NBS SPECIAL PUBLICATION 468

U.S. DEPARTMENT OF COMMERCE / National Bureau of Standards

Prevention of Failures in Coal Conversion Systems

MFPG
24th Meeting

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[2] Located at Boulder, Colorado 80302.

MFPG
Prevention of Failures in Coal Conversion Systems

Special publication, no. 468

Proceedings of the 24th Meeting of the
Mechanical Failures Prevention Group,
held at Battelle, Columbus Laboratories,
Columbus, Ohio, April 21-23, 1976

Edited by

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Washington, D.C. 20234

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FOREWORD

The 24th meeting of the Mechanical Failures Prevention Group was held April 21-23, 1976, at Battelle, Columbus Laboratories in Columbus, Ohio. The program was organized by the MFPG committee on the State-of-Art and Application under the chairmanship of Professor F. F. Ling of the Rensselaer Polytechnic Institute. The State-of-Art committee, the session chairmen and especially the speakers and panel participants are to be commended for the excellent program.

The papers in these Proceedings are presented as submitted by the authors on camera ready copy, except for some minor editorial changes. In addition to the papers, the Proceedings include the discussions of the talks and a panel discussion. The discussions were recorded at the meeting and have been edited to improve readability.

Special appreciation is accorded Battelle, Columbus Laboratories, and in particular to Mr. Robert E. Maringer of Battelle, for hosting the meeting. Mr. Maringer was responsible for the excellent meeting arrangements.

Appreciation is extended to Mr. T. Robert Shives and Mr. William A. Willard of the NBS Metallurgy Division for their editing, organization, and preparation of the Proceedings, to Ms. Susie Armstrong and Mr. Tom Stewart of Battelle for general coordination, to Mr. Paul M. Fleming of the NBS Metallurgy Division for handling financial matters, to Mr. W. Todd Eudy of the NBS Metallurgy Division for photographic work, and to many members of the staffs of Battelle, Columbus Laboratories, the NBS Metallurgy Division, and the NBS Institute for Materials Research for their assistance in many ways. Special thanks are accorded to Mrs. Marian L. Slusser of the NBS Metallurgy Division for her diligent efforts in transcribing and typing the recorded discussions.

HARRY C. BURNETT
Executive Secretary, MFPG

Metallurgy Division
National Bureau of Standards

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SESSION IV: MATERIALS PROBLEMS IN COAL CONVERSION PILOT PLANTS

The following registrants gave presentations at the symposium, but did not submit a manuscript for publication:

G.M. Ugiansky* and C.E. Johnson, "Constant Strain Rate Stress Corrosion Testing of Metals at Elevated Temperatures," Session III

D. Canonico, "Pressure Vessels," Session III

E.R. Fuller, "Effect of Hydrothermal Environments on the Strength of Castable Refractories," Session III

* Indicates speaker when a paper had more than one author.

ABSTRACT

These Proceedings consist of a group of twenty submitted papers and discussions from the 24th meeting of the Mechanical Failures Prevention Group which was held at Battelle, Columbus Laboratories in Columbus, Ohio on April 21-23, 1976. Prevention of failures in coal conversion systems is the central theme of the Proceedings. A series of overview lectures dealing with reliability problems in coal conversion systems, economics of failures in energy generating systems, corrosion, and gaps in engineering data are presented. In addition, failure analysis, materials problems, and related materials research are discussed.

Key Words: Coal conversion; coal gasification; coal liquefaction; corrosion; failure analysis; failure prevention; quality assurance; reliability

UNITS AND SYMBOLS

Customary United States units and symbols appear in many of the papers in these Proceedings. The participants in the 24th meeting of the Mechanical Failures Prevention Group have used the established units and symbols commonly employed in their professional fields. However, as an aid to the reader in increasing familiarity with and usage of the metric system of units (SI), the following references are given:

NBS Special Publication, SP330, 1974 Edition, "The International System of Units."
ISO International Standard 1000 (1973 Edition), "SI Units and Recommendations for Use of Their Multiples."
E380-75 ASTM/IEEE Standard Metric Practice (Institute of Electrical and Electronics Engineers, Inc. Standard 268-1976).

Disclaimer:

SESSION I

OVERVIEW

LECTURES

CHAIRMAN: E. PASSAGLIA,
NATIONAL BUREAU OF STANDARDS

RELIABILITY PROBLEMS IN COAL GASIFICATION AND LIQUEFACTION

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Abstract: Coal gasification and coal liquefaction are two generic techniques for converting coal to environmentally acceptable gaseous and liquid fuels and chemical feedstocks. The two techniques are distinctly different in many ways but, in many others, use numerous similar components.

Significant reliability problems exist in coal conversion. The most important aspect of the problem is the absence of a domestic commercial industry in coal conversion. Reliability data from commercial operations do not exist and must be generated from the next best source: ERDA pilot plants and demonstration projects.

This paper describes both the advantages and problems in reliability resulting from the absence of a commercial industry. Critical interfaces are described between the reliability effort and mission-oriented offices in ERDA responsible for pilot and demonstration projects.

The paper describes failure avoidance progress conducted, under ERDA sponsorship, at the national laboratories, government laboratories and in private industry. Recent progress is described in development of a performance assurance approach to eoal conversion plant design.

Plans for the future are outlined.

Key words: Coal gasification; coal liquefaction; failure analysis; performance assurance system; reliability.

Statement of Problem

One of the most significant aspects concerning coal conversion is that no domestic industry exists. This sole fact has several ramifications which must be appreciated fully to understand the critical role played by the reliability effort in ERDA's plans.

The absence of a domestic industry means there are no commercial plants either gathering reliability data now or that could be candidate sites for gathering such data in the near future. Since data do not exist on which to base reliability (and hence productivity improvements), steps must be taken to design reliability into the equipment, to achieve a satisfactory level of conversion plant reliability.

If reliability and maintainability disciplines had been fully exercised in the design and operation of present-day coal burning power plants, their capacity factors would be substantially better than the ~60% presently demonstrated. This capacity factor is an average value for large ($>390MW_e$) fossil plants owned by a veritable host of public and private utility companies. Power plant data were collected by FEA (Ref.1).

No such data base exists in coal conversion. Foreign technology exists in indirect liquefaction, as for example the coal conversion complex at Sasolburg, Republic of South Africa, but reliability data are not available.

Absence of a domestic industry means that reliability data must be obtained from other appropriate sources and used in design, development and operation of the pilot plants and demonstration projects. Virtually all of these are sponsored wholly or in part by ERDA.

It is appropriate to review the operational purposes of ERDA pilot plants. They are not operated to generate failure and failure analysis data, but rather to generate chemical process data at an operational scale needed by industry to decide upon the commercial viability of a process. For example, in a liquefaction process, the pilot plant would determine the operating conditions and product mixes for different coal sources. From these data, process economics data could be computed and commercial viability determined.

Evaluation of an emerging process at a pilot plant level also generates materials and equipment information. It is necessary to take advantage of all the technological data generated to provide a baseline for improvements and to protect the investment of hundreds of millions of capital dollars for each coal conversion demonstration plant.

The reliability problem is aggrevated by one additional problem stemming from absence of a commercial industry: that suppliers of components can claim that their materials and components will operate in new environments because no contrary evidence exists.

This attitude is true in many cases. The reliability analyst recognizes that the task is to determine, from the standpoint of an integrated plant, those components for which the above conclusion applies and those for which it does not.

A second aspect of the problem of reliability takes advantage of the fact that a commercial industry does not exist. This aspect calls for incorporation of reliability into the original design of future coal conversion plants and a step-by-step action series designed to ensure that reliability procedures are implemented in construction and operation of future plants.

The Need for Reliability

An immediate question arises as to why ERDA requires a reliability program in coal conversion plants when such closely associated industries as petroleum refining generally lack such formalized programs. Coal conversion pilot plants have severe hardware problems that prevent the accomplishment of sustained runs (Ref.2). Many pilot plant runs are considered to be successful if they attain 100 hours of continuous, steady state operation. In contrast, petroleum refineries typically operate for 25,000 hours between shutdowns, and these shutdowns are scheduled. Furthermore, scheduled shutdowns are not for the prime purpose of repairing structural components, but for rejuvenating or replacing the highly sensitive catalyst beds. Admittedly, the shutdown period is used to re-seat valves, resurface pump parts and inspect many areas for the appearance of unsuspected erosion, corrosion and wear.

The problem is how to achieve 25,000 hours-operation in coal conversion. Coal conversion plants might reach this level of operability if the nation could wait 100 years, the period of time required by the petroleum industry to acquire data permitting long-term plant operability.

During that period, plant operability was gradually enhanced in the face of increasingly difficult and complex refining operations.

The benefit of a reliability program is its provision for accelerated path toward optimized plant operation (Figure 1). The conventional development line shows plant operability improving gradually over a period of many years. The accelerated development line shows that high levels of operability are achievable in a much shorter time by efficient utilization of the R&D phase of an emerging technology-based industry.

The nation needs an accelerated program effort because it cannot afford to spend years of precious time on a learning curve. That extended effort must be compressed into one that will give real payoffs by 1985.

Comparison of Operating Conditions in Petroleum Refining and Coal Conversion

Key operating parameters are compiled in Table 1 for selected conversion processes from both currently available commercial technologies practiced in foreign countries and second generation technologies under ERDA development (Ref.3). The conditions are unique; they differ from those encountered in petroleum technology. The environments are abrasive because suspended solids are present in virtually every part of any process. The solids are unreacted coal (char) and ash. Both can be extremely abrasive (Ref.4).

The amount of suspended material is much higher than in petroleum technology. A typical crude petroleum after minimal cleaning (primarily to remove sea water) contains perhaps 0.04% ash. In contrast, the <u>product</u> from the Solvent Refined Coal Process (SRC), an advanced liquefaction process, contains nominally 0.5% ash. This necessarily means that higher ash contents were present at all areas upstream of the final stage in the liquefaction plant. Gasification plants (Ref.4) typically encounter abrasive solids with velocities of ~100 ft/sec (~ 33 m/s). The concentration of suspended solid material can be 0.3 lb/100 scf of gas (Ref.5). Both aspects are more abrasive than current petroleum operations.

The gaseous environment is both unique and corrosive, as shown in Table 1. H_2S causes sulfidation attack on all ferrous and heat-resisting alloys. This attack can be particularly catastrophic in nickel-rich alloys because NiS can form. This compound melts at 800°C (1472°F) and removes any protective scale that may have existed. Evidence that

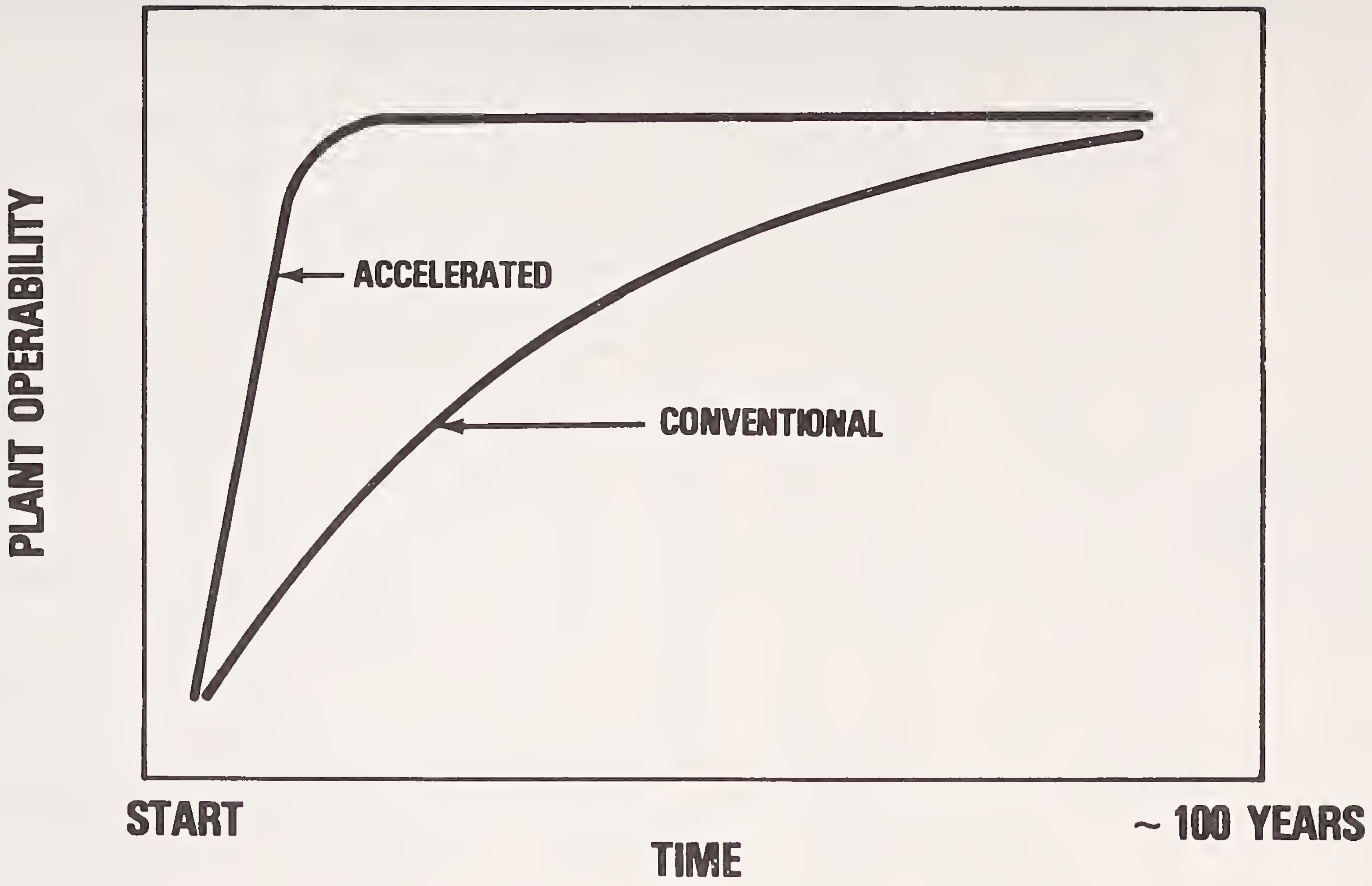


FIGURE 1. CONVENTIONAL VERSUS ACCELERATED APPROACHES TO ACHIEVING OPTIMIZED PLANT OPERABILITY

TABLE 1. CONDITIONS IN COMMERCIAL AND SECOND GENERATION COAL CONVERSION PROCESSES

COAL CONVERSION PROCESS	REACTOR * TEMPERATURE (°F)	REACTOR PRESSURE (psi)	REACTOR ENVIRONMENT (MOLE FRACTIONS)					COMMENTS
			CO	CO_2	H_2	CH_4	H_2O	
COMMERCIAL GASIFICATION								
• LURGI	1400	450	9	15	20	5	50	IN ALL CASES, THE ENVIRONMENT CONTAINS H_2S, NH_3, ASH AND SOME UNREACTED COAL (CHAR). THE AMOUNTS DEPEND ON THE COAL SOURCE.
• KOPPERS-TOTZEK	3300	14.7	50	6	33	0	10	
• WINKLER	1850	14.7	26	16	32	2	23	
ADVANCED GASIFICATION (HIGH BTU)								
• HYGAS	1750	1000	20	19	21	16	21	
• BI-GAS	2700	1500	23	7	13	8	48	
• SYNTHANE	1800	1000	10	18	18	15	37	
• CO_2 ACCEPTOR	1900	300	14	6	45	17	17	
ADVANCED LIQUEFACTION			C	H_2	N_2	S	O_2	
• SRC	815	1000	88	5	2	1	3	

*MAXIMUM TEMPERATURE IN HIGHEST TEMPERATURE STAGE OF REACTOR.

sulfidation is a significant root cause of component failures is accumulating from failure analysis work. (This will be shown later.) H_2O is corrosive, and large amounts of H_2O are present in the off-gas streams of all gasification processes (Table 1). The high temperatures pose severe conditions when it is recalled that the maximum allowable metal temperature in a commercial jet engine is 1600°F and that this level was made allowable only after extensive testing and characterization.

It must be observed that the temperatures in Table 1 are maximum reaction or, in other cases, adiabatic flame temperatures rather than material temperatures. This is fortunate because most iron-nickel-chromium heat-resistant alloys melt near 2250°F (1232°C). They would be totally unacceptable, for example, in the Koppers-Totzek gasifier if it were not for its ceramic liner and good heat transfer.

The chemical environments outlined in Table 1 are new and represent the range of operating conditions to be expected in the new coal conversion industry. The conditions are more corrosive and abrasive than those found in the petroleum industry. It can be expected that many hardware items used in petroleum will be unacceptable in coal conversion.

Comparison of Reliability Needs with Those in Fossil-Fired Power Plants

The FEA has made a concerted effort to determine reasons for large fossil-fired power plants having capacity factors of ~60%. The capacity factors can be improved significantly by reducing the failure frequency and attendant repair times, but knowledge of the real failure causatives is required before equipment reliability and maintainability can be improved. The results of an FEA-sponsored study are summarized in Table 2 and show that the major number of outages and outage hours is due to boiler problems, and not of the turbine or even the electric generator (Ref.1). The utilities seem to generally recognize that the problem, as stated, is in the boilers and that, if allowed to continue, will become a major investment problem. Five power plants will be required to provide the energy supplied by three good plants.

Some failure analyses, including root cause effects, are conducted at ERDA pilot plants, and the results are compiled at the Bureau of Standards (Ref.6). A recent tabulation (Table 3) included an examination and assessment of the failure analysis data generated by ERDA contractors. Several interesting aspects are in Table 3. The most frequent cause of a failure remains "undetermined." The next most frequent is sulfidation, and erosion (physical abrasion) is almost as

TABLE 2. MAJOR FORCED OUTAGES: 600 MW AND LARGER FOSSIL-FIRED PLANTS

AREA OF POWER PLANT	AVERAGE EVENTS PER UNIT YEAR	AVERAGE OUTAGE HOURS PER UNIT YEAR
BOILER-GENERAL	2.0	146
– WATER WALLS	1.1	91
– GENERATING TUBES	0.2	21
– SUPERHEATER	0.4	40
– REHEATER-FIRST	0.3	32
– EXPLOSION	0.04	34
STEAM TURBINE-GENERAL	0.2	38
– BUCKETS	0.07	40
– VIBRATION	0.7	51
– LUBE	0.2	57
– MISC	0.2	31
GENERATOR-GENERAL	0.1	116
– STATOR WINDINGS	0.07	59
– EXCITOR	0.1	32
– MISC	0.1	34
BOILER FEED PUMPS	0.8	32

TABLE 3. FAILURE FREQUENCY TABULATION

CAUSE OF INCIDENT	NUMBER	CO_2	HYGAS	SRC	SYNTHANE	LIGNITE	CLEAN COKE
UNDETERMINED	12	2	4	4	2		
SULFIDATION	11	10	1				
EROSION	10	3		5	1		1
CORROSION	8 (TOTAL)						
WATER	6	1	3	1	1		
$CO\text{-}H_2$	1						1
CARBURIZATION	1						
DESIGN DEFECT	7(TOTAL)						
GENERAL	4	1		3			
THERMAL*	3		2	1			
CHLORIDE ION STRESS-CORROSION CRACKING	5	3	1			1	
FABRICATION	4 (TOTAL)						
WELDING	2	1	1				
RESIDUAL STRESS	2		2				
QUALITY CONTROL	1				1		
REFRACTORY SPALLING	1	1					

*THERMAL - INDUCED STRESSES

high. The data are only now beginning to be compiled at the Bureau, and are updated on a daily basis. The established trend suggests that many failures are simply a result of poor quality control, poor fabrication, and design defects, and can be corrected by implementing a performance assurance system. Alternatively, there also appear to be an even larger number of failures in corrosion, erosion and sulfidation, requiring materials research coupled to strict management control.

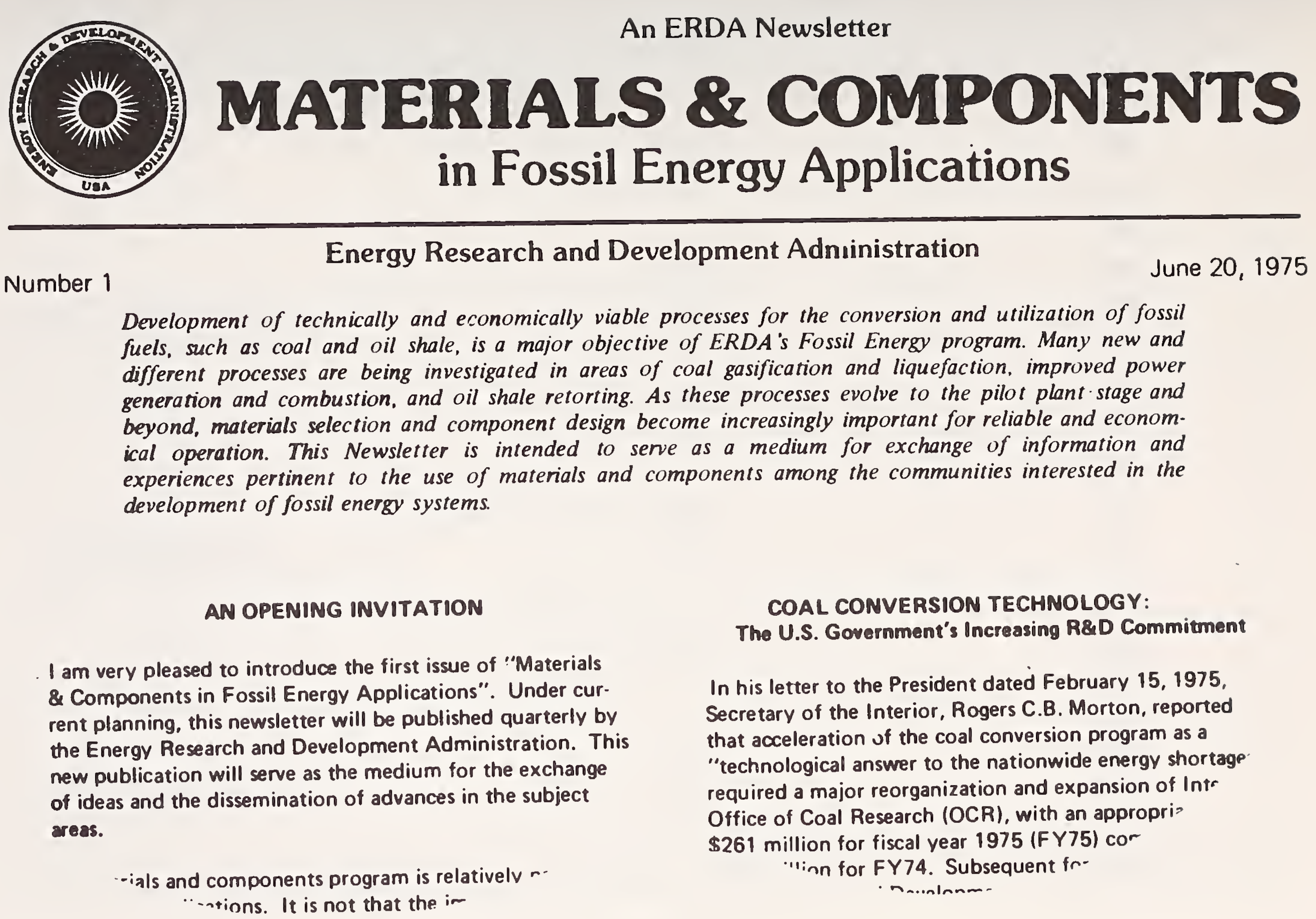
The Bureau's effort is part of an ERDA program to disseminate information on failure root causes. A newsletter (Figure 2) now being published has about 1000 subscribers, over 60% of whom are in private industry.

Test Data

A recent series of short-term burst tests were conducted to determine whether a coal gasification atmosphere (CGA) had any significant effect on the mechanical properties of a heat-resistant steel. Cylinders of 310 stainless steel were pressurized for one hour at 1800°F (982°C) and then further pressurized until rupture occured (Table 4). The gas was air in one case and a CGA in the other. The latter had a distinct effect; the burst strength decreased measurably when the metal was exposed to CGA compared to air, although exposure was only one hour. The data suggest that the materials behavior in a complex H_2O - NH_3 - H_2S - CH_4 - H_2 - CO_2 - CO environment must be determined experimentally.

A Computer Analysis

Another complementary approach was adopted to evaluate the impact of component reliability on operation of an integrated plant. A computer analysis was conducted, and a schematic flow sheet was used to lay out a series of valves, pumps, compressors, pipes, heaters, and to show connections among the components (Ref.7). Because data were absent, failure rates were estimated for each component in the system. Failure rates of 1×10^{-2}/hour, 1×10^{-3}/hour and 1×10^{-4}/hour were then assigned to one component, a hydrogen plant, and the computerized system model was run for each assigned valve. The results (Figure 3) show that the probability of completing 200 hours of operation without failures is nearly zero when the hydrogen plant failure rate is 10^{-2}/hour. The same curve shows it has only a 13% chance of surviving 100 hours of operation without failure. This is because the system being modeled has its components in series, and the low failure rate of 1×10^{-2}/hour for the hydrogen plant component with respect to the other system components has a large impact on system failure rate. By the same token, failure rates of 1×10^{-3}/hour and 1×10^{-4}/hour for the hydrogen plant significantly decrease the failure rate of the system. However, it should be realized



An ERDA Newsletter

MATERIALS & COMPONENTS
in Fossil Energy Applications

Energy Research and Development Administration

Number 1

June 20, 1975

Development of technically and economically viable processes for the conversion and utilization of fossil fuels, such as coal and oil shale, is a major objective of ERDA's Fossil Energy program. Many new and different processes are being investigated in areas of coal gasification and liquefaction, improved power generation and combustion, and oil shale retorting. As these processes evolve to the pilot plant stage and beyond, materials selection and component design become increasingly important for reliable and economical operation. This Newsletter is intended to serve as a medium for exchange of information and experiences pertinent to the use of materials and components among the communities interested in the development of fossil energy systems.

AN OPENING INVITATION

I am very pleased to introduce the first issue of "Materials & Components in Fossil Energy Applications". Under current planning, this newsletter will be published quarterly by the Energy Research and Development Administration. This new publication will serve as the medium for the exchange of ideas and the dissemination of advances in the subject areas.

ials and components program is relatively
ations. It is not that the

COAL CONVERSION TECHNOLOGY:
The U.S. Government's Increasing R&D Commitment

In his letter to the President dated February 15, 1975, Secretary of the Interior, Rogers C.B. Morton, reported that acceleration of the coal conversion program as a "technological answer to the nationwide energy shortage required a major reorganization and expansion of Int
Office of Coal Research (OCR), with an appropri
$261 million for fiscal year 1975 (FY75) co
ion for FY74. Subsequent f

FIGURE 2. ERDA NEWSLETTER

TABLE 4. MECHANICAL PROPERTIES OF ALLOY 310 IN COAL GASIFICATION ATMOSPHERES AT 1800 °F

PROPERTY	AIR ATMOSPHERE	CGA ATMOSPHERE*
BURST STRENGTH (PSI)	13,580 13,110	11,910 12,450 12,270
AVERAGE	13,340	12,210
TOTAL CIRCUMFERENTIAL ELONGATION (TCE)	25 15	23 16 22
AVERAGE	20	20
BURST PRESSURE (PSI)	1500 1450	1320 1380 1360
AVERAGE	1450	1350

*39% H_2O, 1% NH_3, 0.5% H_2S, 5% CH_4, 24% H_2, 18% CO_2, 12% CO

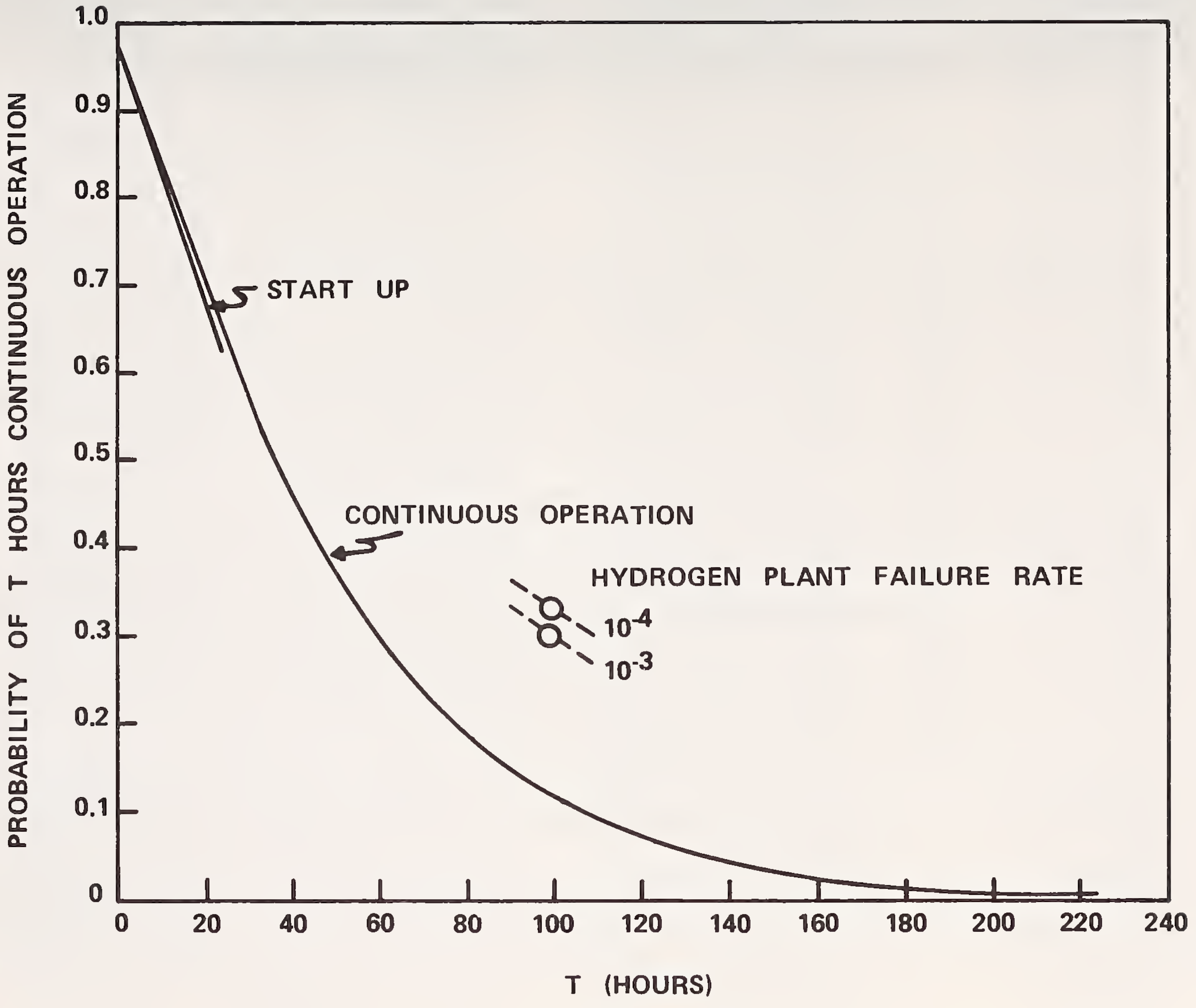


FIGURE 3. PROBABILITY OF A HYPOTHETICAL COAL CONVERSION PLANT OPERATING FOR T HOURS

that failure rates must be extremely low in order to achieve a reasonable acceptable level of system reliability.

The data and procedure used in Figure 3 represent simplified versions of an actual commercial plant.

The results of the study indicate a severe reliability problem requiring detailed evaluation and determination of failure rates for individual components to improve plant operability. Also of great significance is the fact that this type of analysis can be of inestimable assistance in pointing the road to more relevant and significant R&D programs.

A Performance Assurance System

The objective of any failure prevention activity is reduction of the numerous problems in maintaining plant operation. The current failure reporting, analysis, and corrective action efforts disclose problems in real-time operation, and as such are a significant input to the goal of improved plant reliability and operability. However, more can be done to achieve this goal. Efforts can and should be applied during the design and development (and later) phases that will expose potential problems before operation and simultaneously improve the data base and cost aspects of the design.

Once the nature of a problem has been determined, corrective steps can be taken; hence the importance of failure reporting and analysis on operating equipment. However, to avoid the problem, recommendations center on the objective of determining potential failures through early analysis during the program, and incorporating design and/or procedure changes to obviate or reduce the problem. This activity should be followed by feedback from operations to correct problems previously unexposed. The elements of concern are reliability, maintainability, availability, standardization, quality assurance, cost, and service life. These features affect plant or equipment performance. The disciplines and activities controlling these performance features form the major elements of a Performance Assurance System (PAS) that is recommended for application to all ERDA Fossil Energy projects. The success of such a system will depend upon industry's willingness to accept and cooperate. The benefits, as shown in Table 5, are numerous to all concerned.

TABLE 5. PERFORMANCE ASSURANCE SYSTEM FOR DESIGNED-IN-RELIABILITY

ELEMENT	PRIMARY OBJECTIVE
RELIABILITY	CONTINUOUS OPERATION WITHOUT UNSCHEDULED SHUTDOWN.
MAINTAINABILITY	REDUCES COST TO MAINTAIN PLANT AT DESIGN LEVEL.
AVAILABILITY	PLANT OPERATES ON THE SCHEDULE SET BY MANAGEMENT.
STANDARDIZATION	MAXIMUM USE OF COMMON, PROVEN HARDWARE.
QUALITY ASSURANCE	CUSTOMER OBTAINS THE PLANT PRODUCTS HE PAID FOR.
OPERATIONAL SERVICE LIFE	ENSURES THAT HARDWARE LIFE SPAN IS CLOSE TO DESIGN LIFE SPAN.
LIFE CYCLE COSTS	EMPHASIZES TOTAL COST OF OWNERSHIP RATHER THAN ONLY ACQUISITION COST.

The ERDA Program

The reliability problems in coal conversion have been presented. The Materials and Power Generation Group's current programs toward achievement of reliable plant operation will now be presented. There is a continuing need for support of all concerned to accomplish the program goal. Table 6 presents the activities of the Materials and Power Generation Group in Fossil Energy Research. The programs are performed in national laboratories, government laboratories, and in private industry. The following paragraphs summarize the effects.

Failure Analysis and Prevention Program

- Analyzes and catalogs pilot plant materials and component failures
- Advises pilot plants on failure prevention
- Indicates areas for materials and components research and development

Materials and Components Newsletter

- Disseminates information regarding ERDA/FE programs
    1. Highlights coal conversion processes
    2. Announces and updates programs
    3. Distributes failure prevention knowledge
- Distribution free on request - 900-1000 subscribers: 61% private industry: 21% government: 12% universities: 6% government-owned, contractor-operated facilities

Programs Sponsored by ERDA and Performed at the National Laboratories

- Oak Ridge
    1. Feasibility Study on Prestressed Concrete Pressure Vessels (PCPV): determine potential of PCPV in coal conversion systems and establish feasibility of use.

TABLE 6. MATERIALS AND POWER GENERATION GROUP ACTIVITIES

THE MATERIALS AND POWER GENERATION GROUP IN FOSSIL ENERGY RESEARCH:

- SUPPORTS MISSION-ORIENTED PROJECT MANAGERS IN ERDA
    - HYGAS
    - BI-GAS
    - SRC
    - SYNTHANE
    - HYDRANE
    - SYNTHOIL
    - CO_2 ACCEPTOR PROCESS
    - H-COAL
    - SELF-AGGLOMERATING ASH
    - CLEAN COKE
- SUPPORTS MATERIALS EVALUATIONS FOR COAL CONVERSION
- SUPPORTS GOVERNMENT CONTRACTORS IN NDT PROGRAM
- GATHERS AND ANALYZES FAILURE DATA
- DISSEMINATES FAILURE DATA VIA AN ERDA NEWSLETTER
- CONDUCTS MATERIALS DEVELOPMENT WORK

2. Technology assessment of Coal Conversion Pressure Vessel and Piping Materials: assess status of technology in materials selection for coal conversion pressure vessels and piping.

3. Inspection Techniques for Wear- and Process-Resistant Coatings for Use During Fabrication and Operations of Large-Scale Plant: develop equipment and procedures for in situ non-destructive testing of coating materials used in coal conversion processes.

4. Iron and Nickel Carbonyl Formation and Prevention: determine conditions under which iron and nickel carbonyls form when CO is in contact with pipe steels, and develop methods of economically controlling their formation.

- Argonne

  1. Evaluation of Ceramic Refractories for Slagging Gasifiers: evaluation of currently available refractories under conditions existing in slagging gasifiers.

  2. Evaluation of Ceramic Coatings: evaluation of ceramic coatings and inlay refractories for erosion and corrosion resistance in coal gasifiers.

  3. Nondestructive Testing for Coal Conversion Plant Components: evaluation of testing methods that can provide existing pilot plants with life estimates of materials and components, that can be extended for use in demonstration and commercial gasification plants.

  4. Corrosion Behavior of Materials in Coal Conversion Processes: use of thermodynamic data and concepts to develop models for corrosion in coal gasification atmospheres.

  5. Erosion Behavior of Materials in Coal Gasification Processes: formulation of analytical models for predicting material loss due to erosion/corrosion of components used in coal gasifiers.

- Sandia

  1. Formation of Protective Carbide and Sulfide Layers on Alloys in Coal Gasification Environments: explore possibilities of forming protective carbide and sulfide layers by the addition of strong carbide and sulfide formers to existing high temperature alloys.

Programs Sponsored by ERDA and Performed at Government Laboratories

- Bureau of Mines

  1. Development of Improved Valve Materials for Coal Feeding and Char Removal in Coal Gasification Processes: develop improved valve materials using powder metallurgy, casting, electro-deposition, and chemical vapor deposition technique, and subsequent evaluation for wear, abrasion, erosion, corrosion, and shock resistance in atmospheres simulating coal feeding and char removal.

  2. Improved Ceramic Liners for Coal Gasification Process Vessels: determine corrosive effects of the principal constituents of coal gasification atmospheres on selected refractories; determine feasibility of centrifugal casting liners for transfer lines; investigate means of reducing spalling and cracking in ceramic liners.

- National Bureau of Standards

  1. Metal Corrosion: develop and evaluate testing techniques that determine the susceptibility of materials to stress corrosion cracking in environments encountered in coal conversion technology.

  2. Metal Wear: investigate the mechanisms of metal wear from particle impingement, and develop test methods for evaluation of this type of wear.

  3. Ceramic Deformation and Fracture: evaluate strength and crack propagation in various refractories suitable for use in coal gasifiers.

  4. Erosive Wear: evaluate erosive wear of ceramic materials to be used in high wear areas of gasifiers and study of the mechanisms of particle erosion in ceramics.

Programs Sponsored by ERDA and Performed in the Private Sector

- Program to Discover Materials Suitable for Service Under Hostile Conditions Obtaining in Equipment for the Gasification of Coal and Other Solid Fuels (with Metal Properties Council)

  1. Phase I - High Temperature Corrosion: laboratory corrosion testing of various candidate alloys for gasifier internal

components in simulated coal gasification atmospheres at elevated temperatures and pressures.

2. Phase II - Pilot Plant Exposure: evaluation of metallic and ceramic materials exposed to various locations in operating pilot plants.

3. Phase III - Aqueous Corrosion Testing: laboratory corrosion testing of alloys in aggressive environments simulating quench systems in coal gasification plants.

4. Phase IV - Erosion-Corrosion Testing: laboratory erosion/corrosion testing of metals and refractories at high temperatures and pressures in coal gasification atmospheres.

5. Phase V - Engineering Properties: determination of physical and mechanical properties of selected alloys in and after exposure to coal gasification atmosphere at elevated temperatures and pressures.

- Fireside Corrosion

    1. Task I - High Temperature Gas Turbine Engine Components Materials Testing Program (with General Electric Company): determine durability of selected gas turbine nozzle and bucket materials in gas environments from the combustion of coal-derived low Btu gas and coal-derived fuel oil.

    2. Task II - Evaluation of Heat Exchanger and Turbine Materials for use in a Coal-Fired Fluidized Bed Combustion Environment (in contract negotiation process): testing materials suitable for heat exchanger tubes, superheater tubes, and turbine vanes and blades in coal-fired fluidized beds at both atmospheric and elevated pressures.

    3. Task III - Effect of Impurities in Coal Derived Fuels on Service Life of Boiler Tubes for Advanced Power Cycle Application (with Combustion Engineering): both laboratory and field testing of commercial and advanced experimental materials and coatings under design conditions simulating advanced fuel power cycle combinations.

- Sulfidation Resistant Alloy for Coal Gasification Service (with Lockheed Missile and Space Company): design of an iron base alloy having a corrosion rate less than 20 mpy in coal gasification atmospheres with the mechanical properties, fabricability, producibility and weldability of 300-series stainless steels but containing less than 10% Cr and Ni.

- Preparation of a Data Book on High Temperature Oxidation and Corrosion of Metals and Alloys in Electrical Generation Systems (with Battelle Columbus Laboratories and Professor Stringer at University of Liverpool: co-sponsored with EPRI).

Programs Current in Procurement Cycle

- Correlation of the High-Temperature Corrosion Behavior of Structural Alloys in Coal Conversion Environments with the Components of the Alloys and of the Corrosive Environments: establish a statistical correlation between alloy corrosion rates and gas composition, temperature, and pressure and the level of major alloying elements in alloys to be used for internal components of coal gasifiers.

- Determination of the probability of Alkali Degradation and Corrosion of Refractories and Metals in Various Coal Gasification Processes: determine the amount of alkali vapor in the atmosphere of various coal gasification processes in order to assess the probability of alkali degradation and corrosion of refractories and metals in the gasification environment.

- Improvement of the Mechanical Reliability of Monolithic Refractory Linings for Coal Gasification Process Vessels: develop improved materials, lining designs, and operating procedures for monolithic refractory linings of gasifier process vessels in order to reduce or eliminate cracking during curing, drying, and heat-up of the refractory lining.

- Study of Heat Transfer Through Refractory-Lined Gasifier Vessel Walls: develop empirical heat flow/thermal conductivity models for multi-component refractory-lined gasifier walls suitable for use in the design of commercial refractory linings for coal gasification vessels.

- Development of Valves Compatible with Gasifier Plant Conditions: develop and establish the mechanical integrity of families of valves compatible with operating conditions encountered in the various coal gasification processes: discover and describe new problem areas, if any; provide data required for optimization so as to minimize plant costs and maximize reliability and maintainability.

Acknowledgements

The authors acknowledge the assistance of many individuals who provided data and reviewed ideas, budgets and data. Dr. John H. Smith,

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DISCUSSION

D. Livaccari, Coalcon: You mentioned that there are coal gasification projects in some foreign countries. Has there been any effort to obtain data from these foreign sources?

H. E. Frankel: Yes, efforts have been made to get this information. However, you must remember that we are in competition with these people; therefore, they are reluctant to share their data with us. The Lurgi process is by far the major industrial process used in Europe. I can cite an example that may shed some light on the reliability of this process. In the southwestern United States, the El Paso consortium is going to build plants to produce 250 million cubic feet of synthetic gas per day. In order to do this using the Lurgi process, 34 plants would have to be built. Only 27 plants (each producing 10 million cubic feet) would be on line at any given time. The additional seven plants are a reserve or redundant feature. When close to one-fourth of the total plants in a system are inoperative at any one time, I would consider the reliability of the plant to be rather poor.

ECONOMICS OF FAILURES IN ENERGY GENERATION SYSTEMS

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Abstract: Capital equipment costs and efficiency of fuel utilization are well recognized criteria in the selection of new electrical generation capacity. The service reliability of the equipment, as measured by some term which reflects both the productivity of the investment and the consequential cost impacts of failure is an equally important, albeit somewhat more elusive, consideration. The purpose of this paper is to illustrate what forms of 'failure' can be encountered with utility generation equipment and what the consequential impact of these 'failures' is on the cost of electrical generation. This background material, which is specifically developed for large, baseload generation units, is then used to describe some suggested orientations for failure prevention in advanced technology systems.

The economics of equipment failures in utility generation systems are reflected in the cost to the consumer of electric generation. However, the impacts of the failure--and, indeed, the definition of what constitutes a "failure"--must be assessed in several ways that are dependent on the nature of the generation unit and of the system in which it exists. The issue is further complicated because the economic impact will vary significantly depending on when the failure prognosis occurs; failure assessment during acquisition planning is a different situation than true failure during operations.

To unravel this fairly complex introduction for MFPG members, it appears desirable to consider only a large (nominally 800 - 1300 MWe) generation unit intended for true baseload operation. These are the type of units, based on either coal or nuclear energy sources, that are expected to dominate the new capacity market until about the year 2000. A utility will begin the planning and acquisition phase for these units some 10-15 years ahead of need time as defined by a forecast of load and reserve margin requirements. This single unit may represent from 6%-30% of the baseload capacity owned by the utility when its operation is initiated.

Procurement decisions concerning new generating facility additions are usually based on evaluations that determine the lowest overall cost of producing electricity. Initial cost, estimated capacity factor, fuel cost and operation and maintenance cost are expressed by an equation of the form:

$$(1)\quad C_G = \frac{1000 \times A \times B}{8760 \times C_f} + F + (O \,\&\, M)$$

Where: C_G = Generation cost in mills/kwh

A = Annual levelized fixed charge rate (typically 15% - 17%)

B = Capital cost of the unit in $/kw

F = Fuel cost in mills/kwh

O & M = Operation and Maintenance cost in mills/kwh

C_F = Unit capacity factor

Figure 1, a brief parametric study based on equation 1, illustrates the relation between capacity factor and generation cost for a range of equipment and fuel cost combinations believed to be of future interest. The data are normalized to a capacity factor of 0.8.

A utility nominally selects a new baseload unit addition anticipating that it will provide the lowest achievable incremental generation costs for its system. Theoretically, it would like to run this unit "all the time" at full-rated power (CF = 1.0). However, this ideal situation cannot be met. Scheduled maintenance or modification time must be considered; equipment malfunctions will occur; excessive first costs may be associated with high levels of redundancy needed to mitigate the impact of malfunctions. In addition, the utility may anticipate discretionary power reductions such as load following. Thus, the target capacity factor for large baseload units is normally in the range of 0.75 to 0.9 (without provision for load following). Accordingly, the utility will plan a major scheduled outage for preventive maintenance, on the order of once a year, ideally at a time when minimum demand exists. Assuming that the utility has selected a nominal target value capacity factor of 0.8, a failure can be defined as any equipment malfunction, or excessive maintenance time, or regulatory issue mandating modification which will prevent this value from being achieved. This may appear to be an odd definition of failure; but it leads to the correct emphasis on productivity of an investment in generating capacity which is the concern of the utility at this point. There are circumstances where true equipment failures can be avoided by higher frequency of maintenance; there are other circumstances where equipment failures can be repaired without loss of generating capacity. In classical reliability analyses all failures might be given equal weight. Following these cases through to their productivity impact, as measured by capacity factor, provides the more balanced view which is required. Figure 1 permits a direct evaluation of the cost of failures in the aforementioned context. Because the generation cost of current design and future plants appears dominated by capital cost, the impact of capacity factor variation is obviously quite large. Typically, a 10 point capacity factor variation will have a consequential impact of approximately 10% on total generation costs.

Utility procurement evaluation interests focus on determining: the relative capacity factor values for ranking generation alternatives; what failure causes are applicable to each alternative; and finally, what relation exists between first cost adders and the prospects for failure mitigation. Some of these questions are easier to answer than others. However, before considering them in further detail, it is appropriate to consider alternate dimensions of the economic impact of failure.

The approximate situation that a utility faces in its acquisition program is suggested by the pattern of capacity additions in response to increasing customer demand shown as Figure 2. When the new generation unit is first brought on line it may provide a higher degree of reserve margin than actually required in the system. At this time, (point A) the utility might be able to run the unit below its nominal capacity factor without excessive penalty. Later, (point B) when the unit becomes fully committed, an unanticipated outage mandates that replacement power be acquired. The added economic impact of failure now becomes the cost differential which may exist between purchased and generated power. The purchase premium (which results because systems rarely have lowest cost generation output available as surplus) might be from 2-4 times the purchasers own cost of generation. The actual cost for a day's outage of a 1000-MWe unit in this case could easily amount to a half million dollars (assuming a cost of 20 mills/kwh for baseload generation). So the cost of replacement power is the second important dimension of failure economics. It usually becomes dominant when the loss of the unit cannot be accommodated by the owners system reserve.

A related case is a failure to bring the new unit on line at the scheduled time. For a one-year delay, the extreme result might be that the consequential cost of power purchases is 30%-50% of the unit capital cost. In addition, the delay will probably increase unit capital cost by some 5%-10%; the reason for this being that the time-dependent charges (e.g., interest during construction, escalation) currently account for of the order of 30-50% of the plant capital cost.

The preceding examples illustrate how failure to achieve performance on a timely basis yields extremely large consequential costs in the electric utility industry. The factors noted exert a major influence on the character of major programs in which utilities can participate and on the rate at which specific developments will penetrate the utility market. The role of failure prevention disciplines, which is of more immediate interest to MFPG members, can be assessed by reconsidered utility deliberations in the context of Equation 1. The utility would like to know: a) what capacity factor should be applied over the total service life of the unit (nominally 30-40 years), and b) is it reasonable to assume that the capacity factor will follow an orderly predictable pattern, typically with provision for an initial maturing phase, if the system is adequately maintained? On first principles, these questions focus on deterioration modes which might foreshorten service life and on the reliability and maintainability of the operating system.*

Coal-fired units, including advanced approaches such as combined cycle options, are not generally thought of as containing major service life foreshortening elements. The most critical items in this regard are turbomachinery, wherein extremely long duration outages would be dictated should major blading or rotor damage occur. There are many steam turbine assessment programs underway on this basis. These include as examples, stress corrosion behavior of turbomachinery materials in the presence of trace impurities in steam, moisture impingement effects, and cyclic fatigue behavior; all as a means of establishing the least deleterious operating modes. Presumably direct-fired turbines will require analogous efforts as extensions of present activities for smaller-size peaking units.

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*A foreshortened service life would normally be reflected in an increase in the term 'A' in Equation 1. 'A' includes depreciation.

The utility's capability to assess the unit capacity factor is nowhere near as good as it should be. There is much folklore as well as a growing body of technical literature on the subject (1,2) which cannot be easily encapsulated for this paper. However, a brief review of some salient issues may be of interest. Figure 3, a plot of the cumulative lifetime capacity factor of domestic nuclear generation units, is a point of departure. The units represented range from 200 MWe to 1000 MWe, have an 'average' service life of approximately four years and an age distribution covering approximately 1-14 years. What is immediately evident is that an 'average' value does not offer a good description of the situation. Instead, a wide distribution of performance exists. There are good units which meet target expectations. There are also poor performers. When discrimination studies are performed the reasons for the spread do not turn out to be easy simplifications. The age or the size of the unit, for example, are perhaps less important than the perception of value some 10-15 years prior to operation - or the ability to predict a current regulatory climate from this same distant vantage point. Thinking a moment about our own capability to forecast economic and political climates on a similar time scale, it seems difficult to fault anyone for having missed the right (now) trade-off between first cost and maintainability or redundancy. The same assessment applies to regulatory issues, the fairly exclusive domain, until recently, of nuclear units.

The technical results of the discrimination study can best be likened to a complex 'learning experience.' Typically, various 'campaigns' are required to solve those significant engineering problems revealed only through operating experience. Concurrently, the utility is acquiring the information needed to plan and implement maintenance or to diagnose malfunctions. The picture is admittedly 'fuzzy' because several units with the same ills may have been committed while a 'campaign' was in process and because the transfer of operating experience in and among suppliers is not easily accomplished. Nevertheless, a maturity trend can be identified when sufficient statistics become available. Figure 3, which shows evidence of performance improvement following the experience of first refueling, is representative.

At least a partial apology to MFPG members interested in coal gasification is in order for the use of nuclear statistics for illustration; (in part because of the author's background, as well as the pragmatics of more readily available codified data). But, in fact, the conditions are reasonably representative of what any new generation introduction might anticipate--and, at least at the aggregate level, the performance of present large, coal-fired capacity is not notably different than that for nuclear units (Figure 4). One reason for this similarity is that all generation units have many elements more or less in common--and, perhaps to the surprise of some, it is these common elements which are responsible for much of the capacity loss encountered. The approximate historical situation for the locales of nuclear capacity loss shown as Table 1 illustrates this point.

