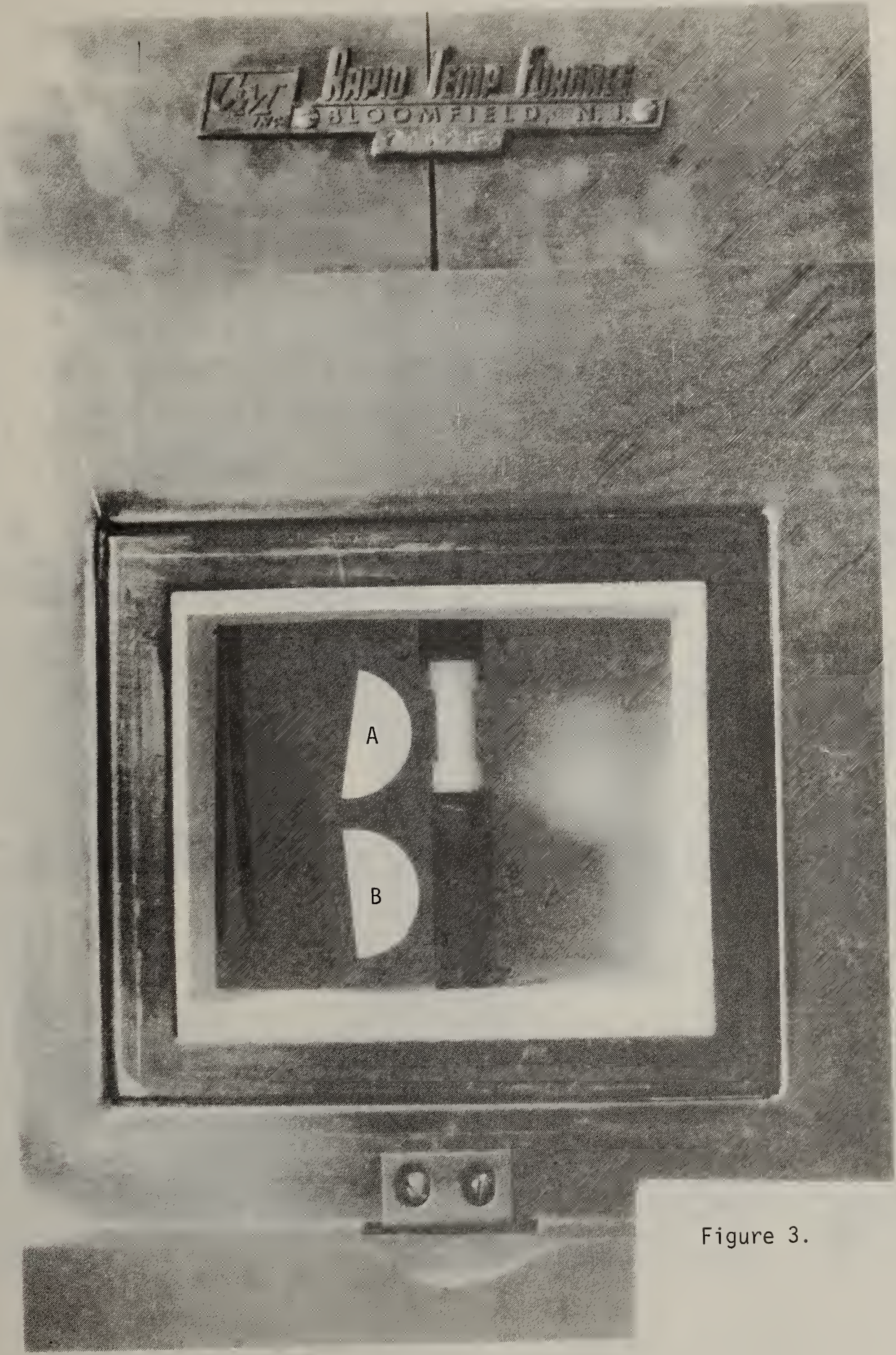


Figure 3.