Also, at the risk of overgeneralizing, the orientation for those interested in failure prevention in advanced systems can also be extracted from either of the trend illustrations. The essential goals of failure prevention are to get the performance distribution function to be as high as practicable with the lowest mean deviation as fast as possible. To do this, it is obviously necessary to emphasize high reliability equipments and high standards of production quality control. But, particularly in the case of the aforementioned common equipments, one is invariably already dealing with items which have achieved such stature (as evidenced by the statistical mean-time-between failures (MTBF) which they can be assigned.) So, the thrust here is to reduce the maintenance time required, to provide the information which permits the most economic spares and replacement policy to be determined, and to provide the technical basis for mitigating the infrequent high impact outage which might occur. Good in-service failure precursor instrumentation is an example of what is needed in the latter case. For those subsystems where 'campaigns' might be anticipated, it is again obviously necessary to insure that the initial design implementation reflects the best levels of technology available. But, it is equally important to provide a sound technical basis for diagnosis and high creditability modification and retrofit for in-service equipment as rapidly as possible.

Many of you will recognize that the preceding comments imply accent on the maintainability aspects of product performance optimization (3) rather than exclusively on those reliability approaches which emphasize MTBF per se. Perhaps the most significant reasons for this assertion is again an economic consideration; in this case, the amount of capital which is placed 'at risk' in a new generation technology before mature operating experience is acquired. This point is made graphically by Figure 5, a representative study of the status of units entering service as a function of calendar time for a fixed construction term and schedule of manufacturing capability build-up. What is most significant is the very large amount of capital which must be committed before any statistically significant, long-term operating experience is available to verify the investment judgment.

One can think of many ways to try and warp this situation, but it invariably turns out that such exposure is inherent in any approach which aims at making an appreciable impact on the nation's electrical generation capability. Given such circumstances, the capability for rapid, incisive analysis of operating experience and feedback to retrofit, design and operating actions is perhaps the highest leverage tool for investment justification available.

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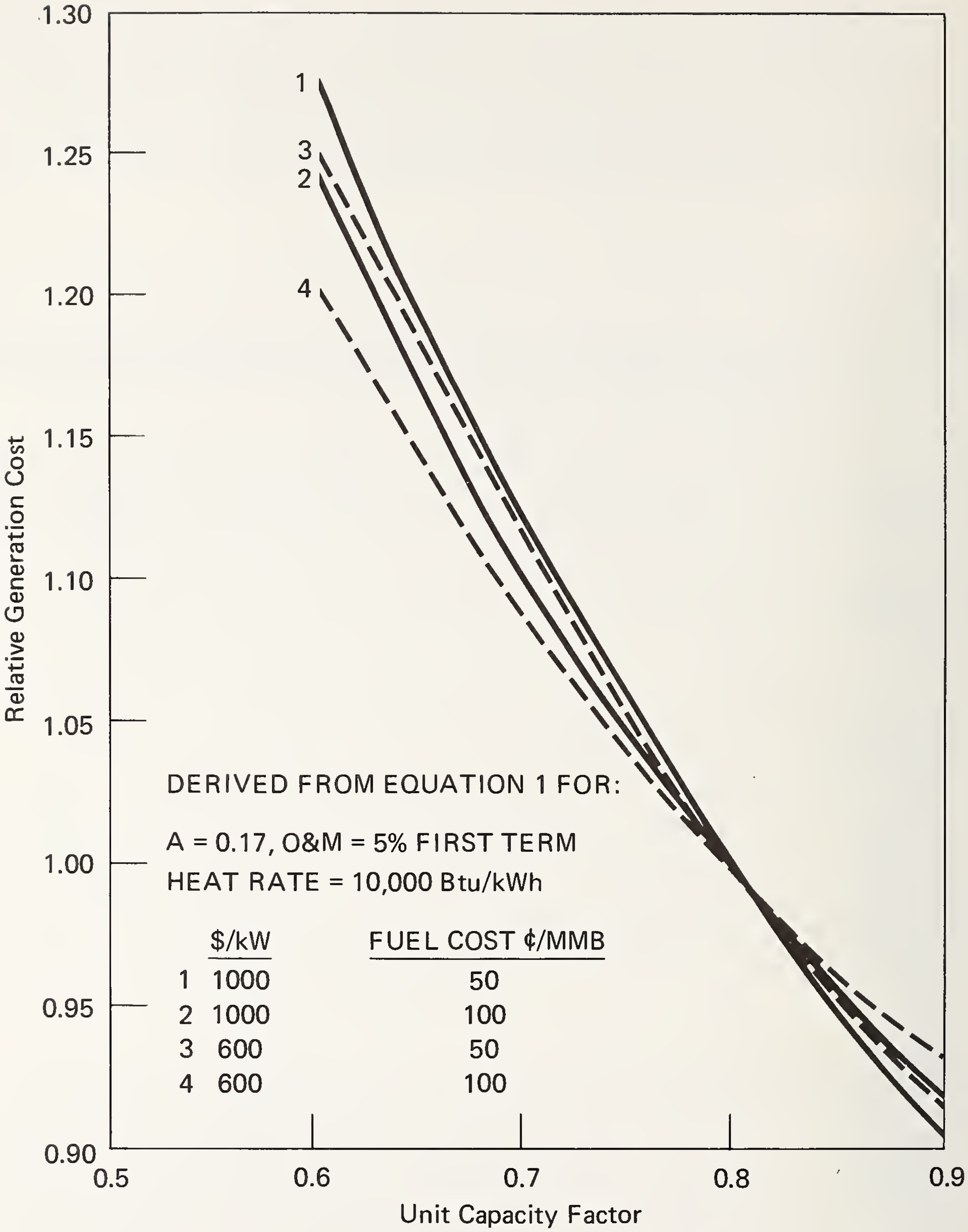


Figure 1 Plot of Relative Cost of Electric Generation Versus Capacity Factor (C_F); Normalized to C_F = 0.8

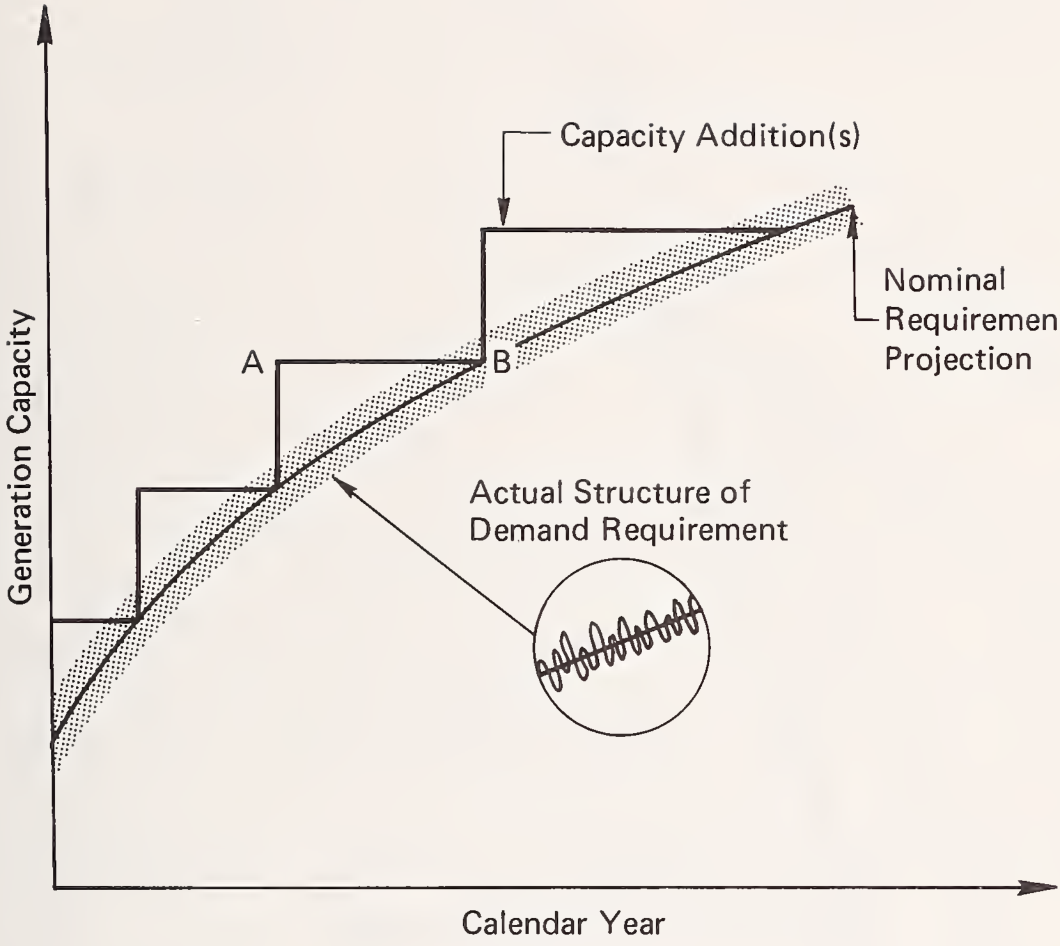


Figure 2 Typical Pattern of Utility Generation Capacity Additions

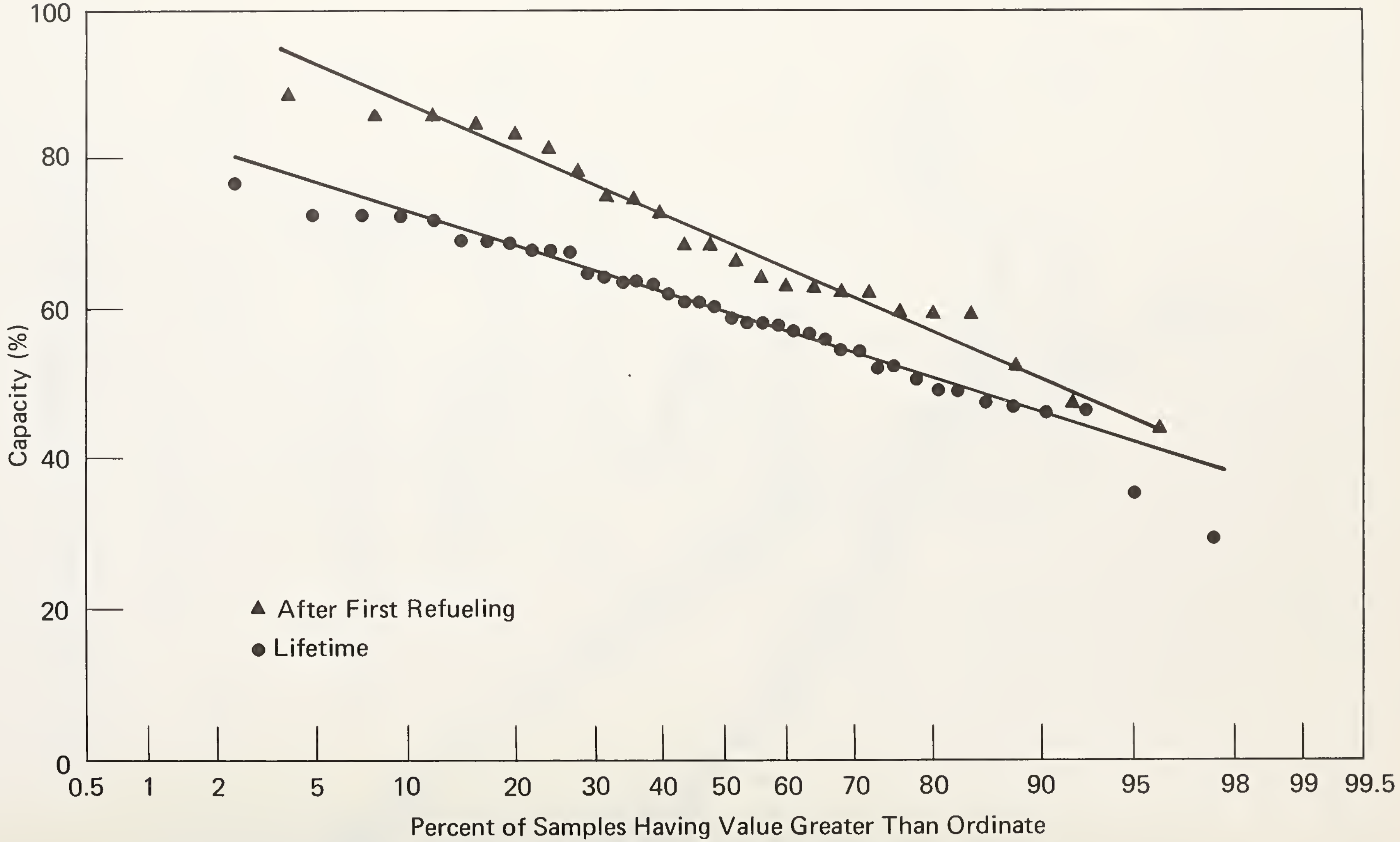
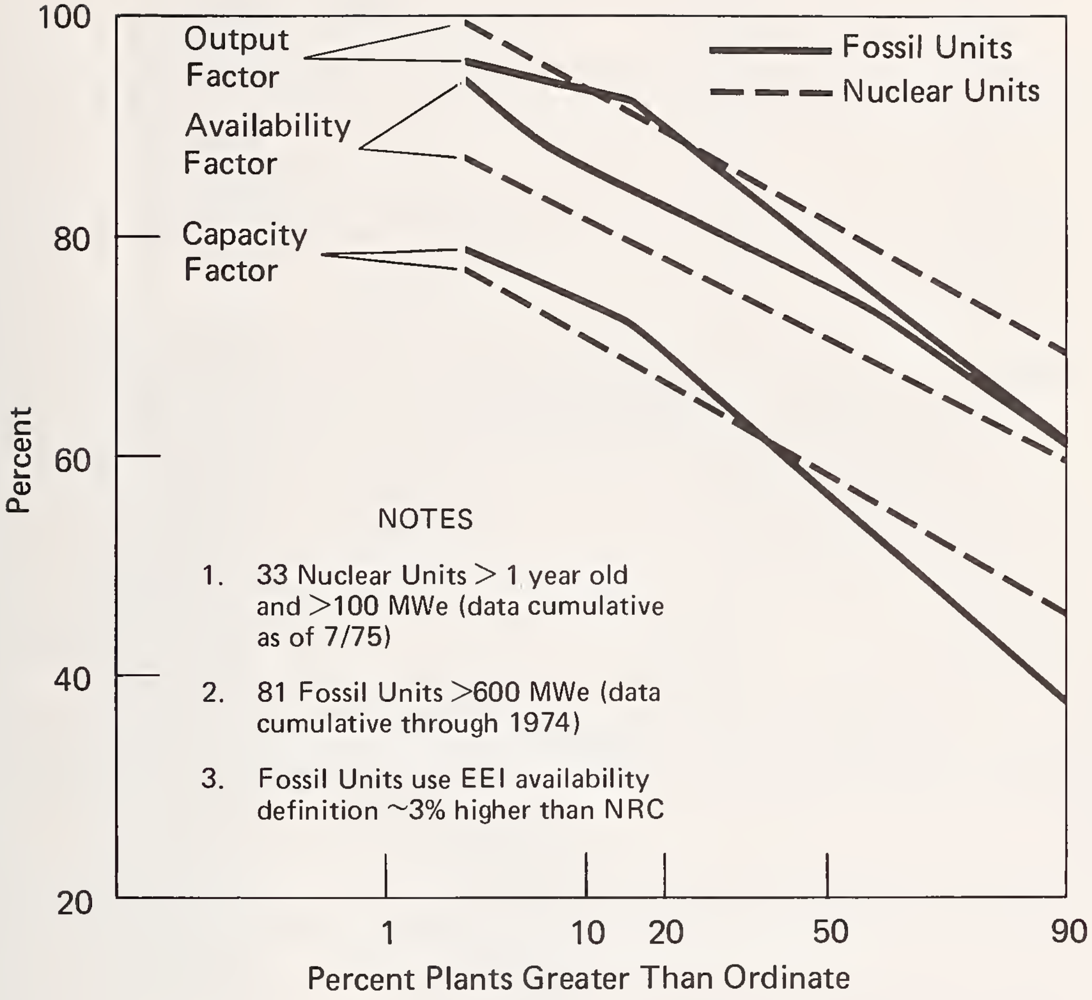


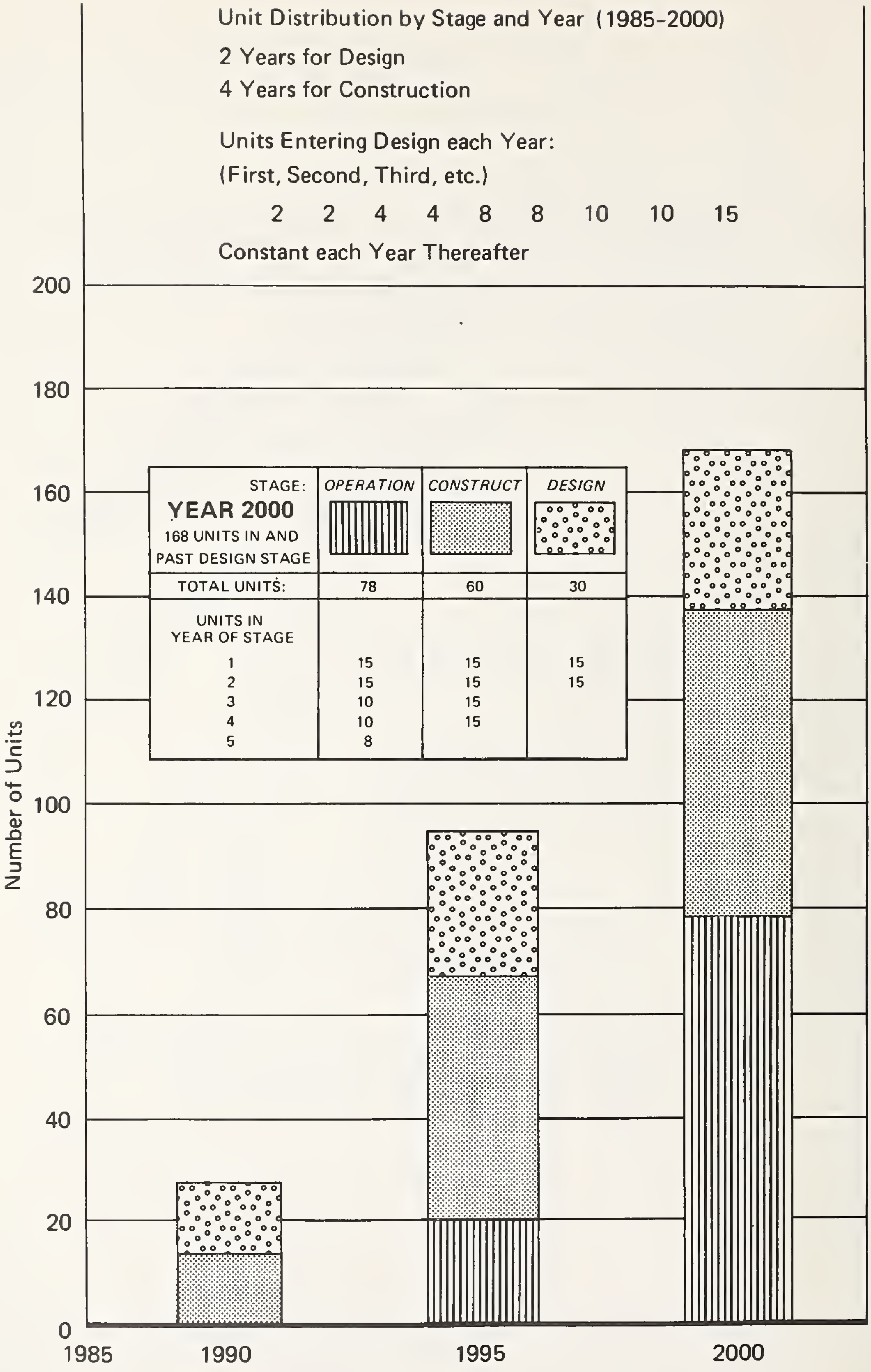
Figure 3 Comparison of Cumulative Lifetime Capacity Factor and Capacity Factor after First Refueling as of January, 1976



NOTES

1. 33 Nuclear Units > 1 year old and >100 MWe (data cumulative as of 7/75)
2. 81 Fossil Units >600 MWe (data cumulative through 1974)
3. Fossil Units use EEI availability definition ~3% higher than NRC

Figure 4 Comparison Plot of Cumulative Lifetime Performance Factors for Large Nuclear and Fossil Units



STAGE: **YEAR 2000** 168 UNITS IN AND PAST DESIGN STAGE	*OPERATION* ▥	*CONSTRUCT* ▒	*DESIGN* ⁘
TOTAL UNITS:	78	60	30
UNITS IN YEAR OF STAGE			
1	15	15	15
2	15	15	15
3	10	15	
4	10	15	
5	8		

Figure 5 Representative Illustration of Build-up of New Generation Capacity

ITEM	DURATION (hours)	% TOTAL
Forced Outage (Equipment Malfunction)		
Turbine/Generator	140	4.7
Condenser	124	4.2
Steam Generator	189	6.3
Pumps	60	2.0
Valves	132	4.4
Vessel & Core	75	2.5
Plant Electrical Distribution	30	1.0
All Other	310	10.4
SUBTOTAL	1060	35.5
Scheduled Outage		
Maintenance	280	9.4
'Refueling'	1500	50.2
Training & Administration	30	1.0
SUBTOTAL	1810	60.6
Regulatory	116	3.9
TOTAL	2986	100.0

Availability Factor (Based on One Year Operation Between Refueling)

$$1 - \frac{2986}{8760 + 1500} = 71\%$$

Partial Power Reductions (Approximate)

	Output Factor Multiplier to Availability
Fuel Defect Related	0.95
Equipment Failure Related	0.98
Regulatory Issues	0.96
Load-Following	0.99
Time to Come to Full Power	0.99
All Other & Unaccounted	0.97
OUTPUT FACTOR (PRODUCT)	0.85

Capacity Factor = Availability x Output Factor

71% x 0.85 = 60%

Table 1 Representative Average Outage Duration and Partial Power Reductions in Nuclear Units Through June 1975

DISCUSSION

S. S. Canja, ERDA, Fossil Energy: I have been looking at some of the FEA reports on electrical utilities. It seems that over the last 3 or 4 years the capacity or availability of new power plants has been decreasing, especially in nuclear power plants. Can you explain why that is occurring?

M. Lapides: I think that is a misconception. I do not think the availability of nuclear units has been decreasing. The availability of fossil units certainly has, for good and obvious reasons. Nuclear power plant availability has been improving quite steadily over the last few years.

S. S. Canja: What incentive does the electrical utility industry have to improve its capacity when, as you say, if there is an outage, a utility simply buys additional power from another utility and passes the costs on to the customer?

M. Lapides: In the late 1960's, I don't think the utility industry had any great motivation to build "high reliability equipment", meaning equipment that would theoretically be available 90 to 100% of the time between maintenance outages and could run at 100% capacity when it was available. The reason for the lack of improvement in the nuclear power plant industry is as follows. The nuclear power plant was a high cost generator. If one went out, the utility could turn to a cheaper coal-fired generating plant. Concerning fossil fueled plants, in the late 1960's and very early 1970's, a utility might be operating a 200 megawatt plant. In order to increase capacity the utility would buy a new unit that might have a capacity of 1,000 megawatts, or five times the capacity of the older plant. With such large reserve capacities, there was little concern for high reliability. Since 1973, conditions have changed. The rate of increased demand is slowing down, the size of the generating unit is nearly constant, utilities are strapped for cash, and they are, of course, aware of the need for energy conservation. They have a very strong motivation now that they never had before. We look for a 10% reliability improvement in the next 10 years in existing plants. That would be equivalent to three billion dollars that utilities do not have to spend. This magnitude of savings is very good motivation. I do not want to defend the utilities, nor do I want to sound glib, but I do not think it is fair to make a 1975 retrospective judgement on plant efficiencies for plants built based on decisions made in 1965.

DEMONSTRATION OF A RELIABILITY METHODOLOGY USING TWO COAL CONVERSION PLANT MODELS

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Abstract: In 1975 the Office of Fossil Energy sponsored a research program to demonstrate the use of a reliability methodology for modeling process flow in coal conversion plants. The methodology was applied to the Institute of Gas Technology HYGAS and the FMC Corporation COED pilot plants. The methodology, called GO, had previously been successfully applied to a variety of complex systems involving electronic, hydraulic and mechanical subsystems. Other applications of GO have included the human interfaces, many of which are common to several subsystems and necessary for proper system operation. This paper presents a brief introduction to the GO methodology and an overview of its application to coal gasification plants.

Key words: Coal gasification; GO reliability methodology.

Coal conversion plants are extremely complex, consisting of thousands of process elements with their associated monitoring and control instrumentation. As conceptual designs are formulated, converted into design specifications, construction is completed and plants enter into operation, continuously updated reliability assessments are necessary to insure that plant reliability goals are achieved and maintained. A highly desirable attribute of a reliability assessment methodology is the capability to model accurately the operation of a coal conversion plant, where there is a high degree of correlation between drawings and schematics and the assessment model. Such a methodology would also provide for rapid revisions in the assessment model in keeping with design changes and proposed engineering change orders. It would permit concurrent reliability assessment of these changes as they are proposed.

Over a period of 15 years a reliability methodology called GO has been developed and successfully applied to a variety of systems involving complex electronic, electrical, hydraulic and mechanical systems including their human interfaces. The GO methodology possesses the attributes previously mentioned. The primary motivation in the development of GO was to produce a computer routine which could, with a minimum of scientific labor, quickly, economically and comprehensively analyze the reliability and safety of complex hydraulic, pneumatic and electromechanical networks involving hundreds of components having two and often three or more modes of operation. Using this generalized approach the computer program, rather than the analyst as in the fault tree or equation writing techniques, systematically creates and retains the various event combinations bearing on both the central problem and all other significant system operational modes. Because the logic, other than the component interactions, is handled automatically, significant savings in scientific labor are achieved, and increased knowledge of system responses is obtained.

The GO program is an event tree procedure. Components are identified by their input signals, output signals, and probabilities of operation in different modes (success, premature and failure). The GO chart is a diagram of component interactions through the signal paths. The modeling required includes selection of the proper standard GO component to represent the physical components and the identification of signal paths. This modeling is direct and immediate since the chart can be drawn in one-to-one correspondence with the schematic, almost as an overlay. Currently there are 11 standard GO components which are used in modeling. Others are available for special applications.

The fundamental steps in a GO analysis are shown in Figure 1. The analyst working with system schematics, wiring diagrams, drawings, etc. constructs the GO chart. From the GO chart an input deck is derived, the data is processed and the results listed. For the moment I am going to skip over a step-by-step discussion of GO chart development and show you an example of one section of the HYGAS pilot plant.

Figure 2 shows a schematic of the HYGAS methanation section. Figure 3 is a GO chart for the same section. There is an almost one-to-one correspondence between the schematic and the GO chart with each major system element being modeled by a GO component. At first glance Figures 2 and 3 may appear to be extremely complex but I assure you that it is relatively simple, particularly when compared with the task of trying to write an equation for the system.

Note in Figure 2 the large number of valves with their associated sensor and control systems. A brief discussion of this combination of valve and sensor will demonstrate that the GO chart is rather simple and easy to use and will also demonstrate how the detail of the modeling can be expanded to the degree desired.

Figure 4 shows a schematic of a valve controlled by a sensor with the flow as indicated. A GO chart for this simple combination is also shown where two Type 1 components are used to model the sensor and the valve. Signal 1 represents the flow of the material. Signal 2 represents the function of the sensor and signal 3 represents the function of the valve. In this discussion let us assume that we are dealing with three time periods. If the sensor functions properly, signal 2 will be generated in time period 1; likewise if the valve functions properly signal 3 will also be generated in time period 1, which also indicates that both the sensor and valve are functioning. If this is the case, the material flow should be allowed to proceed. This is accomplished by the Type 10 component, an "and" gate, i.e., signal 4 which will occur in time period 1 now represents the continuing material flow. This could have been modeled in slightly different fashion by putting the sensor and valve in series with the process flow but our experience has shown that if we follow the schematic it becomes easier to check that all components are modeled and it is also easier to explain to people who are not familiar with GO.

Let us take the example one step further and assume that if the sensor fails a warning light goes on in the control room and an operator is dispatched to the valve and operates it by a handwheel in accordance with instructions received via telephone or two-way radio. During this operation the sensor is repaired. How do we model this back-up mode? The GO chart on the right of Figure 4 shows one approach. Signal 2 indicates the state of the sensor. This is fed into a Type 9 component representing a switch in the annunciation light circuit. Note that the main input into the Type 9 component is signal 3 representing the availability of power. The failure of the sensor triggers the switch allowing power to the warning light. The warning light is noted and if an operator is available signal 5 effectively triggers the operator who then goes to the valve and operates it. Signal 7 representing the operator and signal 2 representing properly functioning sensor are fed into a Type 2 "or" gate. If either signal 7 or signal 2 is present the valve will be actuated and we obtain signal 10 representing the material flow.

When the back-up mode is introduced the system timing can be modeled so that signal 7 will occur in time period 2. The effect of this treatment is to obtain signal 10 in time period 1 representing automatic operation and in time period 2 representing operation in the back-up mode.

A model such as this could be used to conduct experiments to determine the level of operator availability required to achieve some level of successful operation. Another point should be noted, the communication net between the control room and the operator has not been modeled nor is the warning circuit modeled in much detail. If additional detail is required the modifications can be easily and quickly added to the basic model.

Time does not permit a detailed discussion of other examples. Suffice it to say that when we examined the schematics of the two gasification pilot plants for ERDA we did not find any insurmountable problems in modeling them with GO. This is not to say that GO is a perfect modeling device. I doubt if such a thing exists, but it does have features which permit expanded evaluations over those generally available with fault tree or equation writing.

With this brief background let us now turn to results obtained in the ERDA study. Starting with failure rate data for the various elements the reliability for each component for one hour, 10 hours, 100 hours, etc., were calculated. Using these reliabilities as inputs to the GO model the probability of operating for T hours was calculated. A plot of this information is shown in Figure 5.

Figure 5 shows three basic uses of GO results. First, the area under the curve can be integrated to determine the mean time to failure of the total system. In this case the MTTF was about 47 hours. This low MTTF is a direct result of using an arbitrary failure rate of 10^{-2} per hour for a critical non-redundant hydrogen plant. We have been criticized by some people in industry for showing such low MTTF. However, our report specifically states that the 10^{-2} was our assumption. We find it necessary to repeat that we were not attempting to evaluate the reliability of any specific plant - only to demonstrate a methodology. Specific analyses would use refined data.

The next use of the GO analysis is to establish the sensitivity of total plant operations upon the reliability of one critical element or subsystem. In this case we chose the hydrogen plant and decreased its failure rate to 10^{-3} and 10^{-4}. The points plotted at 100 hours on the abscissa indicate the results. The MTTFs associated with the new failure rates are approximately 83 and 90 hours.

GO was also used to evaluate a "start-up" and "continuous operation". There is little difference in these modes, although the continuous operation is slightly more reliable than the start-up mode. Recalling the previous example with the valve it is easy to see how various operational modes can be evaluated.

GO can also be used to assist in the evaluation of continuous operation with various maintenance and repair schemes. The basic evaluation is treated as a discrete-state, discrete-time Markov process with GO being used to develop input probabilities to the general transition matrix.

In summary the project for OFE demonstrated that GO models of the process flow of coal conversion plants typified by HYGAS and COED are applicable and efficient. It has been demonstrated that GO can model various modes of operation such as start-up and continuous operation. The model is also modular in that specific subsystems can be easily omitted or added.

It has also been demonstrated that GO can be used as an experimental tool to evaluate various hypotheses relating to total system operation as a function of a single component reliability change.

In conclusion, the completion of the OFE project within a 10-week period which included significant time for report preparation illustrates that GO can be applied rapidly and economically to provide comprehensive reliability and availability assessments of coal conversion plants.

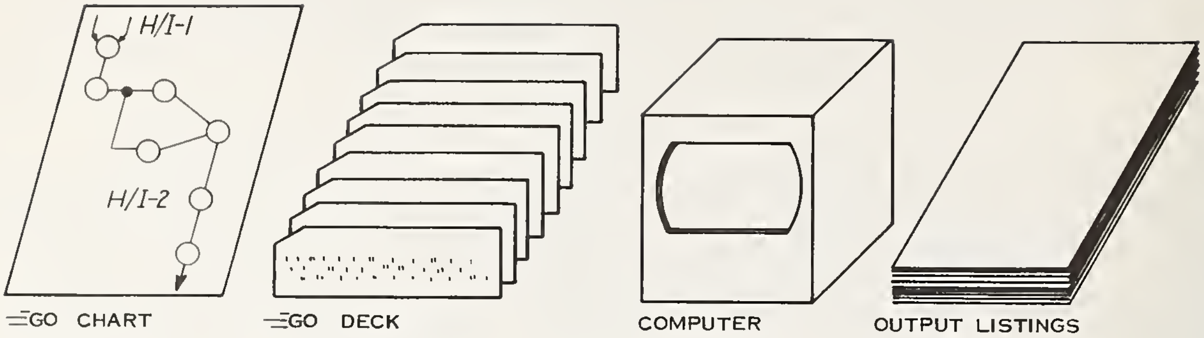

Figure 1

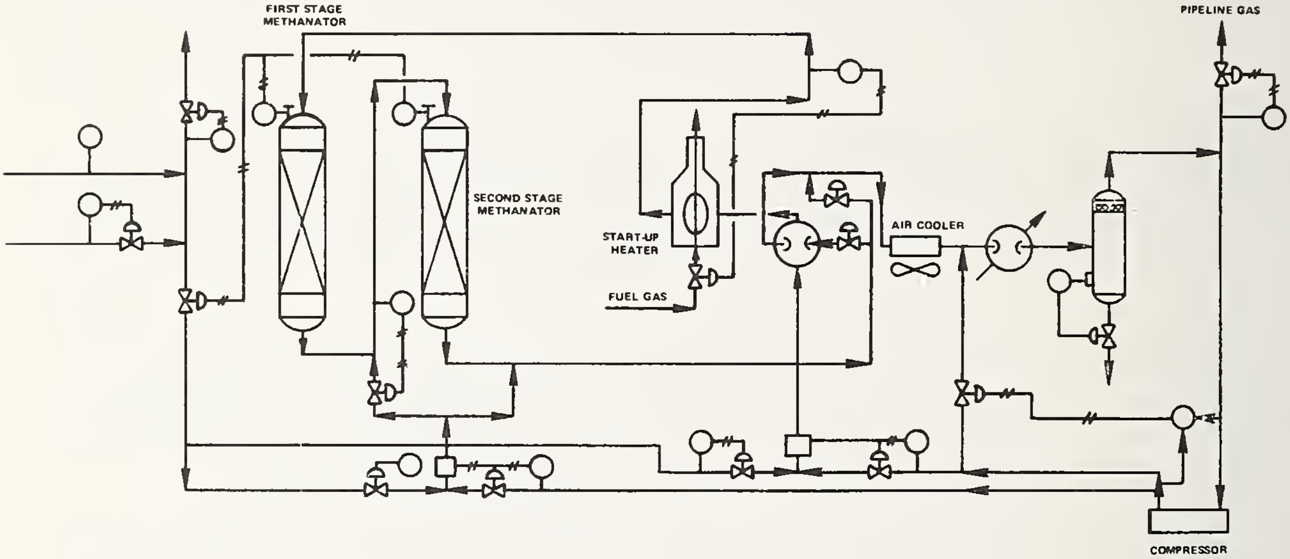

Figure 2

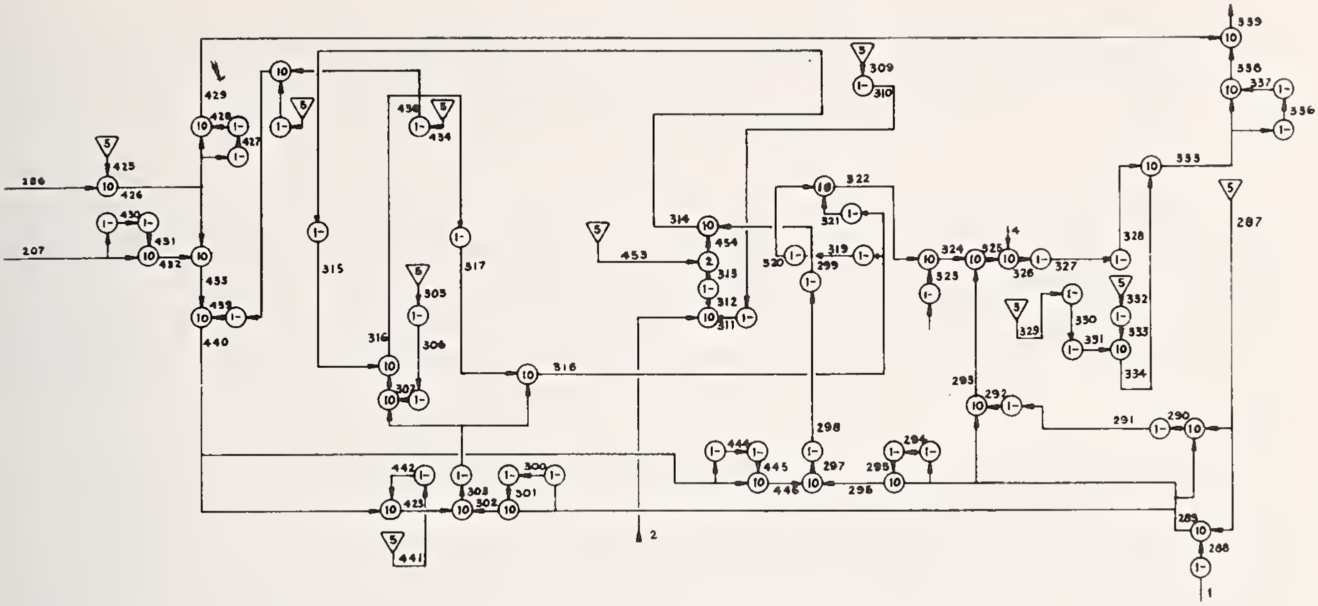

Figure 3

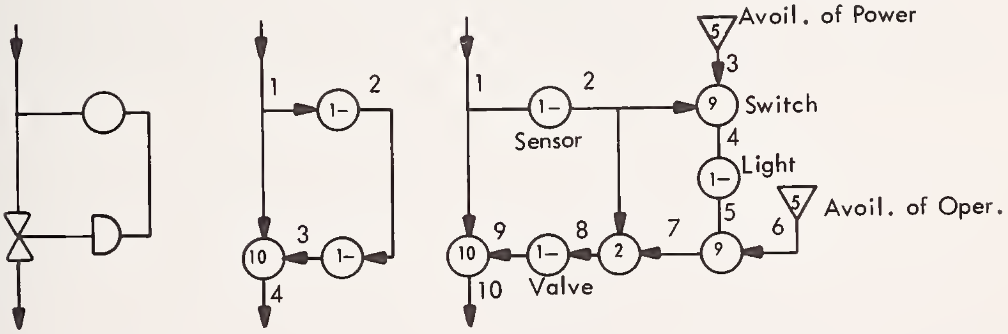

Figure 4

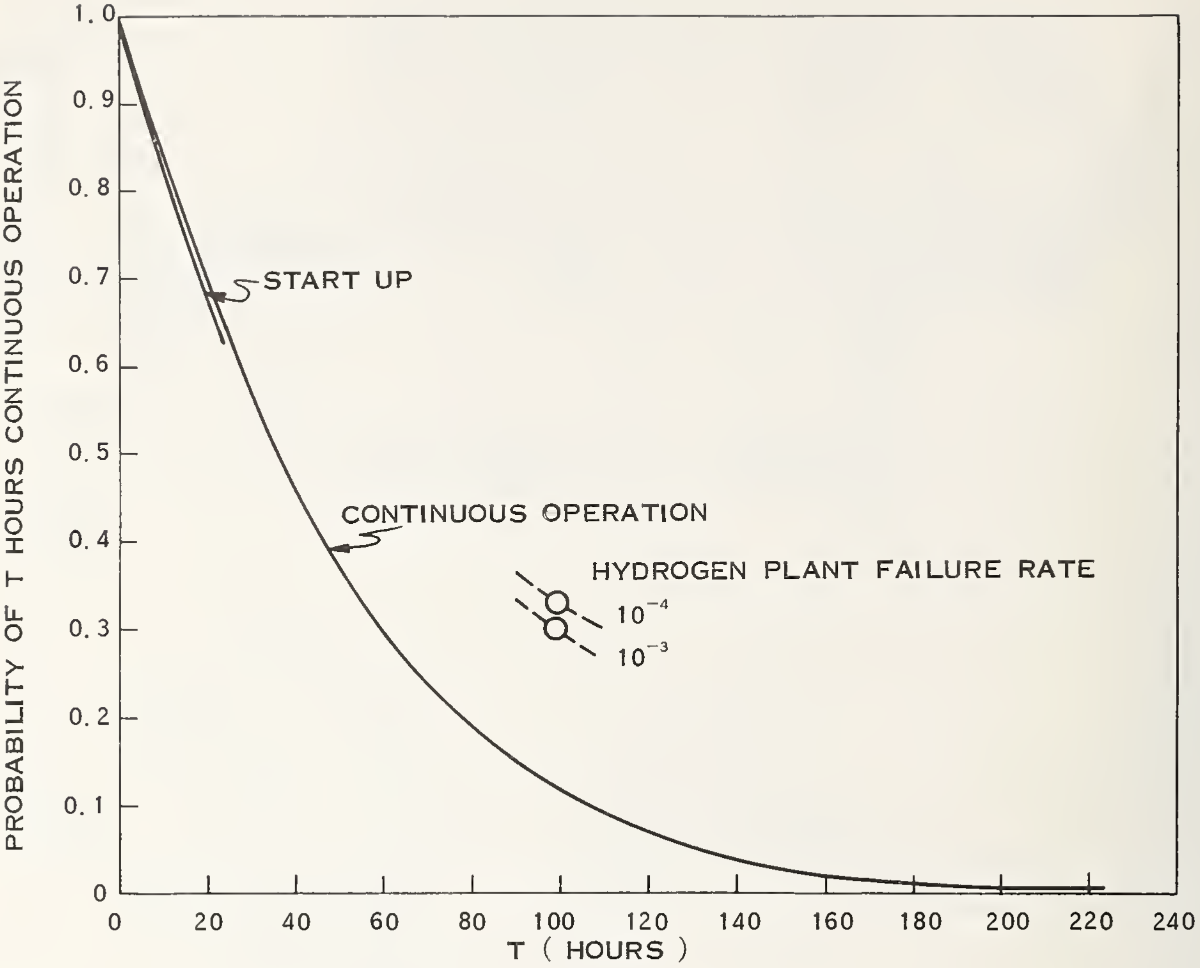
PROBABILITY OF T HOURS CONTINUOUS OPERATION
1.0
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
0
20
40
60
80
100
120
140
160
180
200
220
240
T (HOURS)
START UP
CONTINUOUS OPERATION
HYDROGEN PLANT FAILURE RATE
10^{-4}
10^{-3}

Figure 5

DISCUSSION

E. Passaglia, National Bureau of Standards: Certainly, techniques of this kind must have been applied to other areas of sophisticated technology like that used in the Apollo space program. Can you comment on other programs to which your technique has been applied?

J. R. Hoffman: I am not familiar with the work done on the Apollo. We are modeling the scram system for a gas cooled reactor at Fort St. Vrain for ERDA. At the Fort St. Vrain reactor we modeled about 15,000 components compared to about 300 components at the HYGAS plant. We have found some problem areas that had not been indicated in other analyses.

E. Passaglia: How do your results compare with the Rasmussen report?

J. R. Hoffman: We found nothing that would show that the Rasmussen report was in error in any large degree. We have considered some things that were not covered in the Rasmussen report, for example, operator interactions with the system. The GO technique is a little different from that generally used. Most NASA people that I have talked to are not worried about reliability analysis because there are various ways to circumvent the problem through design. One way is to overdesign. But overdesign with too many redundant systems may lead to trouble by causing premature shutdowns. We found that there were 13 different ways to actuate the deceleration mechanism for the Mercury flights, and in fact predicted prior to that flight that there was a reasonable chance for failure in the automatic system. And in fact, this occurred on one of the Mercury missions. The astronaut had to do everything by hand during deceleration because the reliability was not adequate.

CRITICAL MATERIALS PROBLEMS IN COAL CONVERSION

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Abstract: The stability of engineering materials in coal conversion systems is reviewed with respect to effects of specific chemical species: carbon, sulfur, oxygen, hydrogen, chloride and other species. These chemical species operate both at low temperatures and high temperatures to accelerate the deterioration of engineering materials. These species participate both in general degradation as well as highly localized attack such as stress corrosion cracking, dusting, intergranular attack and break away reaction phenomena. In addition to the strictly chemical influences on the metals, the deterioration of metals in the coal conversion systems is accelerated by erosion and static and cyclic stresses which lead to stress corrosion cracking, hydrogen embrittlement and corrosion fatigue.

Introduction: We start this discussion with the first law of materials application: "all engineering materials are unstable." Thus, the primary efforts of design and materials engineers must be directed toward delaying the inevitable deterioration of materials for sufficiently long times that the material will perform as desired for the intended life of the equipment. A second law having as much validity as the first for large engineering systems is: "The chain is as strong as its weakest link." While the latter seems trite, arising as it does out of someone's long forgotten book of sayings, nonetheless, it is a conceptual cornerstone in the development of large engineering systems. Unfortunately much of the development effort in new systems is placed upon the novel and intriguing problems with little effort given to what is commonly referred to as off the shelf or conventional equipment.

Past history with the fossil and nuclear power industries shows that most of the down time is associated with conventional equipment and not with more exotic components. The implicit suggestion here is that whatever development work is undertaken to construct large coal conversion systems should consider reliability of all components; action

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should be taken to raise the integrity of such equipment in proportion to the consequences of its failure to the performance of the total system.

The full scope of potential materials problems was considered at a meeting in April 1974 at Ohio State University on the subject: "Materials Problems and Research Opportunities in Coal Conversion" (1). This document should be carefully reviewed.

A second and related meeting was held to consider materials for MHD applications: "NSF-OCR Engineering Workshop on MHD Materials" (2). This is also an excellent volume and should be carefully reviewed.

In assessing the expected performance of structural materials in coal conversion systems the approach is little different from that which should be used in any system. One starts first by defining the chemical species which are an implicit part of the system. The second question one asks concerns the operational influences including temperature, pressure, flow, particle velocity, heat transfer, etc. Having defined these one then moves to define acceptable materials or to develop a materials testing program. Unfortunately the common approach is to decide early on the materials of construction and then forget the question of compatibility hoping, by closing ones eyes sufficiently tightly, that any deleterious influence would somehow lie pleasantly dormant for an infinite time.

With respect to the environmental species in coal conversion equipment, these are not difficult to identify. They include primarily carbon, hydrogen, nitrogen, oxygen, sulfur, and chlorine. Impurities such as zinc, lead, tin, and others often are damaging but are unlikely.

Physical influences which are to be considered simultaneously with the chemical influences include the following: temperature, time, thermal cycling, erosion, stress, and stress cycling together with the exacerbation of creep-fatigue and stress corrosion cracking.

The above have implied damage processes which are associated with fracture or corrosion type phenomena. These phenomena may be abetted by prolonged thermal influences on the structure and properties of metals. For example, temper embrittlement operates over an intermediate range of temperatures and affects low alloy steels of the type used in pressure vessels and turbines; stainless steels sustain the sensitization phenomenon at a somewhat higher temperature range; and, finally, high alloy ferritic materials age to produce an embrittling sigma phase.

Having now defined the major considerations in materials performance, i.e., the chemical environments, physical influences, and the change of the substrate with time, now consider what tools and resources are available for describing boundary conditions within which satisfactory performance might be expected. There are four separable categories. The first resource is the large reservoir of empirical data obtained from reasonably similar industrial systems. For example, there is already a fund of information from low BTU coal gasification systems. The petroleum, gas turbine, and chemical industries provide extensive information.

Secondly, there are well developed procedures based on thermodynamic analyses which can be used to predict regimes of stability of materials. These diagrams for stability of materials in environments are utilized in various forms. One of the most common is the simple plot of free energy of formation of a compound vs. temperature.

For the

$$M + {}^{1}/_{2}\, O_2 = MO$$

the free energy of formation is plotted vs. temperature and the curves for the formation of various metal oxides can be readily compared as for Figure 1. Lower values of the free energy of formation indicate the more stable oxides. Such comparative plots as in Figure 1 can be developed for the formation of other compounds such as sulfides, chlorides, carbides, nitrides, etc. all of which are of interest in coal conversion technology.

A useful approach for assessing stability of alloys in high temperature gases is to follow the pattern of Figure 2 where the partial pressures of sulfur and oxygen are plotted with respect to the existence of principal phases of the alloy species. Here regimes of stability of chromium and nickel phases are identified from work by Quets and Dresher (3). The broad range of stability of the protective Cr_2O_3 film accounts for the stability of alloys which contain chromium as a major alloying element. The general approach epitomized in Figure 2 has been discussed by Rapp (4).

The comparably important diagrams for low temperature aqueous corrosion processes are the Pourbaix diagrams (5, 6). These address the question

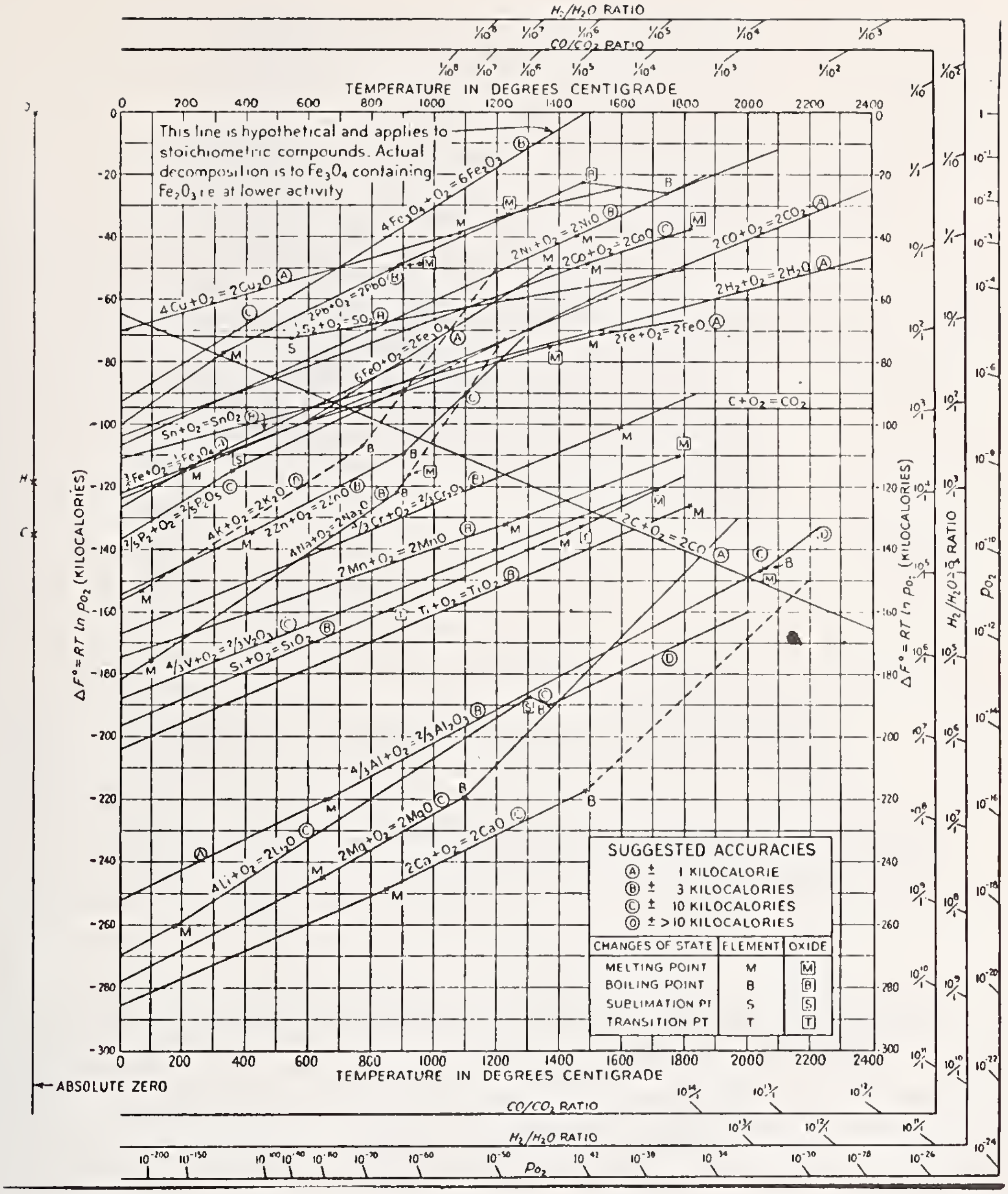


Figure 1. The standard free energy of formation of many metal oxides as a function of temperature. [From F.D. Richardson and J.H.E. Jeffes, substantially as in J. Iron Steel Inst. 160, 261 (1948)]

of stability of pure metals in aqueous solutions. Here important reactions are considered, e.g.

$$Fe = Fe^{++} + 2e \qquad (2)$$

$$Fe_2O_3 + H^+ = 2Fe^{+3} + 3(OH^-) \qquad (3)$$

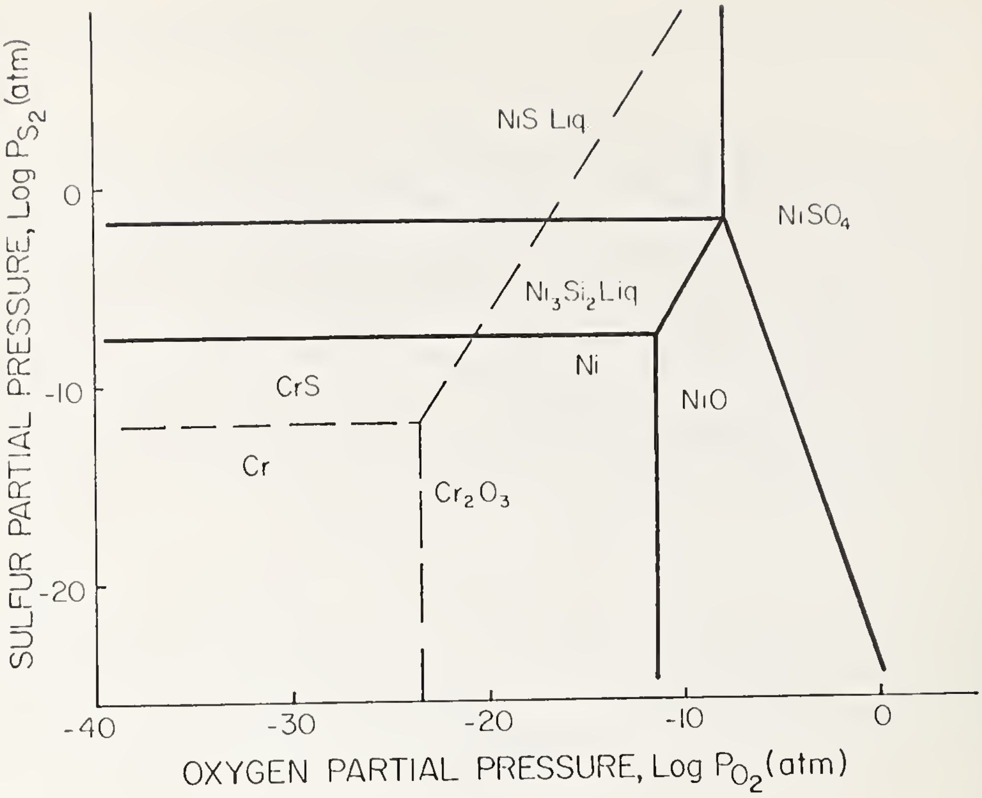


Figure 2. Superposition of Ni-S-O and Cr-S-O stability diagrams at 1200°C (3)

Equilibrium equations for these reactions are developed in terms of the electrochemical potential and pH, and diagrams such as Figure 3 are developed for regimes of stability for various iron compounds. For combinations of potential and pH where ions are stable corrosion is expected; where the insoluble compounds are stable, corrosion is reduced owing to the protective nature of insoluble compounds. Figure 4 compares Pourbaix diagrams for aluminum in water and fused sulfate salts. There is substantial similarity here which, to some extent, is fortuitious but the general applicability of the diagrams is clearly demonstrated for electrolytes in general.

A third approach to assessing the expected performance of materials involves laboratory testing. Advanced approaches take their leads from the diagrams of Figures 1-4 as modified by the expected engineering environments. These laboratory tests would measure general and localized attack under carefully controlled conditions.

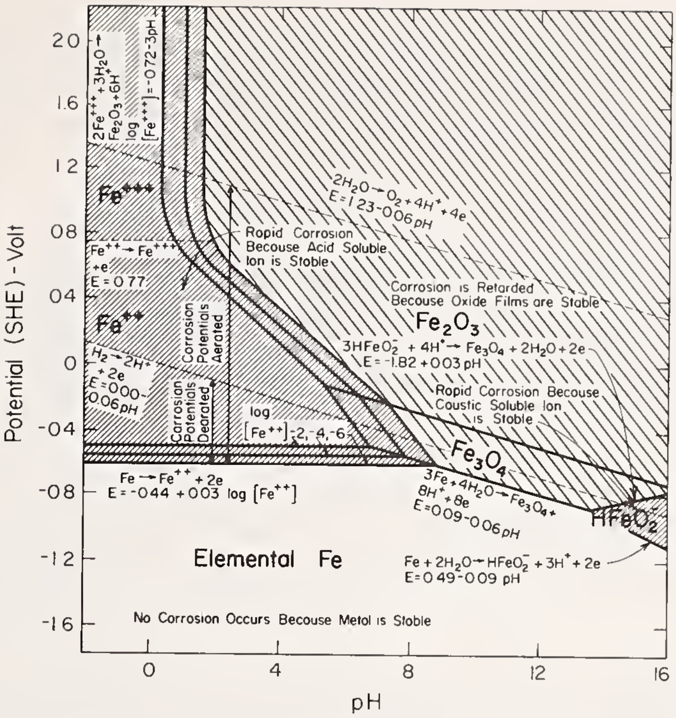
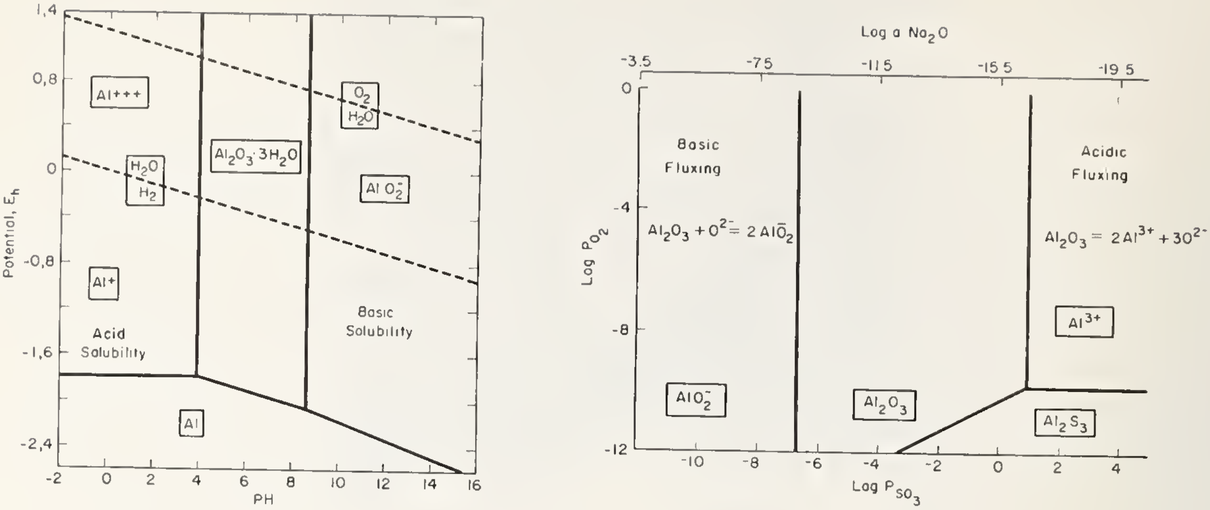


Figure 3. Annotated potential-pH diagram for the iron-water system at 24°C. The hatched regions indicate zones of various reaction types: no hatching indicates that the metal will not corrode; fine hatching means that the metal tends to corrode since the ionic species are soluble; broad hatching shows regions of insoluble product layers where passivity should occur if the layers are protective. The applicable equilibria with their corresponding pH and potential dependencies are placed parallel to the respective lines. Regions are also noted where the corrosion potential can exist depending upon the state of aeration of aqueous solutions. Adapted from Pourbaix.

Fourth, materials testing is conducted in pilot plant facilities and compared with the predictions from laboratory tests.

Each of these four methods for developing an understanding of materials performance produces its own feedback which suggests approaches, analyses, and experiments on the other category.



(a)

(b)

Figure 4. Comparison of thermodynamic diagrams of the stability of aluminum in water at left (a) and in molten sulfates at right (b). The water developed for 25°C and the other for 1000°C. The reversal in location of acid and base regions arises from conventions in the respective technologies.

The following discussion will emphasize problems associated with metallic materials. Problems of ceramic materials in coal conversion systems have been dealt with by Wachtman et al (7). Unfortunately, the corrosion behavior of ceramic materials has never been considered in as much detail as metallic materials. The reason, of course, has to do with the use of bulk of quantities of ceramic materials where small amounts of corrosive activity is not so serious as it is for metallic materials. However, only metallic materials can serve as a pressure boundary; thus, integrity still remains as the principal concern in performance.

The following is divided into brief discussions concerning each of the chemical species; this is followed by comments on the change in substrate, physical influences, and a few commentaries on the so called weak link problem.

I have divided the consideration of the chemical species into high and low temperature circumstances. One is prone to think in the coal conversion circumstances that only the high temperature problem is at issue, and a good deal of work is directed toward the resolution of problems arising in high temperature operations. However, there are inherently low temperature environments which can be equally or perhaps even more debilitating. Such environments, for example, can occur in scrubbers.

Finally, in an overall view Table 1 summarizes the occurrence of failures of components in coal conversion units. This information was prepared by Smith at the National Bureau of Standards and was reported later in the ERDA newsletter "Materials and Components in Fossil Energy Applications" (8).

Carbon: In high temperature gases carbon-containing gas molecules produce two different kinds of phenomena depending upon whether the gases are oxidizing or reducing.

In oxidizing conditions mild steels oxidize in dry CO_2 environments much as they do in oxygen. However, above about 500°C and especially in the presence of small amounts of water the corrosion rate is greatly accelerated. After an initial period where parabolic kinetics--characteristic of adherent films--the protective quality of the films breaks down and a rapid linear rate ensues (9, 10). Figure 5 shows this phenomenon schematically and Figure 6 shows the post breakaway rate as a function of water content and temperature. These conditions are most pertinent to coal gasification. These rapid rates can be reduced by some alloying elements including silicon and by lowering the water concentration.

In reducing environments carburizing conditions may occur and the metal is attacked by two processes. One is simple carburization. Here the surface is embrittled by the inward diffusion of carbon.

In atmospheres where reducing conditions prevail, more serious is the phenomenon of "dusting" which has been reviewed by Hochman (11). The mechanism of the dusting phenomenon is not well established but seems to involve critical surface processes where CO decomposes with the next stop being the inward diffusion of carbon. Particularly characteristic of dusting is a peak in the attack in the range of 500 to 700°C depending upon the alloy and environment. Figures 7 and 8 from Hochman show the effects of environment, temperature, and alloy composition on dusting.

Dusting can be inhibited by adding sulfur species which seem to slow the decomposition of CO molecules on the surface much the same as sulfur

TABLE I

TYPE OF INCIDENT BY FREQUENCY OF OCCURRENCE*

Type of Problem	No. of Items	No. of Incidences	Process A	B	C	D	Significance of Incident To Particular Process	To Coal Conversion Technology	Recommendations
Sulfidation	22	20		2			(1) Thermocouple critical for control (2) Slurry grid – complete shutdown	Major problem causing short life	(1) Use coatings, better alloy, change environment
			18				(1) Severe and continuing problems with thermocouple tube and heater coil not catastrophic but severely limits life of parts	Expect to be the major problem determining life because of high sulfur coal	(2) Alonized Fe Ni Cr alloys are best now available
Corrosion	10	8	5				Unknown	Unknown – many critical areas, possible catastrophic	(1) Need detail diagnostic failure analysis
				3	3				(2) Need critical review of all processes
SCC	9	4	3				Great causes shutdown and expensive repair – all Cl cracking	Great nearly all systems have some areas where possible - identify and correct	(1) Carefully monitor environment (2) Better material selection (3) Some design change possible
				1			Expansion bellows Water Lines		
Erosion/ Wear	8	6	2				Great – short life, shutdown	Unknown – identify areas	(1) Change design/material (2) Misalignment is cause
				1			Not critical – repair during maintenance		
					3	1	Critical – causes shutdown Critical – causes shutdown	Great - urgent problem of seals on pumps	Better pump seals, filters, double seals
Fabrication Defect	3	3		3			(1) Valve (2) Reducer residual stress in welds – shutdown (3) Piping weld crack – total shutdown	Great – short life, catastrophic shutdown	Strict attention to welding procedures and residual stresses
Carbonization	3	1				3	Great – this is the critical materials problem for the clean coke process	Unknown – review other systems to identify critical regions	(1) Add $\underline{S}$, water to environment (2) New alloy (3) Redesign
Design	5	5					(1) Quench pot – short life	(1) Many similar quench areas – critical	(1) Redesign
Thermal Stress	3	3		2			(2) Bellows – total shutdown	(2) Urgent problem – all systems	(2) Redesign
					1		(3) Heat exchanger – total shutdown	(3) Many similar areas – critical	(3) Redesign on basis of thermal stress
Other	2	2			2		(4) Knife shouldn't be problem (5) Sight glass – severe safety hazard	(4) None (5) Critical – all systems use this	(4) None (5) Redesign, new material
Metal Dusting	1	1	1				Generally not a problem $\underline{S}$ in environment prevents	Identify critical areas Extremely rapid, catastrophic failure when present	(1) Need better alloy (2) Add small amounts of $\underline{S}$ (3) Coatings (oxide, sulfide) may help
Refractory	1	1	1				Great significance – major shutdown, long time shutdown, expensive repair	Highest significance – identify critical areas, inspection, Q.C.	(1) Diagnostic Fail. Analysis URGENT (2) Need better refractory
Material Selection or Q.C.	1	1	1				Great – shutdown plant	Requires constant alertness	(1) Need high level of Q.C.
Unknown	2	2	1		1		Great – total shutdown Great – fire, total shutdown	Unknown Unknown	(1) Diagnostic failure analysis needed

*Most failures tabulated here occurred in 1975 and 1976, with some earlier.

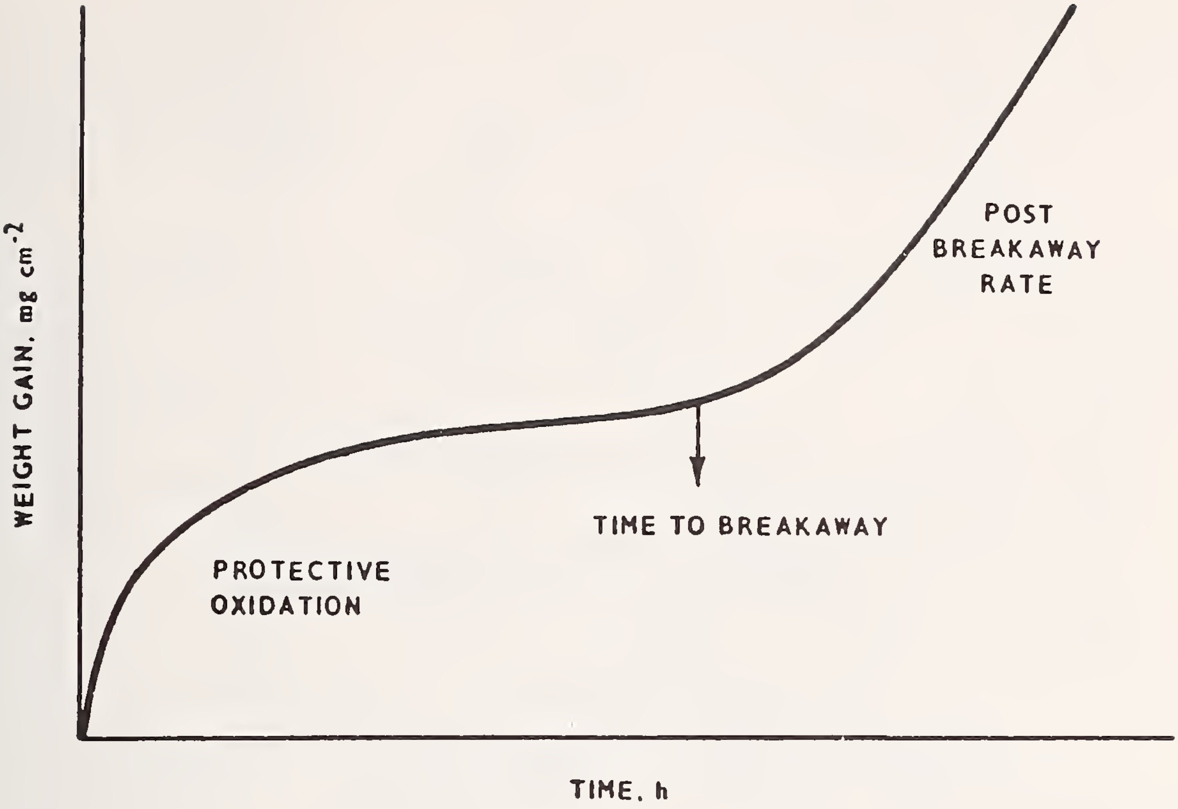


Figure 5. Schematic representation of breakaway corrosion.

prevents other catalytic reactions at high temperatures. Hydrocarbon gases also produce dusting but at higher temperatures. Water vapor and ammonia appear also to inhibit the dusting.

At low temperatures carbon species contribute to acidifying the environment when CO_2 dissolves in water to form carbonic acid. More virulent is the stress corrosion cracking of mild steels which is caused by carbonates and by dissolved CO (9, 10). Results are illustrated in Table II and Figures 9 and 10 from references 12 and 13. The general range of conditions where either of these environments cause accelerated cracking has not been established.

Sulfur: At high temperatures sulfur-containing species seem to exert three important effects. The first involves the so-called hot corrosion

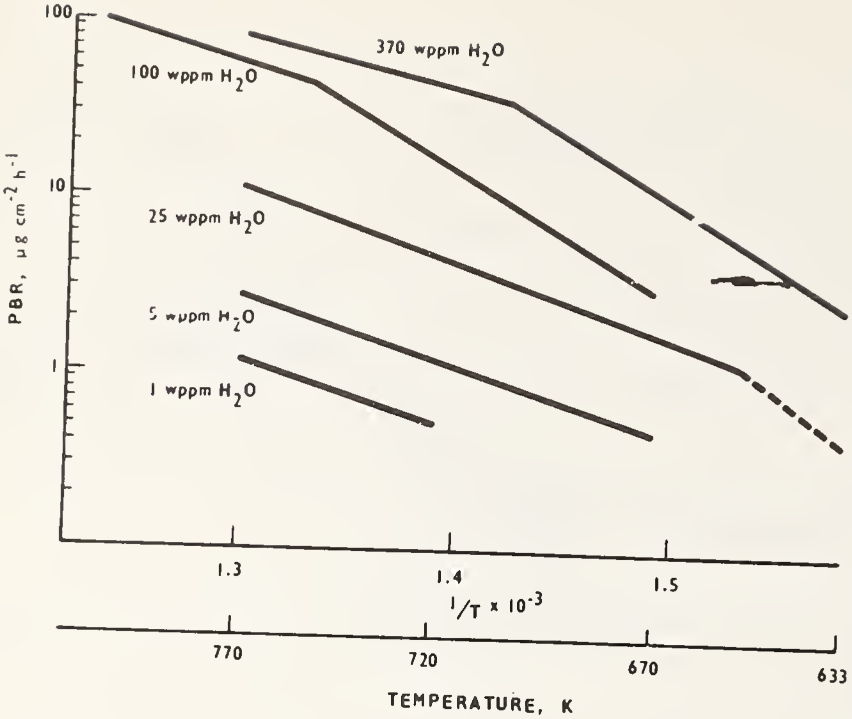


Figure 6. The effect of temperature on PBR at water levels in the range 1-370 wppm of H_2O.

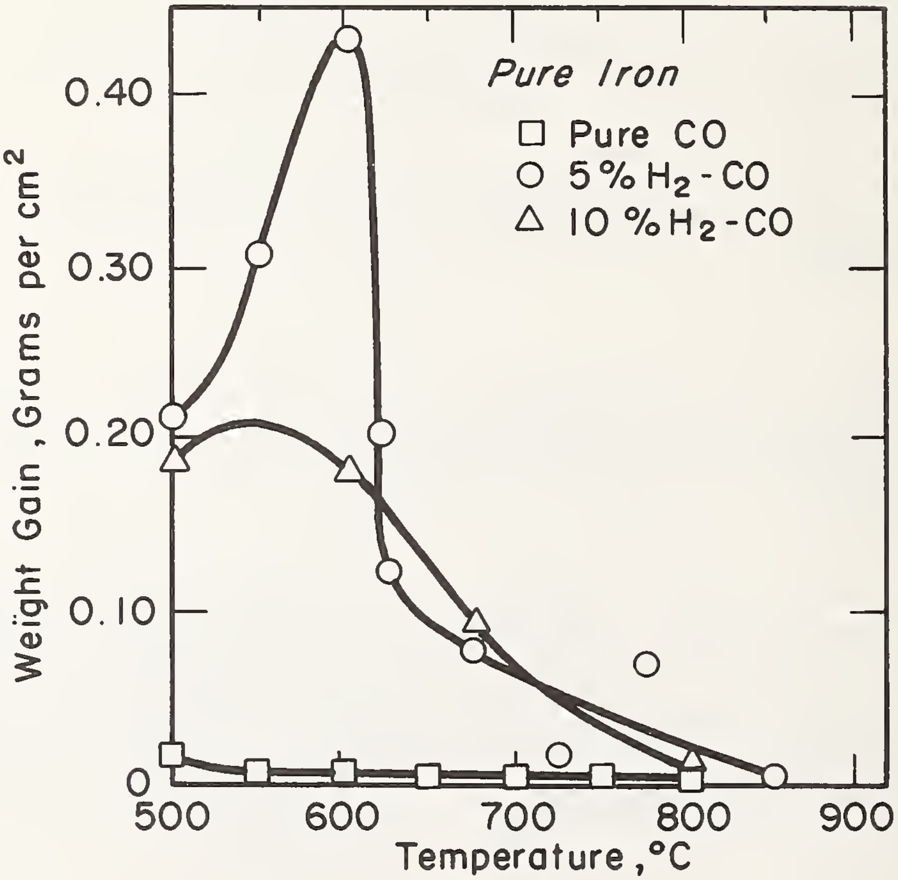


Figure 7. The reactivity of pure iron in pure CO, 5% H_2-CO and 10% H_2-CO environments.

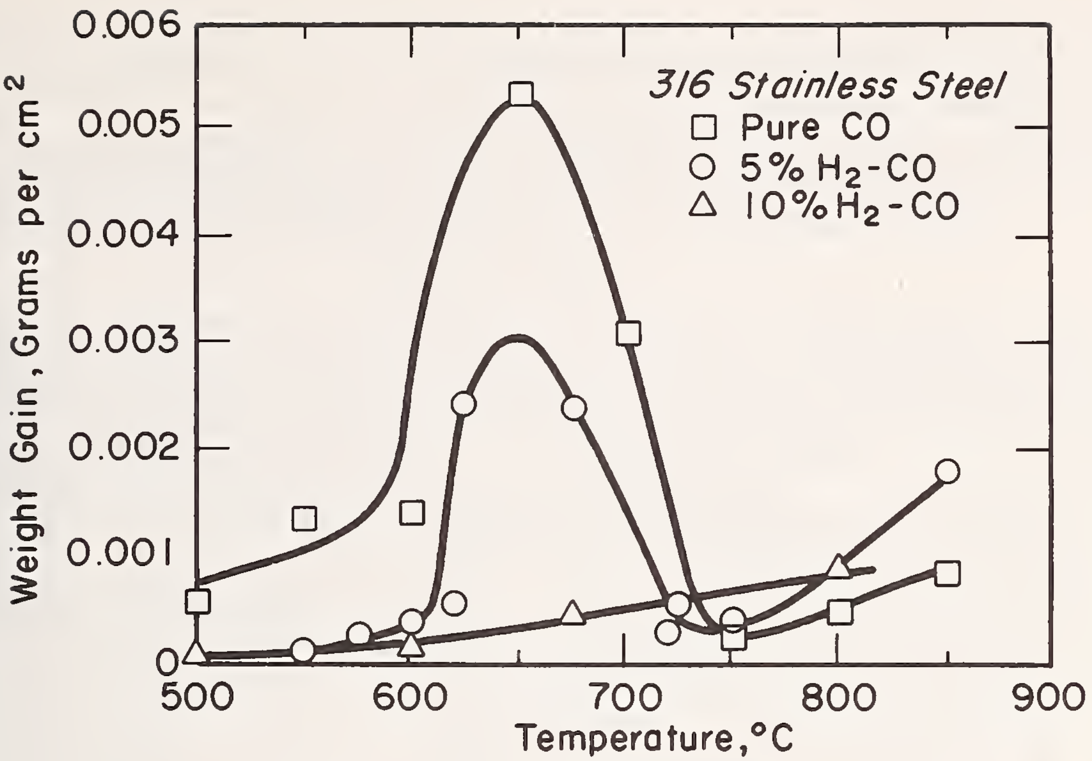


Figure 8. The reactivity of 316 stainless steel in pure CO, 5% H_2-CO, and 10% H_2-CO environments.

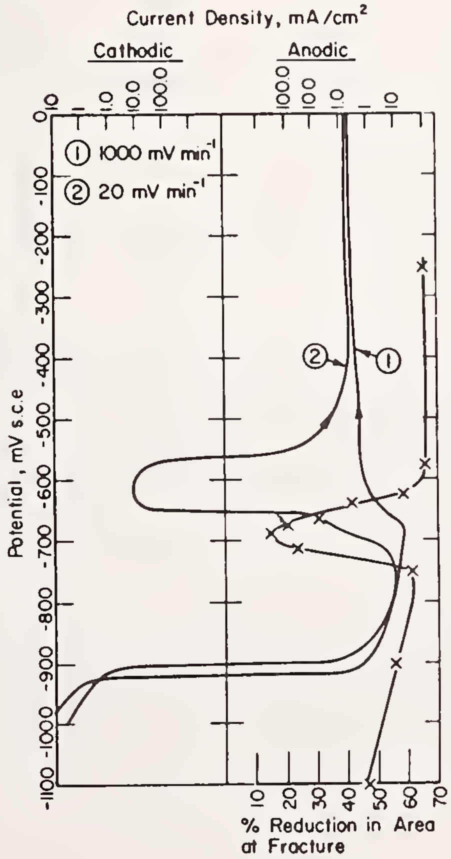


Figure 9. Polarization curves and stress corrosion test results for mild steel in 1N Na_2CO_3 + 1N $NaHCO_3$ at 90°C.

TABLE II

Effect of the Amount and the Ratio of Gas Mixture on Stress Corrosion Cracking of Mn-Steel (O.T.) at 18°C in Moist CO/CO_2

No.	Partial Pressure (kg/cm^2)			Total Pressure (kg/cm^2)	Period Week		
	CO	CO_2	N_2		2	4	6
1	26	--	--	26	0	-	0
2	26	14	--	40	X	X	-
3	26	14	60	100	X	X	X
4	10	16	--	26	X	X	-
5	10	16	74	100	X	X	X
6	5	18	77	100	X	X	-
7	1	17	82	100	X	X	-
8	0.1	16	84	100	-	-	0
9	0.01	16	84	100	-	-	0
10	26	1	73	100	-	-	X
11	26	0.2	74	100	-	-	X

X: Crack   0: No Crack

phenomena which involves attack of metals in environments which contain both sulfur and oxygen. This phenomenon has been extensively investigated in connection with accelerated corrosion phenomenon in gas turbine blades where sulfur is present in the fuel. The accelerative process seems to be associated with the formation of a molten sulfate salt at the metal surface.

Figure 10 shows that the molten sulfate greatly accelerates the corrosive attack on high nickel alloys (14). This attack can be substantially mitigated adding aluminum or chromium to the alloy in order to form a protective film.

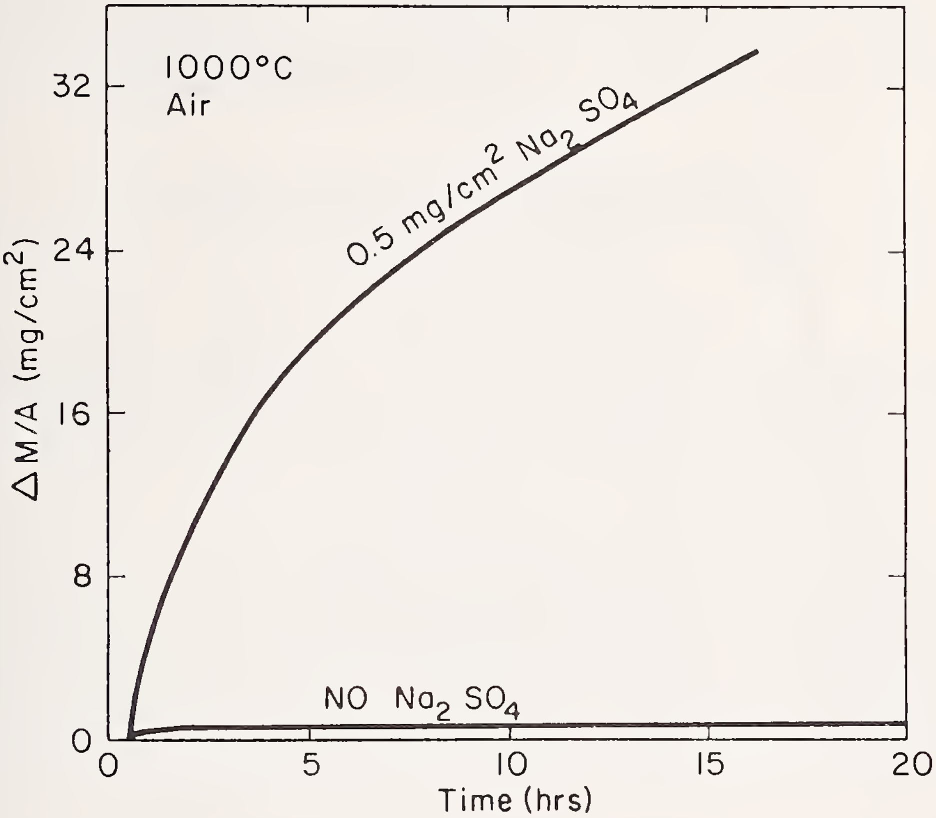


Figure 10. Comparison of the oxidation behavior of the nickel-base super alloy B-1900 (Ni + 8Cr + Ti, 6Al, 6Mo, 4Ta, 10Co, 0.1C, 0.07Zi, 0.01B) with and without Na_2SO_4 coating.

Figure 11 shows the beneficial effect of adding species which improve the quality of the protective film (15).

Sulfur gases at high temperatures seem to attack high nickel alloys rapidly along grain boundaries. For metals exposed to H_2S environments, they are protected generally by increasing the chromium concentration as shown by Yamamoto et al in Figure 12. The beneficial effect of chromium is to be expected here according to the thermodynamic prediction for Figure 2.

At lower temperatures and in aqueous solutions sulfur exerts two important influences leading to accelerated attack--again depending upon the state of oxidation of the sulfur. In the oxidized state dissolved SO_2 acidifies the environment. Additionally, SO_2 raises the dewpoint to the range of 160°C depending upon the concentration of SO_2. The precipitated water with the SO_2 now incorporated to produce H_2SO_4 is very corrosive. Figure 13 shows clearly this correlation between precipitation of water from a SO_2-contaminated atmosphere and the corrosion of iron (17).

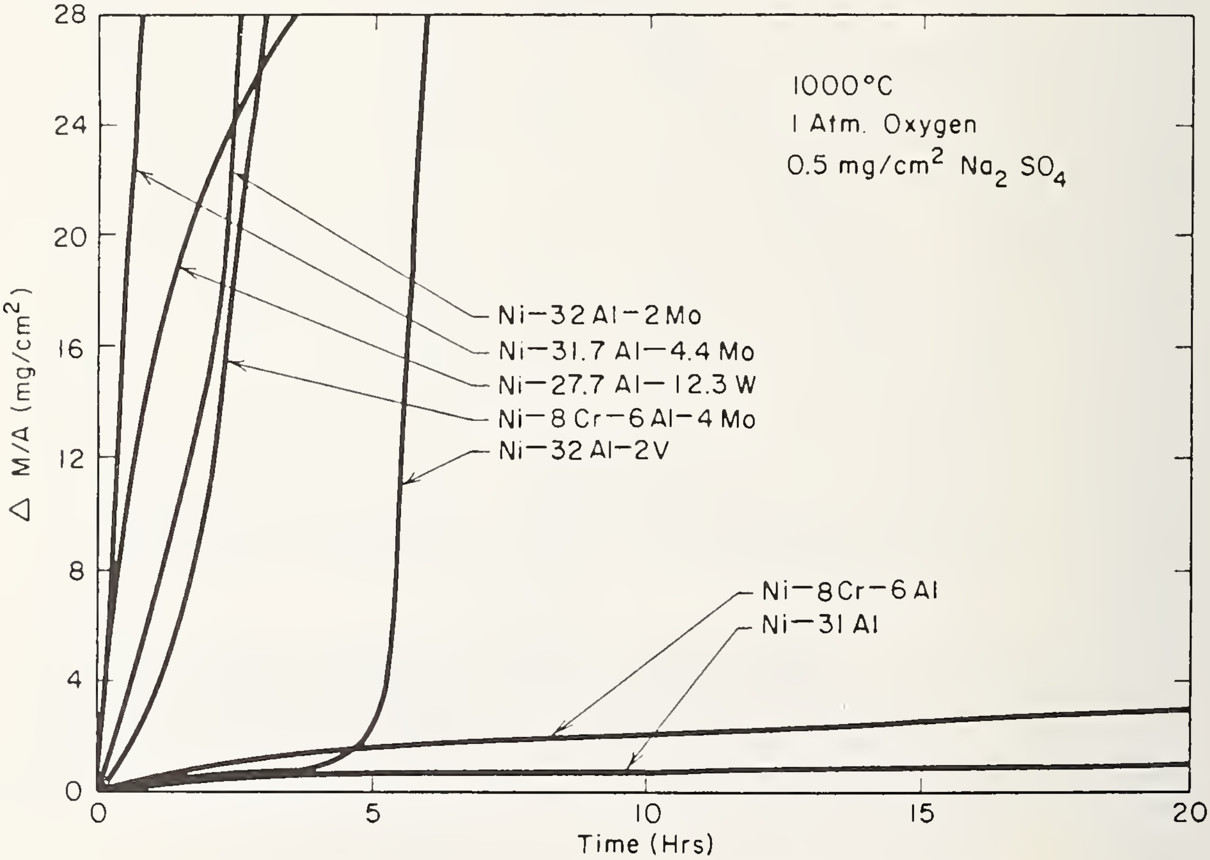


Figure 11. Weight change versus time for the oxidation of Na_2SO_4-coated (0.5 mg/cm^2) specimen of Ni-Al and Ni-Cr-Al alloys as affected by the addition of V, Mo, and W.

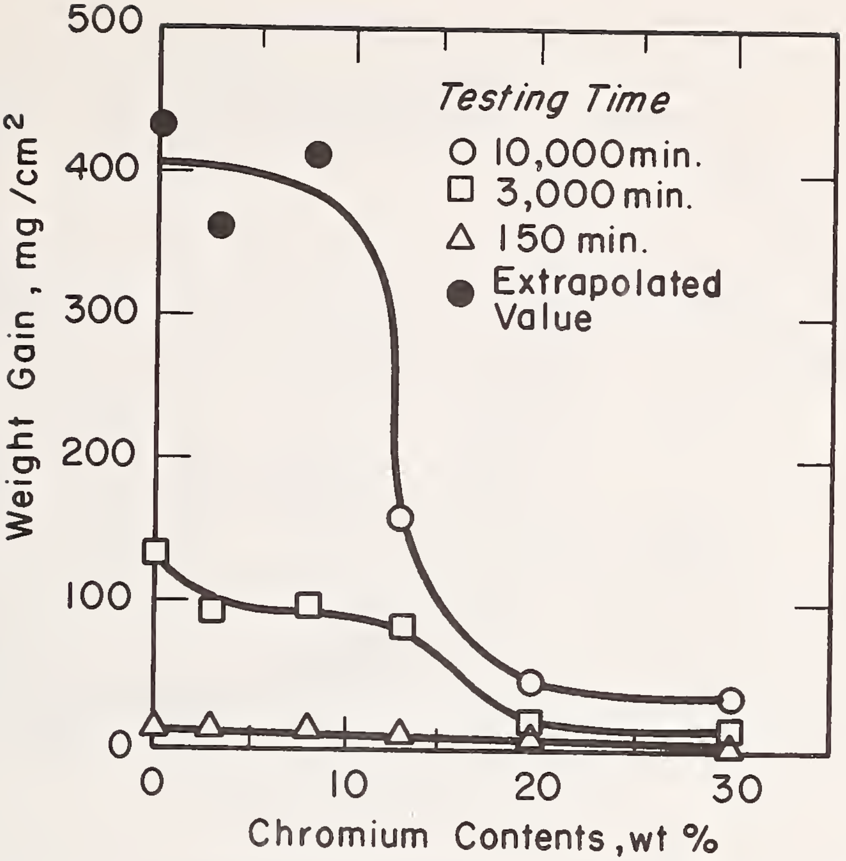


Figure 12. Amount of corrosion products (weight gain) of iron-chromium alloys at 600°C against chromium content of alloys in hydrogen sulfide.

If the sulfur is present as H_2S it substantially aggravates the stress corrosion cracking of iron base alloys as shown in Figure 14; and as the strength of the alloys is increased the tendency for SCC to occur is increased. Further, H_2S accelerates the entry of hydrogen in acid environments and blistering often results.

Dissolved H_2S decreases the corrosion resistance of iron. Together with HCl there is a synergistic effect even at relatively low temperatures as shown in Figure 15 (18).

Hydrogen: At high temperatures the most insidious effect of hydrogen is called hydrogen damage. The engineering data upon which hydrogen damage is based is the Nelson curve shown in Figure 16 (19). This figure defines the combination of temperature and pressure above which a failure will occur for an iron base alloy. This phenomenon seems essentially to result from the formation of methane molecules within the metal and the subsequent formation of blisters. Hydrogen damage

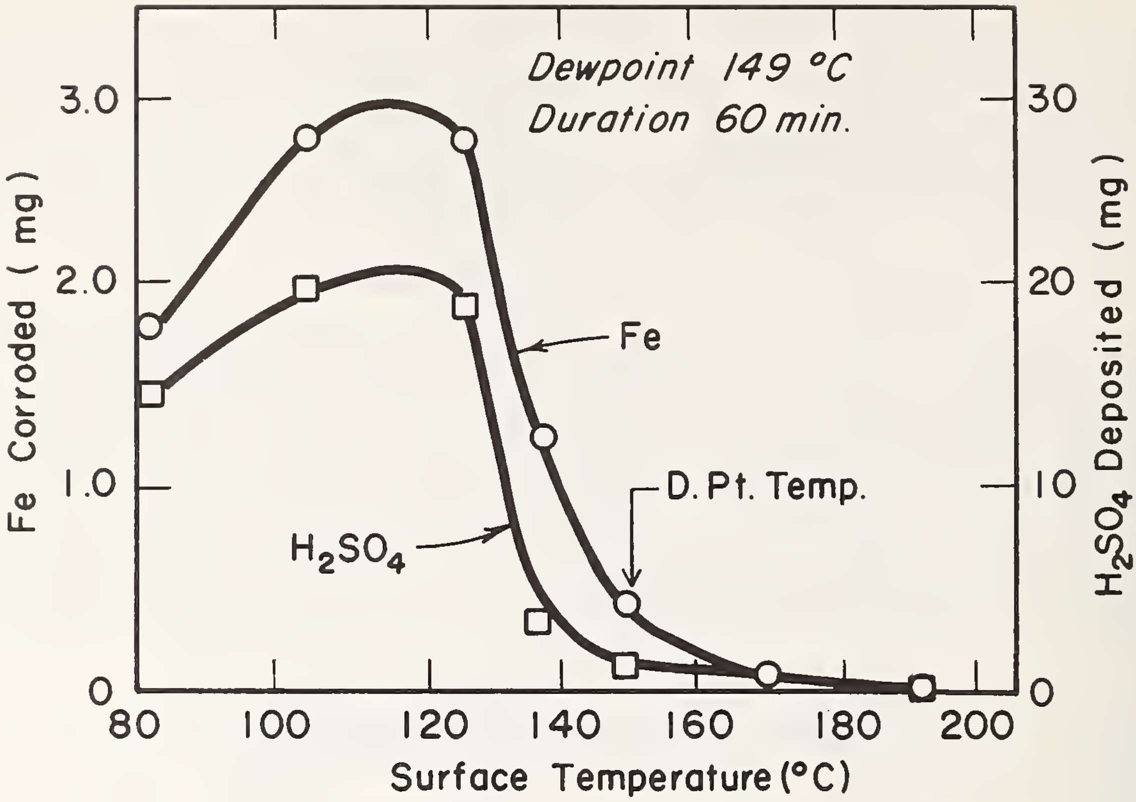


Figure 13. Effect of surface temperature on condensation and corrosion.

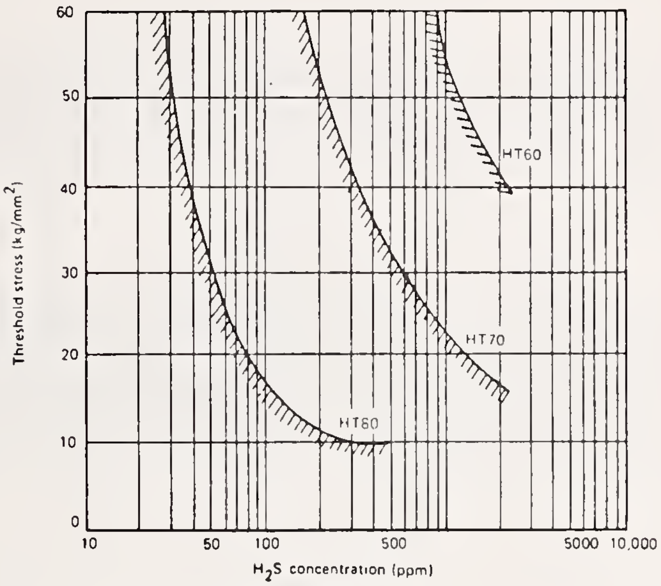
can be mitigated by adding chromium and molybdenum and as a result the failure curve is raised to higher temperatures and pressures.

During thermal cycling of large vessels where hydrogen may have entered the metal there is a further form of deterioration called "flaking." This occurs when the surface temperature is rapidly reduced with the result that the hydrogen is supersaturated. This resulting supersaturation causes hydrogen bubbles to precipitate and force metal flakes off the surface.

In addition, another damage process which can occur at low temperatures and high temperatures is called blistering. An example of blistering is shown in Figure 17. Blistering essentially results from relatively high hydrogen activities. These may result from the presence of hydrogen sulfide or cyanide.

At lower temperatures, hydrogen enters the metal to cause stress

Steel	Chemical composition (%)									TYS (ksi)	UTS (ksi)	H_V
	C	Si	Mn	P	S	Ni	Cr	Cu	Mo			
HT 60	0.12	0.35	1.10	0.019	0.019	0.25	0.15	0.25	0.10	82	97.5	223
HT 70	0.11	0.30	0.95	0.013	0.014	0.85	0.42	0.14	0.32	104	111	238
HT 80	0.14	0.36	1.03	0.010	0.011	1.12	0.47	0.29	0.52	113	119	277



Threshold determined at 30 days on
unnotched specimens loaded in bending

Environment: 0.5% acetic acid solution

Figure 14. Relationship between applied stress and H_2S concentration for initiation of cracking in various grades of high-strength steel (after Ishizuka and Onishi).

corrosion cracking of steels. An early manifestation of this problem in high strength steels especially associated with landing gears was that of delayed failure. This phenomenon was extensively investigated by Troiano (20). More recent work on high strength steels has shown that the critical factor in the mechanism of crack propagation is associated with the entry of hydrogen in the material (21). This phenomenon is exacerbated as the strength of the alloy is increased. The hydrogen can be derived either from water, gaseous hydrogen or other materials such as H_2S, HCl, and HBr. A comparison of the effects of H_2O, H_2, and H_2S is shown in Figure 18 from the work of McIntyre (21). Figure 15 has already shown that H_2S accelerates SCC in lower temperature regimes for relatively low alloy strengths. Two important volumes summarize recent developments concerning effects of hydrogen on mechanical properties (22,23).

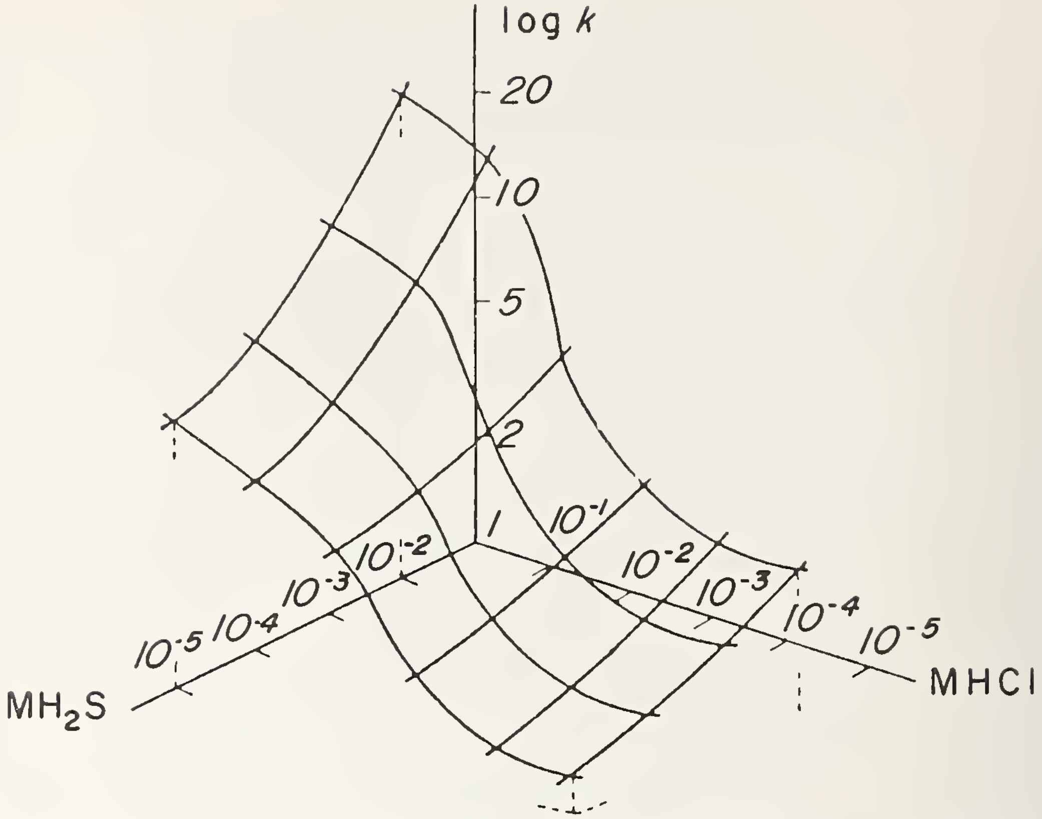


Figure 15. Dependence of steel corrosion on the H_2S and HCl content in the solution t = 25°C K-corrosion loss in $g.cm^{-2}/6$ hrs. 10^{-4}, M-HCl and M-H_2S. Concentration of the components in the system, gram-mole/l.

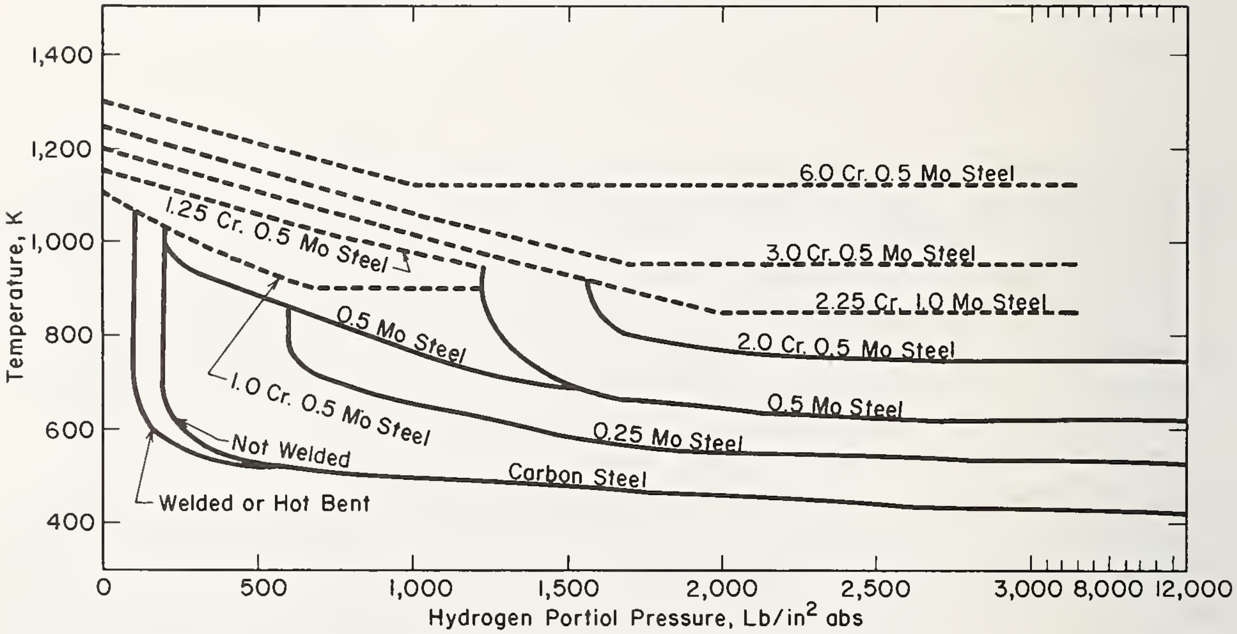


Figure 16. Effect of temperature and hydrogen pressure on the hydrogen damage failure of iron base alloys. Alloys are "safe" below the lines shown and are subject to failure above these lines. Effects of alloy additions indicated. From Nelson (19).

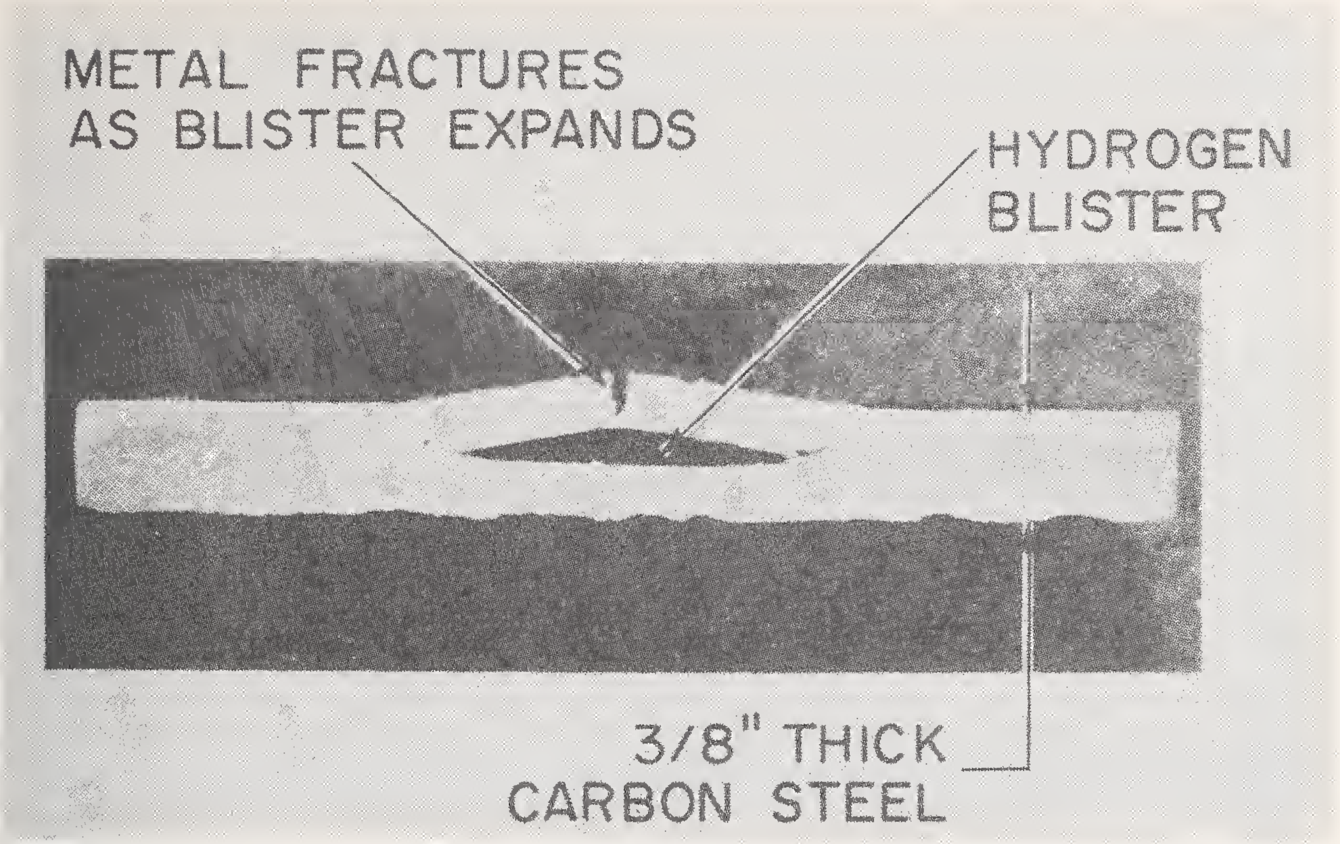


Figure 17. Hydrogen blister in a 9.5 mm thick A-283 carbon steel plate exposed to environment containing H_2S, HCN, NH_4OH, and a trace of NaCl at 130°F.