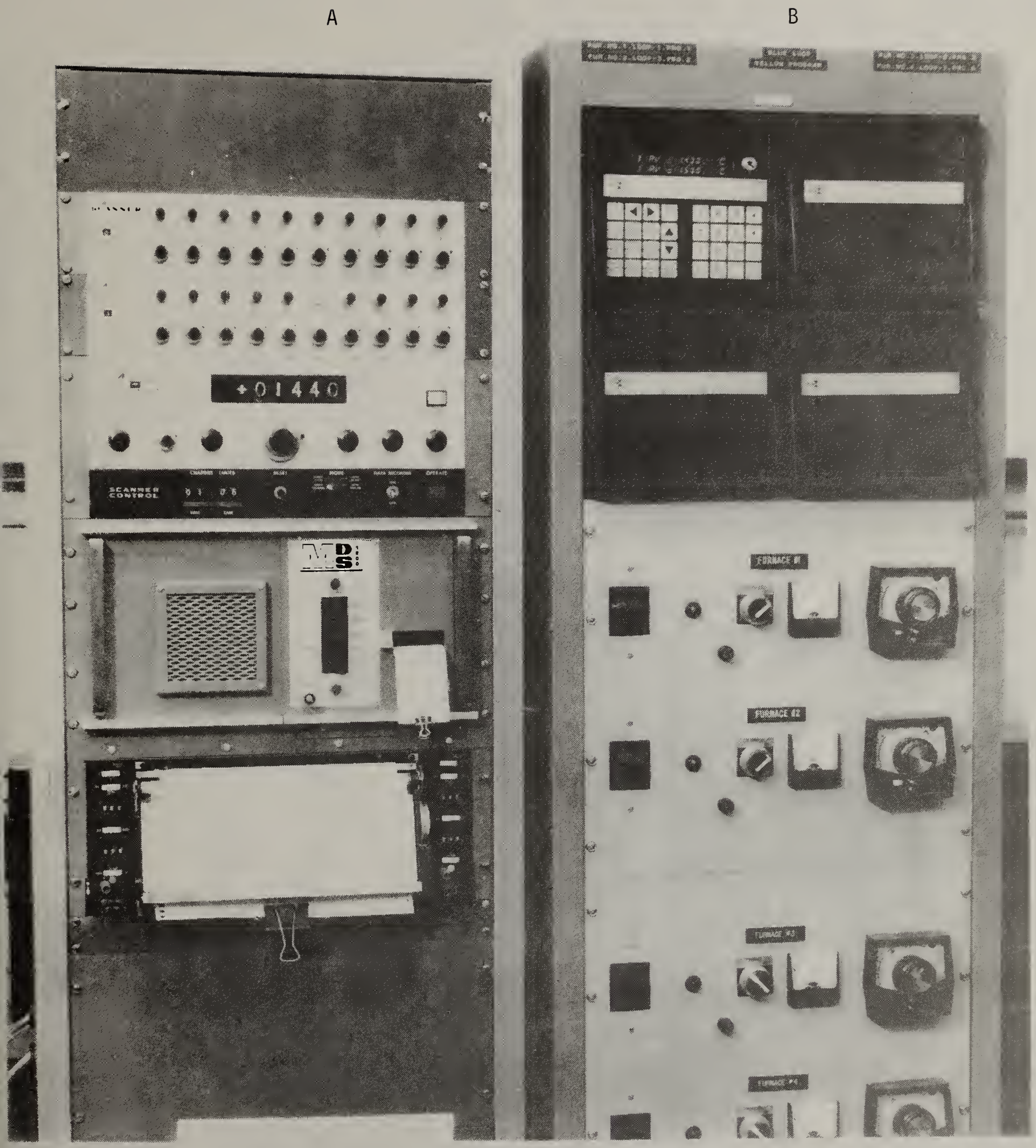

A
B
+01440
SCANNER CONTROL
FURNACE #1
FURNACE #2
FURNACE #3
FURNACE #4

Figure 4.