Chloride: At high temperatures the halidation of metals may occur. This general subject area has been recently reviewed by Rapp (24). However, the concentration of halogen chemicals is not sufficient to be a significant factor at high temperatures in coal gasification environments.

At lower temperatures the effect of chloride ions on the stress corrosion cracking of austenitic stainless steels is well known and has been extensively discussed (25). It is not necessary here to further elaborate except to point out the important interdependence of chloride cracking on the oxygen concentration which is shown in Figure 19 (26). Chloride also accelerates localized corrosion processes such as pitting and crevice corrosion owing to the debilitation this ion has on the passive films.

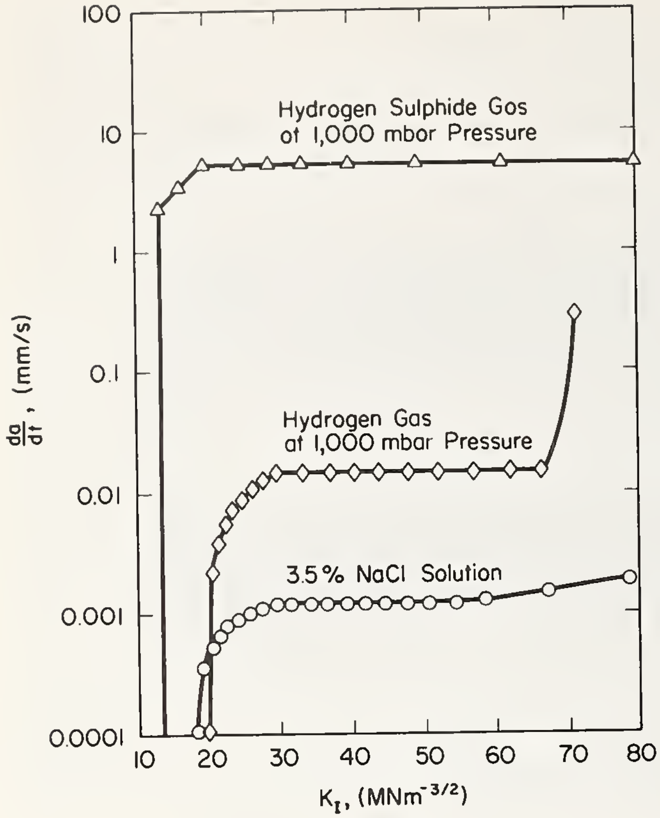


Figure 18. Crack velocity as a function of stress intensity for a 897 M39 steel at 291°K in environments of hydrogen sulfide, hydrogen, and 3.5% NaCl.

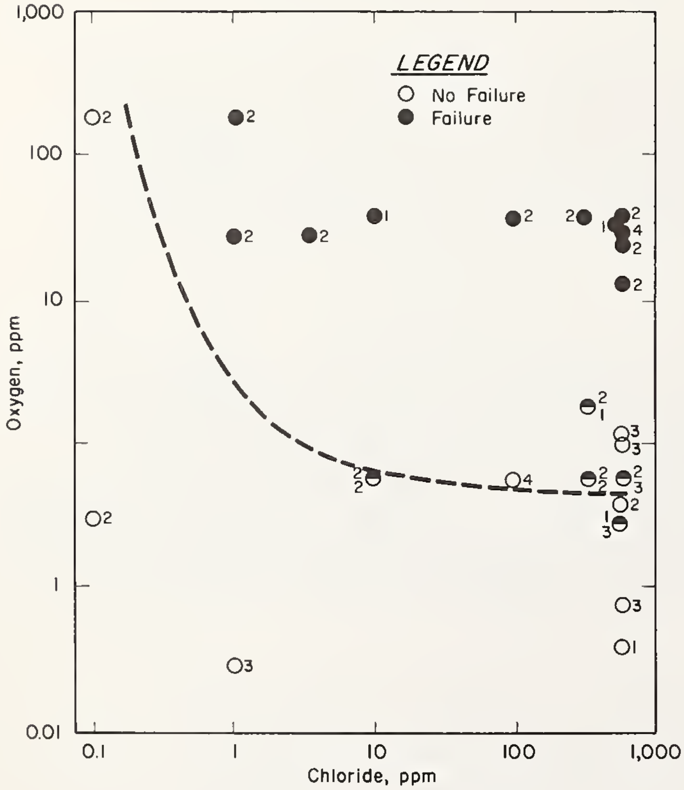


Figure 19. Relationship between chloride and oxygen content of alkaline phosphate treated boiler water for stress corrosion cracking of austenitic stainless steel.

Oxygen: At high temperatures oxygen contributes to the oxidation of metals and is also significant in combination with sulfur, carbon, and hydrogen as discussed above. The high temperature oxidation of metals has been extensively treated and will not be elaborated upon here (27, 28).

At low temperatures oxygen exerts its influence primarily when it dissolves in water. Referring to Figure 3 which shows the Pourbaix diagram of iron, the line for the H_2O/O_2 equilibrium is shown near the top and at potentials substantially higher than the H_2O/O_2 equilibrium. This higher equilibrium potential for the H_2O/O_2 equilibrium is the basis for oxygen raising the potential. The influence of oxygen in exacerbating the chloride-SCC of stainless steel as shown in Figure 19 is directly related to raising the potential into a regime where SCC can occur.

Other Species: Species such as molybdenum and vanadium have been traditionally associated with the phenomenon of catastrophic oxidation which was first studied by Fontana and Leslie (29). This phenomenon has been more recently interpreted in terms of the effect that these species exert on the equilibrium chemistry and the location of these circumstances with respect to Pourbaix diagram stability.

Other low melting species such as lead, zinc, aluminum, and gallium may accelerate damage to either grain boundary attack or a type of liquid metal embrittlement. If substantial quantities of these species can exist in metallic form such problems should be considered.

I have omitted consideration of effects of nitrogen since previous experience suggests that it does not play a significant role relative to the more aggressive reactions involving hydrogen, carbon, chlorine, oxygen, and sulfur.

Physical Influences: The important physical influences in addition to the chemical ones involve primarily the effects of stress and particles as they contribute to wear or erosion.

Stress exerts its influence in a number of ways which vary depending upon the temperature. At high temperatures stress is associated with the phenomenon of creep rupture and environments may accelerate this process although environmental influences on creep have not been extensively studied.

As a variation on the subject of creep, the subject of creep fatigue is receiving significant recent attention as it applies to pressure vessels operating at elevated temperatures (30, 31). While creep fatigue has been of primary interest to nuclear vessels, it will certainly be of greater importance to pressure vessels used in coal gasification. The problem embodied in the subject "creep fatigue" involves the fact that cyclic loading superimposed upon a creep situation accelerates the creep damage; conversely, long holding times between stress cycles also exacerbate fatigue damage, i.e. increases da/dN.

At low temperatures constant stress causes stress corrosion cracking, and in the oscillating circumstances we have corrosion fatigue. Stress corrosion cracking depends upon the synergistic interaction of numerous factors; this interdependence is illustrated in Figure 20. If the product is below an arbitrary constant value, SCC will not occur in a time less than the same value and at some point this time is long enough to be effectively infinite. SCC can be eliminated if any of the factors in the equation is zero or if several factors can be reduced simultaneously.

Corrosion fatigue and stress corrosion cracking are part of the same continuum as illustrated in Figure 21. These two phenomena, which are ordinarily considered as separate, approach each other along the coordinates of cyclic figuring and stress ratio. In the limit of a period which is long with respect to the time required for SCC to occur, corrosion fatigue becomes stress corrosion cracking. On the other hand, as the ratio of the minimum stress to the maximum stress approaches positive unity the condition of constant load is again approached.

With respect to the question of particle induced deterioration consideration should be directed toward two cases. One is the interaction of particles on the wear process. This is particularly significant relative to valves and seals which separate zones of different pressures. There is presently very little information on this process but it deserves careful consideration.

The second problem of erosive wear produced by high velocity particles has been considered more extensively and is expected to be a major issue especially in components such as cyclone separators and turbines. With respect to particles, parameters of size, shape, strength, and angle of impingement are critical. With respect to the substrate, hardness and simultaneous corrosion processes are critical. Here, again, this subject is just now being seriously considered by the technical community and only generalities are available at present.

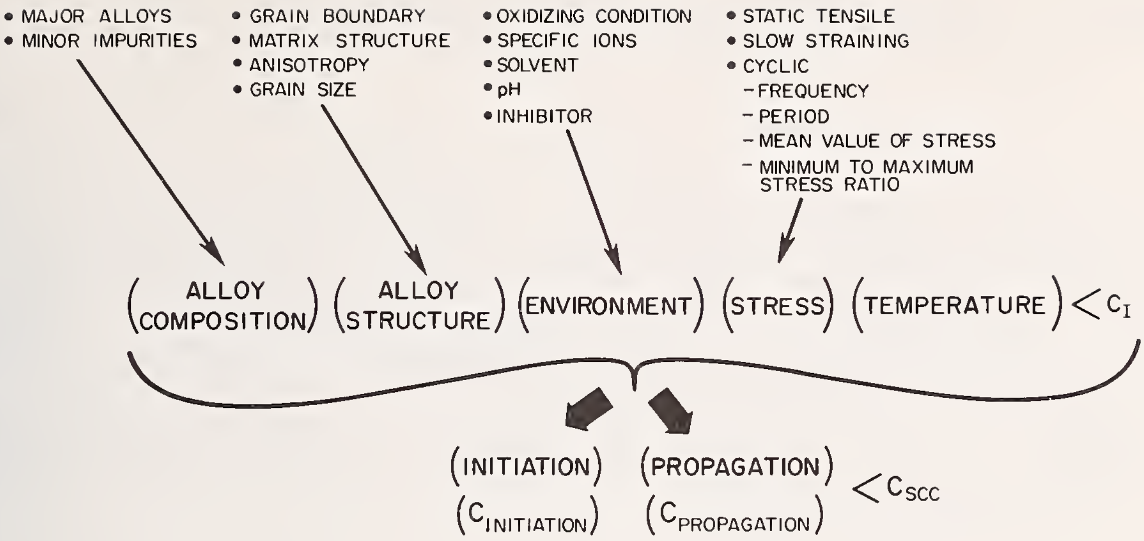


Figure 20. Five factor relationship giving critical components in stress corrosion cracking.

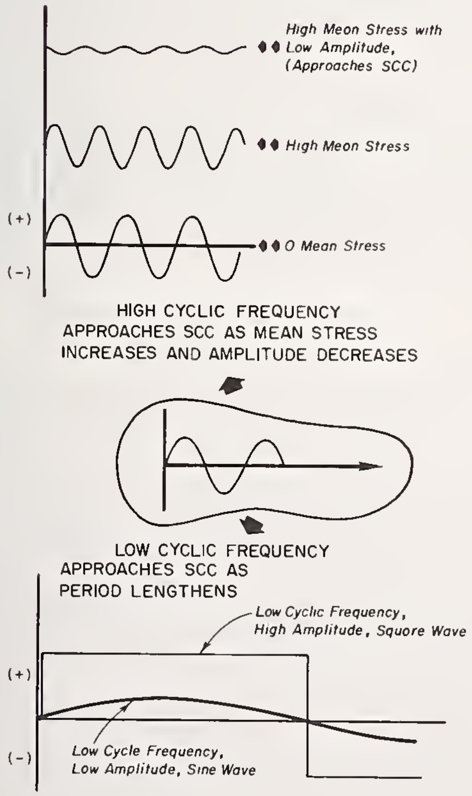


Figure 21. Schematic diagram showing how fatigue parameters approach constant load conditions, i.e., corrosion fatigue approaches stress corrosion cracking.

The general problem of abrasive and erosive wear are discussed generally in the volume from the Ohio State Conference (1).

Changes in Alloy Structure With Time: An implicit part of the performance of coal conversion equipment is its operation at relatively higher temperatures for long times. These temperatures approach those of interest to phenomena such as sensitization of stainless steels, temper embrittlement and sigma phase formation depending upon the alloys. The phenomenon of sensitization in metals has been extensively studied and relationships for the development of the sensitized condition have been determined experimentally (32). Similarly, the phenomenon of temper embrittlement has been extensively studied and the conditions of alloy chemistry, temperature and time, again have been generally developed (33, 34).

These time-dependent changes are important as they contribute to unexpected degradation at a later time in the operation of equipment. This implies a particularly important mandate in the early testing of materials: i.e. that these changed conditions should be evaluated early in laboratory testing for any enhanced susceptibilities to failure which they may imply.

The Weak Link: As engineering systems become larger and operate under progressively more punishing circumstances, the possibility of failure of any of the components increases. Further, there is an increased potential for failure owing to the fact that it becomes progressively more expensive to conduct proof testing on large scale equipment. There is a growing tendency to attempt predicting reliability without going through the prototype stage. Such an approach is generally disastrous but it seems to be the current procedure. Such an approach in this area should be avoided.

Simply stated, the concern of this section is the following: when any one component is shut down, the entire system is shut down; and the length of time necessary to get back on stream depends on the time it takes to repair the failed component. One of the most striking examples occurs in the nuclear industry with the failure of condensers. Condenser failures have contributed substantially to the downtime of nuclear power plants. It would be thought that condensers are a part of "standard engineering;" however, they are not and are frequently subject to leaking. Failures also occur in steam turbines despite the fondest hopes of their manufacturers. Again, these are supposed to be standard items of commerce, but failures are all too frequent. When the turbine or condenser fails the entire system must be stopped until the offending equipment is repaired.

In view of such past history, it seems appropriate that more careful consideration be given to the possibility of such failures. Hopefully, a more balanced approach to design and development would be taken for these large systems.

Acknowledgements: It is a pleasure to acknowledge several contributions to the development of the ideas in this paper. The suggestions to use Table 1 I obtained from Dr. John Smith of the National Bureau of Standards. I discussed the matter of contributions of ceramic materials with Dr. Jack Wachtman also of the National Bureau of Standards. Further, I am indebted to the National Science Foundation and the former Office of Coal Research now of ERDA for its support of the Ohio State Conference on Materials Problems and Research Opportunities in Coal Conversion. Their financial support and encouragement in organizing this meeting got me initially involved in the subject. Finally, I would like to acknowledge the able assistance of Dr. Arun Agrawal, Mr. Gary Welch, and Ms. Catherine Ward in helping with the manuscript.

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DISCUSSION

W. Netter, Lone Star Lafarge Company: You mentioned coating and shot peening. Are these techniques good or bad?

R. W. Staehle: Those were some options that could be utilized for amelioration. Shot peening is a very effective tool and greatly under utilized, especially in the power industry. Unfortunately, at higher temperatures the beneficial effects of shot peening anneal out. We are restricted to using materials that the code lets us use. Unfortunately, many of the standard industrial alloys are very susceptible to cracking. We are going to have to learn how to work within the code and use more ameliorative procedures like coating and shot peening. For example, using a chromate type surface coating in components of the LMFBR's that contact sodium is valuable. It is now clear that chromate is a very good inhibitor for caustic stress corrosion.

THE AGA-ERDA-MPC PROGRAM ON MATERIALS FOR THE GASIFICATION OF COAL

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Abstract: This paper will aim to present an overview of a large program, a portion of which will be described by another speaker. The roles of AGA, MPC, and ERDA and the various contributions of their respective organizations will be described. The attack on the problem, and its division into "Phases" will be explained. The scope of each phase will be clarified. The plans for Phase V will then be described at least as fully as in the latest work schedule. Plans of MPC include ultimate publication of this work in a form and by an organization which will make it possible for the book to be referenced in scientific literature.

Key words: Aluminum alloys; aluminum bronzes; cast iron; coal gasification; corrosion, diffusion coatings, erosion; field corrosion tests; heat resistant steels; liquid immersion tests; nickel alloys; pitting; refractories; steels; stress corrosion; titanium alloys.

I. INTRODUCTION

The AGA-ERDA-MPC Program for Materials for the Gasification of Coal was developed in an Ad Hoc Task Group set up in Subcommittee 8 (Corrosion) of the Technical Advisory Committee of The Metal Properties Council, Inc. The importance of the subject was suggested by Dr. Ab Flowers of the American Gas Assocation. The Task Group was and is still chaired by Mr. William Hulsizer of The International Nickel Co., Inc. The Task Group started its deliberations in 1972. In 1972 a program was formulated duly approved in accordance with MPC practice. The American Gas Association agreed to finance it, and a Contract was placed with the IIT Research Institute of Chicago in 1972.

As of January 1, 1975 the Energy Research and Development Administration assumed responsibility for most of the financing of the program, the American Gas Association retaining a small portion of it, however. The Technical Director of the Fossil Fuel Division, Dr. Henry E. Frankel is ERDA's Technical Director for this program.

II. THE METAL PROPERTIES COUNCIL, INC.

The Metal Properties Council, Inc. is a not-for-profit corporation, organized under the laws of the State of New York, and by whom it is chartered. It was formed in 1966 to make it possible for companies, associations, universities, and government agencies to pool their resources, both technical and financial, to make available reliable information on the properties of metals.

Much data on the properties of metals exists in the literature, and in the several data banks which are available for this work. An unknown quantity exists in company and institutional files. It is costly and time-consuming to extract the data in the latter conditions, and it is also sometimes difficult or impossible to appraise.

Testing programs are costly and time consuming, and the evaluation and arrangement of information is perhaps the greatest problem of all; and the one which must be done by competent workers. It is aimed to publish Metal Properties Council data only after analysis and evaluation by experts, and also after review and approval by a group of representatives of varied interests and backgrounds. The work of the Council is directed to specific areas of need as expressed by industry or government, and is carried out in a number of programs such as the one here under discussion.

III. THE ORGANIZATION OF THE MPC TASK GROUP

The Metal Properties Council, Inc. is an effective organization for accomplishment in its field because it offers a unique combination of a strongly business-oriented Board of Directors, and a Technical Advisory Committee of the best experts in industry. A low-overhead small staff operation efficiently manages the programs of the Council utilizing all funds given to the Council as quickly and efficiently as possible.

The Technical Advisory Committee has a number of technical subcommittees as well as task groups reporting to it. One of these, Subcommittee 8, is responsible for those projects of the Council which are concerned with corrosion. Since it has been in existence under

Subcommittee 8, the Task Group on Materials for the Gasification of Coal has grown to a total membership of 92, and it truly represents all elements of interest in designing, building, and operating pilot plants and the succeeding production units for the stated purpose.

IV. THE PROBLEM IS DIVIDED INTO PHASES

It has been found to be helpful to divide this large program into at least five phases, which may be designated as follows.

Phase I - Gaseous Corrosion - Laboratory Tests
II - Exposure Tests in Pilot Plants
III - Aqueous Corrosion - Laboratory Tests
IV - Erosion-Corrosion - Laboratory Tests
V - Engineering Properties of Materials

The procedure which was followed in developing programs for all of the phases which have been activated so far is indentical in all cases.

After planning the work to be done in the Phase Group, and obtaining the necessary approval from Subcommittee 8 and the Technical Advisory Committee, inquiries were sent out to a number of laboratories, and proposals were received from those prepared to undertake this work. These proposals were reviewed by the Task Group. In some cases visits were made to inspect the facilities and to meet the personnel of the most responsive bidders. Contracts were subsequently negotiated with those bidders considered by the Group to offer the best technical combinations. Contracts for Phases I to IV were awarded to the Illinois Institute of Technology Research Institute. Notice of award of Contract has been issued to Southwest Research Institute for the work planned so far for Phase V.

It is understood that another speaker will describe the programs being conducted at IIT Research Institute for Phases I to IV. The remainder of this paper will describe the work that is being done on Phase V.

V. PHASE V. ENGINEERING PROPERTIES OF PROMISING CANDIDATE MATERIALS - DATA SOLICITATION

The Sub Section for Phase V was assigned the task of developing the information on materials of construction needed by engineers. Phases I to IV are screening a number of candidate materials. Phase V will work on the successful candidate materials as revealed by Phase I to IV. Mention should be made of another Sub Section of the Task Group on Materials for the Gasification of Coal which relates to all Phases of the program. This Sub Section which does not bear an identifying number is charged with the selection of materials for test. It is an important factor in planning the work and in reviewing the claims of all materials suggested for inclusion in the Program. It is chaired by Mr. J. M. Bates of the Union Carbide Corp.

The Sub Section for Phase V conducted during 1974 a search for data on the properties of seven alloys which were at that time considered to be promising. A number of companies, as a result of solicitation by The Metal Properties Council, submitted information from their files.

Alloy 800
Type 310 Stainless Steel
Type 314 Stainless Steel
Rolled Alloys 330
HK - 40
HK - 40 - 3 percent Si
50 Cr - 50 Ni.

Meetings of the Sub Section have been held with engineers to determine what information is needed. Initially, that requested may be divided into three categories - physical properties, mechanical properties, and general characteristics. The information desired must be carefully identified and characterized. It is essential that the product form, complete method of manufacture, dimensions, location and orientation of test specimens be known. The test data desired should permit statistical evaluation. It is sometimes most important to be aware of the range of properties to be expected in materials, as they are available commercially. There follow the properties on which data was solicited in 1974.

Physical Properties

1. Melting Point (or Range)
2. Specific Heat
3. Thermal Expansion (Coefficients)
4. Thermal Conductivity (Coefficients)
5. Density
6. Moduli of Elasticity
7. Poisson's Ratio

Mechanical Properties

1. Tensile Properties, temperatures up to 1850F
2. Creep and Creep-Rupture Properties, temperatures up to 1850F

3. Impact Strength (CV), temperatures up to 1850F
4. Fatigue Strength (several frequencies), temperatures up to 1850F

General Characteristics

1. Weldability - Physical & Mechanical Properties of Weld
2. Castability - Forgeability
3. Machineability
4. Stability
5. Effectiveness of Surface Treatment (Aluminizing, Coatings, etc.)

Response to the solicitation has resulted in some information which has been put into the hands of MPC's analyst, Dr. George V. Smith. It is apparent that available data is not adequate. Isolated, one-heat data, unsupported by characterization of the material, does not enable the engineer to design effectively. It was never anticipated, of course that there would be available from existing files the information needed for materials for the gasification of coal on the effects of the hostile environment on the mechanical properties of the materials to be utilized.

MPC's close relationship with the ASME Committee for the Code on Boilers and Pressure Vessels indicates that there will be a continuing need for more data as experience is gained, changes are made in design, and new materials for construction become available. The important accomplishment to be sought in the conduct of Phase V is to bring to the design engineer a valid body of data supporting certain values of those properties deemed essential. It is hoped that MPC's long experience with the Boiler and Pressure Vessel Code will facilitate this desired end.

Mention should be made of the fact that refractories are being subjected to exposure tests in pilot plants. The program that is reported here will most certainly expand as the screening and pilot plant exposures indicate the value of materials not included in the Phase V tests at this time. It is possible that additional tests will need to be planned on such materials as weldments, coatings, etc.

VI PHASE V. ENGINEERING PROPERTIES OF PROMISING CANDIDATE MATERIALS. THE TESTING PROGRAM

A. Objective

The objective of the Testing Program of Phase V is to generate engineering data on materials which are likely candidates for use in the construction of plants for the gasification of coal. These data include both physical and mechanical properties of the materials in coal gasification environments. Final details of the work schedule for this program are currently being worked out. This report of the current concept of the program may ultimately be modified in details.

B. General Specifications

1. Materials The materials to be tested have been slightly increased in number over those for which data was solicited. They have been divided into groups for convenience in test layout (Table I).

The mechanical and physical property data specified is to be determined for the materials in both Group I and Group II. In addition, selected data will be determined for welded joints produced with the materials in Group I only.

Materials will comply with the applicable ASTM Standards. All materials will be identified by heat number, manufacturing history, product form, size, and chemical composition. All materials will be characterized metallographically by representative photomicrographs. Representative hardness tests will be made and recorded of all materials in the state in which they are tested.

At least three lots (heats) of each material will be procured and tested as specified below. Three specimens will be tested for each condition of test unless otherwise specified.

All welding materials to be used will conform to AWS and ASME standards. All welding filler materials will conform as closely as possible to the chemical composition of the base metal. Welding procedures and operators will be qualified in compliance with the ASME code for pressure vessels to ensure weld integrity and uniformity. All weldments will be fully radiographed and radiographs retained.

All materials and welding processes will be approved for use by the MPC Sub Section which includes the ERDA Technical Director or his representative.

2. Environment - The chemical composition (in mole percent) of test Coal Gasification Atmosphere (CGA) decided upon for the Phase V tests is shown in Table II.

C. Tasks

1. Mechanical Tests

In general, a full set of tests conducted in air is required to serve as a base line for the data developed in the CGA environment. Specimens will be pre-exposed to the CGA at 1000 psi for 1000 hours at various temperatures. Subsequent tests at various temperatures and in prescribed atmospheres shall be conducted as described under each type of test. The contractor is required to have the capability of conducting all tests at a maximum of 2000F if it is subsequently decided to be necessary.

All tests are to be carried out in accordance with applicable ASTM specifications, utilizing standard specimens.

a) Tension Tests

Triplicate specimens from one lot of each material in Group I together with the weldments made from the same lot from Group I will be tested in the condition of the metal as received and in the conditions after exposure in air and in the CGA environment as indicated in Table I. For each test specimen the properties to be determined are (1) yield strength, (2) ultimate tensile strength, (3) percent elongation, and (4) reduction in area to fracture.

The various conditions for which data shall be generated are listed in the Table I. All pre-exposures in the CGA environment will be accomplished at 1000 psi for 1000 hours while the exposures in air are to be conducted at atmospheric pressure for 1000 hours. The tension tests will be run at atmospheric pressure. The effects of various pre-exposure treatments on the microstructure will be documented. In addition, corrosion penetration resulting from the various pre-exposures will be documented from inspection of broken tension test specimens. Tension tests of pre-exposed specimens will be made without removal of any scale.

b) Impact Tests

Charpy V-notch tests will be conducted in triplicate for the same materials for which tension tests are conducted. The results will be reported as fracture impact energy in foot pounds, lateral expansion, and percent shear. The pre-exposure and test matrix for the impact tests shall be the same as that shown in Table I for the tension tests, except that all Charpy tests will be conducted in air and at room temperature.

c) Stress Rupture Tests

Replicate stress rupture tests will be conducted on all lots of material in Group I and Group II and the weldments in both air and the CGA environment. Initial tests will determine the stresses required for rupture in 100, 300, and 1000 hours of exposure in all materials. Subsequently, 10,000 hour tests will be conducted on the four most rupture-resistant materials. Tests will be conducted at 1200F, 1500F, and 1850F in air at atmospheric pressure and in the CGA environment at 1000 psi using 0.357 inch diameter smooth round specimens.

Complete creep curves will be developed for each test together with tables of stress, rupture time, elongation, reduction in area and minimum creep rate.

d) Fatigue Tests

Low cycle fatigue tests will be conducted at 1200F, 1500F, and 1850F in air at atmospheric pressure and in CGA environment at 1000 psi. The specimen geometry and exact loading cycle will be determined by the contractor and approved by the monitoring MPC sub section.

Since the combined effects of creep and fatigue greatly affect the number of cycles to failure in low cycle fatigue at elevated temperatures, it will be required to initially screen several materials to determine the cyclic hold time at which the creep effect saturates. This initial screening program will use specimens from one lot of Alloy 800, one lot of Type 310 stainless steel, and one lot of HK-40.

2. Physical Property Determinations

The following physical properties will be determined by ASTM established methods for three lots of each material (see Table IV).

VII. CONCLUSION

Recognition must be given to the fact that representatives of at least 75 companies have shared in the formulation of this program; and will continue, as their contribution, to take part in the monitoring of the work as it proceeds. Finally, they will appraise and evaluate the results so that the final report will represent the combined thinking of a majority of those who may be expected to participate in this new-old industry, the gasification of coal.

Obviously this program might be expanded as results indicate the advantages of other materials, as pilot plant operations indicate operating environments significantly different from those chosen for test at this time. We are assured of new and significant data, critically evaluated which cannot help but be of value in our current efforts to assure our supply of energy.

TABLE I

Phase V
List of Alloys for Initial Testing Program

Group I	Alloy 800
	Alloy 800, Alonized
	Type 310 Stainless Steel
	Type 310 Stainless Steel, Alonized
	HK-40
	50 Cr - 50 Ni
Group II	Type 309 Stainless Steel
	Type 314 Stainless Steel
	RA-330
	HK-40-3 Si

TABLE II

Chemical Composition (mole percent) Coal Gasification Atmosphere Phase V

CO_2 - 12 percent
CO - 18 percent
H_2 - 24 percent
CH_4 - 5 percent
NH_3 - 1 percent
H_2S - 0.5 percent
H_2O - Balance

TABLE III
Tensile Test Plan

State of Material	Pre-exposure		Test		
	Environment	Temp. (°F)	Air	CGA	Temp. (°F)
As Received	None		x		80
			x	x	1850
Pre-exposed 1000 hrs.	Air (atmospheric pressure	1200	x		80, 1200
		1500	x		80, 1500
		1850	x		80, 1850
Pre-exposed (1000 hrs.)	CGA (1000 psi)	1200) 1500) 1850)	x		80
		1200	x	*	1200
		1500	x	*	1500
		1850	x	*	1850

TABLE IV

Physical Properties
Phase V

Melting Point (Range)
Specific Heat
Thermal Expansion (room temperature to 2000°F)
Thermal Conductivity (room temperature to 2000°F)
Density
Moduli of Elasticity (up to 2000°F)
Poisson's Ratio (up to 2000°F)
Emissivity

TABLE V

Task Group on Materials for the Gasification of Coal

Numbers of Members - April, 1976

The Task Group 91

Phase 1	22	Phase 4	24
2a	29	5	16
2b	16	6	15
3	19		

Steering Committee 14

TABLE VI

AGA-MPC-ERDA PROGRAM on Materials for the Gasification of Coal

Phase I	Gaseous Corrosion
IIa	Pilot Plant Tests - Metals
IIb	Pilot Plant Tests - Refractories
III	Aqueous Corrosion
IV	Erosion-Corrosion
V	Engineering Properties
VI	Special Problems Rotating Parts

DISCUSSION

G. M. Ugiansky, National Bureau of Standards: In our work we have seen tremendous differences in the mechanical properties and the mode of failure at different strain rates. Is any consideration being given to testing these materials at various strain rates?

A. O. Schaefer: Yes, but we think that in the work we are doing now, strain rate should not be a variable. We are watching your work with considerable interest. Incidently, we are purchasing double quantities of material so that this fully characterized material can be tested at various strain rates if such tests are indicated by programs such as yours.

D. Canonico, Oak Ridge National Laboratory: You mentioned that we are concerned with internals in this program, and they are not part of the code. But do you foresee, from a reliability point of view, that we are going to have to set standards for materials used in coal conversion processes that take into consideration the environments to which these materials are subjected?

A. O. Schaefer: Yes, I do. We hope the basis of such codes will be reliable information. We need to get the information fast enough so that these codes will be developed on a sound basis.

D. Canonico: I just wanted to make sure we were not on different tracks. We are considering that indeed someday we will have some sort of standards even for internals to insure reliability.

SESSION II

FAILURE ANALYSIS

CHAIRMAN: E. E. KLAUS,
THE PENNSYLVANIA STATE UNIVERSITY

FAILURE ANALYSIS OF COMPONENTS FROM COAL-GASIFICATION PILOT PLANTS

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Abstract: Construction materials in coal-gasification pilot plants are subjected to a variety of corrosive/erosive environments, ranging from liquids and solids at relatively low temperatures to gas and solid mixtures at high temperatures and pressures. Component failures may be directly attributed to interactions between the materials and the corrosive/erosive environment. Two corrosion failures that have been examined are discussed in detail: (1) Inconel 702 thermocouple sheath exposed to a multicomponent gas environment at 2000°F and 1200 psi, and (2) Types 304 and 316 stainless steel instrument tubing containing superheated steam-oxygen mixtures at 600°F and 1200 psi. The failures were examined in detail using optical metallography, scanning-electron microscopy, and electron-microprobe techniques as well as chemical analyses and nondestructive testing methods. The failure of the thermocouple sheath was attributed to internal oxidation of chromium and aluminum present in Inconel 702. The failure of the instrument tubing was associated with chloride stress-corrosion cracking. Details of the analyses and possible solutions to prevent such failures in the future will be presented. In addition, other ANL investigations of gasification plant components are summarized.

Key words: Coal-gasification plant-component failure; HYGAS plant; Synthane plant, stress-corrosion cracking; internal oxidation; scanning-electron microscope and electron-microprobe techniques.

An early step in the program to use the nation's considerable coal supply in the form of clean gaseous fuel is the design and construction of a series of coal-gasification pilot plants. As these pilot plants continue to come on line, materials-related problems are becoming evident. These problems must be understood and solved before the demonstration plants are reliable for long-term operation in hostile environments, i.e., high temperature and pressure, corrosive chemicals, and abrasive solid, liquid, and gaseous media.

At Argonne National Laboratory we are engaged in a cooperative effort with various pilot plants to examine failed and unfailed components after removal from service. As a result of our examinations and analyses, we expect to be able to recommend changes in materials and/or operating conditions that will lead to improved performance. In addition, through timely and widespread dissemination of results, we hope to make all pilot-plant operators aware of generic problems and so minimize repetition of problems. Since February 1975, twelve investigations have been made of failures and related materials problems at the HYGAS plant in Chicago and the Synthane plant in Bruceton, Pennsylvania (Table I). In the present paper, we will discuss two of the HYGAS investigations in detail and summarize several others.

I. HYGAS Thermocouple Sheath

An Inconel 702 sheathed thermocouple assembly that was exposed to a multicomponent gas environment at 2000°F and 1000 psi in the steam-oxygen gasifier was severely embrittled and cracked after approximately 3-4 months of service. The composition of the gas that was present in the vicinity of the thermocouple sheath is shown in Table II. Chemical analysis showed that the sheathing contained a sulfur concentration of 0.010 wt% (within the maximum specified for Inconel 702) and an oxygen concentration of 0.784 wt% (significantly higher than the specifications for the alloy). Chemical analysis of acid soluble and insoluble fractions demonstrated that the former is predominantly a nickel-rich phase and the latter consists of chromium, manganese, and aluminum. Formation of stable oxides (Cr_2O_3, Al_2O_3, MnO, etc.) in the sheathing material is a distinct possibility in view of the high oxygen concentration and the acid insoluble composition. Chemical analysis of the ceramic packing in the thermocouple assembly showed that magnesium was the major constituent.

X-ray diffraction patterns of the thermocouple sheathing material and the ceramic packing were made to identify the type of phases present in the materials. The powder patterns of the sheathing indicated predominantly nickel (cubic structure) and lines corresponding to Cr_3O_8, Cr_2O_3, Cr_3O_4(noncubic structure). Only traces of chromium sulfate were evident. The powder patterns of the ceramic packing showed that the material was predominantly MgO with traces of $Mg(OH)_2$. Optical microscopy showed that the tubes exposed in the gasifier had significant precipitation of a second phase.

Fracture and metallographically polished surfaces of the tube were examined by means of the scanning-electron microscope (SEM) and analyzed using the SEM in the energy-dispersive X-ray mode. The matrix phase was identified as nickel with small amounts of Fe, Cr, and Al. The second phase was found to be chromium rich, and some aluminum was also detected in the polished specimens.

To establish the composition of second-phase particles, an electron-microprobe analysis was performed. From this analysis, it was shown that the matrix phase was nickel and the second-phase particles were chromium and aluminum oxides. Although extensive examination of the tube wall was conducted to determine the presence of sulfur, none was detected. The composition of the second phase, determined in four locations of the sheathing, is listed in Table III. The quantitative analysis shows that the second-phase regions are predominantly chromium oxides with a small concentration of aluminum oxide.

The results of the analyses presented indicate that the Inconel 702 thermocouple sheathing had undergone internal oxidation of chromium and aluminum in the alloy during its service in the steam-oxygen gasifier. The oxidation characteristics of Ni-Cr alloys have been extensively studied by a number of investigators (1-3) and their results indicate that the process of oxidation of these alloys is a strong function of the chromium concentration of the alloy and temperature. For alloys containing <10% chromium, the oxidation process leads to NiO and $NiCr_2O_4$ at the surface and an internal subscale of Cr_2O_3. In alloys containing >20% chromium, the oxidation process at 1100°C results in the formation of a continuous layer of Cr_2O_3 on the surface of the alloy. The Cr_2O_3 layer is protective in nature and any additional oxidation is significantly lowered due to the slowness of the oxygen diffusion rate through the oxide layer. The chromium concentration required for external scale protection of the alloy increases with a decrease in temperature, requiring $\sim$30% at 800°C. In the chromium concentration range of 10 to 20% and at a temperature of 1100°C ($\sim$2000°F), the alloy is in the transition region between internal oxidation and external protective scale formation.

Wagner(4) has proposed that the transition from internal to exclusive external oxidation should occur when the solute content of the alloy is sufficient to form a critical volume fraction of internal oxide precipitate at the reaction front. The volume fraction of Cr_2O_3, $v_{critical}$, for the Ni-Cr system can be defined as follows:

$$v_{critical} = N^{\circ}_{Cr} \ \alpha \ V_{Cr_2O_3} \Big/ 2V_{alloy}, \qquad (1)$$

where N°_{Cr} is the mole fraction of chromium in the bulk alloy, $V_{Cr_2O_3}$ and V_{alloy} are the molar volumes of the oxide and alloy, respectively, and α is an enrichment coefficient. The value of α is determined by the diffusion rates of chromium and oxygen in the alloy. The volume fraction of Cr_2O_3 phase in the nickel matrix determined in the failed sheathing using an Image Analyzing Computer Company Quantimet 720 yields values in the range of 28 to 34%. Using $N^{\circ}_{Cr} \sim 0.199$ (corresponding to $\sim$15.6 wt% Cr in Inconel 702), $V_{Cr_2O_3} = 29.2$, and $V_{alloy} = 6.6$ in Eq. (1) yields a value of 0.71 for α. The insertion of this value of α in Eq. (1) and calculation of the volume fraction of Cr_2O_3 phase in Ni-20 wt% Cr alloy yield a value of 0.42. This value should correspond to the critical volume fraction at 1100°C, since the Ni-20 wt% Cr alloy oxidizes with the formation of an external scale.

The Inconel 702 thermocouple sheathing exposed in the steam-oxygen gasifier at 1100°C was found to be internally oxidized. The oxidation products were identified as Cr_2O_3 and Al_2O_3 using optical and scanning-electron microscopy, electron microprobe and X-ray diffraction techniques, and chemical analysis. The internal oxidation of the alloy can be prevented by increasing the less noble solute content (i.e., chromium) to a level that exceeds a critical composition whereupon a compact protective layer of solute element oxide is formed at the external surface. This necessitates selection of a nickel-base alloy with >20 wt% chromium or an alloy such as Type 310 stainless steel that is inherently superior in resistance to oxidation. Type 310 stainless steel was substituted for the Inconel 702 and has performed satisfactorily. Use of a nitrogen purge may be contributing to the satisfactory performance.

II. HYGAS Instrument Tubing

Types 304 and 316 stainless steel instrument tubing containing superheated steam-oxygen mixtures at 600°F and 1200 psig failed after brief (hours to days) service. Failures varied from small pinhole leaks to massive tube rupture.

Metallographic examination revealed that cracking originated at the inside surfaces of the tubes and was mixed intergranular and transgranular. Chemical analysis of boiler feed water indicated erratic chloride control. Concentrations of 2.4 and 0.1 ppm were observed. Analysis of the pipe insulation showed that it met nuclear specifications for stainless steel pipe insulation. Energy-dispersive X-ray analysis using the SEM showed the presence of chloride in the scale of the Type 316 stainless steel process piping associated with the instrument tubing, but chloride was not detected in the inside surfaces of the instrument tubing. This is not considered surprising in view of the relatively high detection limit of the analytical technique and the fact that the process piping was exposed to a much greater volume of fluid than the instrument tubing.

The method of plant operation led to the conclusion that failure was associated with chloride stress-corrosion cracking. When the plant is down, the steam system is shut off, thus permitting condensation and possible air in-leakage. On start-up, operating pressure is achieved more rapidly than operating temperature, a condition which also promotes condensation. In fact, due to the length of the lines, it is probable that continuous refluxing takes place in these lines during plant operation. At 1200 psig, the saturation temperature is 569°F. These operating conditions are conducive to intermittent locally high chloride concentrations.

In studies(5) in support of nuclear-fueled superheaters, a variety of alloys were exposed under stress to superheated steam generated from water containing 1.5 ppm chloride, oxygen, and hydrogen. Maximum temperatures varied from 560 to 1250°F, and the temperature was cycled to produce condensation. Austenitic stainless steels failed by transgranular or a combination of transgranular and intergranular cracking. A ferritic stainless steel (Type 406) and high nickel alloys did not fail.

The HYGAS components under investigation operated under similar, although probably less severe conditions. Hydrogen is probably not present but is not believed necessary for the observed failures. Oxygen may not be present in all lines.

Based upon these results and the studies referred to above, Incoloy 800 or Inconel 600 are more suitable materials of construction. Incoloy 800 was substituted for 18-8 stainless steel and has performed satisfactorily in excess of 500 h.

The Type 316 stainless steel process line is considered to be vulnerable, particularly in those areas where condensation can take place, e.g., in horizontal runs. A surveillance program is under way, but to date no incipient cracking has been observed.

III. Other ANL Investigations

A. Type 304 Stainless Steel Purge Lines - HYGAS

These lines carry Chicago city water at 200°F and 1200 psi. They failed by cracking upon startup. The failure was diagnosed as chloride stress-corrosion cracking, and the results of metallographic examination were consistent with this hypothesis. The lines are incompletely drained between runs and evaporation of the remaining water can result in locally high concentrations of chloride upon start-up. (Chicago city water contains about 10 ppm chloride.)

The problem could be alleviated by maintaining the lines always full of water or using an ion exchanger to remove chloride (flow rate is very low, $\sim\frac{1}{2}$ gpm). Based upon our recommendation with respect to the instrument lines, the plant operator chose to substitute Incoloy 800 for the Type 304 stainless steel. Results to date are satisfactory.

B. Carbon Steel Coal Pretreatment Vessel Cooler - HYGAS

The central (water inlet) pipe was severely bowed and the pipe-to-header weld had cracked. Other pipes were less severely bowed. This heat exchanger serves to maintain temperature of the exothermic reaction. Stress analysis indicated the failure was caused by buckling as a result of excessive cooling of the failed pipe section. The excessive cooling was the result of poor temperature distribution produced by the use of six coolers instead of the four for which the process had been designed.

In an associated investigation of a failed thermocouple and thermocouple protection tube from the same vessel, temperatures much in excess of the design temperature were demonstrated to have existed in other parts of the vessel.

C. Type 321 Stainless Steel Bellows - HYGAS

This bellows, part of the start-up burner assembly, was perforated. Metallographic examination revealed extensive melting had occurred. Chemical, energy-dispersive X-ray and Auger electron analysis of deposits found in the bellows demonstrated that coal products had reached the interior of the bellows.

D. Incoloy 800 Bellows - HYGAS

This bellows, part of the main gasifier transfer-line expansion joint, was perforated. Incoloy 800 piping associated with the assembly was cracked, particularly at welds. The failures may be associated with sulfur attack but the investigation is only in the initial stages.

E. Incoloy 800 Thermocouple Protection Tubes and Pressure Taps - HYGAS

These tubes, from the ash agglomerating gasifier, suffered accelerated wastage, apparently coincidentally with change to a high (∿3% in contrast with ∿0.5%) sulfur char feed. Chemical analysis of the scale revealed a sulfur content of ∿14%. On the assumption that sulfur attack is responsible for the observed wastage, the Incoloy 800 has been replaced with higher chromium Types 310 and 446 stainless steel alloys. The performance of these materials will be monitored.

F. Type 316 Stainless Steel Pipe to Type 304 Stainless Steel Flange Weld - Synthane

The initial purpose of the investigation was to determine the cause of the cracked weld in the socket-welded assembly. This was determined to be the result of mechanical stresses, on a weld that had inadequate penetration.

In the course of the investigation, cracks were also observed in the pipe and diagnosed as chloride stress-corrosion cracking. The source of the chloride was the pressure testing fluid. This fluid was not completely drained from the system and locally high chloride concentrations could be produced when the fluid evaporated and the system was subsequently refilled. Chloride was detected on crack surfaces, inner surface of the pipe, and weld socket.

Acknowledgments

This work was carried out under the auspices of the Fossil Energy Branch of ERDA. Many individuals in the Materials Science Division and Chemical Engineering Division, in addition to the authors, participated in the investigations. The work could not have been as successful as it has been without the complete and wholehearted cooperation of the pilot-plant operators.

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TABLE 1. Summary of Failure Investigations

Plant	Component - Problem	Status
HYGAS	Cracking of 304 SS purge tubing*	Diagnosed as stress-corrosion cracking. 304 SS replaced with Incoloy 800. Satisfactory operation.
	Cracking of 304 and 316 SS instrument tubing**	Diagnosed as stress-corrosion cracking. 304 SS replaced with Incoloy 800. Satisfactory operation.
	Wastage and cracking of main gasifier Inconel 702 thermocouple sheathing**	Diagnosed as internal oxidation. Inconel 702 replaced with 310 SS. Satisfactory operation.
	Cracking of carbon steel cooler for coal pretreatment vessel*	Cracking caused by mechanical buckling resulting from poor temperature distribution. Number of coolers reduced. Satisfactory operation.
	Excessive oxidation of 316 SS coal pretreatment vessel thermocouple protection tube and melting of thermocouple	Related to above failure. Hot solids reacted with thermocouple protection tube. New method of operation results in more uniform temperature and satisfactory operation.
	Perforation of 321 SS start-up burner expansion joint bellows*	Failure due to melting. Coal products reached bellows.
	Perforation of Incoloy 800 main gasifier transfer-line expansion joint bellows*	Investigation in progress. Sulfur attack possible.
	Cracking of Incoloy 800 main gasifier transfer-line expansion joint	Investigation in progress.
	Excessive wastage of Incoloy 800 ash agglomerating gasifier thermocouple protection tubes and pressure taps*	Sulfur attack indicated by high ($\sim$14%) sulfur concentration in scale. Substitution of 310 or 446 SS recommended. Investigation continuing.
Synthane	Identification of piping system materials and evaluation of weld joints	Consulted on materials identification and weld evaluation. System satisfactorily modified.
	Fractured coal-elevator bucket	Investigation terminated because necessary components not available.
	Cracked weldment and pipe in 304 and 316 SS transfer line*	Weld cracked as result of mechanical stresses on joint with inadequate penetration. Pipe crack diagnosed as chloride stress-corrosion cracking.

*Summarized.

**Investigations discussed in detail.

TABLE II. Gas Composition (% by Volume) in the Vicinity of the Failed Thermocouple Sheath in the Gasifier[a]

H_2	20
H_2O	50–51
CO	9–12
CO_2	15–17
CH_4	2
H_2S	∿0.1

[a] The gas composition is that obtained by room-temperature analysis using gas chromatography technique.

TABLE III. Microprobe Analysis of the Second Phase in Thermocouple Sheathing (Concentrations in wt%)[a]

Location	Cr	Ni	Al	O (By difference)
ID	56.0	8.1	7.3	28.6
Center-ID	66.5	4.1	1.7	27.7
Center-OD	62.7	2.7	3.60	31.0
OD	43.3	8.1	16.2	32.4

[a] The oxygen concentrations corresponding to CrO, Cr_2O_3, CrO_3, and Al_2O_3 are 23.5, 31.6, 48.0, and 47 wt%, respectively.

DISCUSSION

P. J. Birbara, Coalcon: When the stainless steel instrument lines were replaced with Incoloy 800, there were no failures after 700 hours. How long did it take for the stainless steel lines to fail?

S. Greenberg: The stainless steel lines failed in a matter of hours to days.

EROSION PROBLEMS IN LETDOWN VALVES

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Abstract: The coal-derived slurries associated with all types of liquefaction processing present serious wear problems for many of the system components. In particular, the throttling valves used to let down the ash-containing slurry from the high processing pressure to near-ambient pressure experience relatively short service life as a result of erosive wear of the internal components. The wear life of cemented carbide trim components in pilot facilities ranges from only about 2 weeks to 2 months. Other components of the valve body have lasted only a few days when not properly protected from direct impingement by the rapidly moving slurry. Design modifications are continually being made to minimize the wear problem and thereby lengthen the time between component changes. The design of trim components is discussed and examples are presented of worn components and some of the valve design modification which have been evaluated. Wear of the critical cemented carbide trim components is shown to occur by the mechanism of removal of the soft metal matrix from between the hard, wear-resistant carbide particles, and the associated pullout of the hard particles. Improvement in the life of trim components is expected to come with minimized metallic binder content or adoption of hard, single-phase ceramic materials.

Key Words: coal liquefaction, letdown valves, wear-resistant cemented carbides, valve trim, wear problems.

Presentation: The present and projected shortage of petroleum has prompted extensive development of the technology for converting coal to useful forms of fuel by the liquefaction approach. In addition to providing much needed fuels, this technology will also yield desirable organic compounds for the chemicals industries. The development of this technology in the United States was started in the late forties by the Bureau of Mines at Louisiana, Missouri. The purpose at that time was to demonstrate that American coals could be processed into liquid fuels in a manner similar to that done earlier in Germany. Despite considerable equipment difficulties this goal was basically achieved, but because of the then ample sources of petroleum the effort was discontinued. During the past decade, however, the need for such technology has been expressed through the establishment of several pilot and experimental facilities in the United States.

All of the presently operating liquefaction facilities are experiencing many of the equipment problems which were encountered in the first demonstration plant. Pumps, seals, valves and piping are being worn by the flowing coal-derived slurries which contain inorganic matter, a large part of which is ash. The inorganic contaminants normally constitute 5 to 15 percent of the coal.

The equipment component which suffers most from erosion by the ash is the letdown valve through which the slurry passes from the zone of high reaction pressure to one of near-ambient pressure. In Figure 1, which shows a flow diagram of a solvent extraction process, the letdown valve is located in the line between the dissolver and the gas-liquid separator. At this point the coal has been fully reacted, and the resulting slurry consists of dissolved coal products and gases and the undissolved erosive agents which are held in suspension. The slurry is typically at a pressure of 1000 to 3000 psi and at temperatures as high as 600 to 800 F. Throttling control is required to maintain the large drop of operating pressure during letdown.

Figure 2 shows the basic design of a typical valve used for slurry letdown. This type of valve has several desirable features over the many other types and designs of valves available, many of which have been tried. One important feature is that the stem and seat trim components are replaceable. They can be removed without breaking a line connection by simply removing the bonnet. The flow direction, which is the reverse of normal for such valves, allows the slurry to enter the stem-containing chamber and to exit through the seat into the larger line-size opening. The expansion of the slurry resulting from volatiles and dissolved gases is thereby more readily accommodated. The exiting slurry does not have to change direction rapidly as would be the case for flow in the opposite direction. Wear is common at surfaces which force the change in direction under conditions of rapid flow.

The trim components of the valve which experience the highest flow rates with associated direction change and turbulence undergo the most severe problems of erosion. Figure 3 shows a typical trim set for throttling control. The stem member may be gradually tapered or else be cylindrical with a tapered flat to provide a changing cross section. In operation, the stem is unseated and withdrawn until the appropriate size of opening is obtained. The stem position during letdown changes as wear and associated change in opening size occur.

Solution of the trim wear problem has been sought primarily through changes in materials of construction, with relatively minor changes in trim design in a given valve. The evolutionary changes in materials have followed a trend from stainless steel to shock-hardened steel to Stellite alloys to cast carbides and finally to the presently used cemented carbides. Evolution in the cemented carbides was generally from the machining grade of tungsten carbide with high cobalt binder content to wear-resistant die grades and finally to specially designed wear-resistant grades containing a more corrosion-resistant cobalt-chromium binder alloy. With the change from metal alloys to the best grades of cemented carbides the service life of trim components increased from a value of a few hours to up to as long as 2 months.

The most wear-resistant carbides have been found to be less tough, and users have experienced a much higher rate of breakage during installation and operation. Stems break under bending loads resulting from misalignment or particle jamming. Seats are broken as the result of tensile stressing at the time of valve closure.

Figure 4 gives examples of the severity of wear that a coal slurry can produce on alloy components within a relatively short time. The bulk of such wear can occur over a period covering anywhere from hours to a few days of operation.

Figure 5 shows a carbide stem member from the H-Coal Pilot Plant in Trenton, New Jersey. It has a typical life span of 2 weeks in letting down a coal slurry from a pressure of 3000 psi. The tapered flat on the 1/4-in.-diameter stem is grooved by wear to a depth of about 1/16 in. The groove pattern varies among stems. The corresponding seat member undergoes comparatively little wear. The stem is believed to experience the major wear because it forces the flowing slurry to reverse direction as it enters the trim set.

Figure 6 shows a stem obtained from the Solvent Refined Coal (SRC) Pilot Plant in Wilsonville, Alabama. This stem is about 3/8 in. in diameter and is shown after 2 months of service in letting down coal slurry from 2000 psi. The wear pattern is quite uniform, with fine wear grooves which appear to relate to the initial pattern of grinding lines. The wear of other replacement stems in the same valve, however, is not always as uniform nor do they have the same length of service. Figure 7 shows a similar stem member which exhibits nonuniform erosive wear. Localized gouging occurred along the flow path, while undercutting in the form of a ring occurred at the junction of the cylindrical and angular sections. The irregular gouging is believed to result from turbulence in the stream of slurry. It has been noted that the seat member, which usually experiences little attack, is attacked and gouged when the stem member is unevenly attacked. This could be expected under turbulent flow conditions. Figure 8 shows a third type of erosive wear in the same type of stem member. The wear is in the form of localized gouging on the cylindrical surface on the side opposite the tapered flat, a location where there is usually no apparent wear problem. The cause of this type of attack is not obvious.

The mechanism of wear of the cemented carbides has been tentatively identified as resulting from erosion of the softer metal matrix from between the hard carbide particles, and subsequent pullout and loss of the hard particles. Figure 9 is a scanning electron micrograph of an eroded surface of a fine-grained cemented carbide. The carbide particles stand well above the underlying metal matrix. The particles are well defined in shape and have, in general, retained their sharp edges. Figure 10 shows an eroded surface of a specimen containing some large carbide particles. The absence of binder can be seen clearly at the exposed surfaces of the large particles.

The wear problem in letdown valves is not solely restricted to erosion of the surfaces of the trim components. One of the problems commonly encountered occurs at the exit side of the trim set where slurry expands and sprays out against the walls of the valve. Since standard designs of valves are used in the larger pilot operations, the exit chambers are of a normal, moderately small size. The exiting slurry strikes the walls with such high energy that it can erode through in times as short as a few days.

The general approach to preventing this type of erosion problem has been to incorporate a cylindrical sleeve of cemented carbide at the exit of the trim set. The wear-resistant sleeve, which is of moderately small diameter, collimates the slurry and directs it axially into the exit line. This works well, if no openings develop between the sleeve and trim set which allow shunting flow between the sleeve and valve body. The spray from this type of leak will also readily cut through the alloy valve body at an accelerated rate. To prevent such leaks, high pressure loadings are required between the sleeve and seat member, and with the more brittle wear-resistant grades breakage can occur, which permits a similar problem to appear. Figure 11 shows an example of erosion of the exit side of a valve seat holder which occurred following breakage of the adjoining carbide sleeve. The broken surface of the sleeve caused the slurry to be directed back in a direction reverse from which it came, thereby causing penetrating attack of the seat holder and ultimately complete penetration of the valve body.

An attempt was made to avoid the above problem of breakage by making the sleeve extension integral with the seat member, thus eliminating the fitup problem between two separate members and the need for application of an axial force. This, however, was not effectively evaluated because of the occurrence of another already experienced problem. A leak path developed between the carbide member and the seat holder where a braze is used to join and seal the members. The location of leakage is shown in Figure 12. The leak at that location occurs as either the result of improper brazing or disruption of the braze caused by the difference in thermal expansion rates of the components. Figure

13 shows the results of the leak on the back side of the assembly. The stainless steel sleeve which surrounds the carbide extension has been grossly penetrated, thereby allowing the slurry to attack the surrounding valve body.

The erosion problems experienced in letdown valves in coal liquefaction service point up the need for development in at least two major areas, namely (1) improvement in the basic materials from which trim components are fabricated and (2) design of valves specifically for this type of service with consideration given to the requirements of the trim material involved. The apparent cause of erosive wear of the cemented carbides presently used suggests that improvements in trim materials will come through the use of hard, probably single-phase ceramic-type materials without a soft binder present or with the binder content significantly reduced. With this trend, the materials may become more fragile and potentially unbrazable. The valves into which these new materials will be incorporated will consequently have to be designed to suit the requirements of those materials.

We acknowledge our appreciation of samples of worn trim supplied by the H-Coal and SRC facilities in support of a program of trim materials development sponsored by the Electric Power Research Institute and monitored by Mr. Howard Lebowitz of that organization.

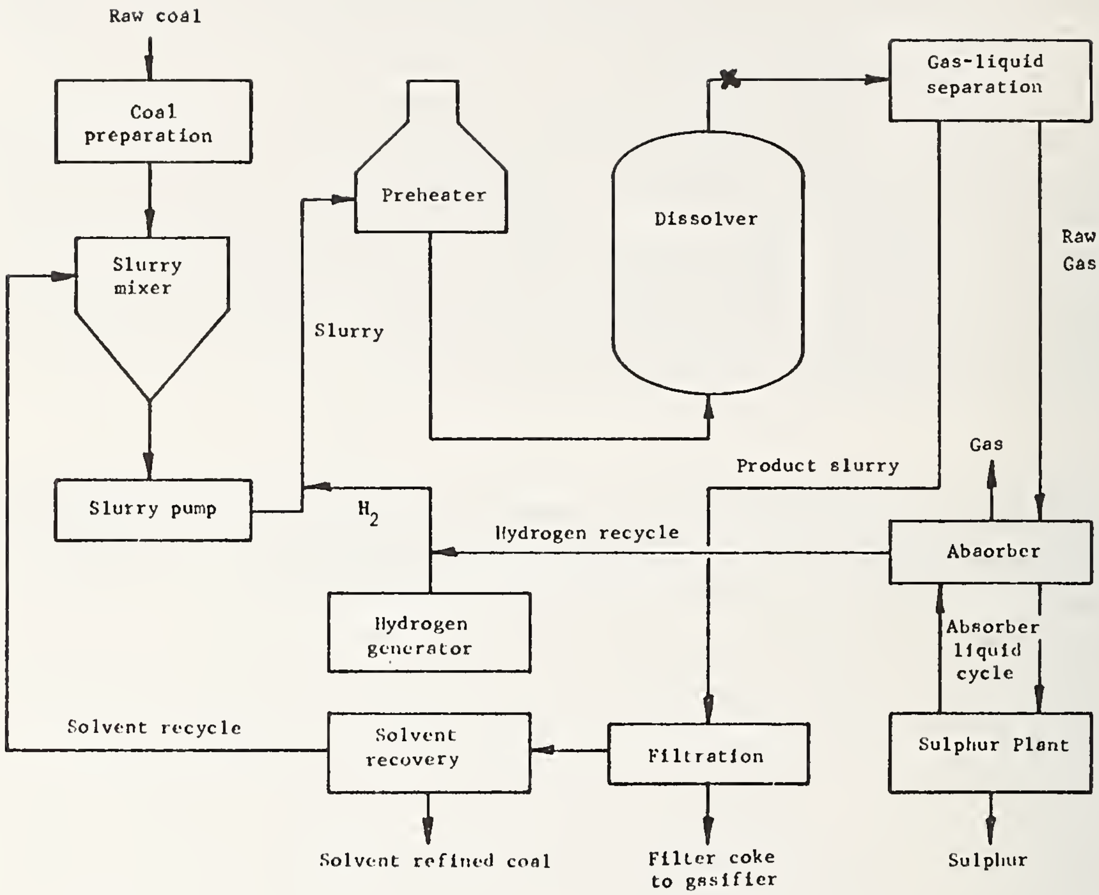

FIGURE 1. FLOW DIAGRAM OF TYPICAL LIQUEFACTION PROCESS

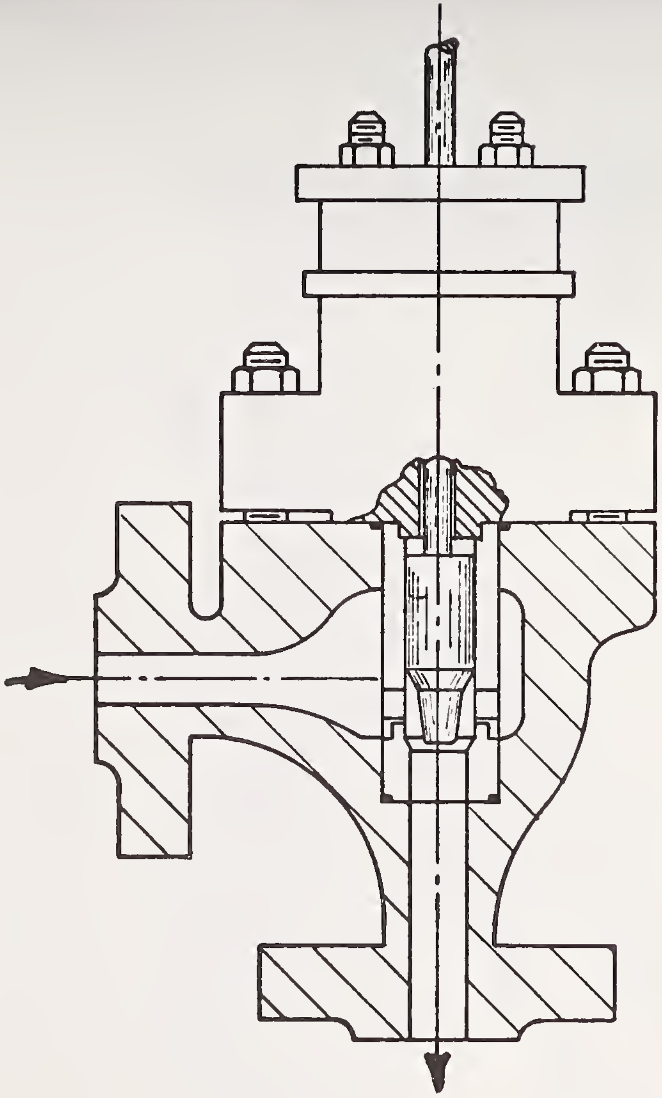

FIGURE 2. DESIGN OF VALVE USED FOR LETDOWN OF COAL SLURRY

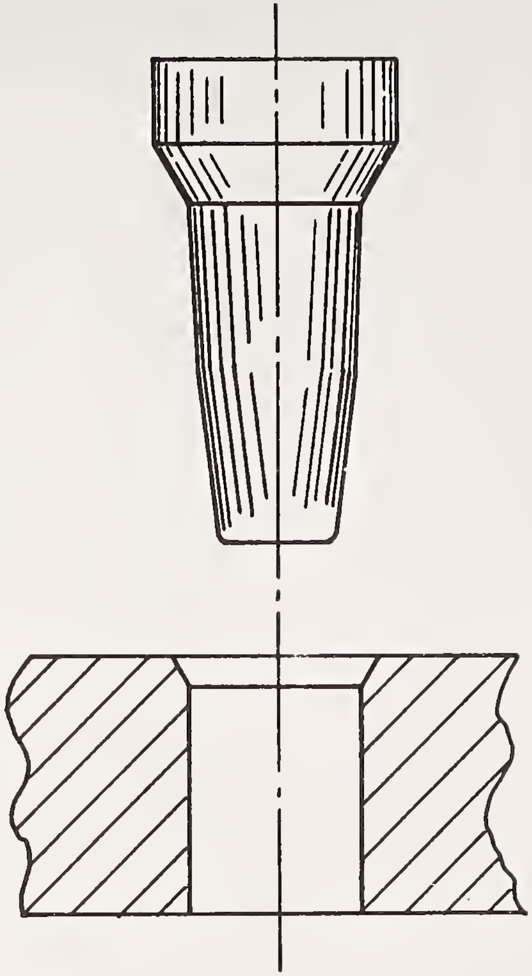

FIGURE 3. DESIGN OF VALVE-TRIM SET

FIGURE 4. ERODED ALLOY TRIM SET

FIGURE 5. WORN CARBIDE STEM MEMBER FROM H-COAL PILOT PLANT

FIGURE 6. WORN CARBIDE STEM MEMBER FROM SRC PILOT PLANT

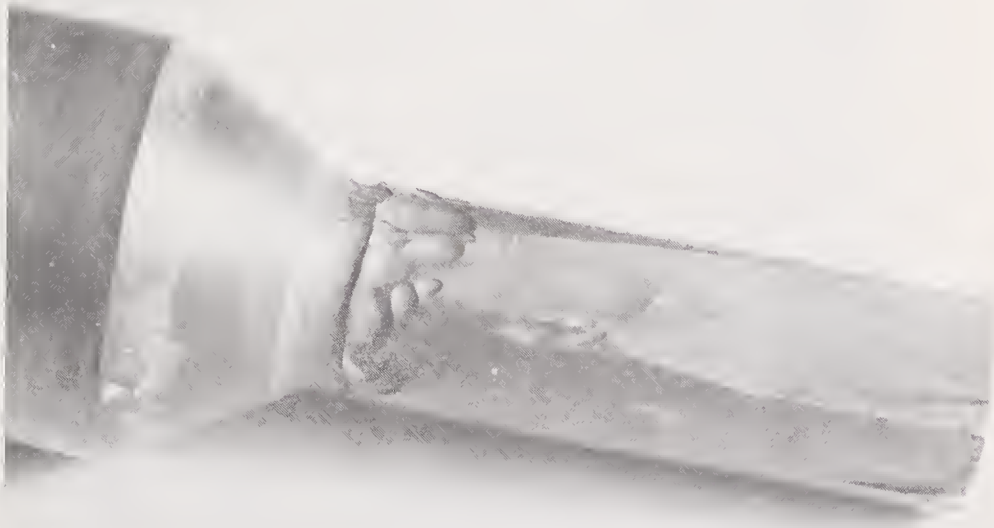

FIGURE 7. UNEVENLY WORN STEM MEMBER

FIGURE 8. LOCALIZED EROSION ON BACK SIDE OF STEM MEMBER

FIGURE 9. SCANNING ELECTRON MICROGRAPH OF WORN SURFACE OF FINE-GRAIN CEMENTED CARBIDE

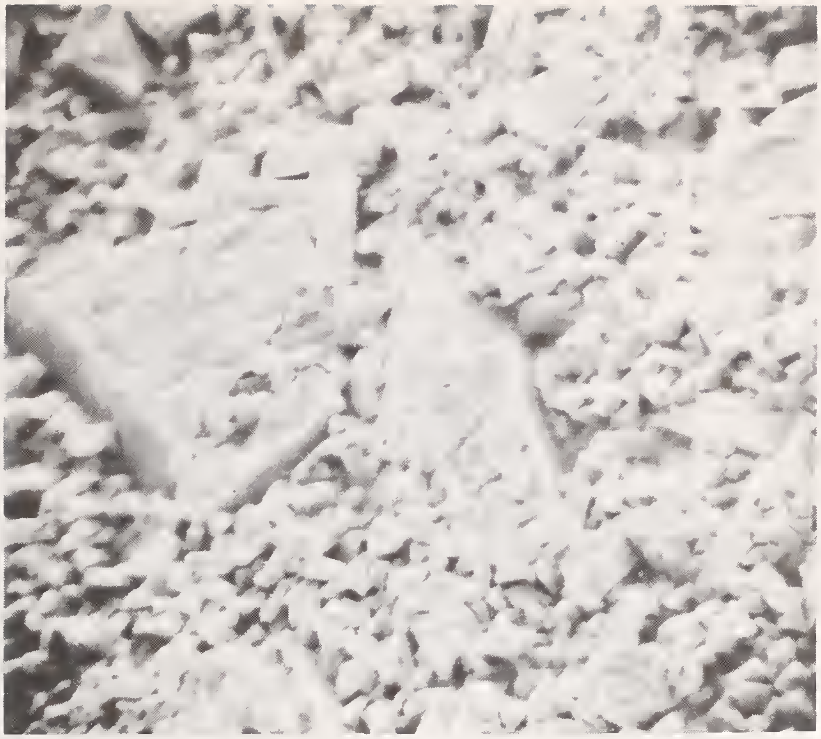

FIGURE 10. SCANNING ELECTRON MICROGRAPH OF WORN SURFACE OF CEMENTED CARBIDE CONTAINING LARGE CARBIDE PARTICLES

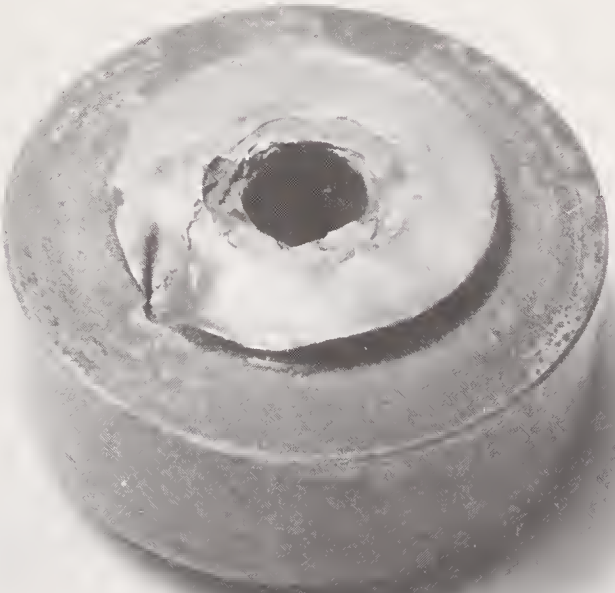

FIGURE 11. ERODED SURFACE OF EXIT SIDE OF SEAT HOLDER

FIGURE 12. SITE OF LEAKAGE BETWEEN SEAT AND HOLDER MEMBERS

FIGURE 13. RESULT OF LOCALIZED LEAKAGE BETWEEN SEAT AND HOLDER MEMBERS

DISCUSSION

W. Netter, Lone Star Lafarge Company: Have you consulted with the cemented carbide producer about particle size packing to minimize the binder?

J. J. Mueller: We developed a wear test to enable us to evaluate some straight ceramic type materials to be used as a short term immediate fix. We believe that these will possibly work. Then in our own program we will consider particle size.

W. Netter: What kind of ceramics have you been looking at?

J. J. Mueller: Zirconium diboride, high density boron carbide, high density alumina, aluminum nitride, silicon nitride, some very high quality bearing materials and others.

E. E. Klaus, The Pennsylvania State University: Would you describe the wear test you developed?

J. J. Mueller: We tried to duplicate the conditions that the actual letdown valve sees. We are working with a typical coal slurry, using anthracene oil as a solvent for the unfiltered SRC product from Wilsonville. This product contained about 32% ash. We are using in this system a cemented carbide orifice to direct a very fine slurry beam at the end of a specimen and then we can evaluate for depth and type of wear. We used a bellows type pump because we thought that the piston pump would suffer erosion damage. This works very well except that in a small system, if all the slurry is pumped out from the reservoir, the bellows can blow itself up. We had good success with it and succeeded in boring the straight machining grade carbide K3H to a depth of 300 microinches in 45 minutes. We feel that we have a sucessful test now for evaluating wear in materials.

R. Perkins, Lockheed, Palo Alto: Chromium carbide is quite widely used as a coating and as a bulk material for sliding wear resistance. I am interested in seeing its application in the slurry. Do you have any feel from the work you have done as to why chromium carbide looks good? What are the essential characteristics of a material that make it an excellent wear resistant material? Is it just hardness, or is it a unique combination of hardness, modulus and toughness?

J. J. Mueller: I cannot answer you from the standpoint of properties. The chromium cobalt matrix cemented carbides are made not by adding cobalt and chromium but by adding cobalt and chromium carbide. The amount of cobalt, I believe, is lower. The trend is toward a minimized, soft matrix.

S. M. Wolf, ERDA: Have you considered using coatings of a single phase ceramic deposited onto the commercially available cemented carbides, and if so, would you tell me what type of coating characteristics -- such as thickness -- you would be looking for?

J. J. Mueller: We considered using single phase ceramics, but we feel there are solid materials that can work. I don't think there is a good reason not to use them except that coatings have been tried, although usually on too soft a substrate. This resulted in catastrophic erosion of the trim when the coating breaks. As an example, stainless steel trim was coated with tungsten carbide. When the coating was penetrated, the stainless steel stem member wore out in about 2 to 5 hours.

S. M. Wolf: Have you any feel for the desired hardness of the surface of the material?

J. J. Mueller: The general feeling is that the surface should be as hard as possible, but then there will be problems with thermal shock resistance and other properties. Perhaps Ian Wright can comment.

I. G. Wright, Battelle Columbus Laboratories: One of the factors that seems to have been causing the high wear of the cemented carbides has been the difference in hardness between the carbide phase and the matrix. The matrix has been eaten out whereas the carbide has not been attacked at all. We simply would require a uniform hardness of the surface, as in a single phase material.

ERDA INFORMATION CENTER FOR FAILURE PREVENTION IN FOSSIL ENERGY PLANTS

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Abstract: The ERDA Fossil Energy Failure Prevention System is a centralized system for collecting, evaluating, and disseminating information about operating experiences and component failure experiences in coal conversion pilot plants. The objectives of this program are to assist plant operating and design personnel in preventing plant shutdowns and in extending the useful life and reliability of plant components.

Centralized coordination of the failure prevention activities will ensure that adequate and accurate diagnostic failure analyses are conducted and will enable the identification of significant and recurring problem areas common to many coal conversion pilot plants. This will provide a means of sharing and exchanging information between pilot plants about component failure problems and successful corrective measures to prevent future problems.

The National Bureau of Standards is responsible for collecting and evaluating all detailed information from the pilot plants and from laboratories conducting diagnostic failure analyses. This information is stored and catalogued according to coal conversion process, material type, failure category, and component type so that the information is readily accessible. The information will be made available to operating and design personnel through direct contact with the Failure Prevention Information Center and through published reports describing specific problem areas and recommended corrective measures in coal conversion pilot plants.

Keywords: Coal conversion pilot plants; computerized data system; failure analysis; failure prevention; fossil energy; information retrieval.

The major function of the ERDA Fossil Energy Failure Prevention Program will be to coordinate all the failure prevention, failure analyses and research programs now underway at Argonne National Laboratory, IITRI, Oak Ridge National Laboratory, various pilot plants, etc. The purpose will be to ensure: 1) that adequate and accurate diagnostic failure analyses are conducted, 2) that significant problem areas are identified, 3) that corrective measure and/or development efforts required are identified. These programs will be accomplished by collecting information on component failures, material evaluations, research projects, etc.; the evaluation of this data for completeness and accuracy; and the dissemination of this information to the users. An overall view of how the program functions is shown by the information flow diagram (Fig. 1). It is expected that the information from this program will be useful in avoiding unexpected plant shutdowns, in extending the life of plant components and in the conservation of materials of construction.

The Failure Prevention Data Center will serve as the centralized base for the collection, evaluation, and dissemination of information on operating experiences and component failures from all pilot plants, process development units and future production plants.

Information Collection. Information will be gathered from many sources (Table I). The ERDA Materials and Component Failure Reports are sent to the Materials and Component Division of ERDA by the plants' operating managers. These reports are the key to an effective failure avoidance program since they can be used to identify recurring and particularly troublesome problem areas. The plant operating manager will make the decision to initiate a failure analysis of the failed component. These analyses can be performed at company laboratories or sent to ERDA designated research laboratories. The plant operator may decide against doing an analysis due to a lack of time, money or interest. To help alleviate some of the expenses involved in doing necessary failure analyses, ERDA has supplied funds to several research laboratories to assist the pilot plants. Argonne National Laboratory, Oak Ridge National Laboratory, Sandia Laboratory are the designated organizations which will perform failure analyses. Detailed failure analyses reports are the essential keystone of the failure prevention system. They are essential in building up the data base needed to improve component reliability and performance.

Information Evaluation. All of the failure analysis reports from the various laboratories are evaluated at the National Bureau of Standards for completeness, accuracy of diagnosis and correctness of the data. Analyses are performed by specialists in areas of metals, ceramics, erosion, corrosion, etc. Once all of this material has been collected a failure mode analysis can be conducted to identify significant problem areas. Table 2 presents some of the problem areas which have been uncovered to date. As expected the major problem areas are with corrosion and erosion. The hostile environments associated with the coal conversion processes and the necessity of moving coal slurries have led to many operation problems.

Information Dissemination. All of the information collected and analyzed must be made available to the users rapidly and in a suitable format. A brief description of the operation of the ERDA Failure Prevention Data Center will show how this function is performed. All the information items mentioned in Table I that are received at the data center are assigned an identification number. This number is recorded in a logbook along with a short description of the item. The item is analyzed for completeness and accuracy and a detailed technical abstract is prepared. The item is classified, for cross-indexing purposes, into several categories, such as coal conversion process, type of material, component, failure mode, author, (as shown in Table 3). The original report is then filed by information number into a master file. Initially the procedure was to type the information on 5 x 8 cards and cross-filed by the information in a manual retrieval system. This procedure is inadequate to handle a large volume of information. Therefore, the information is being transferred onto a computerized (CCA Model 204) data base management system which will enable information to be more readily available for failure mode analysis and will ease the dissemination of information to the users. The most important function of this data center is to get pertinent information into the hands of people who can use it, in a useful form and in time to be of value. Some of the methods to be used for information dissemination are shown in Table 4. As recurring problem areas are uncovered reports will be written and sent to interested users to alert them of the problem. Through technical meetings, publications in the ERDA Newsletter and in technical journals, information from the data centers will be disseminated to a wide range of interested users.

The ERDA Failure Prevention Data Center has only just gotten underway. Additional information is being received continuously on component failures and material problems so that the data base is being enlarged. The sharing of operating experience between all the pilot plants and process development will significantly reduce the time required for these plants to reach full operation and to establish which materials and components will perform satisfactorily in the coal conversion environments. This information will permit designers to design more efficient, dependable plants and plant operating personnel to improve the operation of the plants and minimize downtime. This program will enable the ERDA Fossil Energy program to reduce the time required to get production plants on-stream and start producing significant amounts of energy in gaseous and liquid form from coal.

Failure Avoidance:
Information Flow Diagram

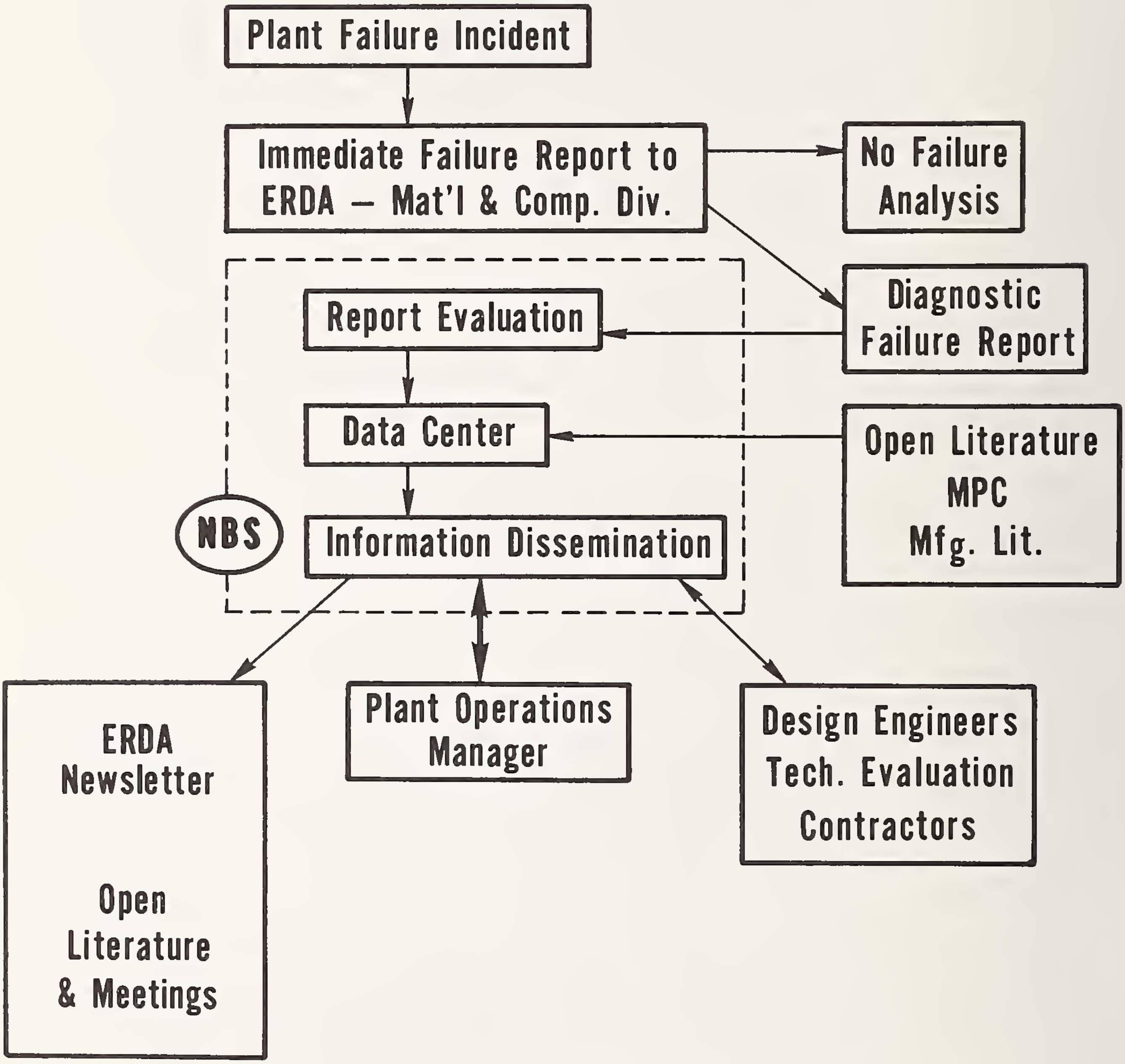

Figure 1

Table 1

—INFORMATION GATHERING—

—SOURCES—

—ERDA MATERIALS AND COMPONENT FAILURE REPORTS

—DIAGNOSTIC FAILURE ANALYSIS REPORTS BY DESIGNATED LABORATORIES

—SERVICE EXPERIENCE OF OPERATING PLANT MANAGERS

—PUBLISHED LITERATURE AND MANUFACTURERS DATA

—SPONSORED RESEARCH PROJECTS

—TECHNICAL SOCIETIES

- METALS PROPERTY COUNCIL
- NACE
- AIME
- ASM

Table 2

PROBLEM AREAS

- —HIGH TEMPERATURE SULFIDATION
- —CHLORIDE STRESS-CORROSION CRACKING IN STAINLESS STEELS
- —EROSION
- —THERMAL FATIGUE/THERMAL STRESSES
- —CARBONIZATION
- —FABRICATION DEFECTS
- —CORROSION—CAUSTIC,METAL DUSTING
- —DESIGN
- —IMPROPER MATERIAL SPECIFICATION/QUALITY CONTROL

Table 3

—INFORMATION WILL BE CROSS INDEXED TO INCLUDE:

-COAL CONVERSION PROCESS
-TYPE OF COMPONENT
-TYPE OF MATERIAL
-FAILURE CATEGORY
-INFORMATION SOURCE

Table 4

—INFORMATION DISSEMINATION—

—REPORTS DESCRIBING SIGNIFICANT PROBLEM AREAS AND CORRECTIVE MEASURES

—PERSON-TO-PERSON COMMUNICATION WITH WORKING PERSONNEL

—PUBLICATION IN THE ERDA MATERIALS AND COMPONENTS NEWSLETTER

—PUBLIC TECHNICAL MEETINGS

—PUBLICATION IN OPEN TECHNICAL LITERATURE

DISCUSSION

D. Livaccari, Coalcon: How can I get access to your data file? Can I make a phone call and obtain information on a particular problem?

J. H. Smith: That is a good question. We hope to get some thoughts from people like you as to the form in which this data would be most useful. Right now, all we have done is to abstract and cross-index the information so we can find it. What we want to have is a quick retrieval system so that the people who need the information can call us directly and we can give an immediate answer covering everything in our files on that particular subject. Further down the line we will be writing essentially interpretive reports that describe certain problem areas. These reports will probably be published in the ERDA Newsletter. I would like to solicit comments as to what kind of information is needed, in what form it can best be used and on what time scale it would be needed. I think with this information we could then mold our system better to serve you at the early stages of your design and materials evaluation.

D. Livaccari: Well, I think that if we could call in and talk to you people that would be a big help to us.

S. M. Wolf, ERDA: I think this type of information is very useful. Are you also planning to incorporate in your data bank information from accelerated engineering testing programs such as those at IITRI and those that Schaefer described yesterday?

J. H. Smith: Yes.

D. M. Bailey, The Lummus Company: Getting this information down to the operating facilities as soon as possible is going to be of great significance. We all don't want to reinvent the wheel. In the Synthane plant, we have already worn out most of our pumps, nozzles, etc.

L. G. Samuels, SRC Pilot Plant, Ft. Lewis: One of our problems is that we are constantly besieged by people who want the same information. We cannot report our new information because of so many requests for the old information.

J. H. Smith: This is an extremely good point. How do you suggest that we bypass or eliminate some of these requests?

L. G. Samuels: I would suggest that the information be sent directly into ERDA and let them disseminate it.

J. H. Smith: All of our information comes through Frankel's office. We are not trying to set up another system.

L. G. Samuels: In addition, we have to prepare a very detailed monthly report.

J. H. Smith: These reports have been one of our best sources of information.

STEEL PLANT COKE-OVENS:
AN ENERGY CONVERSION PROCESS WITH WARPAGE PROBLEMS

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Abstract: A brief discussion of the warpage problems existing in the armour of steel plant coke-ovens and the proposed research to develop upgraded designs/materials to eliminate these problems.

When Professor Ling became aware of the research Battelle is doing on the metallic components of by-product coke-oven batteries, he expressed an interest in having a paper by Battelle at this Symposium. Our research is still in the early stages, but with the interest of this group in coal-conversion systems, you may be interested in the failure problems we have found and how we plan to overcome them. There may in the future be a relationship between these problems, and their solutions, and the problems being encountered in new coal-conversion processes.

Long before there was an interest in coal conversion or coal gasification processes, there existed batch, low-temperature and high-temperature processes for coal distillation or coal carbonization. In particular, the high-temperature by-product coking process is alive and well today, producing blast-furnace and foundry coke from 85 million tons of coal per year. Each ton of coal used also produces about 10,000 SCF of gas having a heating value between 500 and 600 BTU/SCF, plus tars, oils, and ammonia chemicals.

Coke ovens typically measure about 18 inches wide by 10 to 20 feet tall and 40 to 50 feet long. Usually about 25 to 80 ovens are stacked side by side (like books on a shelf) to share common separating walls that contain flues for heating the coals to coking temperature (about 2000 F). A coke battery of 25 to 80 ovens is essentially an arrangement of high-conductivity silica brick, plus insulating brick, held together with steel tie rods, buckstays, and other structural parts. Battelle's interest is in the metallic armour on these batteries, with particular emphasis on the end-closure equipment. Members of this audience may be interested in the fact that this batch process is 90 to 95 percent thermally efficient, with the probability that this percentage can be improved.

Because coke ovens produce solid product in a batch operation, each end of the 12,000 or more coke ovens is closed by a heavy refractory-lined removeable steel or cast iron door. After the coal in a particular oven has been coked, the doors on each end are removed so that the incandescent coke can be pushed from the oven into a railroad car. For the next charge, the doors are latched back onto each oven. The seal is a flexible stainless steel strip pressing against the cast iron frame on each oven. These seals leak and release hydrocarbon particulates and gases into the atmosphere. Our first research effort was directed towards improving the sealing of these doors against the metallic armour of each end closure.

In June of 1974, Battelle's Columbus Laboratories was awarded a research contract to "Study Concepts for Minimizing Emissions from Coke-Oven Door Seals". This contract was funded jointly by the Control Systems Laboratory of EPA and by the American Iron and Steel Institute. One of the tasks in this completed first-stage study was to "define the problem". To complete this task we made field measurements and did field testing plus laboratory experiments. It was found that the primary cause of the leakage was the degree of warpage that has occurred on most (if not all) of the 25,000 or more cast iron jambs in everyday operation. In addition it was found that the metal contact seals were not flexible enough to adjust to the degree of out-of-plane warpage of the jambs. Given warpage and relatively inflexible seals, the result is gaps or openings in the sealing system, resulting in leakage from the ovens. Table I is an example of the warpage found on operating jambs.

Table I

AN EXAMPLE OF THE DEGREE OF INWARD AND OUTWARD BOWING (OUT OF PLANE) OF JAMBS ON COKE-OVEN END CLOSURES AS REFERENCED TO THE JAMB CORNERS (Numbers are in 32nd's of an inch. A minus 5, for example, is 5/32 inward bowing)

Measurement Location	Left Side				Right Side			
	Oven Number				Oven Number			
	1	2	3	4	1	2	3	4
Top	0	0	0	0	0	0	0	0
Quarter Point	-5	0	-22	-12	-3	-1	-15	-10
Middle	+1	-1	-8	-8	+4	+3	-12	-2
Quarter Point	-1	0	-8	-2	+8	+5	-9	+4
Bottom	0	0	0	0	0	0	0	0

The recommendations made by Battelle as the result of this preliminary study were:

1. More-flexible and more-heat-resistant metal seals should be developed further in a follow-on program.

2. Design work should be preceded by experimental effort to analyze the temperature distribution and thermal-stress patterns in existing systems and designs. These analyses would serve as valuable input to the design and material-selection process.

3. Because the basic emission-causing problem is the distortion that has occurred at operating jambs, the factors causing this problem should be analyzed quantitatively. A technical analysis should indicate what steps can be taken in design and materials to develop a more dimensionally stable jamb for both new coke-oven batteries and replacement of some jambs at existing batteries.

The American Iron and Steel Institute and its member companies, and the EPA accepted these recommendations and asked Battelle-Columbus to submit a research proposal to complete the follow-on project. This proposal has been submitted and includes:

- Interaction between a detailed mathematical modeling, physical modeling, and field experimentation program, leading to
- Full-scale unit design and testing of parts, and
- Fabrication, installation, and field testing of new designs for jambs and seals.

It is possible that the analytical portion of this proposed project may be useful in the materials problems that may surface in the development of other coal-conversion processes.

DISCUSSION

J. Kelly, Rolled Alloys, Inc.: It is my understanding that people make ingot molds out of gray iron rather than nodular iron because the gray iron warps less. Has that been considered or studied?

A. O. Hoffman: It is part of our analysis to find out if this is true. We don't have enough information at present. There are only a few nodular cast iron jambs in our system, and they have only been in use for a short time, so we don't know if they are showing a warpage pattern similar to that of gray cast iron.

J. Kelly: And, more in line with my company's interests, what coefficient of expansion would be required in the stainless steel spring?

A. O. Hoffman: As low as possible, depending on how it is fastened. The most important part to us is its strength at temperature.

J. Kelly: What sort of temperatures?

A. O. Hoffman: We expect that these seals may hit 900°F at times when an oven is left on without coal in it.

J. Kelly: Then you could almost use a martensitic steel.

A. O. Hoffman: Possibly.

SESSION III

RELATED MATERIALS RESEARCH

CHAIRMAN: F. F. LING,
RENSSELAER POLYTECHNIC INSTITUTE

SELECTION OF MATERIALS USED IN COAL GASIFICATION PLANTS

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1. INTRODUCTION

Early review of the projected environments of coal gasification pilot plants clearly showed that the temperatures, pressures, and gas compositions in the different components of the plants were extremely hostile. There was no literature or any real experience with the behavior of materials of construction in such hostile environments. In 1972 the American Gas Association saw the need for some reliable data on the corrosion behavior of alloys (and ceramics) of construction in the projected environments of pilot plants. The AGA initiated this work with the Metal Properties Council whose Task Group on Materials for Coal Gasification developed a well-planned series of experimental programs to gather the needed data. Beginning in 1975 ERDA assumed the funding of the programs through a contract with MPC. The work is subcontracted to IIT Research Institute in Chicago and is being regularly monitored by the appropriate subcommittees of the Metal Properties Council.

The major areas of materials degradation in coal gasification equipment, along with some of the environments, are summarized in Table 1. It is apparent from this chart that unless the "right" materials are selected for construction of the equipment, one may be faced with frequent shutdowns for repairs. During the limited experience with the operating pilot plants most of these problem areas in coal gasification equipment have been observed. The most severe conditions exist, of course, in the main gasifier and the transfer lines which see high temperature, high pressure, corrosive gases, and high-velocity char and ash particles which cause severe erosion. There is very little information in the literature related to high temperature, high pressure erosion. Another area of concern is the severe corrosion of alloys in the aqueous quench tower system. Here again, there are no data in the literature to draw from.

Based on these considerations, the American Gas Association and ERDA initiated a five-phase experimental program. The first phase is to screen alloys in laboratory tests in a high temperature, high pressure gaseous environment typically found in the gasifier. The second phase is a comprehensive one in that all materials of construction, both metals and ceramics, are to be suspended in selected locations of each of the pilot plants for varying amounts of time and the results to be compared with appropriate results from each of the other three phases. The third phase is concerned with the screening of alloys of construction in the environment expected in the aqueous quench tower systems with and without inhibitors in the environment. The fourth phase is a complex version of Phase I in that the best alloys from Phase I will be examined for high temperature, high pressure erosion/corrosion resistance in the environment containing the gasifier gas as well as high concentrations of char, ash, and dolomite particles moving against the samples at high velocities. The fifth and final phase is being organized to collect reliable design engineering data on the best alloys and ceramics found in the first four phases.

2. PHASE I - HIGH TEMPERATURE AND PRESSURE GASEOUS CORROSION

This phase involves laboratory corrosion testing for exposure times up to 5000 hr, primarily in an atmosphere typical of that which would exist in a gasifier but containing three different hydrogen sulfide levels at temperatures of 900°, 1500°, and 1800°F and at a pressure of 1000 psi.

Two reactors were designed and built to operate at a pressure of 1500 psi with a temperature capability of 2000°F. The reactor is surrounded by a pressure vessel with a controlled argon pressure so that the differential pressure across the reactor wall is only a few pounds per square inch. The specimens are spaced on a sample tree, and the tree is suspended from a ceramic tube which also forms part of the gas circulation system. A circulating fan, driven by a magnetic drive unit, causes efficient interaction of gas with the metal surface. The gas flow is arranged so as to obtain a change of gas volume every 8 min.

The specimens are 1 x 1 x 0.25 in. coupons. After exposure these samples were cut and metallographically polished and etched. Measurements were made of the scale and penetration. From the data, calculations were made to express corrosion rates in terms of percent loss of sound metal in inches per year.

Reproducibility tests were carried out in both reactors using all-309 specimens and also a six-alloy series. The results show that, statistically, it makes no difference to the corrosion result which reactor is used or which portion on the tree is selected for test.

To date about 31 tests have been completed. New alloys now being tested include cast and wrought high chromium alloys, cobalt-base alloys, and stabilized high chromium stainless. Future plans include corrosion effects on weldments and on refractories.

3. PHASE II - PILOT PLANT EXPOSURE TESTS OF METALS AND REFRACTORIES

The laboratory tests planned in the laboratory phases will be corroborated with exposures of alloys and ceramics in various test locations in four of the pilot plants constructed for coal gasification. The cumulative exposure durations are planned approximately at one month, three months, and six months. It is expected that comparison of the laboratory test data with results of the pilot plant exposures will give a high level of confidence for the corrosion rates of all the alloys and ceramics.

It should be pointed out that all the alloys selected for exposures in Phase II have a logical counterpart in one of the laboratory phases. It should also be noted that the metallic samples are in the form of corrosion coupons, welded coupons, and stressed coupons. After the samples are exposed in each location for a specified duration of time, they will be examined metallographically and otherwise in the same manner as in Phase I, III, or IV.

The pilot plants involved in this program are Hygas, Consol, Synthane, and Bigas.

The sample racks were specifically designed for each location and welded in place. The main requirements are rigidity and minimum interference with gas flow in the component.

To date, exposures for only a limited number of pilot plant tree locations have been completed. These test locations are at the Consol and Hygas plants. With such limited corrosion results available as yet, an extensive correlation of laboratory and pilot plant data is not possible, but the two sets, in general, compare well thus far. Significant differences have been observed in a few cases in high-temperature locations at Consol. These differences in corrosion behavior can be attributed to a complex corrosion environment in the pilot plant, and resolving them will require additional corrosion data from both laboratory and pilot plant.

4. PHASE III - AQUEOUS CORROSION IN QUENCH TOWER SYSTEMS

Hot gases coming out of the gasifier are quenched in order to remove most of the harmful impurities. Here the temperatures can reach 475°F and pressures up to 1250 psi. The gases contain major impurities such as H_2S, NH_3, SO_2, HCN, oils, tar acids, and chlorides. In this environment, one can anticipate general and pitting corrosion, crevice corrosion, stress corrosion, weld cracking, and hydrogen embrittlement.

To determine the required corrosion data in the proper environment several autoclaves are employed. Plain and U-bend samples (welded and nonwelded) are suspended in the gas phase, placed at the gas-liquid interface, and immersed in the liquid. The data recorded include weight change, maximum pit depth, stress corrosion susceptibility, etc.

The initial group of alloys selected for Task I indicated that all these alloys cannot be tested in a single test chamber at the same time. The 316L lining from the reactor also interfered with some of the test alloys. The lining is now Teflon coated and seems to be inert to the test materials. After many experiments, it was finally possible to group the alloys in such a way as to eliminate the interference.

Statistical analysis of the data indicated that, because of the strong correlation existing between the variables, it would not be possible to uniquely identify the corrosion potential of each variable. To determine the effect of these variables, a statistically designed experiment was recommended by the MPC Phase III Committee.

In Task II of this phase, a statistically designed test series was started. This is a modified subset of the experimental design suggested by IITRI to the MPC Committee. Basically it is a series of 50 hr and 150 hr tests intended to uniquely determine the individual and interaction effects of the various components in environments, some of which correspond to the Task I environments 2J, 2H, etc. Initially, only four steels will be tested, and these carbon steel and stainless steels 304, 316, and 410.

5. PHASE IV - EROSION/CORROSION TESTING

In parts of the gasifier where gas velocities are appreciable, erosion may also become a problem. This effect may be important in cyclones, transfer lines, valves, and other areas. Laboratory tests are planned which include combined erosion/corrosion testing for periods of 50, 100, and 1000 hr in a Phase I gas atmosphere. Temperatures of 1500 and 1800°F will be used at atmospheric and 1500 psi pressures with erodents of char, ash, or dolomite.

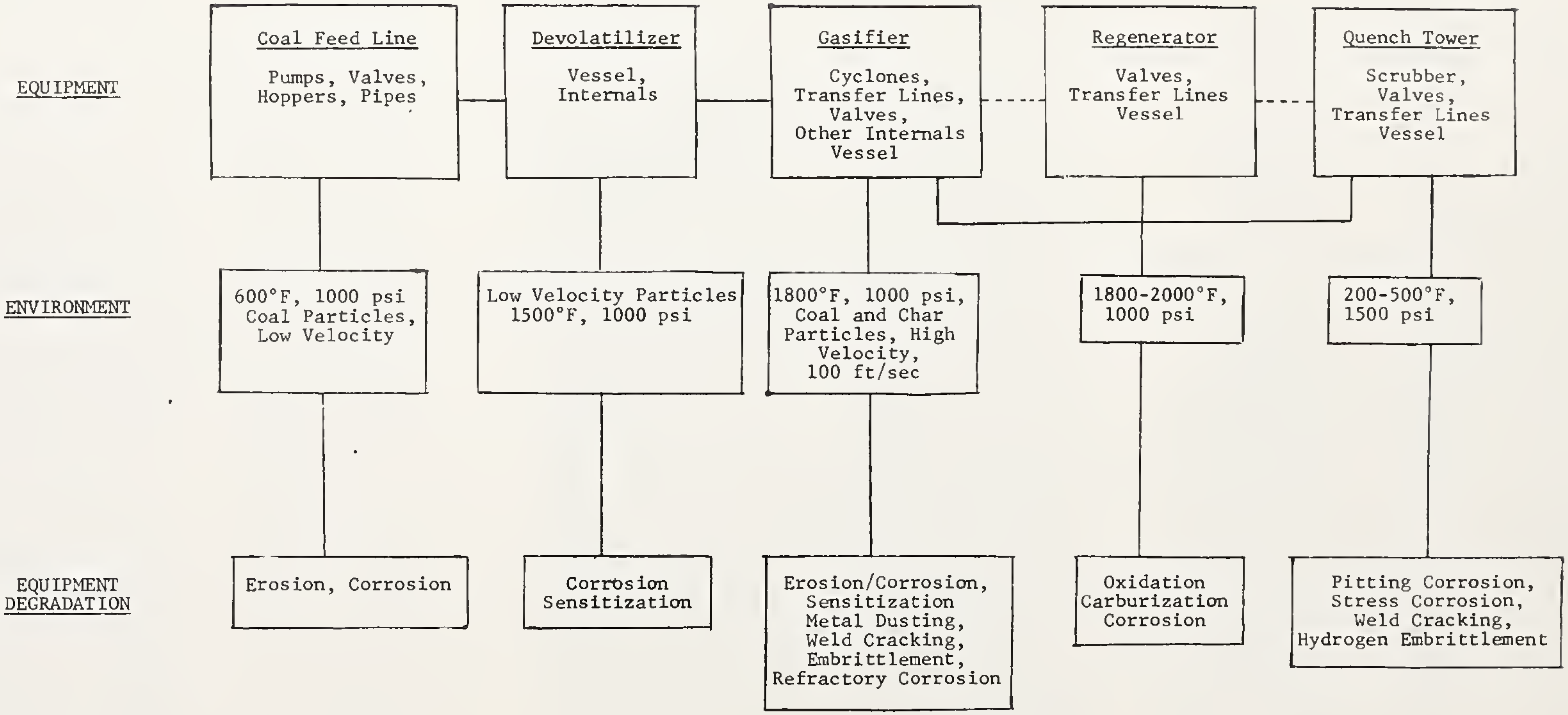
Three reactors are in various stages of construction. The atmospheric pressure equipment is completed, and tests have commenced. The high pressure equipment is expected to be completed in the near future.

6. SUMMARY

A large number of materials of construction are being evaluated for their resistance to different environments expected in various components of coal gasification pilot plants. Several alloys have been found to be promising for application in the high-temperature, high-sulfur environment. The pilot plant exposure tests are now well under way with test coupons installed in four plants and plans made to include three others. Results are now available from the aqueous program, and a statistically designed test sequence is now being commenced. The erosion/corrosion test program is in the early stages, and three reactors are under construction. When the data become available and their reliability and long-term reproducibility characterized, the information will be an invaluable aid in designing and costing commercial coal gasification plants.

Table 1

PROBLEM AREAS IN COAL GASIFICATION EQUIPMENT



EQUIPMENT	Coal Feed Line Pumps, Valves, Hoppers, Pipes	Devolatilizer Vessel, Internals	Gasifier Cyclones, Transfer Lines, Valves, Other Internals Vessel	Regenerator Valves, Transfer Lines Vessel	Quench Tower Scrubber, Valves, Transfer Lines Vessel
ENVIRONMENT	600°F, 1000 psi Coal Particles, Low Velocity	Low Velocity Particles 1500°F, 1000 psi	1800°F, 1000 psi, Coal and Char Particles, High Velocity, 100 ft/sec	1800-2000°F, 1000 psi	200-500°F, 1500 psi
EQUIPMENT DEGRADATION	Erosion, Corrosion	Corrosion Sensitization	Erosion/Corrosion, Sensitization Metal Dusting, Weld Cracking, Embrittlement, Refractory Corrosion	Oxidation Carburization Corrosion	Pitting Corrosion, Stress Corrosion, Weld Cracking, Hydrogen Embrittlement

DISCUSSION

D. W. Short, Lawrence Livermore Laboratory: In the results from your aqueous corrosion tests, you found that there was a greater corrosion rate in the lab tests than in the pilot plant tests. Did you look at the effect of galvanic coupling?

M. A. H. Howes: The difference in the results came about because conditions in the pilot plant were quite different from those in the laboratory. The conditions are enumerated in the table of results. They are quite a lot more severe in the laboratory, mainly because of differences in pH.

G. M. Ugiansky, National Bureau of Standards: Have you seen any cases of stress corrosion cracking in any of your specimens either in the lab or in the pilot plants?

M. A. H. Howes: As you know we do have U-bend specimens in three phases of the program and so far we have not seen any evidence of stress corrosion. The information we are getting from the U-bends, particularly the welded U-bends, at the moment is principally the difference in corrosive attack around the welds and the heat affected zones.

R. De Angelis, University of Kentucky: Have you compared any of your corrosion rate data with data in the literature on the same alloys to see what the effect of the environment is?

M. A. H. Howes: Yes, we have done a fairly thorough literature survey to compare the other data that are available with ours, but the other data seem to be so far removed from the coal gasification environment that we really can't make a meaningful comparison.

S. M. Wolf, ERDA: You mentioned you were planning to do mechanistic evaluations as well. How are these evaluations coming along, particularly in the transient stages of corrosion, since you indicated that erosion-corrosion attack may be more significant than erosion itself?

M. A. H. Howes: These studies really haven't started. We have done a few experiments on the samples that we have, but it was only at a recent meeting that the scheme was laid out. We have a matrix now that covers all four phases. Specimens of about eight materials will be compared from the plants and laboratory studies.

D. Canonico, Oak Ridge National Laboratory: I noticed that some of the U-bend specimens were tested at very high temperatures. Therefore, would you not expect relaxation? And if there is relaxation, what good are the tests?

M. A. H. Howes: Exactly. That's what we said for a long time. The U-bend specimens are of particular value in phase three in the aqueous corrosion study. They should be in any of the lower temperature portions of the plant. I've always questioned putting U-bends in the gasifier section. The answer always comes back that it is the welded specimens that we are interested in because what we want to see is what happens to the weld after it is stressed and then exposed to the environment. I think it is of some value because it does give us an indication of the corrosion on the heat affected zones and on the weld metals themselves. U-bends probably don't have to be used -- the specimens could be straight.

PARTICLE EROSION MEASUREMENTS ON METALS AT ELEVATED TEMPERATURES

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Abstract: Measurements of the erosive wear of metals resulting from impact by abrasive particles under reactive environmental conditions are being conducted. Specimens of several stainless steel and nickel alloys have been exposed to a gas stream containing abrasive particles. The particle flux, angle of attack, particle velocity, particle size, surface temperature and other significant variables were controlled. Different types of abrasives were used including Al_2O_3, SiO_2, SiC and pumice. Two different erosion test apparatus have been developed and used to obtain particle velocities from 10 to 90 m/s and temperatures up to 1000°C. Different gaseous atmospheres have been established during the erosion exposures. Weight loss measurements and penetration depth measurements were conducted on the specimens after exposure. Multiple alloy specimen packages were used in some experiments to obtain relative erosion data. Erosion rates were determined in the temperature range of 25 to 1000°C. Erosion depended on particle velocity over the range V^2 to V^3, increased with particle size, increased with particle hardness, and was maximum for an angle of attack around 20°. The effects of temperature and environment vary in a complicated way among the different alloys.

Key words: Abrasive particles, erosion, erosive wear, metals, oxidation, wear.

Introduction: Studies of metal wear by erosion under various conditions have been conducted [1-6] at room temperature and in a few cases at elevated temperatures [7,8]. Industrial applications, for example in the petrochemical industry and in new industries such as coal gasification [9], require data on metal erosion under conditions of high temperature and reactive environments. Much of that information still needs to be acquired. Erosion of metals at room temperature has been found to depend on particle velocity in the range v^2 to v^3 [1,2]. The angular dependence of erosion exhibits a peak at about 20° angle of attack [1,4] and the effects of different particle sizes have been investigated [2,5]. Erosion can either increase or decrease with increasing temperature [7,8] depending on the metal and the atmosphere. The mechanisms of erosion have been studied using single particle impact techniques [10]. The present paper describes recent erosion measurements conducted on metals at room and elevated temperatures up to 1000°C in different gaseous atmospheres.

Experimental: Two different erosion testing apparatus have been used in the present work. One system was based on an instrument developed previously at this laboratory [11]. That instrument had been used principally to determine the durability of coatings.* Considerable attention was paid during its development toward achieving reproducibility of operation. Modifications introduced in the current program have been designed to extend the operating capabilities to elevated temperatures while maintaining that reproducibility. A schematic diagram of the system is shown in Fig. 1. In order to obtain data at elevated temperatures, the metal specimens were heated directly by passage of a dc current through them during erosion testing. Current values up to 200 A at 10 V were used to reach an operating temperature of 500°C and above. The metal specimen size was typically 3 cm long x 1 cm wide x 0.5 mm thick. Battery clips (gold plated) were used to achieve electrical contact at each end of the specimen during heating. A thermocouple pressed in contact with the underside of the specimen was used to determine the test temperature. The propellent gas-particle mixture was also heated. After injection of abrasive particles, the mixture passed through an eight foot length of 1.6 mm inside diameter stainless steel tubing that was heated by the passage of up to 60 A ac current along its length. The gas-abrasive particle mixture was discharged through a right angle tungsten carbide nozzle, 0.5 mm inside diameter. The exit gas temperature was adjusted so that the specimen temperature did not change during the test although it may have been as low as 300°C at high flow rates when the specimen was maintained at 500°C. The propellent gas used in the tests reported here was 99.9% CO_2. The exposure time during each test was 2 minutes. Table 1 lists some of the characteristics of this test apparatus.

Table 1

Range of Operating Parameters for Roberts Erosion Unit

Gas: CO_2,O_2,CO, mixtures

Particle sizes: 5 to 50 μm (0.5 mm nozzle)
5 to 150 μm (1.25 mm nozzle)

Gas Pressure: 20-70 psig (0.14 - 0.49 MPa)

Particle velocity: 10-60-m/s

Solids concentration: 0.5 to 3 g/l

Particle flux: 0.5 to 3 g/cm^2-s

Specimen temperature: ambient to 500°C+

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[*] Roberts Jet Abrader, Kameras Instruments. This instrument is identified only for the purpose of adequately specifying the experimental procedure used.

The second erosion system has been described previously [12] and permitted test temperatures of 1000°C to be achieved. Briefly, a propane-oxygen-air burner was used to direct a stream of hot combustion gases containing entrained abrasive particles down a 41cm tube to impact on the specimen. The stream was approximately 2 cm in diameter. By changing the oxygen and propane flows, the composition of the gases impinging on the specimen could be varied. When oxygen was supplied in excess of the amount needed for complete combustion, an atmosphere composed of N_2, O_2, CO_2 and H_2O was produced. When excess propane was introduced, CO, H_2 and hydrocarbons were obtained in addition to the above gases. Test times were usually 60 minutes. Table 2 describes the operating features of this unit.

Table 2

Range of Operating Parameters for High Temperature Erosion Unit

Gas: Combustion gases; air

Particles: Al_2O_3, SiC

Particle sizes: 50-200 μm

Particle velocity: 10-90 m/s

Solids concentration: 0.005 to 0.02 g/1

Particle flux: 0.04 g/cm^2-s

Specimen temperature: ambient to 1000°C

Table 3

Parameters of Abrasive Particles Used in Erosion Tests

Type	Size (μm)	Hardness (HK@25°C)
SiC	150	2500
Al_2O_3	5	2100
Al_2O_3	50	2100
SiO_2	5-50	820
F-Pumice	5-100	560
4F-Pumice	5-50	560

The abrasives used in the tests reported here are listed in Table 3. Room temperature hardness values are listed. The Al_2O_3 abrasives are metallographic polishing grade material having a narrow size distribution. A collection of the 5μm Al_2O_3 particles is shown in Fig. 2. The particles passed only once through the system and were then discarded. The particle velocity was measured using the rotating disk, time-of-flight method [13]. Velocity measurements were conducted at the test temperatures.

Weight loss values were determined by direct weighing after exposure and cleaning. Microscopic inspection of the surface indicated whether particle adhesion to the specimen was significant. If so, attempts were made to mechanically remove the adherent particles by light brushing. Since scale formation occurred at elevated temperatures, it was difficult to remove all adherent abrasive and yet not affect surface scale. Dimensional measurements of erosion crater depth and of specimen thickness loss were made using microscopic or micrometer methods with a precision of the order of ±1 to 10 μm depending on surface roughness. Examination of the eroded surfaces was conducted using optical and scanning electron microscope (SEM) methods.

Results

Studies at 25°C and 500°C: Examples of isolated particle impacts are shown in Fig. 3 after a low flux exposure of an SAE 52100 steel specimen. The rim of each erosion crater consists of exposed material that has been deformed plastically and which is susceptible to removal on subsequent particle impacts. That material is also more susceptible to attack in a chemically reactive environment. Measurements have been conducted at room temperature using the modified Roberts unit with different abrasive particle velocities. Relative weight loss and penetration measurements on 304 stainless steel are shown in Fig. 4 for 90° (normal) incidence, using two sizes of Al_2O_3 particles. The erosion loss depends on the velocity raised to about the 2.8 power in these tests. In general, we find that erosion is proportional to velocity in the range of $v^{2.5}$ to v^3 at room temperature. A greater erosion loss is seen here for the larger particles, 50μm Al_2O_3. The comparison between weight loss and penetration distance is usually complicated by such factors as erosion beam geometry changes when the abrasive type and size are changed. It is preferable to use the same erosion measure when comparing different metals.

The relative weight loss as a function of angle of attack α, the angle between the specimen surface and the incident particle stream, has been determined for 304 stainless steel at both 25°C and 500°C. The particle flow and velocity were maintained constant as the specimens were exposed at different angles to the particle stream. At 25°C a peak erosion loss occurs at about 20° followed by a gradual decrease as the angle is increased up to 90°. The angle dependence of erosion at 500°C is different. The low angle peak occurs at about 10°, followed by a rapid decrease and then little change occurs in the range 40-70°. The loss appears to increase again as 90° impingement is approached. At 500°C oxidation is more rapid than at room temperature, hence, the incident abrasive particles experience a different surface condition. The increased relative loss at 90° may be connected with the known erosion characteristics of brittle materials where the peak loss occurs at normal incidence. The oxide film or scale probably behaves in a more brittle manner than the ductile underlying metal.

A group of eight different alloys have been examined at 25°C and 500°C using this apparatus. Results on the erosion loss of the alloys using two different sizes of Al_2O_3 particles are shown in Table 4. The particle velocity was 30 m/s at 45° angle of attack using CO_2 propellent.

Table 4

Erosion Measurements on Alloys Using Two Sizes of Al_2O_3 Particles at 30 m/s and 45° Incidence

Alloy Type	Wt. Loss(mg)/Wt.Abr.(g)				Penetration(μm)/Wt.Abr.(g)			
	25°C		500°C		25°C		500°C	
	5 μm	50 μm	5 μm	50 μm	5 μm	50 μm	5 μm	50 μm
18Ni-250MS	0.08	0.12	0.04	0.95	10	9	0.5	16
AISI 446	0.05	0.05	0.01	0.20	10	6	0.6	11
AISI 304	0.05	0.47	0.02	0.26	7	26	0.2	12
AISI 316	0.04	0.10	0.01	0.21	7	9	0.8	13
AISI 310	0.03	0.09	0.02	0.20	5	9	0.8	13
Incoloy 800	0.06	0.32	0.01	0.26	8	20	0.4	18
Inconel 601	0.06	0.30	<0.01	0.19	8	20	0.4	14
Inconel 671	0.02	0.11	0.01	0.15	4	9	0.4	13

Both relative weight loss and penetration are shown. It is clear that erosion increases significantly with particle size at both temperatures and for all alloys. This size effect is particularly large for the 500°C tests where a decreased erosion loss is seen for all alloys for the 5 μm particles (relative to 25°C). Apparently the developing oxide scale on the surface offers significant protection but only for the small particle size. The data at 500°C using 50 μm size particles varies among the alloys in comparison with the 25°C results. At 500°C the alloys having larger chromium content in general show the least erosion loss (although exceptions are present). Oxide thicknesses measured on specimen cross sections after testing at 500°C for 2 minutes ranged from about 2μm down to less than 0.5 μm, depending on the alloy. More data needs to be acquired at other exposure conditions of velocity, particle size, and environment before definitive conclusions can be reached concerning relative material erosion rates.

A comparison between the results obtained with different abrasives is shown in Table 5. The data refer to 304 stainless steel in air at

Table 5

Erosion Measurements on 304 Stainless Steel Using Different Abrasives at 30 m/s and 45° Incidence

	Wt. Loss (mg)/ Wt. Abr. (g)		Penetration (μm)/ Wt. Abr. (g)	
Abrasive (Size)	25°C	500°C	25°C	500°C
Al_2O_3 (5 μm)	0.5	0.2	7	0.2
Al_2O_3 (50 μm)	0.47	0.26	26	12
Pumice 4F (5-50 μm)	0.21	0.04	29	2.7
Pumice F (5-100 μm)	0.10	0.03	8	1.7
Silica (5-50 μm)	0.50	0.21	45	12

30 m/s particle velocity at two temperatures, 25°C and 500°C. The increased erosion loss for 50 μm Al_2O_3 relative to 5 μm Al_2O_3 particles is apparent. The two grades of pumice show somewhat reduced losses relative to 50 μm Al_2O_3 that are probably associated with the reduced hardness value. However, a detailed study of these and other data shows that factors other than abrasive hardness are significant. These include "cutting efficiency" at elevated temperatures, tendency to fracture, hardness at temperature, etc. Such details can only by understood as a result of comprehensive studies of the various abrasives. Another relevant abrasive characteristic is the tendency for certain abrasives to sinter to the eroded surface and hence offer protection.

Studies of the effect of surface hardness of the metal on erosion resistance are underway. The results of one series of tests are shown in Fig. 5 using SiO_2 abrasive and type 18Ni-250MS steel hardened to different levels. Relatively little effect on erosion rate is seen as the metal hardness varies; that observation has been reported previously by others [14].

Studies at 1000°C: Experiments conducted in the second erosion system have used either 150 μm size SiC abrasive or 50 μm Al_2O_3 particles. Two different types of experiments were conducted. In one, multiple alloy specimen packages were used in order to obtain data on relative erosion for the alloys. In the other, a single alloy specimen was used for each test. Figure 6 shows an example of a multiple alloy specimen package after exposure in the test unit at 1000°C for 60 minutes using

150 μm SiC particles at a velocity of 50 m/s. Excess oxygen conditions were maintained during the test. Each specimen in the package was 0.5 mm thick and was fabricated to have two surfaces exposed to the particle stream at angles of 90° and 45°. Fiducial marks were placed on each specimen surface in order to measure the dimensional loss after testing One other edge, not exposed to the abrasive particles but exposed to the heated atmosphere, provided information on the oxidation process. While this specimen design provides an opportunity for obtaining relative data between the alloy samples in the same atmosphere, there can be interferences between neighboring specimens in the package, particularly if the erosion rates differ greatly. In Fig. 6 the specimens in Inconel 601, 671 and 310 stainless steel exhibit the least erosion. On cooling to room temperature the oxide scale spalled from the 304 stainless steel specimen. Little or no spalling occurred on the other alloys.

Erosion data obtained from two tests using the multiple specimen package under different environmental conditions are shown in Table 6.

Table 6

Erosion Measurements on Alloys at 1020°C in Combustion Gases at Approximately 55 m/s Using 150 μm SiC Particles

	Equivalent Wt. Loss (mg)/Wt. Abrasive (g)			
	Excess Oxygen[1]		Excess Propane[2]	
Alloy Type	90°	45°	90°	45°
AISI 446	0.41	0.35	0.73	1.1
AISI 304	0.59	0.37	1.6	1.5
AISI 316	----	----	1.2	1.3
AISI 310	0.32	0.28	0.58	0.64
Incoloy 800	0.42	0.32	0.56	0.64
Inconel 601	0.29	0.24	0.23	0.49
Inconel 671	0.26	0.28	0.15	0.25

1. Typical Analysis: O_2=13%, CO_2=10%, N_2=64%, H_2O=13%
2. Typical Analysis: H_2=8%, CO=12%, H_2O=17%, CO_2=5% N_2=52%, Other=6%.

Examination of the results in the excess oxygen atmosphere indicate a small difference between the 45° and 90° surface erosion losses. However, under excess propane combustion conditions a greater difference develops between the erosion rates at these two angles. The increased loss at 45° associated with the ductile behavior is clear under excess propane conditions. The difference in erosion rate among the different metals is also more apparent under those conditions. The most resistant alloys appear to be those containing larger chromium concentrations. That same characteristic appears to apply in more strongly oxidizing atmospheres. An example of the surface conditions that develop at 1000°C after erosion is shown in Fig. 7a for 310 stainless steel exposed under excess oxygen conditions. Much roughening due to impact damage is evident on the surface and some abrasive particles (or fragments) are seen imbedded in the specimen. A cross section through this specimen is shown in Fig. 7b and indicates the oxide film present.

Summary

1. Two sets of test apparatus have been developed and applied to erosion measurements on metals in the temperature range of 25°C to 1000°C. The atmosphere, particle size and velocity, specimen orientation and other significant variables can be controlled.
2. Significant effects on the erosion rates of the metals studied are attributed to the oxide film or scale that forms on the surface. The chromium content of the alloy is one important parameter.
3. Abrasive hardness is a significant factor in erosion rate although other abrasive characteristics may be equally important. Hardness for a given alloy may not be an important parameter.
4. The maximum rate of erosion occurs at angles of 10° to 20° at temperatures up to 500°C, suggesting that cutting processes are a prominent metal removal mechanism.

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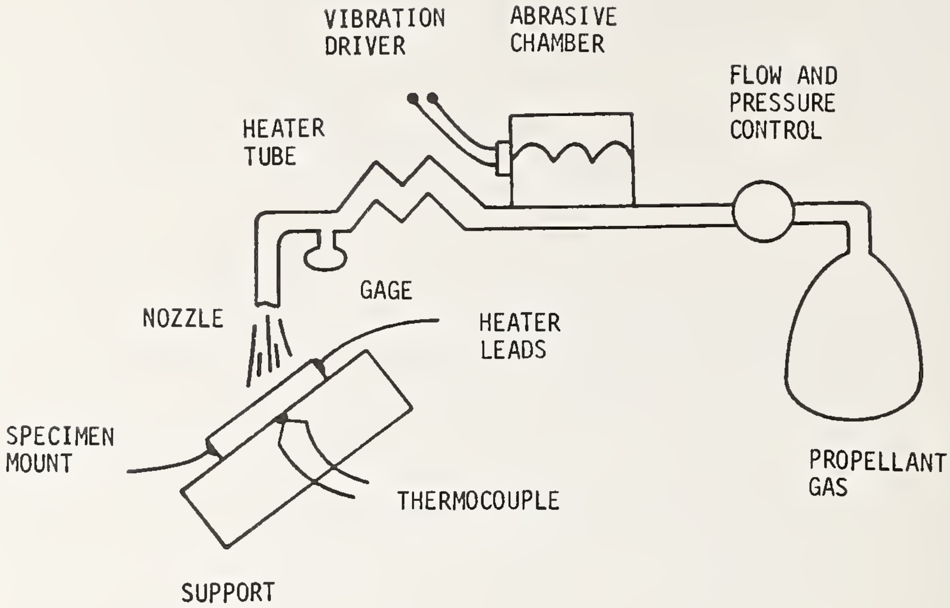

Fig. 1 Schematic representation of the modified Roberts erosion apparatus.

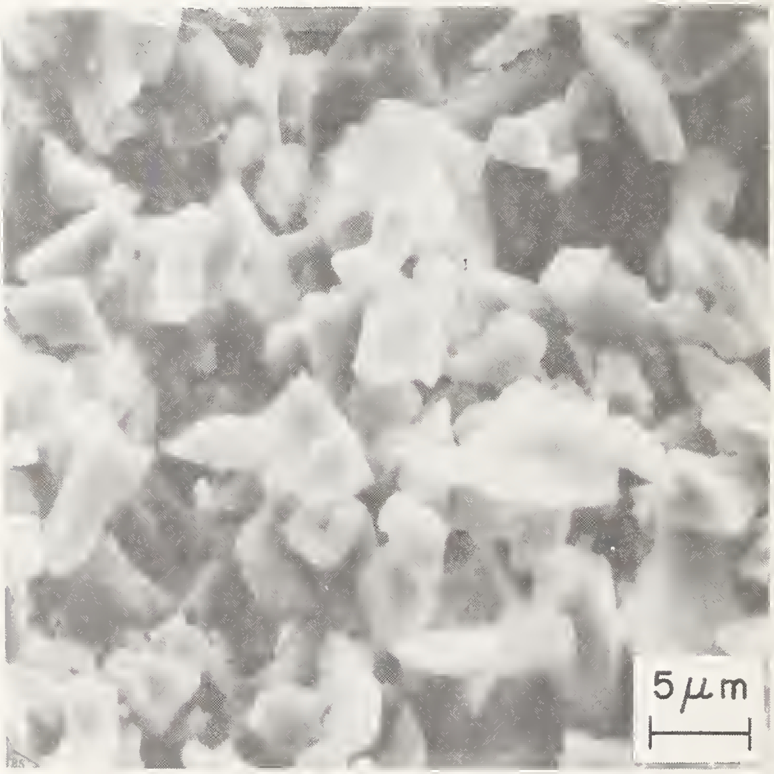

Fig. 2 Examples of 5 µm sized Al_2O_3 abrasive particles.

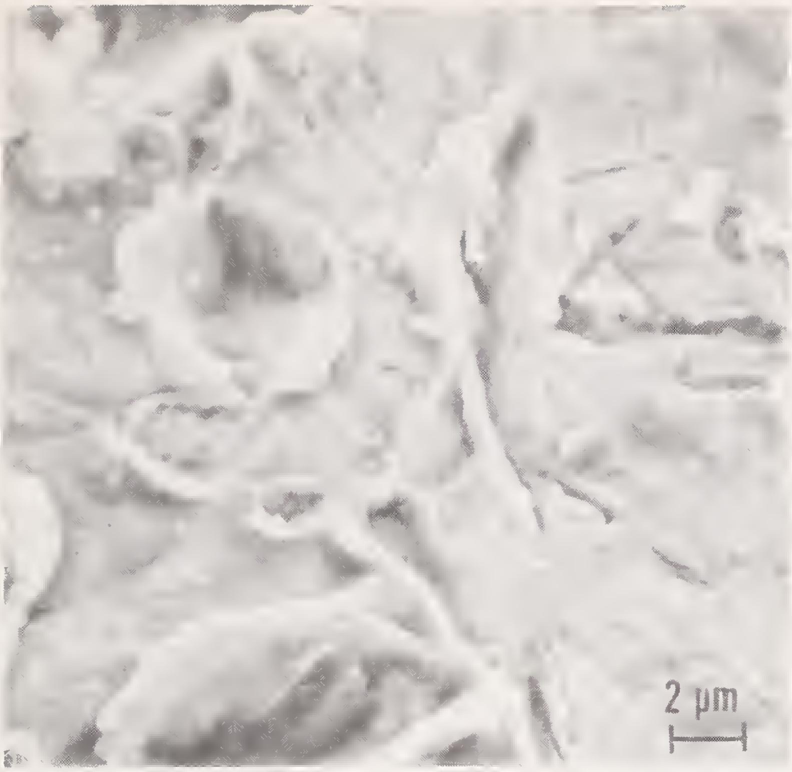

Fig. 3 Erosion impact craters on steel surface.

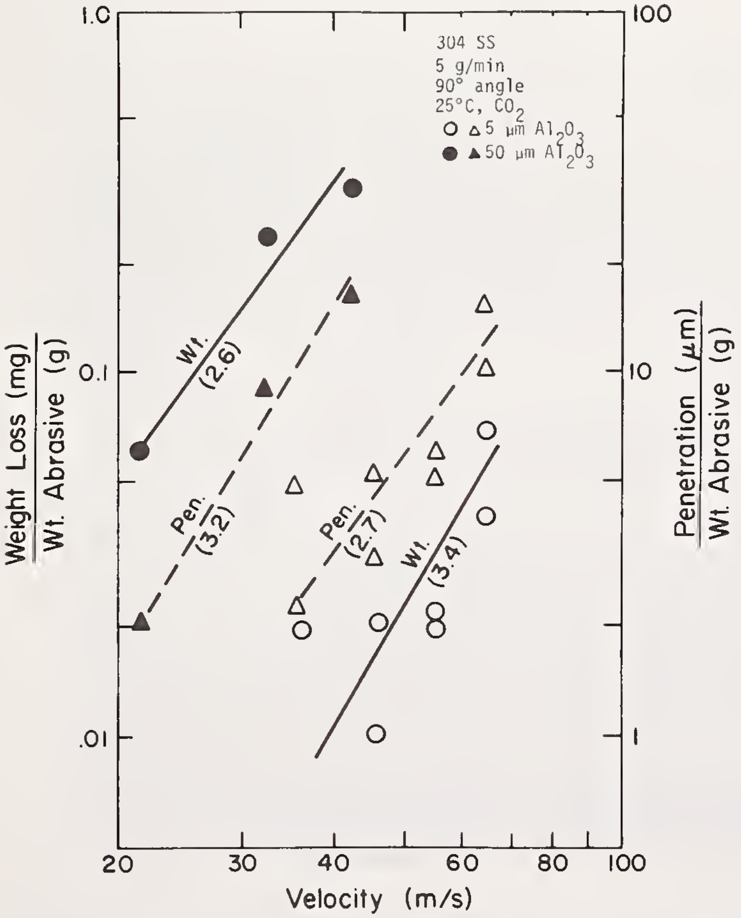

Fig. 4 Velocity dependence of erosion loss at 25°C for two particle sizes.

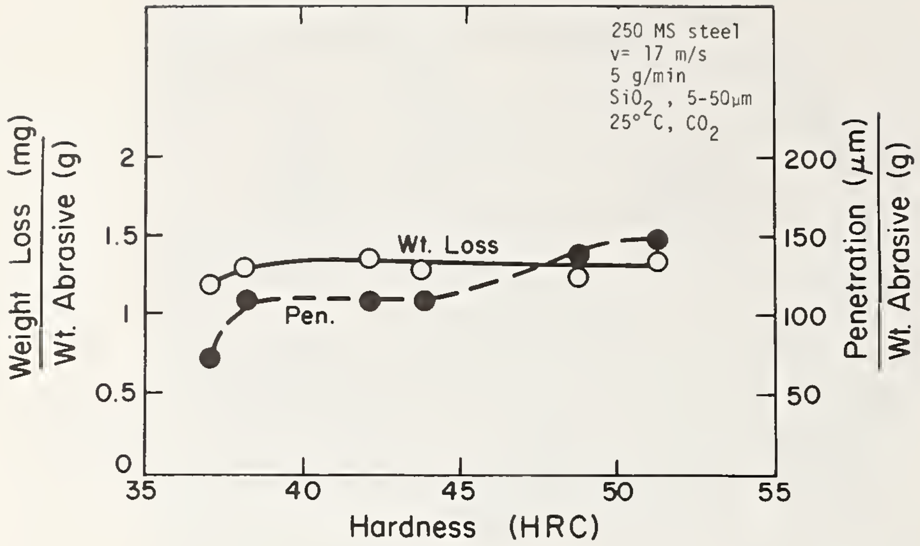

Fig. 5 Variation of erosion loss for different hardness levels in type 18Ni-250MS steel.

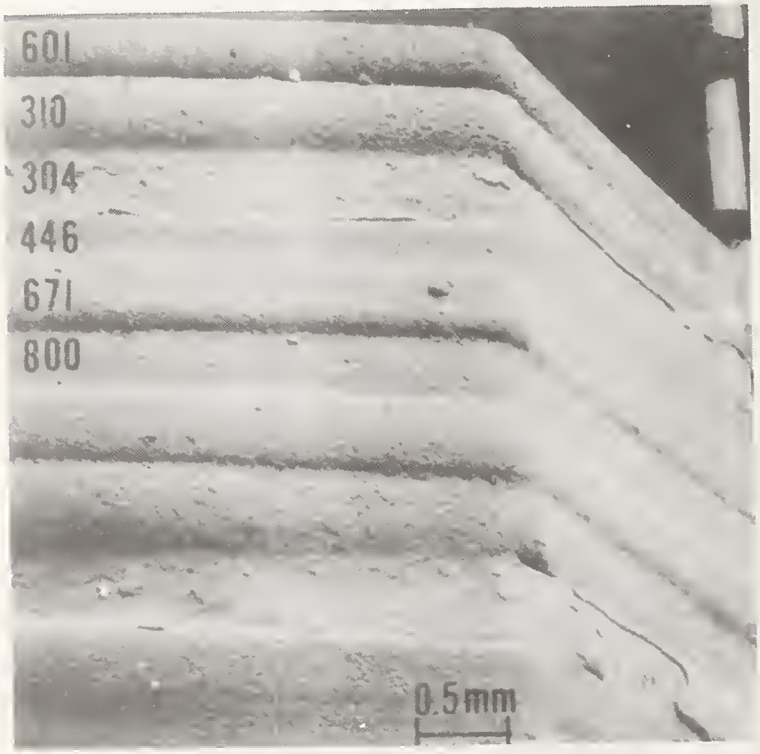

Fig. 6 Multiple alloy specimen package after erosion test at 1000°C. Two exposed surfaces 45° and 90°, are shown.

Fig. 7

(a) Surface of 310 stainless steel after erosion test at 1010°C.

(b) Cross section of specimen showing oxide film and erosion craters.

DISCUSSION

I. G. Wright, Battelle Columbus Laboratories: Can you tell me the size range of the particles used in the jet abraser test?

L. K. Ives: These were commercial abrasive materials used for polishing. I believe the size range was fairly narrow, but I don't have data on the exact size range.

I. G. Wright: What was the most convenient method you found for measuring the depth of penetration?

L. K. Ives: We simply focused a measuring microscope on the bottom of the depression and then on the upper surface and determined the difference.

HIGH-TEMPERATURE CORROSION

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Abstract: Materials considered for application in the high-temperature gaseous environments that are present in various coal-gasification processes must possess good structural integrity at elevated temperatures and inherent resistance to the corrosive atmospheres. A thermodynamic approach has been developed to characterize the gas environment in different pilot plant coal-gasification processes. The analysis is used to examine the thermodynamic stability of iron- and nickel-base alloys in these process environments. The results show that the sulfur and oxygen potentials in all processes are in a region where chromium oxide will be thermodynamically stable. In the absence of a continuous oxide layer, sulfidation of the alloys is expected which can lead to severe corrosion rates, particularly when liquid nickel sulfides are formed. It is concluded that a protective Cr_2O_3 layer is essential for an effective application of these alloys in coal-conversion processes.

INTRODUCTION

Much of our knowledge concerning the high temperature corrosion behavior of commercial iron- and nickel-base alloys is derived from studies of high-purity metals and simple alloys in well-characterized binary and ternary gas mixtures. Even though the results from the simple experiments aid in the establishment of the relative corrosion behavior of different metals, the usefulness of such information in the prediction of the corrosion rates of multicomponent alloys in complex gas environments is very limited. Studies on fundamental processes such as oxidation, carburization, sulfidation, and nitridation are generally conducted over a relatively short time period with the objectives to identify the most stable phases formed and to determine the mechanism and rate-limiting step for the process. Thermodynamic considerations have been used to rationalize the experimental observations in terms of the reaction potential of the gas species at the temperature and pressure of interest. Gulbransen and Jansson(1) have reviewed the thermodynamic analysis of product phase stability for metals in 2 component reactive gases, e.g., $SO_2 + SO_3$, $H_2 + H_2S$, $CO + CO_2$, and $H_2 + H_2O$, by means of what is commonly known as Ellingham diagrams. These diagrams depict the stability of the condensed phases for a given metal as a function of thermodynamic activities of the two reactive gaseous components. A similar approach for the evaluation of the corrosion behavior of metals in multicomponent gas mixtures is usually limited to the determination of stable compound(s) that are formed as a result of exposure of the metal to the gas mixture.

The combustion gas composition in coal-conversion processes generally depends on the type of process and feed stock analysis in addition to operating temperature and pressure. The gas mixtures usually consist of CO, CO_2, H_2, H_2O, CH_4, H_2S, and NH_3; as a result, the simultaneous presence of reactive elements such as O, C, H, N, and S needs to be considered in the evaluation of the material behavior in the gas environment. The room-temperature compositions of the gas mixtures that are anticipated in different pilot plant processes are listed in Table I.(2) The compositions given are based on the use of low-sulfur coal in the processes. Also given in the table is the composition selected by the Metals Properties Council (MPC) for use in the experimental research programs for the corrosion evaluation.(3) It is evident that the processes can have a wide variation in the gas composition and therefore, materials performance in different processes is not expected to be the same even for identical conditions of temperature and pressure. It is also evident that in order to cover the entire spectrum of gas composition variation, the experimental program will be time consuming and expensive, especially if long-term corrosion rates under high pressure conditions are required.

GAS COMPOSITION CHARACTERIZATION

When studying reaction kinetics or reaction equilibria between complex gases and condensed phases, the chemical potential of the reacting elements C, O, H, and S needs to be established and their competing interactions with the alloys must be considered. For this purpose, a computer program was developed by which the equilibrium gas partial pressures could be evaluated at elevated temperatures for a given room-temperature composition. For a gas system that consists of CO, CO_2, H_2, H_2O, CH_4, H_2S, and NH_3, the following gas equilibria may be written:

$$C + \frac{1}{2}O_2 = CO \qquad K_1 = P_{CO} \Big/ P_{O_2}^{1/2}\, a_c \tag{1}$$

$$C + O_2 = CO_2 \tag{2}$$

$$C + 2H_2 = CH_4 \tag{3}$$

$$C + \frac{1}{2}S_2 = CS \tag{4}$$

$$C + S_2 = CS_2 \tag{5}$$

Table I. Estimated Gas Compositions in Various Coal-gasification Processes with Low-sulfur Coal Feedstock.

	Hygas	CO_2 Acceptor	Synthane	Battelle-Union Carbide	Bigas	MPC Selected
H_2	17	52	19	48.8	15	24
CO	21	11	9	26	12	18
CO_2	21	6	21	5	13	12
CH_4	15	3	12	6	7	5
H_2O	25	28	36	14	52	39
H_2S	0.1	0.03	1.1	0.3	0.5	0-1.0
NH_3	1	1	1	1	1	1
P(psi)	1200	300	1000	100	1000	-
T(°F)	1750	1600	1800	1800	1700	-

$$H_2 + \frac{1}{2}O_2 = H_2O \tag{6}$$

$$H_2 + \frac{1}{2}S_2 = H_2S \tag{7}$$

$$\frac{1}{2}S_2 = S \tag{8}$$

$$\frac{1}{2}S_2 + \frac{1}{2}O_2 = SO \tag{9}$$

$$\frac{1}{2}S_2 + O_2 = SO_2 \tag{10}$$

$$\frac{1}{2}S_2 + \frac{3}{2}O_2 = SO_3 \tag{11}$$

$$CO + \frac{1}{2}S_2 = COS \tag{12}$$

$$\frac{1}{2}N_2 + \frac{3}{2}H_2 = NH_3 \tag{13}$$

where $K_1, \ldots$ are equilibrium constants, a_c is carbon activity, and p_{co}, $p_{co_2} \ldots$ are the partial pressures of CO, CO_2,...etc. The equilibrium constants for the reactions (1) through (13) are given by

$$K_j = \exp\left(-\Delta F^\circ_j/RT\right), \text{ for } j = 1 \text{ through } 13 \tag{14}$$

where

R = gas constant, 1.987 cal/mole °K

T = absolute temperature,

and

ΔF°_j = free-energy change for the jth reaction, cal/mole.

In a given complex gas mixture, the total number of gram-atoms of carbon (ΣC), sulfur (ΣS), oxygen (ΣO), hydrogen (ΣH), and nitrogen (ΣN), were obtained from the sum of the gas species present.

For the initial mixture:

$$\Sigma C_i = \left(n_{CO_2}\right)_i + \left(n_{CO}\right)_i + \left(n_{CH_4}\right)_i \tag{15}$$

$$\Sigma O_i = 2\left(n_{CO_2}\right)_i + \left(n_{CO}\right)_i + \left(n_{H_2O}\right)_i \tag{16}$$

$$\Sigma H_i = 2\left(n_{H_2}\right)_i + 4\left(n_{CH_4}\right)_i + 2\left(n_{H_2S}\right)_i + 2\left(n_{H_2O}\right)_i + 3\left(N_{NH_3}\right)_i \tag{17}$$

$$\Sigma S_i = \left(n_{H_2S}\right)_i \tag{18}$$

$$\Sigma N_i = \left(n_{NH_3}\right)_i \tag{19}$$

where n is the number of moles of the gas indicated by the subscript.

For the mixture at elevated temperature:

$$\Sigma C_f = n_{CO_2} + n_{CO} + n_{CH_4} + n_{CS} + n_{CS_2} + n_{COS} \tag{20}$$

$$\Sigma O_f = 2n_{O_2} + n_{CO} + 2n_{CO_2} + n_{H_2O} + n_{SO} + 2n_{SO_2} + 3n_{SO_3} + n_{COS} \tag{21}$$

$$\Sigma H_f = 2n_{H_2} + 4n_{CH_4} + 2n_{H_2O} + 2n_{H_2S} + 3n_{NH_3} \tag{22}$$

$$\Sigma S_f = 2n_{S_2} + n_{CS} + 2n_{CS_2} + n_{H_2S} + n_S + n_{SO} + n_{SO_2} + n_{SO_3} + n_{COS} \tag{23}$$

$$\Sigma N_f = n_{NH_3} \tag{24}$$

If the total number of moles of the gas species CO, CO_2, CH_4, S, S_2,...etc., is denoted by Σn, the partial pressure of any species j is given by

$$P_j = \frac{n_j}{\Sigma n} P \tag{25}$$

where P is the total pressure in the system. Since the respective values of ΣC, ΣO, ΣH, ΣS, and ΣN in the initial and final states are the same, it can be written

$$\Sigma C_i = \Sigma C_f \tag{26}$$

$$\Sigma O_i = \Sigma O_f \tag{27}$$

$$\Sigma H_i = \Sigma H_f \tag{28}$$

$$\Sigma S_i = \Sigma S_f \tag{29}$$

$$\Sigma N_i = \Sigma N_f \tag{30}$$

$$P = \Sigma p_j \ (j \rightarrow \ CO, CO_2, \ldots etc.) \tag{31}$$

Equations 26-30 along with the total pressure relationship given by equation 31 comprise a set of nonlinear equations that were simultaneously solved with a digital computer to obtain the gas compositions at elevated temperatures. Table II lists the values of pO_2, p_{S_2}, and carbon activity a_c, calculated for the gas mixtures given in Table I for different coal-gasification processes. The results show that at a temperature of 1000°C, the oxygen partial pressures range from 7.2×10^{-17} to 4.5×10^{-15} atm and sulfur pressures from 8.4×10^{-12} to 6.1×10^{-9} atm in these processes. The results also indicate that the MPC selected gas composition (with 0.1% H_2S) is approximately the same as that anticipated in the Synthane coal-conversion process.

The sulfur partial pressures calculated in these processes can be used to evaluate the sulfidation behavior of materials in the gas environment. In the absence of a protective oxide, the calculated sulfur pressures (or p_{H_2S}/p_{H_2}) for different processes can be superimposed on the isocorrosion-rate curves for austenitic stainless steels reported in the literature.(3) Such a comparison is shown in Fig. 1. The values of p_{H_2S}/p_{H_2} for all processes except CO_2-Acceptor are in the range 1.8×10^{-3} to 4.6×10^{-3} and are insensitive to temperature. Under these conditions, the steel should corrode probably at a rate of 20 to 100 mil per year at a temperature of 800°C. The p_{H_2S}/p_{H_2} ratio calculated for the CO_2-Acceptor process is in the range 4×10^{-4} to 6×10^{-4} and this should result in a negligible sulfidation attack of the steels. At a temperature of 980°C (∿1800°F), the metal protection by the formation of Cr_2O_3 layer on the steel surface is minimized due to formation and subsequent vaporization of CrO_3. Under these conditions, the MPC program at IIT Research Institute reported(4) a sulfur corrosion rate of ∿125 mils/1000 h for Types 304 and 316 stainless steel which is in agreement with the values predicted from this analysis.

CORROSION BEHAVIOR IN PROCESS-GAS ENVIRONMENT

The oxidation behavior of Fe-Cr and Ni-Cr alloys show that Cr_2O_3 will be a thermodynamically stable oxide at all chromium concentrations in the alloy. Under these conditions, the simultaneous presence of O_2 and S_2 should be considered in evaluating the alloy behavior. Figures

Table II. Calculated Values for Oxygen, Sulfur, and Carbon Potentials for Gas Compositions in Different Coal-gasification Processes with Low-sulfur Feedstock.

Process	T = 1000°C			T = 700°C		
	P_{O_2} atm	P_{S_2} atm	a_C	P_{O_2} atm	P_{S_2} atm	a_C
Hygas	4.6×10^{-16}	1.2×10^{-10}	0.011	1.4×10^{-22}	3.2×10^{-15}	0.657
CO_2 Acceptor	6.3×10^{-16}	8.4×10^{-12}	0.004	2.8×10^{-22}	2.2×10^{-16}	0.168
Synthane	1.5×10^{-15}	1.8×10^{-10}	0.004	4.6×10^{-22}	4.1×10^{-15}	0.221
Battelle-UC	7.2×10^{-17}	7.0×10^{-10}	0.024	4.8×10^{-23}	2.4×10^{-13}	0.93
Bigas	4.5×10^{-15}	6.1×10^{-9}	0.002	1.5×10^{-21}	1.3×10^{-13}	0.072
MPC Selected (0.1% H_2S)	2.3×10^{-15}	2.1×10^{-10}	0.003	7.2×10^{-22}	4.6×10^{-15}	0.147

2 and 3 show the Ellingham diagrams for Fe-Cr-O-S and Ni-Cr-O-S systems at 1200°K (∿1700°F) along with the calculated partial pressures of oxygen and sulfur in different coal-conversion processes. The open symbols in these figures are the calculated pressures for the gas compositions listed in Table I and the closed symbols are for a H_2S concentration of 1.0 vol. % in the process gas environment. The analysis shows that chromium oxide is thermodynamically stable in Ni-base alloys and that the oxides in Fe-base alloys will be a mixture of iron and chromium oxides. The effect of an increase in the sulfur concentration in the coal feedstock (Appalachian coal in contrast to Western coals) is to shift the process gas compositions in the direction of increasing p_{S_2}. Under such conditions, there exists a distinct possibility for the formation of Fe- and Ni-sulfides. Since the sulfide layers are not protective in nature and the growth rates of sulfide layers are orders of magnitude larger than the oxide layers, the corrosion rates of these alloys may become prohibitively large. Furthermore, in the case of Ni-Cr alloys, an increase in sulfur potential in the gas environment can lead to formation of Ni sulfides that are liquids at this temperature and can result in catastrophic corrosion rates. Therefore, an effective utilization of Fe-Cr and Ni-Cr alloys requires that a protective Cr_2O_3 layer be established upon exposure of the materials to coal-gasification environment. Even though Cr_2O_3 is thermodynamically stable at all chromium concentrations in Ni-Cr alloys, a large chromium content in the alloy is required for the oxidation process to change from internal oxidation to formation of an external protective layer. The chromium concentration that is required for the external scale formation is a function of temperature and oxygen potential in the environment and these functional relationships are yet to be established. It has been reported that a value of 20-25 wt % Cr in the alloy is necessary for the application of Ni-Cr alloy at 900°C and 0.1 atm oxygen pressure.(5)

Extensive high-temperature corrosion studies are being conducted at Argonne National Laboratory to evaluate the behavior of commercial iron- and nickel-base alloys in a complex multicomponent gas environment with a wide variation in composition. Experiments are also planned on high-purity Fe-Cr-Ni alloys in ternary and multicomponent gas environments to understand the corrosion mechanisms under different conditions. Microstructural evaluations and thermal cycling effects form a large part of our investigations. The effect of the corrosive-erosive environment on the stability of the protective layers will also be evaluated.

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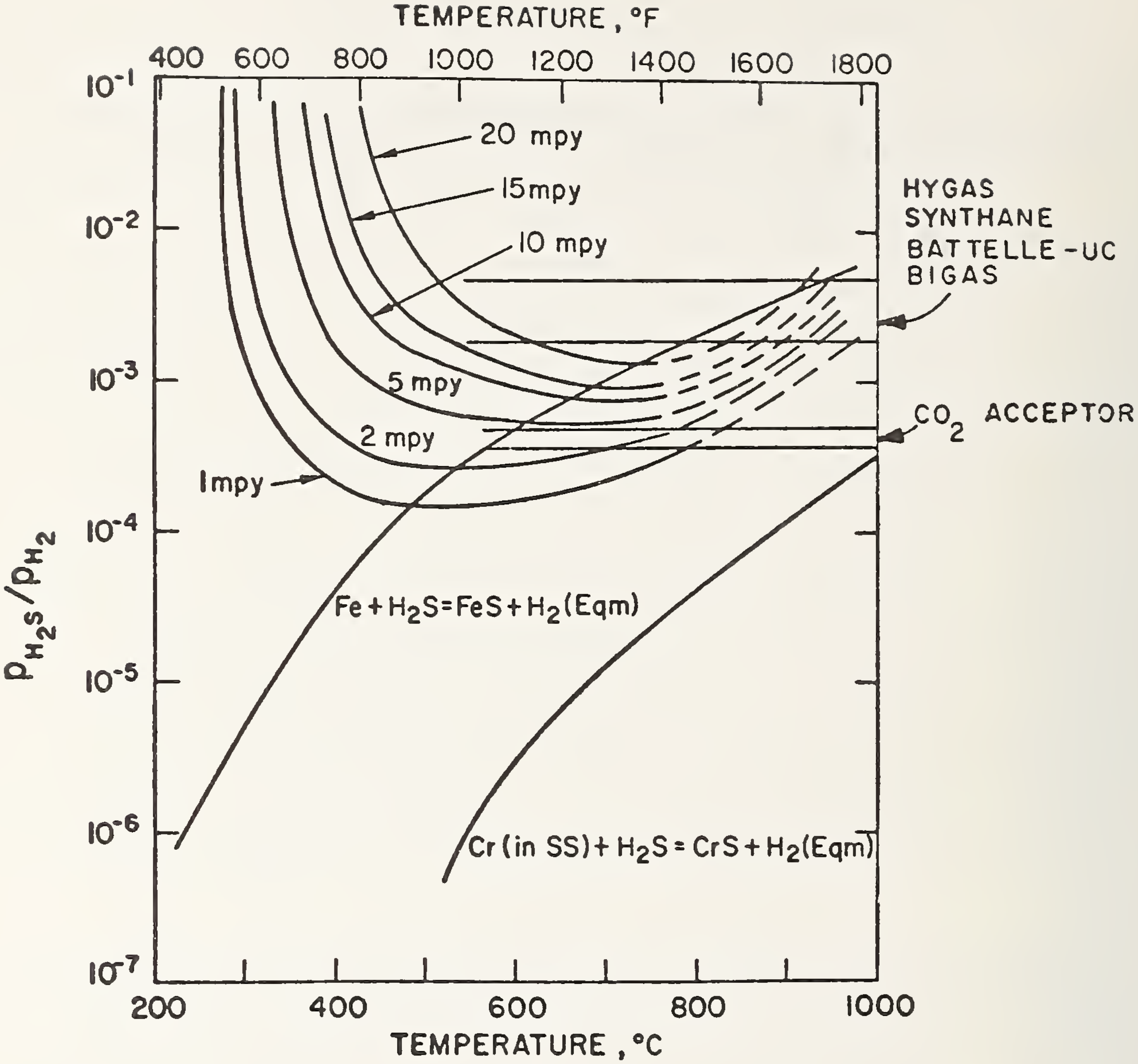

Fig. 1. Superimposition of Sulfur Potentials in Pilot-plant Processes on Isocorrosion-rate Curves for Austenitic Stainless Steel.

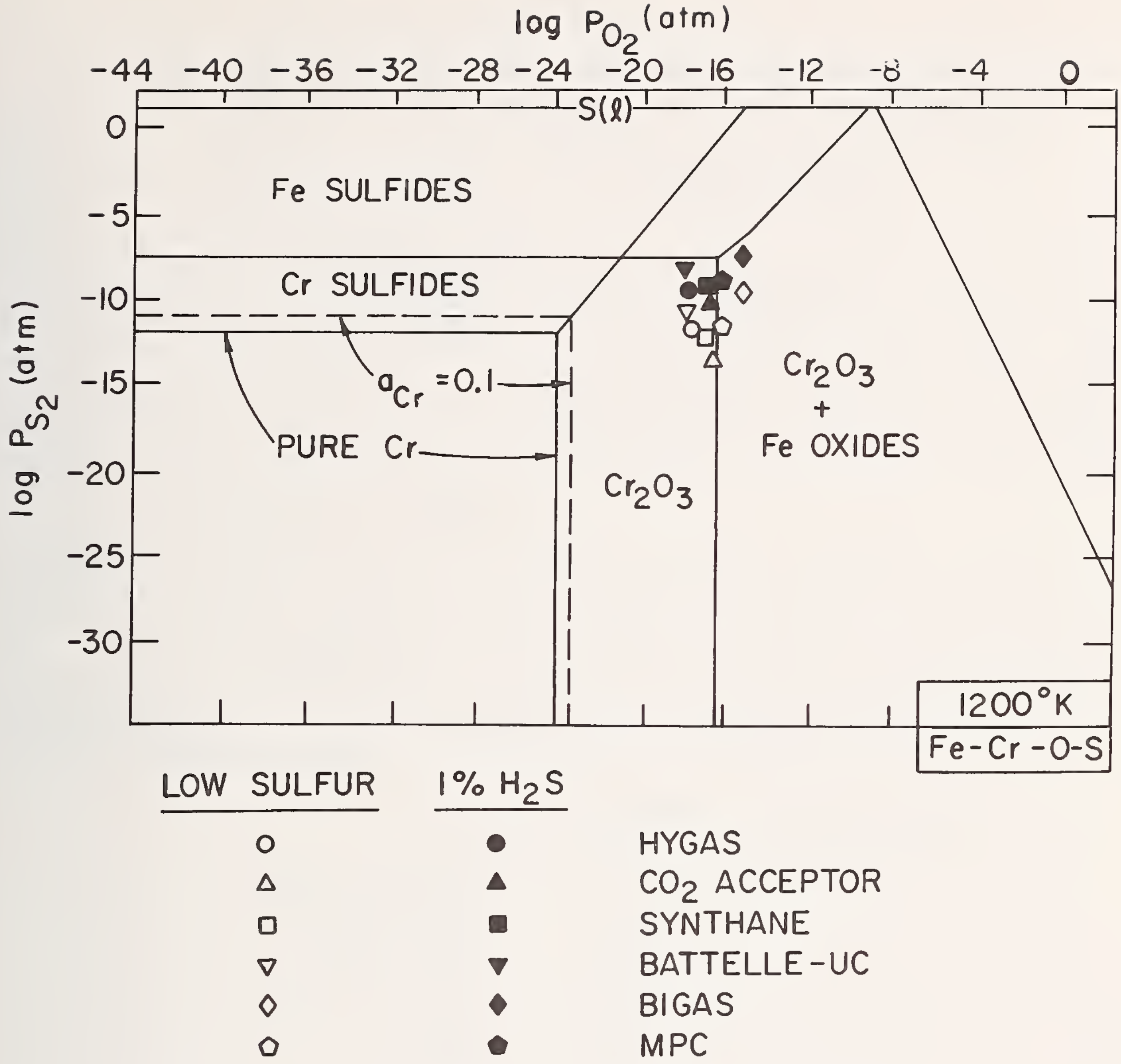

Fig. 2. Thermochemistry of Fe-Cr-O-S System at 1200°K. The gas environments in pilot-plant processes are indicated by different symbols.

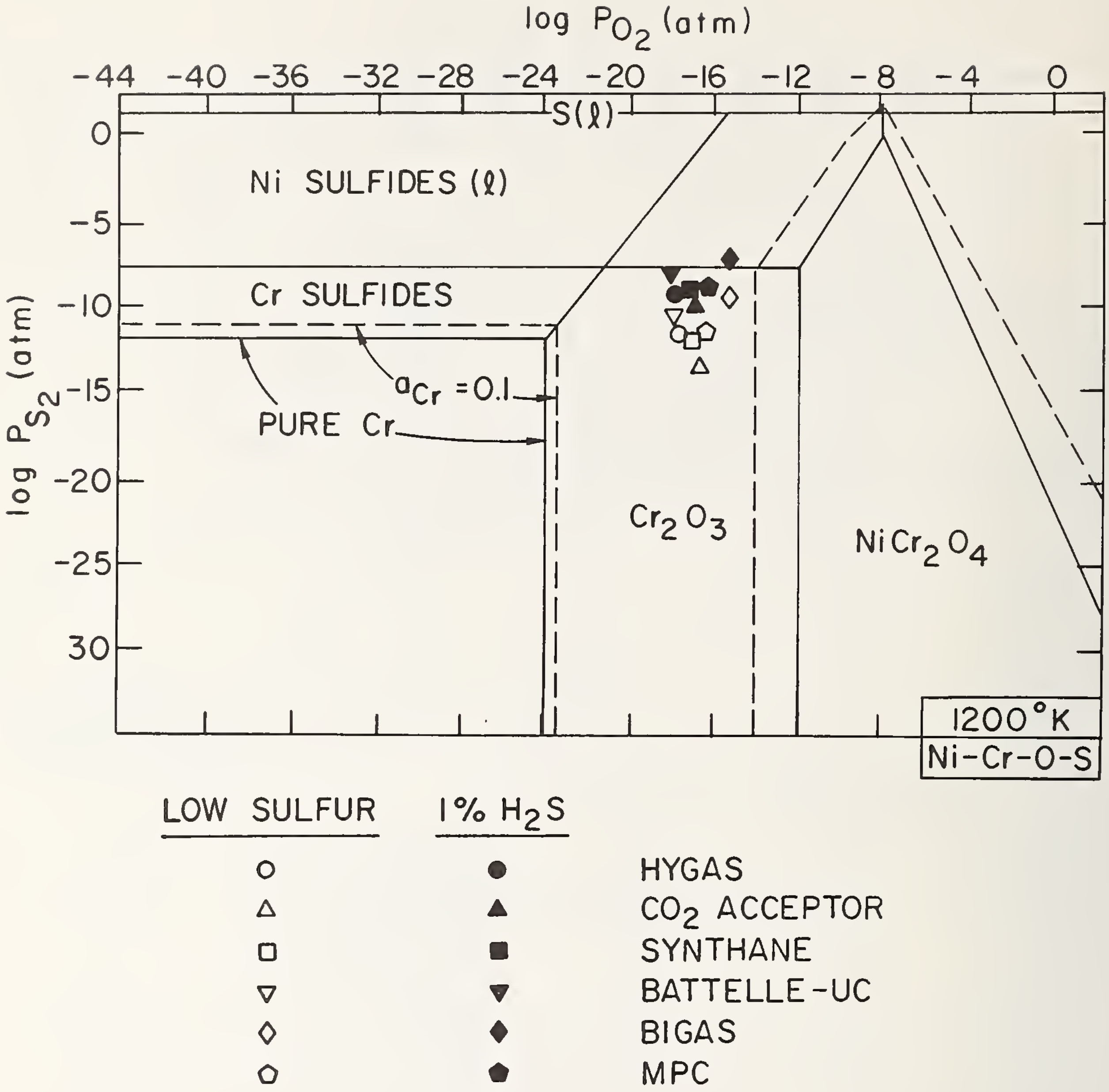

Fig. 3. Thermochemistry of Ni-Cr-O-S System at 1200°K. The gas environments in pilot-plant processes are indicated by different symbols.

DISCUSSION

A. J. Mac Nab, C. F. Braun and Company: I would like to compliment Nat on this work. This kind of analysis is badly needed. The fact that he has suggested getting away from just the Y-direction study and going in the X-axis is also a fine approach. As you rightly pointed out, we are working with only one gas composition and that may not be too meaningful. There are different gases, different types of coal, and different operating conditions, all of which are going to affect gas composition. Would it not be possible to complement what you are doing with a process-type study to pick out gas compositions that could occur in commercial units. It would be nice, if possible, to narrow down the number of different compositions. The charts showing corrosion rates and the thermodynamic cutoff data with the gas compositions from the different plants looked like the data published by Backensto. They are a little controversial at this time in regard to the validity of the corrosion rates. There are now considerably lower values for sulfiding rates from the petroleum industry.

K. Natesan: I don't mean to indicate that that is absolute data. The one thing I wanted to point out was that with 0.1% H_2S and 1% H_2S, the C type 304 and 316 stainless steels measured 125 mils corrosion in 1000 hours. That is right in the same ball park as the data show at 1500°F. The curves may be shifted a little either way, but the approach is there. How effectively it is used depends on the feedback from the plant to the researchers.

DEVELOPMENT OF NONDESTRUCTIVE EVALUATION METHODS FOR COAL-CONVERSION SYSTEMS*

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Abstract: Coal-conversion processes require the handling and containment of high pressure, high temperature, corrosive and erosive gases and liquids often containing particulate loadings. These severe environments cause materials failures that reduce successful and long-time operation of coal-conversion systems. The determination of the material and component response and development calls for proper nondestructive examination methods, equipment, and techniques. This paper briefly describes the nondestructive development efforts in high-temperature, wall-thickness measurements for in situ erosion data, passive infrared imaging applications for thermal profiles,gamma radiographic applications for crack and erosion detection and acoustic methods for failure prediction.

Key words: Nondestructive evaluation; coal gasification; ultrasonic inspection; infrared imaging; refractory liners.

Nondestructive evaluation (NDE) methods for coal-conversion systems must of necessity encompass a broad spectrum of applicable technology. Transfer lines, for example, may be refractory lined or unlined, depending upon the requirements of the location, i.e., composition, temperature, and pressure of the flow. Preoperational inspection and online monitoring systems that measure the material response of materials systems require different nondestructive examination approaches. In nonrefractory-lined transfer lines, ultrasonic pulse-echo systems can be developed to measure wall thinning caused by erosion/corrosion at high temperature. However, the porosity of a refractory, acoustic impedance of a refractory/steel interface, and poor acoustic transfer properties do not allow ultrasonic pulse-echo methods to be used on refractory-lined components typical of coal-conversion process systems. Gamma radiography or passive infrared imaging with appropriate thermal models is necessary to determine the material response and/or structural integrity of refractory-layered structures. In addition, some components such as the lock-hopper or pressure let-down valves have a high initial cost and are time consuming to replace. Nondestructive evaluation methods to assist in determining the optimum time for replacement of such components are of value. The broad-based ERDA/FE sponsored nondestructive evaluation development program at Argonne National Laboratory is designed to address the above areas.

The ultrasonic monitoring of the erosion of high-temperature steel transfer lines requires a waveguide design that must consider: (a) signal-to-noise ratio, (b) energy transfer, (c) material attenuation, (d) appropriate interface geometry (including attachment mechanism), and (e) a satisfactory cooling mechanism. These considerations have led to a delay-line design that will shortly be employed on several coal-conversion pilot plants. A schematic of the delay-line design and associated temperature decay curve is shown in Fig. 1. Ultrasonic thickness measurements require a reflection at the inner and outer wall surfaces to determine the time of flight of the pulse and hence the wall thickness. A typical amplitude decay of the back-wall reflection is shown in Fig. 2. This is a critical reflection because part of the ultrasonic system is triggered by the back-wall amplitude, and an unsatisfactory amplitude would cause the system to fail.

A complete material wall-thickness measurement system using this delay-line design is being implemented to monitor real-time erosion of the main coal feed line of the Synthane coal-gasification plant. Figure 3 shows the coal feed elbow and array of 31 transducers that will be used to establish real-time erosion. A large number of transducers are required on this initial system for a complete mapping of the erosion pattern on this critical component. Clearly, such a large number of transducer sites is not required in all applications, and methods have been developed that will allow the ultrasonic delay line to be attached to existing piping for in-place monitoring.

Gamma radiography has been shown to be capable of clearly visualizing the bore of refractory-lined transfer lines. Figure 4 shows a double-wall gamma radiograph taken with ^{60}Cobalt and Eastman Kodak Type AA film. The dark bore region is sharp, and thus time sequential images could be used for erosion-rate measurements. Figure 5 is a schematic diagram of the transfer line showing the refractory thickness and bore diameter for the double-wall radiograph. The refractory in this case is KAOTAB, which is a high-density cast-alumina refractory with a density of $\sim$150 lb/ft^3.

The high temperatures and high pressures of most gasification systems have also demanded the use of refractory-lined pressure vessels. The most common methods used to install refractories are gunning or casting. The protective refractory lining is usually monolithic or layered with low-density insulation covered by a high-density hot face. These refractory-steel structures are used with or without water-cooling jackets. Schematic diagrams of typical dry-wall and water-cooled wall pressure vessel sections are shown in Figs. 6 and 7.

Thermal cycling and the resultant moisture condensation during start-up and shutdown can cause degradation and cracking of the refractory. This can result in sufficient refractory spalling to expose so that the steel shell would be exposed to high temperature and pressure. The thickness of the installed refractory and the uniformity of the refractory density is important for long duration runs. Gamma radiography has been shown to be a viable method to not only locate cracks but also to map refractory thickness variations. Figure 8 is a plot of normalized radiographic film density as a function of refractory thickness for KAOTAB refractory (of uniform density) on a 3/4-in. steel plate. The data were normalized by means of a steel step wedge and the characteristic amplitude transmission-exposure (t-E) curve for Eastman Kodak AA radiography film (see insert on Fig. 8). In the linear range of the t-E curve, the density D can be related to the exposure E as follows:

$$D = \gamma_n \log E - D_o, \tag{1}$$

where

D = photographic film density,

γ_n = slope of line curve,

E = exposure = IT,

I = intensity,

and

T = time of the exposure.

The results of the photographic density versus refractory thickness were verified by a field application on the Battelle-Columbus coal-gasification process development plant.

Additional work is being conducted on the use of remote thermal-sensing and pattern-recognition techniques through the use of passive infrared imaging systems to obtain thermal profiles on critical components. Expansion bellows, necessary in long, high-temperature transfer lines, are in dynamic states of design, and full field thermal mapping is useful in determining design effectiveness, i.e., indicate particulate buildup, erosion, or gas by-pass flows. Figure 9 shows the general exterior geometry of an expansion bellows and a typical real-time isothermographic image. Such images are being obtained by the use of a commercially available AGA Model 750 portable infrared scanning camera that produces a complete image in 40 ms. The use of pattern-recognition methods by means of digital computers will be employed to compare the thermal patterns generated by particle erosion.

Other work is also being conducted on acoustic emission as a means of detecting crack initiation and propagation in refractory-layered vessels.

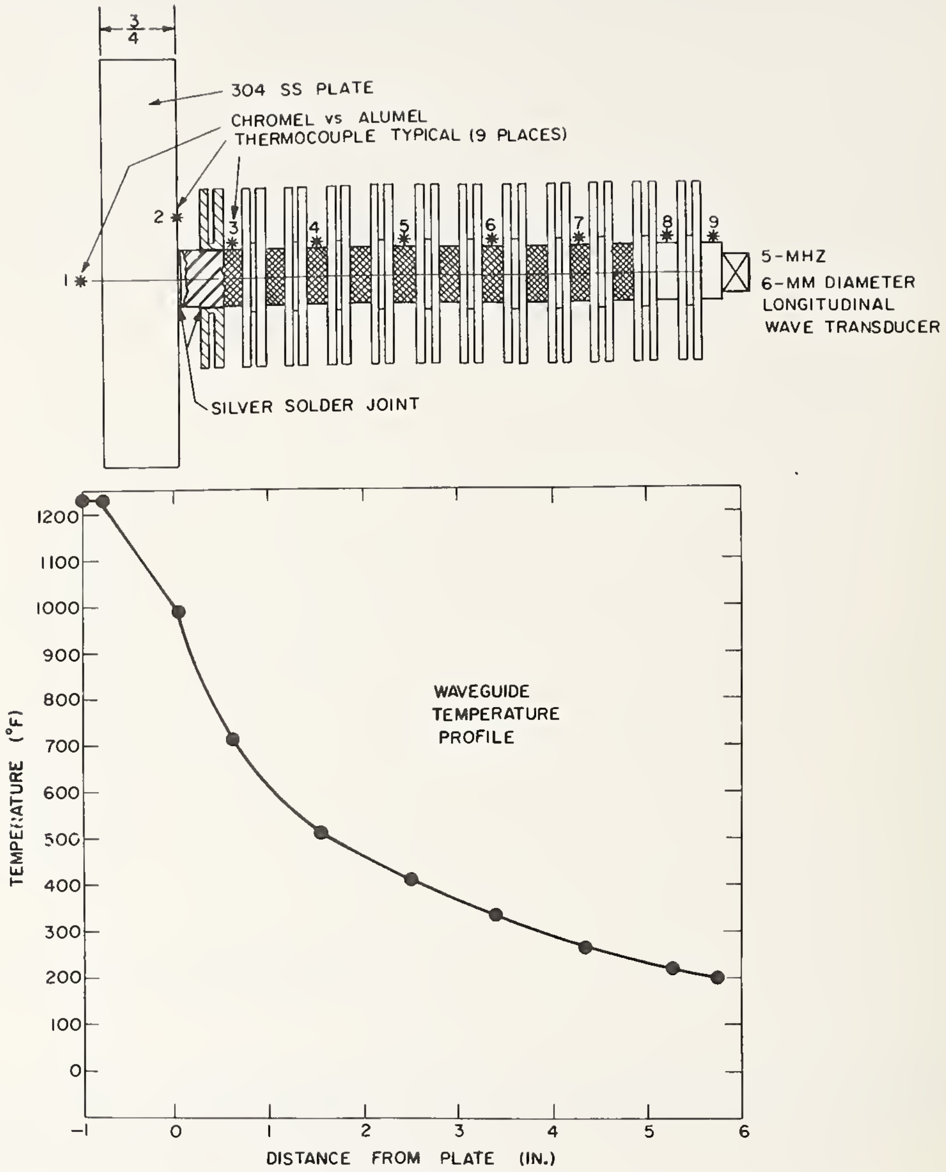

Fig. 1. Air-cooled Waveguide and Temperature Decay Curve with Block Diagram of Instrumentation.

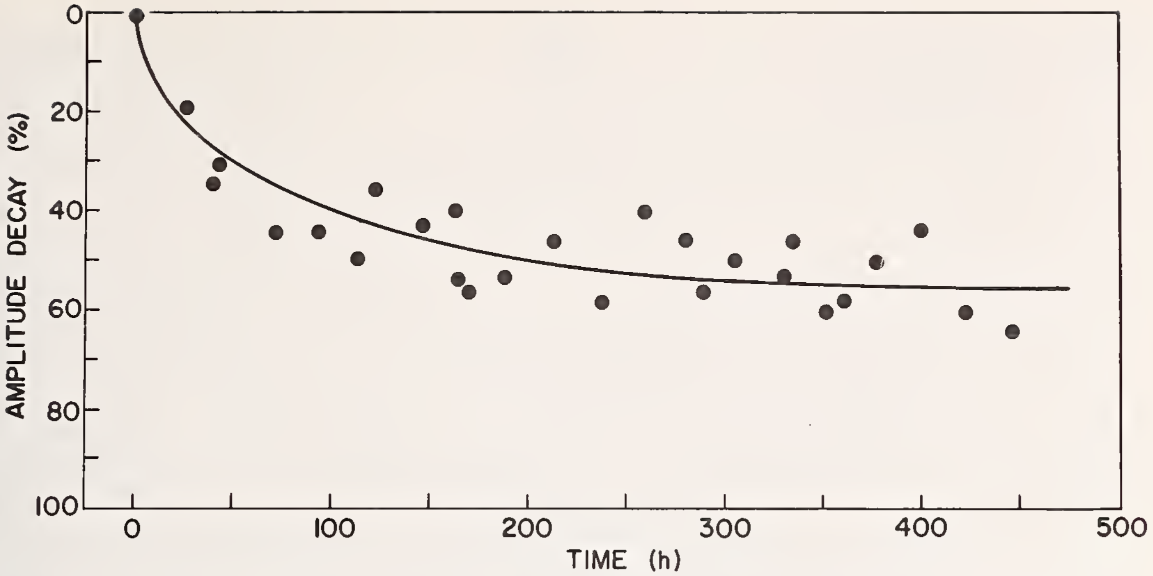

Fig. 2. High-temperature Ultrasonic Backwall Signal Amplitude Decay Curve.

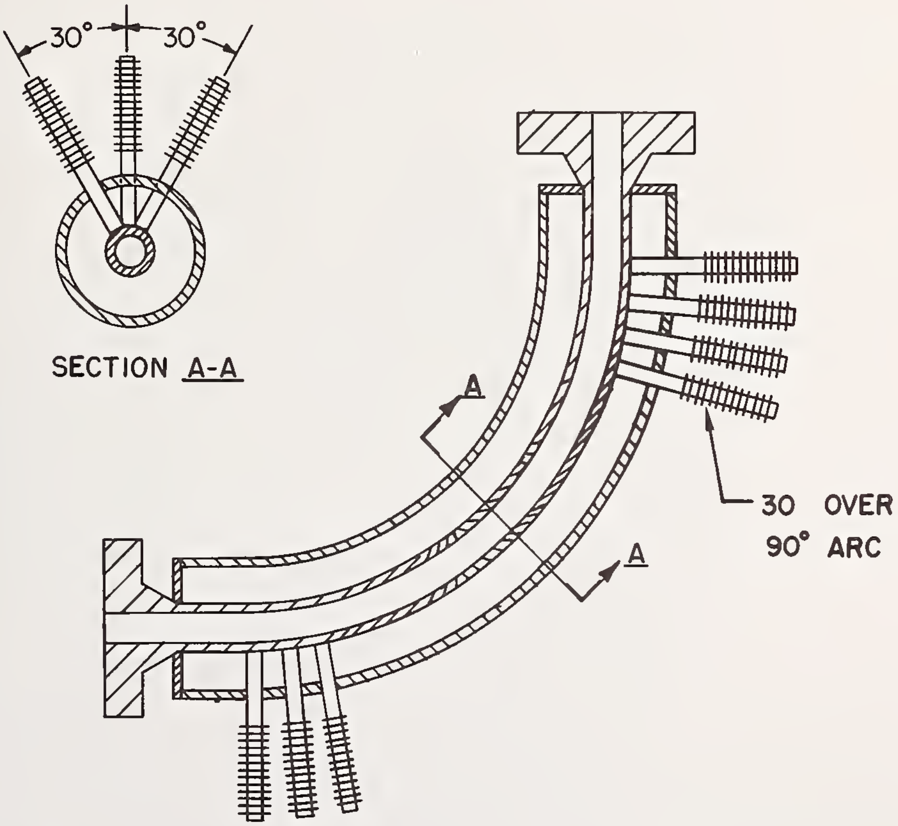

Fig. 3. Line Drawing of Coal Feed-line Replica. Waveguides are axially staggered ±30° to cover a 60° cross-sectional sector.

Fig. 4. Typical Gamma Radiography Taken of Refractory-lined Transfer Line.

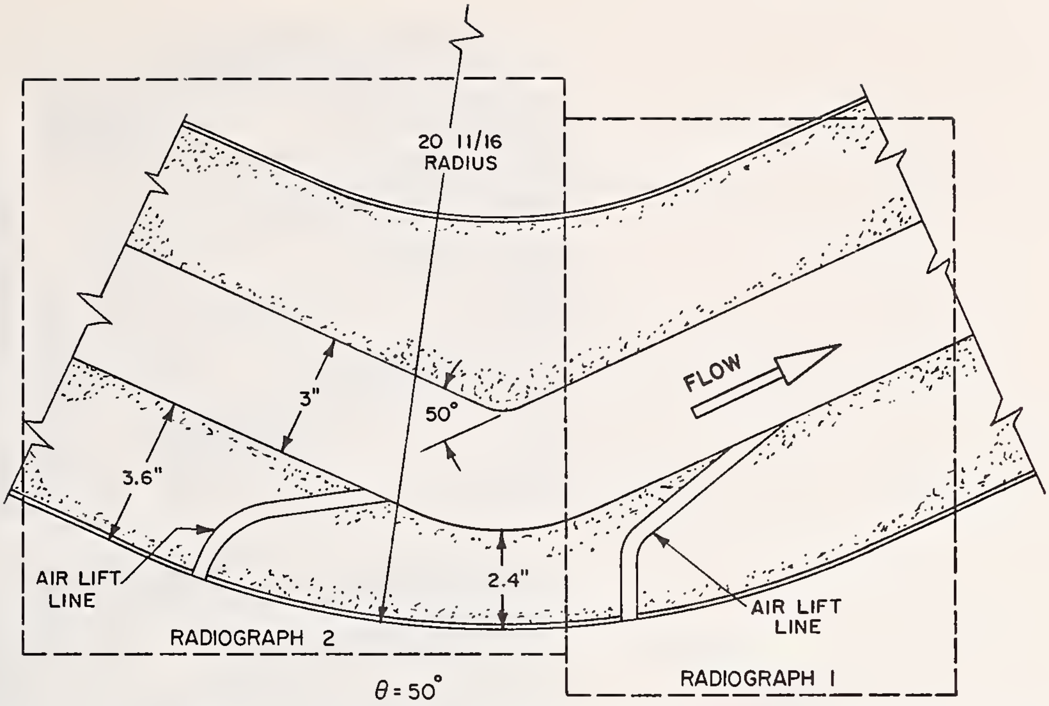

Fig. 5. Schematic Diagram Showing Refractory Thickness for Double-wall Radiograph.

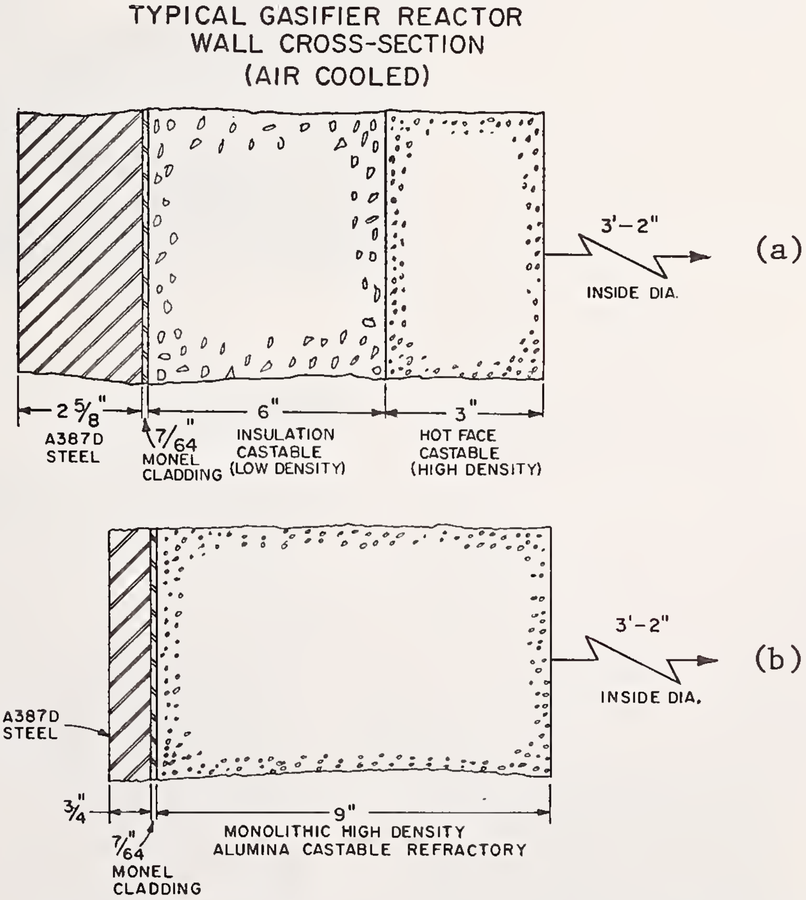

Fig. 6. Schematic Diagrams of Typical Dry-wall (Air Cooled) Refractory-lined Pressure Vessels. (a) Two-component refractory and (b) monolithic refractory.

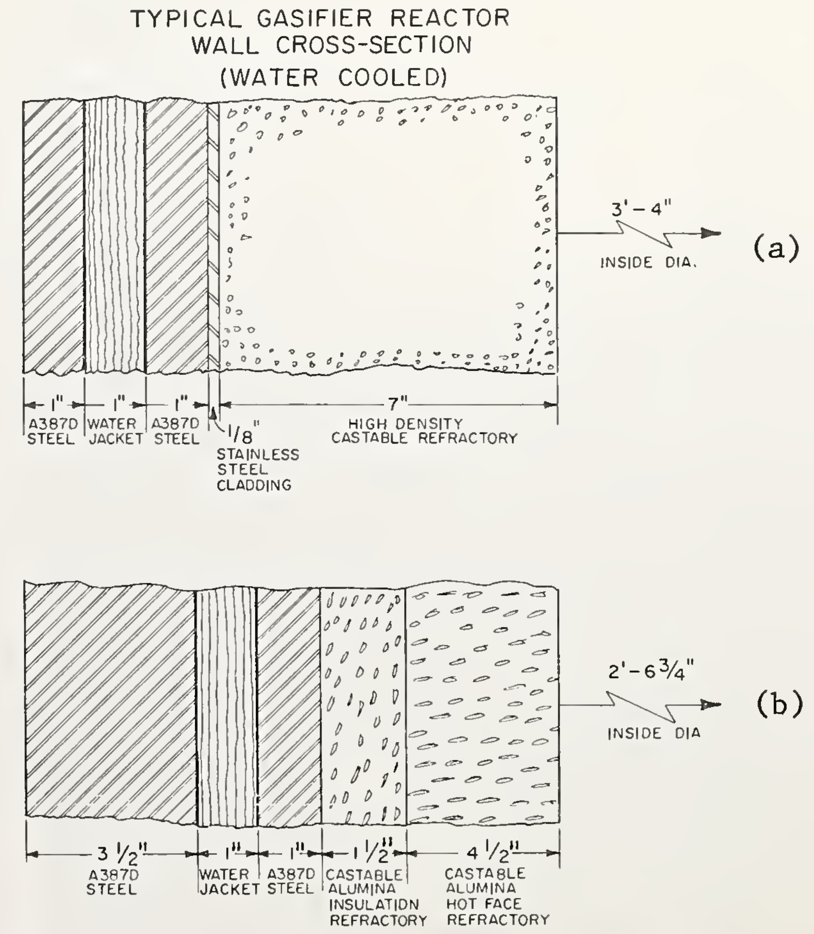

Fig. 7. Schematic Diagrams of Typical Water-cooled Refractory-lined Pressure Vessels. (a) Monolithic refractory and (b) two-component refractory.

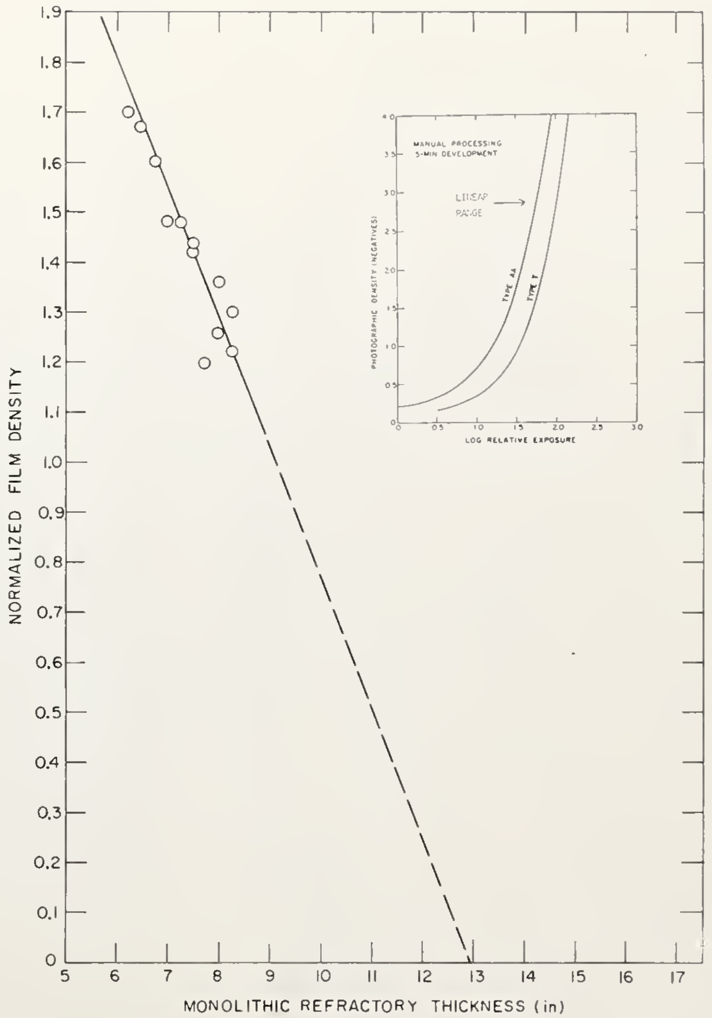

Fig. 8. Normalized Radiographic Film Density as a Function of Monolithic Refractory Thickness on a 3/4-in. Steel Plate.

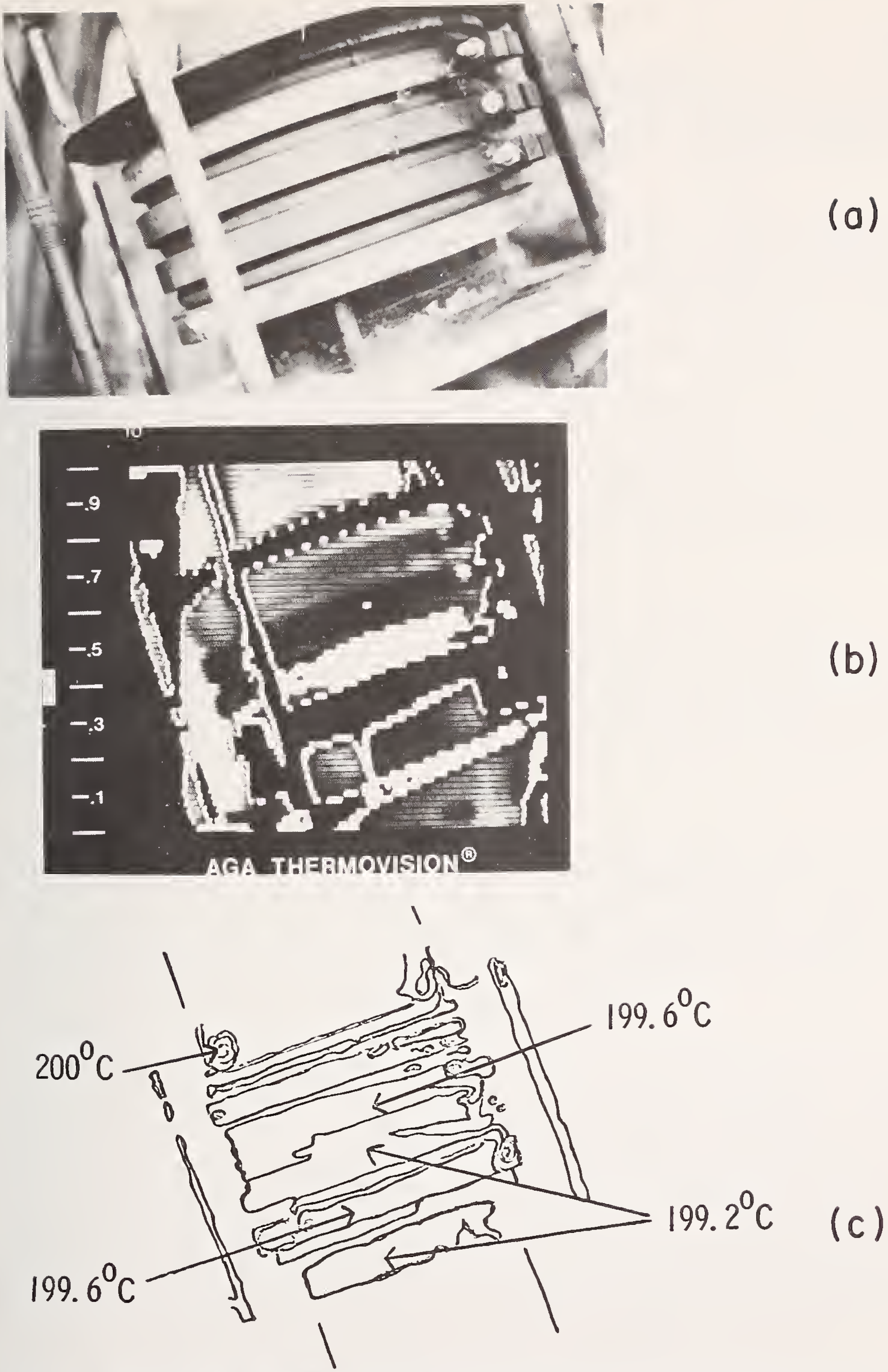

Fig. 9. Infrared Thermal Profiling of Bellows Expansion Joint of Bellows Expansion Joint of CO_2 Acceptor Coal-gasification Pilot Plant. (a) Conventional photo of bellows expansion joint, (b) isothermogram image of bellows expansion joint, level at 199°C, and (c) composite thermal profile map of expansion joint.

DISCUSSION

E. R. Fuller, Jr., National Bureau of Standards: Are you planning to use the acoustic emission spectrum analysis technique instead of ultrasonics to monitor erosion of the elbow joints?

W. A. Ellingson: We are planning to use this technique to monitor erosion in the cyclone. One of the problems when mapping the cyclone wall thickness is knowing whether the bore is concentric. An apparent wall thinning may have been due to a nonconcentric bore rather than erosion.

POTENTIAL MATERIALS PROBLEMS IN COAL GASIFICATION SYSTEMS

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Abstract: Potential problems with structural materials in those coal gasification systems designed to produce a make gas for conversion to pipeline quality gas are discussed. The following problem areas are covered: corrosion and erosion/corrosion; wear and abrasion; hydrogen effects; creep and creep-rupture; temper embrittlement of steel; thermal fatigue; component design and manufacture; material quality; and assembly of components and equipment. Both metallic and ceramic materials are discussed in terms of these potential problem areas.

Key words: Abrasion; ceramics; coal gasification; component design; corrosion; creep; creep-rupture; erosion/corrosion; fracture toughness; hydrogen attack; hydrogen effects; hydrogen stress cracking; material quality; metallic materials; refractories; stress raisers; structural materials; temper embrittlement; thermal fatigue; wear; welding.

The objective of this presentation is to highlight structural materials problems that are being, or may be, encountered in coal gasification plants. Our interest, however, is not in all types of plants but is directed entirely at those plants that are designed to use the advanced processes currently under development to produce a synthesis gas suitable for conversion to pipeline quality gas.

These processes differ considerably among themselves as to chemistry, operating conditions, and component and equipment design. Nonetheless, they have much in common and some general statements can be made regarding materials and equipment problem areas. First, it is clear that process conditions are unique and, in a number of process steps, the environments experienced by the structural materials are extremely severe. Again, equipment and component failures seldom are ascribable exclusively to the material; they usually involve other factors such as component design, operating conditions, component manufacture, and equipment fabrication, or a combination thereof.

METALLIC MATERIALS: For convenience, we will separate problems involving metallic materials from those involving ceramics, and we will discuss them by phenomenological category starting with corrosion.

Corrosion and Erosion/Corrosion: No unusual or severe corrosion problems are to be expected in the handling, storage and preparation of the coal to be consumed in the plant, unless unsuitable materials are selected. For example, if an overzealous contractor constructed coal pretreatment equipment of austenitic stainless steel instead of carbon steel, he might invite chloride stress-corrosion cracking because moisture would be present and coal contains chlorides.

The greatest opportunity for corrosion appears to be elevated-temperature attack of exposed penetrations in such equipment as gasifiers, regenerators and combustors. The attack is likely to take the form of oxidation, sulfidation, carburization or combinations thereof, depending on the composition of the environment. Whenever the atmosphere is turbulent and contains particulate matter, the situation will be complicated by the added phenomenon of erosion which will work hand-in-glove with corrosion to promote material deterioration. The problem is one of material and/or coating selection coupled with design aspects aimed at reducing turbulence. Coatings to be considered in such cases would be abrasion-resistant types capable of withstanding high temperatures.

The erosion factor probably becomes more pronounced in the case of the exposed metallic components of valves for transfer lines and let-down systems. These assemblies will handle streams heavily laden with particulates often at very high temperatures. Careful material selection and clever value design are required to alleviate the likely problems.

Equipment for wet scrubbing and quenching of the product gas from the gasifier constitutes another locale for potential corrosion problems, the nature and severity of which will depend on the composition of the gas and the materials of construction and perhaps on the design and construction of the equipment. It could be stress-corrosion cracking, pitting corrosion, crevice corrosion, general corrosion, or even galvanic corrosion.

Wear and Abrasion: Lines handling dry or slurried coal may be subject to wear and abrasion. The same holds for the valves used in such lines, for the controlled-rate feed devices used to inject coal into gasifiers and combustors, and for the pumps that handle slurries. The ensuing problems should be resolvable by proper material selection, component design, equipment selection, or alteration of operating conditions.

For example, as to equipment selection, plunger- and piston-type reciprocating pumps are preferred for handling slurries. Liquid-end parts are quickly and easily replaced. Centrifugal pumps are impractical for transporting slurries at high pressures because of the large number of wear parts and close tolerances required.

As to materials, standard gray cast-iron components may be unsatisfactory and white irons may be required; or, structural steels may have to be replaced by abrasion-resistant (AR) grades. Again, ceramics or rubber may be superior to metallic materials or coatings for certain components.

Hydrogen Effects: Economic considerations strongly favor use of structural carbon or low-alloy steel for the principal structural elements of the gasifier, such as the shell, heads, nozzles, and internal jackets or coils. However, during operation, the internal gaseous environment will gain access to the ID surfaces of these components either by diffusing through the refractory lining or by penetrating cracks in the refractory. Thus, to prevent corrosion of the steel by condensed vapors the temperature of the shell must be maintained above the dew point of the process stream. At the same time, to avoid excessive scaling, sulfidation, and degradation by hydrogen as well as loss in strength, the shell should be held to a moderate temperature, i.e., in the order of 600 F. The major contributors to hydrogen problems would be the H_2 and H_2S in the internal atmosphere, while H_2S would be the sulfidizing reagent.

Of course, the steel shell may be internally coated with an epoxy or other organic material to protect it from corrosion by condensates, or it may be clad, lined, or overlaid with a corrosion-resistant alloy such as stainless steel.

Superficially, it would appear that the situation regarding the steel components is well in hand. However, even though these components may be coated or clad, hydrogen will be able to enter them at the temperatures and over the time periods they are expected to experience in service. Moreover, the interaction of hydrogen and steel at high pressures in the temperature regime of 450 to 600 F or so is not well documented and is a matter of great concern because of the danger of unexpected catastrophic failures.

Problems resulting from dissolved hydrogen that could be encountered in shell and duct components are: (1) loss in tensile ductility on cooldown to room temperature, (2) hydrogen stress cracking that may occur on cooling to room temperature during a shutdown, (3) blistering, and (4) hydrogen attack which results in decarburization with attendant loss in strength and formation of internal fissures, cracks and sometimes blisters. In coal gasification the concern over degradation of structural components by hydrogen is both with the effect of hydrogen at temperature and, perhaps more importantly, with the events that could occur on cooling down to room temperature during a shutdown. These types of questions are now under investigation, but the area requires much more research than is now underway.

Other Phenomena: Creep-rupture failures, or failures caused by excessive creep without rupture, should not occur in load-carrying components under normal conditions. They would not be operating in the creep regime. However, failure of a section of refractory lining in, for examle, a high-pressure gasifier undoubtedly would trigger these failure mechanisms in the steel shell by exposing the metal to the high temperature of the internal environment. A way to reduce the potential damage is to coat the steel shell with temperature indicating paint, to place thermocouples at strategic locations on the shell, or to monitor the shell's temperature with a scanning infrared thermocouple, and have water hoses available to cool the shell when a runaway overtemperaturing situation is indicated.

Carbon and low-alloy steels used in combustors, gasifiers, and regenerators may gradually become brittle during long-time service at operating temperature. The petroleum industry has become concerned about the problem of aging or temper embrittlement of chromium-molybdenum steels during service for long periods at intermediate temperatures. The industry uses this type of steel in many heavy wall reactors where resistance to hydrogen attack and high elevated-temperature strength are required. Research on the phenomenon is in the early stages.

Thermal fatigue is another problem that may arise. However, it is far more likely to be encountered in process demonstration units than in full-scale commercial operations because the former experience a great many more start-ups and shut-downs than the latter. Thus, although thermal fatigue failures are frustrating, their prevalence in process demonstration units is not necessarily indicative of performance under full-scale conditions.

CERAMIC MATERIALS: Coal-gasification processes currently being investigated will place severe demands on the refractory linings of gasifiers as well as those of combustors, regenerators, cyclones, and connecting piping. It is these materials that must withstand the brunt of the corrosiveness, erosiveness, and general reactivity of the hot, high-velocity, high-pressure, particulate-laden process gas stream. The severest condition is considered to be in the gasifier.

The refractory problem is compounded by the fact that there are no closely analogous applications. Thus, considerable extrapolation from state-of-the-art experience is required to meet the coal-gasification situation. The nearest analogs seem to be the secondary reformer in the manufacture of ammonia, ethylene pyrolysis, blast furnaces for production of iron, and the cyclonic boiler. These operations encompass various combinations of gas reactivity, gas velocity, erosion, pressure, and temperature but none combine reducing conditions, high pressures, high temperatures, high velocities, and erosion--the conditions that distinguish the gasifier environment. Therefore, many questions arise with respect to the performance of refractories in the unique and severe environment of the gasifier. It is clear that much needs to be learned about the performance of refractories in coal gasification environments. The same goes for insulating materials. Some specific problem areas are as follows:

(1) Chemical interaction between refractories and the CO, H_2, H_2O, H_2S, char, and unreacted coal in the atmosphere

(2) Effect of hydrogen on the properties of insulating materials

(3) Erosion of refractories by steam and solids in the atmosphere

(4) Effect of impurities on the properties of refractories and insulating materials operating in reducing environments

(5) Spalling and disintegration of refractories

(6) Compatibility of refractories with coal ash, including slagging, penetration of the refractory, and alteration of the refractory's properties

(7) Effect of acid condensation on the integrity of refractories and insulating materials

(8) Reliability of thermal-physical property data for candidate refractory and insulating materials

DESIGN, MANUFACTURE AND ASSEMBLY: As has been noted, intelligent and innovative design can alleviate or even solve materials and equipment performance problems. On the other hand, faulty design can do the opposite. For example, designing mechanical notches into a load-carrying structure is intolerable. Likewise, overdesign may be self-defeating; for instance, increasing wall thickness may increase thermal gradients and hence, transient stresses. Again, a structure which is to be assembled by welding should be so designed and not treated as monolithic. Likewise, castings should be designed to take into account the constraints of the casting process, rather than being designed according to concepts based on manufacture by forging or machining.

In the manufacture of critical components, steps should be taken to insure that the quality of the material used is high enough to provide adequate fracture toughness. In this regard, in structural steels, important considerations include: minimum adequate carbon content; low sulfur content; chemical homogeneity; low nonmetallic inclusion count; and good quality surfaces.

Assembling components into a structure, especially a structure so massive and demanding as a full-scale pressurized gasifier, is a critically important activity. Such a vessel may be as much as 250 feet high by 30 feet in diameter and weigh 3000 tons.

For the metallic components the job is accomplished principally by welding. Careful planning and close supervision are required. Among other things, every precaution must be taken to insure that assembly operations do not promote structural failure. This means close attention to details even to such seeming trivia as avoiding arc strikes, avoiding nicking and gouging of surfaces, avoiding rough welds and excessive crowns on welds, and a host of other actions that produce stress-raising notches in the structure which, in turn, may become points of origin of catastrophic failures.

Finally, another area fraught with potentially catastrophic problems should be noted. It is the refractory linings for such equipment as combustors, gasifiers, regenerators, and connecting ductwork. As indicated earlier, collapse of sections of such linings may bring about failure of the pressure-containing metallic piping or shell by creep buckling or creep rupture. The cause of such a collapse could well lie in improper installation of the refractory. For most installations, castable refractories will be used. Great attention must be directed to supporting them and anchoring them in place.

SESSION IV

MATERIALS PROBLEMS IN COAL CONVERSION PILOT PLANTS

CHAIRMAN: H. E. FRANKEL,
ENERGY RESEARCH AND
DEVELOPMENT ADMINISTRATION

LOW DUCTILITY, HIGH TEMPERATURE FAILURE OF AUSTENITIC MATERIALS

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Gulf Oil Corporation is actively engaged in coal conversion research and development with a Solvent Refined Coal (SRC) process pilot plant in Tacoma, Washington, and laboratory studies at Merriam, Kansas and Harmarville, Pennsylvania. They are also conducting pilot plant development of the Catalytic Coal Liquefaction (CCL) process at Harmarville, Pennsylvania.

An interesting and pertinent problem that we have seen several times involves low ductility intergranular fracture of austenitic alloys operating in the 1100 to 1200°F temperature range.

One specific problem that confronted us was a crack that developed in a ten inch Incoloy 800 type 2 transfer pipeline. Incidentally, this piece of piping was made of rolled plate that was manufactured in England. It was shipped to a fabricator in France who rolled it into pipe sections and welded it. It went to another fabricator in France for fabricating into a piping detail. From there it went to a unit in Spain.

This pipeline operated at about 1200°F under 400 psig in a clean environment--that is, little or no sulfur was present. There were essentially no contaminants in the system. The temperature was too high for the presence of chlorides, but the fracture was obviously brittle. The fracture passed through parent material--not weld material. We worked at solving this problem for five years. It was concluded that the cracking was caused by a strain aging phenomenon which led to low ductility at high temperatures. We had similar low ductility intergranular fractures occurring in Types 304, 316 and 321 stainless steel that had been operating in the 1100 to 1200°F range.

A modest research effort testing prestrained specimens has shown that a 10% prestrained specimen aged for 1000 hours can be notch sensitive and fail with less than 5% elongation. The cause of failure was identified as prestrain followed by loading at temperature. The failure mechanism has been characterized as strain age embrittlement. There is formation of a precipitate and there is grain boundary depletion. Solution annealing after all fabrication has been an effective corrective measure.

It seems that this is the kind of problem that we are going to have in the coal conversion industry when working with austenitic materials at high temperature.

THE SRC PLANT

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Plant Background

The SRC (solvent refined coal) plant at Tacoma, Washington is a 50 tpd (feed rate) pilot plant located on Fort Lewis, Washington. The plant was originally financed by The Office of Coal Research, Department of Interior. It is presently funded by ERDA and operated by The Pittsburg & Midway Coal Mining Co., a Gulf subsidiary. The process design was developed by The Pittsburg & Midway Coal Mining Co. and the initial plant design was by Stearns-Roger Corporation. Construction was by The Rust Engineering Company under the direction of The Pittsburg & Midway Coal Mining Co.

Construction began July, 1972 and was completed in the summer of 1974. The first coal was introduced September 30, 1974. The first year of operation was spent making material balance runs at different conditions of reaction flow, residence time, pressure, and temperature and sorting out other reaction area parameters. In September, 1975 a concerted effort to produce 3,000 tons of SRC for a burn test was begun. The first four months of this production run resulted in approximately 100 tons per month production due to mechanical problems in the filtration area and processing problems in the solvent fractionation areas. During February, 1976 approximately 500 tons of SRC was produced and during March production amounted to about 400 tons. Improvement to 500 or 600 tons per month now seems possible.

Process Description

In the process, coal is dried and pulverized to about 70 percent through 200 mesh. The coal is then blended with a solvent derived from the process at a solvent to coal ratio of approximately 1.5/ 1. The solvent/coal slurry is then mixed with hydrogen and fed through a preheater to a dissolver which provides sufficient contact time at approximately 1,500 psig and 850°F to complete the reaction.

In the reaction, hydrogen (about 2 percent by weight of the MAF coal) adds to the coal molecule, which dissolves in the solvent. After reaction, the coal solution is let down in pressure and fed to a pressure precoat filter operating at approximately 150 psig and 450°F to remove the ash. Flash gases are scrubbed to remove H_2S and a portion is recycled. The cake from the filter is dried to recover solvent in an indirect fired rotary dryer.

The solution of SRC coal product and solvent is fed to a vacuum flash unit operating at 600°F and approximately 3 psia. Solvent is recovered and fractionated for use in the process. The heavy ends (process solvent) is used for slurrying with feed coal while the middle cut (wash solvent) is used for washing on the pressure filter. Light ends from the pilot plant are disposed of through local contractors. The molten SRC product from the bottom of the vacuum flash drum is solidified by cooling on a flaking belt.

Other facilities at the pilot plant include a hydrogen generator which is a conventional steam-methane reforming unit. Desulfurization of recycle gas is accomplished by absorption in a diethanolamine scrubber. H_2S is converted to elemental sulfur using a Stretford process.

The plant is also equipped for slurry recycle from the flash step before filtration back to the slurry blend tank. This is expected to render product as liquid rather than solid at ambient conditions.

The SRC product has a heating value of approximately 16,000 Btu/lb and solidifies around 350°F. The SRC product contains 0.1-.2 percent ash and 0.6-0.8 percent sulfur, depending on type of coal fed.

Problem Areas Encountered to Date

(1) Slurry preheater feed pump valve erosion. This was solved using 200 chrome spherical valves instead of spring loaded hemispherical valves.
(2) Raw coal feed chute pluggage when feeding wet coal. Solved by changing plant chutes, air locks, etc..
(3) Early concern because of high erosion rates of 347SS reaction area piping has diminished to the point of being inperceptable with ultrasonic gaging equipment.
(4) Erosion of reaction area tungsten carbide letdown valve trim every 15 to 30 days of operation remains a problem to be addressed before commercial scale-up.
(5) Overstressing of high pressure-high temperature slurry to air exchangers was solved by removing this unit from service and replacing it with water quench, flowing directly into the slurry.
(6) High-temperature, pressure filtration has caused the most problems, but we are confident we now have most of these problems solved on the pilot plant level. Commercial size scale-up of these units still poses many potential problems, including capital and maintenance cost.
(7) Coking of the vacuum flash preheater and downstream piping has been minimized by removing light ends prior to preheating and vacuum flashing.
(8) Product solidification problems have been eliminated, however there are now serious product dusting problems to solve.
(9) Pump and pump seal life has been greatly improved but much work remains to be done in this area.
(10) Mineral residue drying to recover solvent from the filter cake discharge is a severe operational problem and the disposal of the dry mineral residue is an even worse problem due to its pyrophoric nature and the potential pollution hazard due to contained phenol

and sulfur.

A Challenge to Manufacturers

Manufacturers and others may help develop this process by being willing to do the unusual in terms of design, delivery, and in some cases initial pricing of their special product.

Those of us on the energy crisis "firing line" need, from manufacturers, quick solutions to "hairy" problems, even if it is on a very small scale and in fact may be unprofitable and seemingly undesirable business. When this, or some other process, is commercialized, those with foresight enough to have been willing to help, (I mean really help by giving our needs priority in your production and R&D shops) will be at the top of the recommended vendor's list. More importantly, they will be in a position to provide workable equipment based on first hand knowledge of the process. Gaining further knowledge through participation in early synthetic fuels problems is the most economical and logical approach to fulfill the mission of this group, the Mechanical Failures Prevention Group.

I believe I speak for the entire coal conversion industry when I say, we welcome all the help we can get, but be prepared to perform (and perform quickly) before a proposal is made. When a $20 million plant is idle with a problem, we can't wait 42 weeks for a $5,000 solution. By providing quick action and delivery, manufacturers may gain invaluable insight into a new industry. A few "handsprings" by an engineering force and/or a production department can accomplish as much as 50 super-salesmen working night and day.

HIGH BTU GASIFICATION PILOT PLANT EXPERIENCE

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In developing a process from laboratory to commercialization the usual justification for a pilot plant stage is to demonstrate continuous, integrated process feasibility. Process considerations therefore receive top priority. Secondary benefits include the gathering of process information for operational control and for scale up to commercial size. Materials performance usually takes a low order priority and is of concern only as it affects the safety or operability of the pilot plant. Pilot plant construction therefore tends to be expedient and self serving and does not necessarily reflect the best or even reasonable materials selections for commercial plants.

Such is the case with the high Btu gasification pilot plants. In a few areas where the environments are particularly severe the best materials selections presently available have been made. Thus, in a sense, these selections can be regarded as feasibility studies. But so far there has been no attempt to operate the units to establish operational limits on materials or to determine the long term outlook for materials life. Indeed the incentive to do so may be lost if the present materials perform well and do not significantly affect the capital investment for a commercial unit. That is to say the next plants built, either demonstration or commercial units will copy the pilot plant materials of construction and the limitations will be learned by experience.

The above discussion covers events that are fairly normal throughout industry, but are an essential background to the following discussion on pilot plant materials failures.

RELIABILITY: The significance of any pilot plant component failure must be valued against its reliability requirements in commercial service. There are three main aspects of reliability, (1) Safety, (2) Operability, and (3) Economics.

While all three items are of utmost concern in any successful commercial venture, primary and mandatory attention must go to safety. Component failures that endanger personnel or property cannot be tolerated. Engineering practices concerning the design and operation of most components in this category are, of course, governed by law as reflected in the various Codes.

Even so, there are unpredictable events that can occur which lead to hazardous situations. For example, an entirely new mode of failure previously unrecognized, or, failure of a minor component which triggers a series of events leading to disaster. Pilot plant component failures of this type are of course completely accidental but have the morbid compensation that they occur only once preventing larger and possibly more numerous occurrences.

Component failures that interfere with operability receive prompt attention although the solution in a pilot plant tends to be more expedient than optimum. The economics of component reliability is harder to define. Component size, replacement cost, frequency of repair or replacement, effect on plant operation, predictability of failure and so on, are all factors that need to be taken into account in assessing the economic impact of a failure.

PILOT PLANT FAILURES: In the light of the above discussion then it should be clear that any pilot plant component failure must be carefully analysed to determine the long term consequences. The following questions must be asked,

- is the component of commercial significance ?
- has the proper material been used ?
- has the component been used properly or exposed to meaningful environments ?

Very often the answer to these questions are obvious and an immediate decision can be made with regard to the disposition of the problem. But there will also be situations which are not as readily resolved and which require the best judgement of experienced operators, materials engineers and materials scientists. It is in dealing with problems of this type that the failure reporting and analysis systems discussed in this conference can be extremely useful. The dissemination of materials performance information to all concerned with development of these plants at this time is of particular importance in view of the National goals at stake. Economic competition frequently prevents private industry from revealing pilot plant information, except where safety is concerned. Matters relating to safety however should be, and are, openly discussed. It might be argued then that in matters of national economy affecting us all, the same attitude should be taken. The only problem appears to be at a plant level where initial recognition of a problem is vital to the system. Or, conversely can we afford to investigate every failure.

PILOT PLANT EXPERIENCE: Up to the present time only two of the pilot plants have operated under self-sustaining conditions. The IGT Hygas pilot plant has been in operation since 1971, and the Consol CO_2 Acceptor pilot plant since 1972. The total operating time is of course far less than the calender years, probably around several thousand hours a year, and the time at actual operating conditions much less than that. So while it can be said that in general there have been no major safety problems in those plants, the time frame only allows us to discuss short-term effects. The long-term effects of corrosion or cumulative exposure have yet to be evaluated.

However this is not insignificant. The plants have been subjected to extraordinary cyclic conditions under high pressures of hydrogen without adverse effect so far. This improves our confidence in the present designs.

Numerous component failures have been reported for both plants, some of which involved severe mechanical or environmental effects. In almost every case there has been an available solution, or the problem does not relate to commercial operation. For example, severe sulfiding of the Consol recycle gas heaters has been a serious operating and economic problem in the pilot plant. However, the recycling of sulfide containing raw gas through such heaters is a pilot plant feature only. The information obtained is useful but may not have serious implication in a commercial design.

Failure of stainless steel by stress corrosion cracking has been reported in minor components at both plants. This is a well known problem throughout industry and in most cases can be eliminated by proper design or materials selection. Occasionally, unpredictable chloride ion contamination occurs that must be traced down and eliminated. As indicated above, however, not all the usual options are available to pilot plant designers and failures of this type can be expected.

SUMMARY: Pilot plant component failures require careful analysis to maximize the information on materials performance. The key to any successful system of analysis would seem to be at the plant where problem recognition is needed.

CO_2 ACCEPTOR PROCESS

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I represent Conoco Coal Development Company and am involved with the Consol CO_2 Acceptor Pilot Plant at Rapid City, South Dakota.

To understand the metallurgical and mechanical problems, a brief description of the process might be helpful.

The CO_2 Acceptor Process, a fluidized bed system to convert lignite or subbituminous coal to pipeline gas, is undergoing testing in a 40-ton per day, $10,000,000 pilot plant at Rapid City, South Dakota. The demonstration program is being carried out by Conoco Coal Development Company under a contract with ERDA and the American Gas Association.

A schematic diagram of the CO_2 Acceptor Process is shown in Figure 1. There are two fluidized bed reactors, a gasifier, and a regenerator which operate at a pressure of 150 psig. Lignite or subbituminous coal is fed to the bottom of the gasifier where, after rapid hydrodevolatilization, gasification of fixed carbon with steam occurs. The gasifier temperature is in the range of 1480°F to 1550°F. Heat for the gasification reactions is supplied by a circulating stream of lime-bearing material called acceptor. The acceptor, which can be either limestone or dolomite, supplies heat needed for gasification by the exothermic CO_2 acceptor reaction.

$$CaO + CO_2 \longrightarrow CaCO_3 \qquad (77°F)$$

$$\Delta H = -76{,}200 \text{ Btu/Mol.}$$

The CO_2 Acceptor reaction is reversed in the regenerator at about 1850°F where heat is supplied by burning the residual char from the gasifier with air. Ash is removed from the regenerator by elutriation and collected via an external cyclone and lockhopper system. Seals between the gasifier and regenerator are maintained by purged standlegs or solids.

Since the acceptor loses reactivity to the CO_2 acceptor reaction as it circulates between the reactors, some of the circulating inventory purposely is withdrawn from the gasifier and replaced with fresh stone makeup. The makeup is added to the acceptor which is returning to the regenerator.

The above describes the essence of the process. Necessary auxiliaries and process steps not shown include a coal preheater, pressured coal feed lockhoppers, gas cleanup and quench system, and a methanation step, wherein the CO and H_2 are converted to methane (CH_4).

To date we have had a total of 33 runs. The most recent one was a very successful 12-day run under full control at full design conditions. The run included a 10-day period making a high Btu gas--most of it above 900 Btu/cu ft. The run was terminated when a small piece of refractory lodged in a control valve.

Many of the earlier runs were aborted because of mechanical and metallurgical failures. As one can readily see, we are dealing with aggressive atmospheres, at high temperatures, along with a potential erosive condition because of the solids that are present.

A brief list of some of the hardware and metallurgical problems that cause stoppage would include:

(1) Cracking and spalling of refractories in the reactors.

(2) Lockhopper valve failures. Seal areas become scratched and permit flow of gases.

(3) Loss of internal cyclone in the gasifier due to a weld failure.

(4) Failure of solid transfer line control valves.

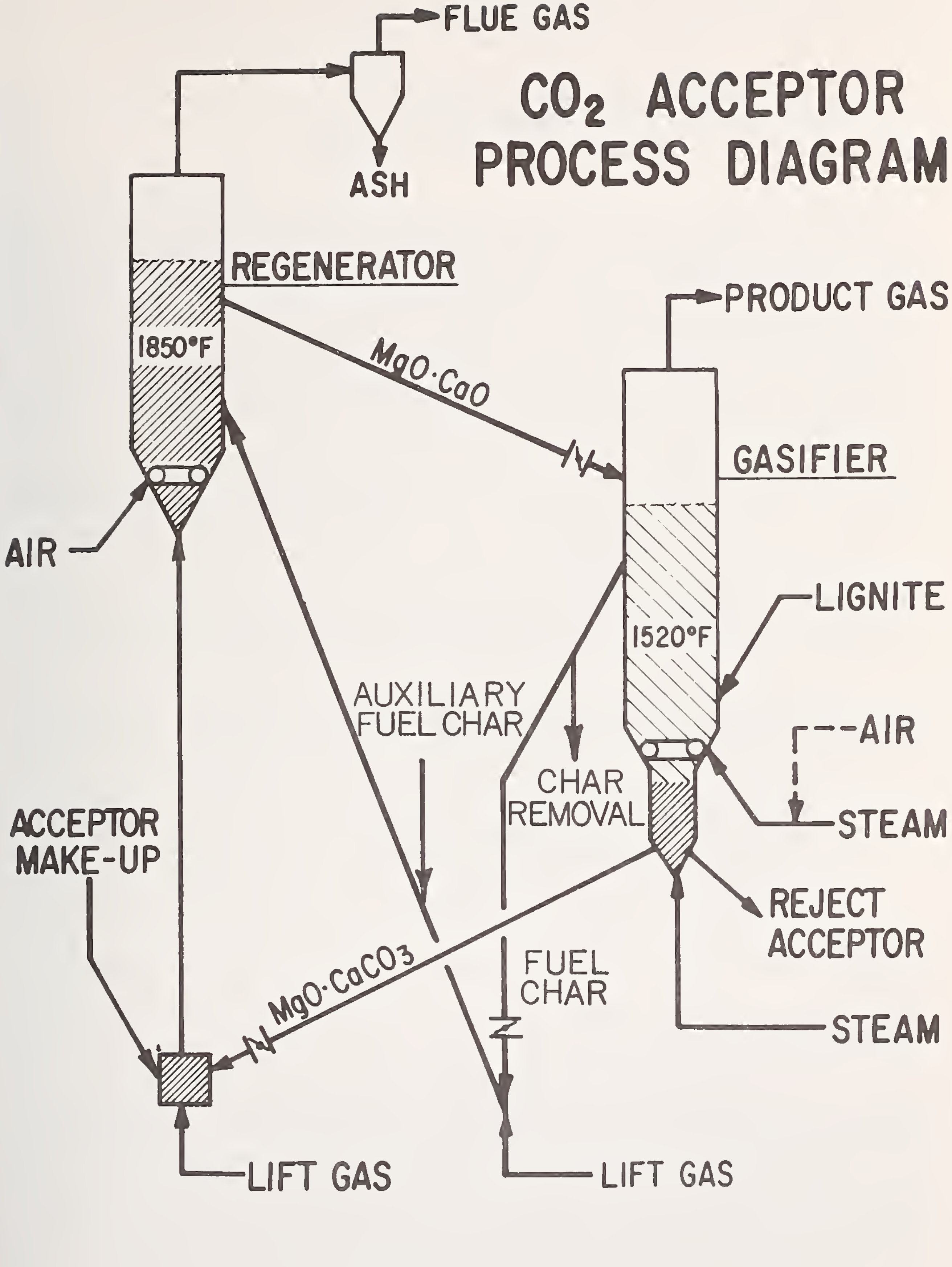
(5) Erosion of solids transfer lines, particularly in the area of expansion joints.

(6) Stress-corrosion failure of cold expansion joints.

(7) Carburization and sulfidization attacks on gasifier heater coils.

The last mentioned item was a frequent cause of run failure in earlier runs. The furnace coils are in a recycle system which is a pilot plant requirement only. Subsequently, we have changed metallurgy, reduced temperature, added a sulfur removal system, and also added an oxidant (steam) to the quench gasifier recycle stream.

FIGURE 1



CO$_2$ ACCEPTOR PROCESS DIAGRAM

CONSOL PILOT PLANT MATERIALS PROBLEMS

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Abstract: Recycle gas heaters suffered severe sulfidic corrosion of Incoloy 800 tubes prior to desulfurizing the gas stream with a ZnO system. In a sulfur free recycle gas stream 321SS and Incoloy 800 tubes encountered rapid pitting attack by metal dusting until 50% steam was introduced into the recycle gas stream. Large vertical cracks in the inner castable refractory liner of the gasifier and regenerator vessels occurred because a light weight insulation between the refractory and the vessel shell lacked strength to support the refractory layers when the system was pressurized. Erosion of the expansion joints of an inner liner of a transfer line pneumatically carrying solids and the stress corrosion cracking of the 304SS expansion bellows of the outer pipe are other problems experienced.

Key words: Erosion; metal dusting; process vessel refractory-insulation design; stress corrosion cracking; sulfidic corrosion.

The chief materials problems encountered with the operation of the Consol CO_2 Acceptor Coal Gasification Pilot Plant have been:

.sulfidic corrosion
.carburization - "metal dusting"
.refractory-insulation failures
.erosion
.stress corrosion cracking

A brief description of the essential aspects of these problems follows:

Sulfidic Corrosion

The most severe corrosion problems have occurred in Incoloy 800 recycle gas heater coils operating in the vicinity of 1500°F and higher. The principal form of sulfur corrodents have been H_2S and to a lesser degree SO_2 and COS. The normal level of H_2S is about 500 ppm with concentrations up to several times this level at some occasions such as during start-up periods. Thick layers of metallic sulfides form on tube walls within 150 hours operating time and have occasionally caused complete tube blockage. Liquid nickel sulfides have formed in higher temperature zones of heaters and have been carried downstream, solidified and agglomerated into chunks and balls in lower temperature locations.

To control this problem sulfur was eliminated in the recycle gas streams by the installation of a ZnO desulfurizer system.

Carburization - Metal Dusting

With the virtually complete removal of sulfur from the gasifier recycle gases severe pitting attack of the recycle gas heater type 321SS and Incoloy 800 tubes was experienced by the metal dusting reaction. This attack was chiefly in the 800-1200°F temperature range. During normal operation with char and steam in the gasifier, the recycle gas downstream of the desulfurizer contains 13% CO, 10% CO_2, 6% N_2, 60% H_2, and 11% CH_4. The problem was solved by adding steam to the recycle gas in an amount equal to about 50% of the whole.

Refractory - Insulation Failure

The lignite gasifier vessel and acceptor regenerator vessel were originally lined with 7 to 17 inches of J-M superex block insulation and then 3 inches of low density castable insulating refractory A. P. Green VSL-50 and finally 3 inches of a harder higher density castable A. P. Green KS4V. Large vertical cracks developed in the cast layers because the block insulation did not have sufficient compressive strength to support the castable layers when the system was pressurized. The cracks permitted channeling and gas flow within the insulation blocks thereby causing excessive heat loss to the vessel shells. Corrective measures were the replacement of the diatomaceous silica block insulation with higher crushing strength castable and vapor stops of high density hard-face castable were installed at several levels to prevent bottom-to-top circulation paths within the insulation thickness.

Erosion

Piping which carries solids at high temperatures consists of an outer steel pipe, an inner alloy liner with the annular space between the two filled with vacuum packed ceramic fiber insulation and diatomaceous silica insulation. Thermal expansion of the inner tube is accommodated by an enlarged end section bell-and-spigot type slip joint. Severe erosion of the inner tube occurs if there is misalignment of the tube at flanged joints and at the slip joints because of flow turbulence at these locations. Metal spray and plasma arc spray of chromium carbide and nickel aluminide coatings of the slip joint areas provided marginal improvement. Hardface weld overlay with Stellite 12 of the slip joints and close attention to alignment have greatly reduced erosion problems. A new ceramic lined slip joint was recently installed and work is in progress on the design and fabrication of a new solids line completely lined with formed ceramic shapes. The velocity in this line is now maintained at about 50 ft/sec, previously the velocity ranged up to 100 ft/sec.

Stress Corrosion Cracking

The expansion joints in the outer carbon steel solids handling pipe were 304SS bellows type design. Several of the stainless steel bellows have failed through chloride stress corrosion cracking. The original 304SS expansion joints are being replaced with solution annealed Incoloy 825 expansion bellows which should minimize this problem.

These have been the chief problems experienced in the pilot plant operation. In a commercial size unit heat loss considerations would not require recycle gas heaters or such exotic insulation designs. This would minimize many of the problems briefly described in these introductory remarks.

THE SYNTHANE COAL TO GAS PILOT PLANT

Robert Lewis
Synthane Coal to Gas Pilot Plant
Pittsburgh, Pennsylvania 15213

The Synthane plant is the only pilot plant in the United States that is 100% government funded. Furthermore, the Synthane process is a government developed process. The Government, however, does not operate the plant. It is operated by the Lummus Company under contract to the Government. There is one other unique feature of the Synthane plant--in the early days, it was the only plant that had full time government personnel.

The plant has been in a debugging, startup type operation for approximately ten to twelve months. We have started up and operated all utilities and the coal handling and grinding system. The coal feeding system, the char handling system, and the filtration system have been operated. Coal has been fed to the gasifier, fluid beds have been established and maintained, and some coal feed rates have been measured. Various useful data have been obtained. Coal has been successfully removed from the gasifier through the char handling system. Coal has not yet been ignited and we therefore have not tested the gas cleanup system under process conditions.

We have had various kinds of failures during the past twelve months, despite the fact that we have yet to produce any process gas. The failures can be categorized into two general types--those that are primarily related to the coal gasification process, and those that are not specifically related to coal gasification and involve materials or equipment common to other industrial or manufacturing operations.

Failures in the first category have been limited to only a part of the plant since there are many portions of the plant that have not yet seen process conditions. We had a major problem with a leak at the welded gaskets at the gasifier main body flange. We had an internal stainless steel head deform under excessive pressure in the gasifier itself. This head served as a diaphragm to give us, in effect, two vessels in one. We have had a number of erosion problems with slurry pumps, mixing venturis, spray nozzles, agitators, piping, etc., in coal slurry handling facilities and we have had some minor instrument problems, although most of these belong in the second category.

Failures of conventional industrial equipment not specifically related to the coal gasification process have been numerous. There were 29 types of instrument failures involving perhaps 50 to 100 individual instruments. Most of the other problems relate to rotating equipment that does not handle coal or gasification products, and consist of mechanical seal failures, cracked and broken pump casings and blower casings. There were difficulties with our large four-stage carbon dioxide compressors, such as valve leakage and excessive wear on pistons and piston rings. We have had problems with overloading of motors on various pieces of equipment. We had a broken chain on our raw coal bucket elevator and on our char conveyor. There were numerous problems with explosion doors opening prematurely on a dry coal dust collector. We had packing failures on the rotating leaf vacuum filters. There was a major failure in the low pressure gas holder revolving around improper functioning of the piston and rupturing of the diaphragm.

I do not think that the second category failures should be glossed over because they occur with standard industrial items. It is my unsubstantiated and very subjective opinion that the number and variety of this type of failure are greater than they used to be when process units were started up. This relates to problems of quality of material and equipment, and can be attributed in part to misapplications, problems with procurement, rigid adherence to the principle of low bidder, poor vendor quality control, and lack of appropriate guidelines for field storage and handling during construction and startup.

Gasification pilot plant failures are difficult to anticipate. The best ways to avoid or alleviate most of them were not available at the time the pilot plant was engineered and constructed. I refer specifically to the need for improved communications, centralization of information, greater distribution of information and more interplay between those engaged in projects where these problems tend to occur. Many people are trying hard to improve the situation and I am happy to see that there have been some positive steps taken along those lines. There are several things that could be done. One is to establish a priority system. When we need 316L stainless steel tubing, for example, we have to compete on the open market. We have no special priorities because we are government funded.

I would like to add that these last comments about improved communications are not intended to be critical. I believe meetings and symposia such as this are an effort to cope with this problem and should be encouraged. I also know that there have been difficulties in attempting to implement some of these concepts. Further efforts along these lines should pay dividends, particularly in view of the large expenditures being planned for the near future.

PANEL DISCUSSION

Chairman: Henry E. Frankel, ERDA, Fossil Energy

Panel Members: Fred B. Hamel, Gulf Science and Technology Company
Larry G. Samuels, SRC Pilot Plant
Adrian J. Mac Nab, C. F. Braun and Company
Carl A. Schulz, Conoco Coal Development Company
Jene F. Leterle, Continental Oil Company
Robert Lewis, ERDA, Synthane Pilot Plant

F. B. Hamel: I was listening to some of the talks yesterday about reliability and how reliability could be designed into plants. The oil industry is very concerned with reliability. Several years ago I was involved with a decision to go to an integrated refining installation. We knew we could obtain 95% reliability or availability in a given unit without too much trouble. Our management people wanted the plant designed to be able to run for four years with a one-month shutdown. This looks like 98% availability. But you fast come to the conclusion that from a design, construction and operational basis, there is only one thing to be done--do everything possible to get 100% reliability during the operating interval. In order to have 100% reliability, all components in a system must be working right. Obviously, some of these components will malfunction at times. Most of my work has been involved in correcting these malfunctions. A malfunction must be corrected as quickly as possible on an emergency basis. There is no other way of getting reasonably good availability.

When we try a new process, we like to have a turn around--that is, a period when the equipment is shut down and examined. We try to run at least six months before turn around. If everything is going well, we will stretch it out to a year or maybe two. We don't go for a four-year turn around with an unfamiliar process. We want 100% reliability for the operating interval before a turn around and that becomes the objective of our engineering work. Basically, this is the kind of philosophy we use.

L. G. Samuels: I assumed that there were going to be some manufacturers and vendors of equipment here, and from looking at the registration list, I see only three or four. I believe we have to have more participation from these groups if we are ever going to solve the energy crisis. We have had some outstanding examples of research papers presented this week, but they are not of much immediate value to someone like myself who faces immediate problems everyday in the plant.

To give you a little insight into what I am talking about I would like to describe part of a typical two-week period at the SRC plant. We discovered that there was a leak in one of the wash solvent overhead condensors. We quickly determined that the thin 1 inch diameter, 8 foot long carbon steel tubes on the air cooled condensor were leaking at the inlet tube sheet, and further inspection disclosed that all 76 tubes were very thin at the inlet tube sheet and that they would have to be replaced. It was Friday afternoon. We have 64 operators, 16 on each of four shifts, we have about 40 maintenance personnel plus perhaps 20 or 25 technical people. When the plant is not running, we feel the taxpayers are not getting their money's worth. So we quickly called Tulsa where the exchanger had been made and we found out that we could get some thin carbon steel tubes made up on Saturday and air freight them from Tulsa to Tacoma. We inquired about 304 and 316 stainless steel, but it would take 4 to 6 weeks or longer for the stainless steel tubes, so we ordered carbon steel tubes and began arranging for a crane to remove the exchanger for retubing. About Tuesday of the following week, we had it down off the roof, retubed, and back up on the roof. But just as that was finished, we discovered that the apparatus that drys the mineral residue that is filtered out of the coal was coked up. It takes about three days of jackhammer work to decoke this 36 inch diameter, 40 ft. long vessel. Further, we had 1100 feet of chain in the dryer that we added to rub on the sides to forestall this coking. These had broken loose and become matted. So three days of jackhammer work and 1100 feet of chain later, we thought we were going to run, but just as that was being completed, the wash solvent column sprung a leak. This is a 30 inch diameter carbon steel vessel, 3/8 inch thick, which operates at 10 to 15 psi and 600°F. It is rated at 75 psi and 750°F because we did not know the operating conditions when the plant was built. The plant inspector examined the column with an ultrasonic tester and decided that it was going to take many large patches to repair it. Again it was Friday--the plates were rolled in anticipation of applying several half circle patches around this column on Saturday and Sunday. On further investigation we found there were large craters, and by that I mean craters up to 2" in diameter. We also discovered general corrosion in at least one square foot of every tray in this column. On Saturday we decided to remove the trays and overlay the column from the inside, but by Monday, that became impractical because, as we started to remove the trays, we found that all the tray supports and downcomer supports had corroded off. On Tuesday, it was decided that we were going to replace the top 18 feet of this column. We initially decided to replace the column with 316 stainless steel, but finally decided on 316L because the column was to be welded to part of the original carbon steel column. Fortunately, the material was available from a local fabricator. The old section was cut out and the new section was fabricated, installed, pressure tested, inspected, recoated and back in operation in four days.

The basis for the 316L selection was an article in Metals Handbook concerning naphthenic acid corrosion. Now our lab had not been able to verify that we have naphthenic acid corrosion, but it looks like a perfect textbook example. The condensor tubes that I first mentioned should have been 316 stainless steel also, but there wasn't any 316 available. So we used carbon steel because we feel that it is more important to keep the pilot plant running then to determine which materials give best performance. We must strive to get as much information as possible from the pilot plant with what is available, not waiting for what we can get next month or next year. Direct contacts with some of the people in this room would be a big start in the right direction.

H. E. Frankel: You mentioned that you saw very few vendors present at this meeting. This, indeed, highlights a major problem facing the whole coal conversion industry. The fact is, there really is not a commercial coal conversion industry. It is not worthwhile for vendors to make specialized pieces of equipment. We are just going to have to bear with this situation for many, many years until an industry does come on stream.

A. J. Mac Nab: Of the failures about which we have heard so far, there are only a few that I feel are directly significant in the development of commercial gasification processes. Very often a material is used in a system simply because it is available at the time it is needed. Sometimes it is deliberately put in, but the process situation changes. The operating conditions are not as the designer was told. Minor component failures are going to occur, and they are going to occur on commercial systems. That type of failure to me is not terribly important in a pilot plant as long as we understand why it happened and it can be corrected in a commercial design.

A problem that needs attention involves the failure reporting systems and failure analysis systems. The weakest link in the chain is the reporting at plant level. That is not to say that we don't have competent people at the plant level, we certainly do. But, very often they are not metallurgists or they are not familiar with the full gamut of metallurgical problems and therefore could often overlook a significant piece of information. If we are leaving the reporting of the failure to the first observer, he can classify it as not important or important as it strikes him at that time. For instance, an internal failure in a reactor could be dismissed as a routine mechanical problem such as a poor weld, whereas in fact, a component may have a fatigue life 1/3 below the expected level for that material under unexposed conditions. This is an important piece of information but it is neglected.

C. A. Schulz: In line with Adrian's remarks, gas recycle heater failures are not really significant except for historical interest because gas recycle heaters will not be needed in the commercial plant.

H. E. Frankel: So far in this session, we have just discussed our failures. There have been some notable successes. Perhaps Carl can address himself to some of the pieces of equipment and components that have been successful. These are perhaps more important than the failures.

C. A. Schulz: We have had a lot of successes. We have a solids control butterfly type valve that has operated successfully for about 5000 hours at 1850°F with dolomite--a very hard solid--passing through it. Our pressure vessels themselves have never been attacked despite the refractory failures. We have a cyclone in the gasifier that performed satisfactorily for 5000 hours in a corrosive and erosive environment. It even fell to the bottom of the gasifier once because of a failure in a weld in the discharge pipe, but it survived. There are many good things that can be proven in pilot plants, and perhaps we could learn from the surviving components as well as from the failures.

R. Lewis: It appears to me that we are being let down by many of the vendors who are supplying shoddy equipment and lousy workmanship, and failing to meet even promised deliveries. I do not like to take the position that American industry is not what it used to be, but we have a pretty strong feeling that when you buy something, you do not get what you thought you were going to get. We have had a lot of trouble with slurry pumps and slurry pipelines. When you start asking questions, however, you find that there are solutions to many of these problems. We found that Black Mesa pumps a coal slurry about 300 miles with positive displacement pumps. They do not have any problems. Even in industry, there is not the kind of feedback there should be.

L. G. Samuels: One of our initial problems was pumping slurries with positive displacement pumps. The pump was designed for 2400 pounds of pressure at about 200 or 300°F, but we were only operating at about 1500 pounds. These spring loaded hemispherical valves would erode in a matter of hours. These valves cost about $200 apiece and we were going through about 12 per set every other day. We kept going back to the vendor who gave us good service. But everything he sent was worse than what we had. Finally, after we had exhausted every means available to us that the vendor could suggest, we called IGT (Institute of Gas Technology, Chicago). They suggested putting a 2 3/4 inch diameter chrome ball in the valves. In less than a day, we had these $35 balls that you could buy off the shelf installed and those pumps operate for months now without maintenance. There was interchange between government contractors.

R. Lewis: You are also backing up my point about some of the vendor problems.

C. A. Schulz: Bob, you point out something. You are three miles from me. We were the developers of the world's first pipeline. All the technology of Black Mesa came from Library, PA. You should have consulted with us.

R. Lewis: Carl, we talked to you, but the problem was that the designers did not talk to you. The slurry system was designed four or five years ago by a big engineering company who said they knew how to design a coal handling system. Our slurry pumps are recommended by everybody you talk to--they are standard slurry pumps.

C. A. Schulz: Larry, that is a mud pump that your vendor recommended. I am sorry that you had so much trouble with your pumps. We learned about the ball valve some ten years ago. Specifying a pumping system is a complex problem. You have to know something about the hydraulics of the system, the slurry concentration, the size consists, the carrier, etc. I do not think the vendor has that process know-how.

R. Lewis: Sometimes an apparently insignificant and unnoticed design change, usually in the interest of saving a few cents, can wreak havoc with a system. As an example, we had a $100,000 four-stage reciprocating conventional compressor to pump carbon dioxide. We suffered repeated failures with the unloaders. It took four weeks to determine that the problem was that the gasket material had been replaced with an inferior material. Problems of this type are very hard to diagnose.

C. A. Schulz: Your point about equipment manufacturers and fabricators is a very valid one. I think that integrity has gone out of the fabricating business. We recently built an addition to our CO_2 Acceptor Plant. We decided to spend the extra money to sit down with the vendor to make sure he really understood the specifications. We also spent a lot of time and money visiting the plant to make sure that everything functioned properly. It was worthwhile to spend the extra money and manpower because this pilot plant is the only one in many years that worked properly from the beginning.

S. S. Canja, ERDA: I am ERDA's Systems Reliability Engineer. I work on the staff of Dr. White, the Assistant Administrator for Fossil Energy. We are in a very infantile stage in reliability quality assurance in fossil energy programs. Dr. Frankel has begun the work in the area of materials and failures. He and I are to work together in developing what we call a performance assurance system. When we talk about performance assurance, we are not in any way trying to develop processes. We are trying to reflect on all of the activity necessary to assure ourselves that we are going to have a working system at the lowest possible cost. The primary elements of a performance assurance system as we see it are a reliability program and a maintainability program. We feel that by beginning performance assurance procedures in the process development unit (PDU) stage, maybe we can predict some of the problems that may arise in the pilot plant and in the demonstration plant. One of the problem areas in the coal conversion industry is a lack of standardization. Everybody today seems to design a pilot plant independently of everyone else. It would seem to be appropriate to provide designers with information as to what components operate with a high degree of reliability at a good cost, etc. For instance, wouldn't it be reasonable to have several plants using the same kind of pulverizers? When there are problems, repair parts would be more plentiful. Problems could be predicted from the experience of others. So we feel that standardization is a very important element in our total program.

Life cycle cost--we are looking at this from the standpoint of what it costs to operate equipment, not how many tons of coal go in and how many BTU's of gas come out. This is not a performance assurance problem; it is a processor's problem. We are also concerned with the service life prediction--how long the various elements of the plant will continue to function properly. That is a very quick overview of the total performance assurance system that we would like to formalize and enable the ERDA program managers and project managers in fossil energy to apply to their contracts.

In order to get a performance assurance program underway, we talked to our program/project managers and we were told by many of them that reliability was already written into our contracts. In order to see if this was really true, we screened 280 contracts. We identified 45 contracts that were prime candidates for performance assurance. These contracts were for late PDU plants, pilot plants, and demonstration plants. Only one contract had an appreciable number of elements concerning performance assurance. Seven referred in some way to reliability or quality assurance and some stated that there will be a reliable process. The remaining 37 contracts did not even have the word reliability in any part of the work statement. Most of ERDA's request for proposals had no provisions for reliability, quality assurance, availability, etc. ERDA is now beginning to realize that in order to get quality, reliability and performance assurance into its programs, it is going to have to pay for them. Finally, there are not in existence at this time any formal procedures, specifications, or any other program elements which will act as a guide for performance assurance within the fossil energy milieu. Performance assurance has proven itself in the Department of Defense and NASA. I have been directed to prepare some performance assurance provisions to be included in future contracts. We are preparing interim specifications and procedures to formalize the program. We must develop our own procedures because often those developed by other agencies are not suitable. Some of the discussion we heard this morning indicates that we are attacking our problems on a piecemeal basis. We must apply some techniques of failure prediction in the early stages of design.

Another subject that I would like to discuss is the possibility of fossil energy programs joining what is known as "GIDEP". GIDEP is the Government-Industry Data Exchange Program. It was originally started by the Department of Defense (Navy) in 1959. The ERDA fossil energy program has been invited to join at no cost. If we join GIDEP, the program should complement the data collecting program at the National

Bureau of Standards. Right now, GIDEP is primarily electronics oriented, but there is much information on materials and components that is common to fossil energy programs. GIDEP is willing to create whatever categories of data that are needed and Dr. Smith is currently working on a program to identify these categories of information and how we would put them into a data retrieval system like this. The whole point of GIDEP is the possibility of our using it instead of creating a separate system that would cost us many hundreds of thousands of dollars when this one is free. Two of the data banks in GIDEP are the failure rate and failure experience data banks. You can join only one bank if you wish, but you still have, as a user, access to any of the other data banks. I think that the most compelling reason for joining GIDEP is cost avoidance.

A. J. Mac Nab: The overall problem seems to be that we are trying to upgrade the quality of standard industry. Things like weld-procedure qualification, welder certification, etc., are standard procedures and should not present a problem in gasification. In other words, there is no reason at this time to demand any additional quality over what we already have in other industries. What must be decided is how far should one go in gasification in improving quality assurance and quality control at every level of operation from design through the vendors and in the reporting of information.

F. B. Hamel: Frankly, Mr. Canja, your remarks frighten me somewhat. If we implement the programs that you are talking about, I think that we would double the cost of the R & D programs. The documentation that implements a performance assurance system is gigantic. There is one major technical problem that we have in common with the space people; that is, building one-of-a-kind of something. Reliability data cannot be obtained on one-of-a-kind equipment. It is high time we recognized the prime purpose of pilot plants and demonstration units. It is not to develop hardware, it is to prove that we have a process that will work. We make changes in our Tacoma plant every day. We feel that the implementation of a performance assurance program to improve the operability of a demonstration unit which was not intended to run all the time anyway would be a waste of time and money. Some of our equipment was bought with the recognition that the plant in Tacoma was not going to run forever. It has a finite life. We have to keep this kind of thing in mind. Of the many failure analyses discussed at this meeting, very few will prove to be of any significance in the development of equipment for commercial plants.

I agree that we need better technical information retrieval systems in our industry. I am heavily involved with technical information retrieval in my company. We all are fighting this thing.

To implement a performance program, there is the problem of working with nationally accepted standards (most of which are accepted by OSHA) and then there are government regulations that tell us we have to buy from the lowest bidder. As soon as we write a special standard we raise the price. But I defy you to show me, on something you have never built before, how you can show any payoff in doing anything but the lowest cost job that you can do.

O. Decker, Mechanical Technology, Inc.: I did not think the purpose of pilot plants was just to prove the process.

F. B. Hamel: I was oversimplifying an issue.

E. B. Bell, TRW: Once an extensive data base is established, equipment and plant availabilities can be calculated. This data base can be developed from pilot plants and demonstration plants, and in some cases from PDU's. When we have this data base, we can apply computer techniques to put redundance in a system to improve availability and reliability. It is imperative that this data base be established as soon as possible.

F. B. Hamel: My experience in the oil refinery business indicates that most failures are one-of-a-kind. It is hard to establish a meaningful data base from these one-of-a-kind failures.

E. B. Bell: If in fact over a period of time you are able to standardize some of the equipment in your plants, then maybe you wouldn't see as many one-of-a-kind failures.

F. B. Hamel: Standardization is a good idea, but very difficult to implement. For instance, several years ago we tried to standardize heat exchanger sizes. It did not work out because if you needed an exchanger just slightly larger than one standard size, you would not be willing to pay for the next larger standard size which would probably be much larger than needed.

R. Lewis: The statement was made earlier, and I generally agree with it, that pilot plants are basically built to demonstrate that process chemistry is valid and is capable of being scaled up to a commercial size installation. The hardware that most of the pilot plants have is basically commercial, off-the-shelf hardware. Now when it comes to scale-up to demonstration and full scale plants, it is an altogether different ball game, and here is where you get into questions of standardization. We do not do many things in our pilot plants that you are going to do on a commercial scale. We are grinding 5 tons of coal a day in a little hammer mill, a standard conventional piece of equipment. Now, no commercial gasification plant is going to have equipment like that. I know there are people who are looking at ways of handling 20,000 tons of coal, so the reliability factor on my little 5 ton a day coal mill is irrelevant. All I want to do is run the equipment long enough to get some coal in the gasifier and gasify it. I do not care what I have to do to grind that coal. The situation is similar with heat exchangers, cooling towers, etc. Our problem is that we have been working now for close to a year and we have not made any gas yet. The reason we have not is because we cannot get past this commercial hardware we bought and get it to work long enough to put some coal in the gasifier.

E. Passaglia, National Bureau of Standards: I think Bob Lewis hit on the key point with respect to materials reliability in pilot plants. Clearly a pilot plant is designed to study the process because you want to know if you are going to make money or not. However, when the materials reliability question prevents you from getting your operating data or prevents you from getting it on time, then I think you are in trouble. I do not know if we are in trouble with respect to these coal gasification plants. Are we having more troubles with coal gasification plants, particularly with the standard equipment, than one would normally have in a pilot plant in the oil industry? Bob, you seem to feel that you do. Others of you may not. If we are, why, and what can we do about it? Mr. Canja's program might help.

There is a related comment that I am prompted to make contrasting the results of this particular meeting with the meeting that was held in Columbus just about two years ago--the workshop on coal gasification. At that meeting, materials people were being asked to consider what kind of materials problems were likely to arise in coal gasification systems. People hypothesized, on the bases of their experience and on the kinds of atmospheres that the metals and ceramics were supposed to be experiencing in these plants, that the main problems would be erosion and corrosion. Based on results we have to date, those predictions are very largely borne out as would have been expected. From what people are saying at this meeting, the problems seem now to be concentrated in areas where the materials people thought the problems were solved--such as coal buckets and slurry pumps. Are these problems unique to the coal gasification pilot plants? Are we experiencing the same kind of thing we would experience in plants in other industries?

A. J. Mac Nab: There have been an unusual number of failures in standard equipment on the pilot systems from what we have seen so far, and the operating times are short. But this is really tied in, to some extent, with the fact that they are pilot plants. I believe it has to do with the economic incentive, the monetary value of pilot plants. There is not much money invested in them, the budgets are usually fairly tight, and they spring out of process development groups without a big engineering organization backing up the design information that is transferred between them and contractors. You get what you pay for. Contractors take a lot of blame for plant quality, whereas in fact, a lot of shortcomings are due to poor specifications. A customer frequently does not say what he wants. It is the nature of pilot plant construction to have something less than the big guns working on the job because of the economic incentive.

C. A. Schulz: I think that the petroleum industry is experiencing the same problem as we are with ordinary standard equipment. Our pilot plants are different from theirs in that they are much larger. Our plants are unique mostly because we are handling solids.

S. S. Canja: It has been alleged that performance assurance adds at least 50% to the cost of a project. Performance assurance cost the U.S. Government about 14% of the total cost of putting a man on the moon. Now, remember that we were really concerned about his health and safety. We calculate the cost of the desired performance assurance in the coal conversion industry to be 3 to 5%.

I am distressed by the fact that people tell me pilot plants are to prove out a process. What was the purpose of the process development unit? Well, to prove out a process. What was the purpose of the demonstration plant? Well, to prove out the process on a bigger scale. The question is, when do you start looking at components? When do you start designing them with scale-up in mind? One of the problems in the electrical utilities industry is scale-up. There are more problems on a 600 megawatt and above generating plant than anticipated because of the scale-up problem. But I think if you start scaling up from PDU to pilot plant to demonstration plant, then the next step to a commercial plant will be easier.

R. Lewis: I agree with Carl in regard to the size of our pilot plants vis-a-vis the petroleum industry. These pilot plants are really not pilot plants at all. They are misnamed. Somebody arbitrarily said a PDU is up to 10 tons a day, and a pilot plant is up to about 200 tons a day. We are talking about coal input now, and a demonstration plant is a single train of a commercial-sized unit. Now, we are not really going through all of these steps. Most of the large pilot plants have 70 to 100 tons a day capacity. These plants are scale-ups from the PDU's on the order of 200 times. Probably the next size which is the demonstration unit is a scale-up of 200 times more. Our pilot plants are pretty substantial operations and very little of our equipment is designed specifically for use in a coal gasification plant. Most of the equipment consists of things like standard heat exchangers, standard piping, etc., that are all bought commercially. We cannot prove the feasibility of the process in the pilot plants because of continuing problems with this standard, off-the-shelf equipment.

A. J. Mac Nab: Standardization has come up a couple of times and, as Bob has pointed out, both pilot plants and commercial plants are built using standardized piping, valves, pumps, etc. Standardization often leads us into trouble. In order to pick a standard size or standard piece of equipment, we oversize or undersize and create problems. This can be as much of a pain as having nonstandardized equipment.

C. A. Schulz: Specification standards have progressed from the overly elaborate bookshelf size volumes that were formerly used in the petroleum industry to the almost nonexistent ones currently in use for the coal conversion systems. I do not propose that we go back to the elaborate procedures once used, but a definite upgrading of current specification standards is needed.

O. Decker: Why, in a pilot plant, can't you both demonstrate the process and at the same time run parallel programs to develop critical components so that by the time you get to a 2500 ton a day demonstration plant, you will have equipment that gives the kind of service you need?

R. Lewis: We had this in mind when we designed our compressor. One of the requirements was that it must be capable of being scaled up.

F. B. Hamel: That is terrific. But we have seen problems in just doubling the size of a machine.

O. Decker: I think the Synthane Plant is looking at this problem in just the right way.

F. B. Hamel: If the process does not work, the equipment is not needed, so my point is that the process is governing.

O. Decker: Government contractors possibly have an advantage that commercial companies do not enjoy. That is, they have more flexibility. They can have programs to develop equipment while studying a process. I am suggesting that ERDA should consider equipment development when issuing contracts.

H. E. Frankel: You have to remember that every single pilot plant was designed, and indeed built, long before such things as performance assurance or reliability were considered. So let's not fault the pilot plant for trying to prove a process. What we are trying to do is to get as much information as possible from an existing pilot plant. The demonstration plants that are at least 6 or 7 years away have time for the instituting of this philosophy. I happen to be in agreement with the performance assurance philosophy as you well know. We are all taking pot shots at the pilot plant. I think they have done a good job. What we are trying to do is make them aware that, while they are trying to prove a process, they are also proving out materials, good, bad, or indifferent.

R. Lewis: Materials yes, Henry, but not so much equipment.

H. E. Frankel: That is true.

L. G. Samuels: Our biggest problem with standardization is that we are forced to purchase equipment from the lowest bidder. Similar equipment from two different manufacturers may do the same job, but the parts, fittings, etc., probably will not be interchangeable forcing us to stock items for each. So, although standardization is nice, our necessity of dealing with the low bidder does not allow us to take advantage of it.

Another problem with the low bidder is that you may not be able to buy equipment you are familiar with and that you know works properly. The low bidder may say his equipment works, but our experience indicates that that is not always true.

O. Decker: We must not forget the fact that equipment will be needed for demonstration plants that is not available today.

R. Lewis: Adrian Mac Nab's Company, C. F. Braun, is very deeply involved with that problem. They are looking at various systems, schemes, equipment, etc., which could be used on very large scale operations.

E. B. Bell: In terms of equipment operating times, how do you schedule maintenance in a plant if you do not have a pretty decent handle as to the life or operating time of the equipment? And if you keep good records of operating times on equipment and these records indicate that the operating times are inadequate, I think this would be a very strong case for not buying from the same vendor or not buying from the low bidder. Without this information, you do not have the handle to apply leverage to change contractors or to get a more expensive piece of equipment if the cheaper equipment does not perform properly.

R. Lewis: That is awfully hard to prove.

C. A. Schulz: They hang you on minor changes and variables. You have a pump that does not work, so you go back to the vendor. It turns out that you said you were going to use a 30% concentration but, in fact, you ran a 35% concentration through the pump. The change in concentration may have nothing to do with the failure, but the vendor will claim you are using the pump out of specifications.

E. B. Bell: Are you saying that you are operating out of specs?

R. Lewis: We are not building a million units all alike on an assembly line. These are research and development installations. If 5 years ago we said we were probably going to run with a 30% concentration, and now we find out that the process works a little better with 35%, that is hardly operating out of specs.

E. B. Bell: I still do not see how you can approach a good maintenance program without a good knowledge of equipment operating times.

R. Lewis: We do not do any maintenance, we are doing repairs all the time.

F. P. Williamson, Panhandle Eastern Pipe Line Company: We want to build a coal gasification plant in Wyoming and it is going to cost us about a billion dollars. We are looking at the Lurgi technology and I would like to have the people here give me some advice. How can I insure that, after my contractor has done the detailed engineering and supposedly guaranteed that things will operate properly, when I push the button six years from now, the project is going to work. I cannot take a year or two to debug it like we do on the pilot plants.

R. Lewis: You ask that man to give you a half a floor in his engineering building and you put about 20 or 30 people in there and you watch everything that he does.

F. P. Williamson: In other words, you check his engineering effort?

R. Lewis: You look over his shoulder at everything he does. That is the only way you are going to be sure that the job is done right.

C. A. Schulz: Do not assign the young, green engineer to oversee the project. You need someone with experience in there.

A. J. Mac Nab: Every customer comes in asking for the moon, and when he finds out what it costs, he backs off rapidly.

F. P. Williamson: Well, that is the problem. You wrestle with how much money you spend before you push the button versus how much money you spend after you push the button.

R. Lewis: There is a calculated risk involved. How much risk are you willing to take and how much are you willing to pay to minimize it?

F. P. Williamson: These are all good points. I agree that you need to put the effort in to monitor your engineering contractor because he has people working on the project who are really not familiar with it.

One other point, we need to get mechanical engineers involved in evaluating equipment bids and equipment specifications to make sure we are getting the right piece of hardware to do the job.

L. G. Samuels: I am a mechanical engineer. We are sitting here batting back and forth the purpose of pilot plants. Every morning at the 9:00 a.m. meeting at our plant, there is a big argument about what the purpose of the pilot plant should be--to prove the process or prove machinery. Someone asked if we are operating out of specs? Yes, we are. The SRC plant was designed for a 3 to 1 solvent to coal ratio, but we proved that the process will work with 1 1/2 to 1. That cuts the size of the next plant way down. We built the plant with two dissolvers with four foot thick walls. These are very expensive vessels. Even for the pilot plant, they cost about $200,000 apiece. Now we are operating with one half of one, which means that we have saved 3/4 of the cost of the dissolvers. We have a preheater to heat the slurry to about 800°F at 1000 lbs pressure. Right now we are operating the preheater with half the coils insulated, which cuts down the cost of the unit. We are doing more than proving a process--we are cutting down the cost of plants and proving out machinery.

Somebody said that private companies like Gulf cannot run research and development programs like the Government, but we are trying to develop concurrently other equipment in the plant. We are trying to do basic research right in our plant to develop a different kind of filter with no moving parts.

Since last September, we have been under a mandate to produce 3,000 tons of SRC. Some of us thought we could not do it, but our management said we were going to do it and we are half way there. We are running our pilot plant like a production plant.

D. Livaccari, Coalcon: I wonder how much money is lost in pilot plant down time that could possibly have been saved by a simple alert system among pilot plants. We have heard some discussion about the poor quality of American equipment. The function of quality assurance is to put quality in the equipment that you buy.

R. Lewis: I do not quite see how you can put quality in the equipment you buy by quality assurance when you are forced to buy the cheapest thing on the market.

D. Livaccari: I disagree that you are forced to buy. You are forced to evaluate costs, but you are also forced to evaluate a vendor's expertise, experience, engineering, etc.

R. Lewis: No, sir. If you are buying a conventional item, you must take the low bid.

R. Perkins, Lockheed, Palo Alto: Another change is taking place in American industry that I have not heard mentioned here. We in the aircraft industry have become painfully aware of this change. Years ago, you could get either a materials producer or an equipment producer to do anything if you paid him for it. But today, chances are he will say no. There is a danger in assuming you can get an improved quality product or an improved material by simply paying more for it. We are being forced in many cases to even go out of this country to buy materials because the producers of materials in this country say there is not enough profit involved. A manufacturer producing pumps by the thousands will not be willing to change his basic production procedures.

A. J. Mac Nab: Very good point, Roger. We are buying a large amount of steel these days from Japan. One of the things that impresses us about the Japanese is that they implement their research. You cannot ignore that, to say nothing about their cost and delivery times. Temper embrittlement, for example, is much talked about and worked on in this country, but the producers do not do much about it.

F. B. Hamel: You must remember that you get what you pay for. If you put a much greater technical effort into any of these plans, there will be a major return in quality. The big problem is to justify the higher costs of quality to management.

S. S. Canja: I do not really feel that I have to defend performance assurance because, as I said, DoD and NASA have been using this scientific technical approach to develop good quality material for many, many years. I am delighted to have had the kind of controversial discussion that we had today. We do not want to sell the whole program to pilot plants, but if we can start doing little things day by day in performance assurance, I think that whenever we built commercial plants, we will have improved reliability. So we ask everyone to keep an open mind--do not close your mind to performance assurance.

H. E. Frankel: I am really gratified by this morning's discussion. We have heard many problems associated with pilot plants. It also has to be recognized, and I think it has been brought out strongly, that the basic and prime purpose of the pilot plant is not to prove materials or components, but processes. But, hopefully by meetings such as this, what materials and component information that has been developed can be gathered and used in the design of demonstration plants. I think this meeting has been eminently successful and it is one of the most productive meetings I have attended in many years.

MECHANICAL FAILURES PREVENTION GROUP

24th MEETING

Prevention of Failures In Coal Conversion Systems

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Handbooks—Recommended codes of engineering and industrial practice (including safety codes) developed in cooperation with interested industries, professional organizations, and regulatory bodies.

Special Publications—Include proceedings of conferences sponsored by NBS, NBS annual reports, and other special publications appropriate to this grouping such as wall charts, pocket cards, and bibliographies.

Applied Mathematics Series—Mathematical tables, manuals, and studies of special interest to physicists, engineers, chemists, biologists, mathematicians, computer programmers, and others engaged in scientific and technical work.

National Standard Reference Data Series—Provides quantitative data on the physical and chemical properties of materials, compiled from the world's literature and critically evaluated. Developed under a world-wide program coordinated by NBS. Program under authority of National Standard Data Act (Public Law 90-396).

NOTE: At present the principal publication outlet for these data is the Journal of Physical and Chemical Reference Data (JPCRD) published quarterly for NBS by the American Chemical Society (ACS) and the American Institute of Physics (AIP). Subscriptions, reprints, and supplements available from ACS, 1155 Sixteenth St. N.W., Wash. D. C. 20056.

Building Science Series—Disseminates technical information developed at the Bureau on building materials, components, systems, and whole structures. The series presents research results, test methods, and performance criteria related to the structural and environmental functions and the durability and safety characteristics of building elements and systems.

Technical Notes—Studies or reports which are complete in themselves but restrictive in their treatment of a subject. Analogous to monographs but not so comprehensive in scope or definitive in treatment of the subject area. Often serve as a vehicle for final reports of work performed at NBS under the sponsorship of other government agencies.

Voluntary Product Standards—Developed under procedures published by the Department of Commerce in Part 10, Title 15, of the Code of Federal Regulations. The purpose of the standards is to establish nationally recognized requirements for products, and to provide all concerned interests with a basis for common understanding of the characteristics of the products. NBS administers this program as a supplement to the activities of the private sector standardizing organizations.

Consumer Information Series—Practical information, based on NBS research and experience, covering areas of interest to the consumer. Easily understandable language and illustrations provide useful background knowledge for shopping in today's technological marketplace.

Order above *NBS publications from: Superintendent of Documents, Government Printing Office, Washington, D.C. 20402.*

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NBS Interagency Reports (NBSIR)—A special series of interim or final reports on work performed by NBS for outside sponsors (both government and non-government). In general, initial distribution is handled by the sponsor; public distribution is by the National Technical Information Services (Springfield, Va. 22161) in paper copy or microfiche form.

BIBLIOGRAPHIC SUBSCRIPTION SERVICES

The following current-awareness and literature-survey bibliographies are issued periodically by the Bureau:

Cryogenic Data Center Current Awareness Service. A literature survey issued biweekly. Annual subscription: Domestic, $20.00; Foreign, $25.00.

Liquified Natural Gas. A literature survey issued quarterly. Annual subscription: $20.00.

Superconducting Devices and Materials. A literature survey issued quarterly. Annual subscription: $20.00. Send subscription orders and remittances for the preceding bibliographic services to National Bureau of Standards, Cryogenic Data Center (275.02) Boulder, Colorado 80302.

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