**Coal Technology Program
Progress Report for May 1976**

MASTER

OAK RIDGE NATIONAL LABORATORY

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Printed in the United States of America: Available from
National Technical Information Service
U.S. Department of Commerce
5285 Port Royal Road, Springfield, Virginia 22161
Price: Printed Copy \$4.00; Microfiche \$2.25

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Contract No. W-7405-eng-26

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PROGRESS REPORT FOR MAY 1976

JUNE 1976

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Oak Ridge, Tennessee 37830
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COAL TECHNOLOGY PROGRAM PROGRESS REPORT FOR MAY 1976

ABSTRACT

This report - the twenty-second of a series - is a compendium of monthly progress reports for the ORNL research and development projects that are in support of the increased utilization of coal as a source of clean energy. The projects reported this month include those for hydrocarbonization research, solid-liquid separations, in situ gasification, analytical chemistry, engineering evaluations of the Synthoil and Hydrocarbonization processes, materials, and coal-fueled MIUS.

1. SUMMARY

J. P. Nichols

Highlights of our progress in May are summarized below:

° In our Hydrocarbonization project, two successful experiments were completed in the bench-scale hydrocarbonizer. The first, HC-3, involved feeding 13.2 lb/hr of Wyodak coal to the reactor at 910°F and 300 psig H₂ pressure for a period of 8.1 hr. The second, HC-4, involved feeding 14.0 lb/hr of Wyodak coal to the reactor at 1000°F and 300 psig H₂ pressure for a period of 7.3 hr. The system will be operated in a routine manner to complete a campaign of experiments with Wyodak coal.

° In our Solids-Liquid Separations project, the bench-scale settler was modified by covering the surface with a conductive copper layer and embedding the calrod heaters in Thermon heat transfer paste. It is being installed and will be operational by June 9. Initial filtration runs on the bench-scale filter employed a 40-μ metal disc filter. Filtration rates for PAMCO-SRC at 500°F were as high as 165 lb/hr·ft², but decreased rapidly with cake thickness.

° High-temperature pyrolysis studies of large coal blocks were significantly interrupted by equipment problems which developed during the latter part of the report period. Several tests at 800°C were completed, however, with the continued production of pyrophoric chars at external heating rates of 0.3 and 3.0°C/min. Detailed analyses of condensable pyrolysis products collected during these and other tests are in progress.

° In Analytical Chemistry, whole samples of Synthoil, shale oil, crude petroleum, and by-product waters from oil shale retorting and Synthane coal gasification have been chemically fractionated and are being examined for

biological activity. Normal alkanes in oil shale and by-product retort water have been determined. Polycyclic aromatic hydrocarbon (PAH) fractions have been prepared from extracted organics removed from shale oil by-product water and Synthane condensate water. Twenty-five PAH compounds have been measured in the shale oil water while ten were measured in the Synthane water. Feasibility studies are progressing to determine the utility of a separation scheme employing a Sephadex gel.

° In our conceptual design study of the Synthoil process, preliminary figures indicate that the overall capital cost estimate for the commercial-scale (100,000 bbl/day) plant will be in the neighborhood of \$2 billion. Work continued on the estimation of operating costs. The computer program for calculating product price as a function of coal cost and rate of return on investment was extended to produce parametric runs over any desired range of coal prices. Three companies were contacted to verify the cost estimate (\$125 million) and optimum module size (six 2000-T/D units) for the oxygen plant. Cost estimates for selected pressure vessels and pumps were obtained from vendors. An outline of the final report was prepared and discussed with ERDA/FE representatives.

In our Hydrocarbonization conceptual design study, the major effort was placed upon capital and operating cost estimation. Utilities requirements were completed except for the requirements of the fluid bed combustor, which will be available by mid-June. An estimate of the total overall thermal efficiency of the process indicated a value of about 70.3% based upon the heating values of the inlet coal and all the combustible products. The economic evaluation of the process is continuing and preparation of the final report is underway.

° In our Pressure Vessel and Piping Technology project, we continued our assessment effort with evaluations and discussions at Chicago Bridge and Iron and at Taylor Forge. ERDA/FE personnel conducted program reviews on May 5-6 and on May 12 for our Materials Programs.

Critical angle ultrasonic studies show some potential for inspecting intermetallic erosion resistant coatings but does not work for ceramic coatings.

Conceptual design work was continued in our Prestressed Concrete Pressure Vessel Studies. A finite-element computer program is being implemented to rapidly and cheaply make design evaluations for the HYGAS and Synthane gasifier.

Electron microscopic examination of four candidate steels for fluidized bed applications is in progress. These alloys are Incoloy 800, Incoloy 825, 310 and 316 stainless steel.

° In Coal-Fueled MIUS, stainless steel and Inconel 600 tubing for the furnace was delivered. Because of excellent progress in preparation of tube bundle drawings and specifications, it was decided to hold the furnace housing bid request in order to send out a combined furnace and tube bundle bid package. Placement of the turbine modification contract with AiResearch is awaiting receipt of additional funds. Tube vibration frequency vs air velocity was measured in the cold flow model, and endurance testing of the coal feed system continued.

2. HYDROCARBONIZATION RESEARCH

H. D. Cochran, Jr.

2.1 Bench-Scale System

H. D. Cochran, Jr., G. L. Yoder,
P. R. Westmoreland, and R. L. Andrews

2.1.1 Work Accomplished

Design and Review. Specifications for the 80-atm hydrogen booster compressor have been let out for bids. Design effort for the 80-atm reactor and preheater has been temporarily curtailed pending final decision on the design pressure rating.

Operation. Routine experimentation continued in May with two hydrocarbonization experiments as follows:

- HC-3 - Uniformly fluidized bed reactor at 910°F and 300 psig H₂ pressure, Wyodak subbituminous coal fed at 13.2 lb/hr for 8.1 hr.
- HC-4 - Uniformly fluidized bed reactor at 1000°F and 300 psig H₂ pressure, Wyodak subbituminous coal fed at 14.0 lb/hr for 7.3 hr.

Cleanup and sampling procedures have been established. Char is recovered both from the char receiver and from the reactor. Three phases of scrubber liquid (light oil, an aqueous phase, and bottoms) are physically separated, weighed, and sampled. Aqueous and organic phases from the cold trap are similarly separated and sampled. Tars are filtered from a water rinse of the scrubber and weighed, and both the scrubber and cold trap are rinsed with trichloroethane (solvent is gently evaporated from a sample and the residue is analyzed). Tars collected on the in-line high-efficiency filter and tars wiped out during cleaning of lines are weighed.

2.1.2 Work Forecast

Design and Review. Effort will continue on modifications for 80 atm operation of the bench-scale system.

Operation. Routine operation of the bench-scale reactor will continue.

2.2 Residue Carbonization

H. D. Cochran, Jr., J. B. Gibson, and J. L. Rose

2.2.1 Work Accomplished

Three residue carbonization runs - RC-4, RC-5, and RC-6 - and four agglomeration tests were performed in May. The residue carbonization runs were all at 1200°F while the first two agglomeration tests were at 390°F and the latter two at 300°F.

Run RC-4 was terminated after 15 min when a plug in the scrubber exit caused system overpressure. Run RC-5 ran intermittently for 4 hr with frequent shutdowns due to fluctuations in system pressure. These fluctuations were caused by partial plugs in the scrubber. A total of 1.2 lb of residue was fed in RC-5. During cleanup after RC-5, large agglomerates were found in the reactor. It also appeared that a 1/2- to 3/4-in.-deep layer of liquid residue had accumulated on the distributor plate. The bottom 6 in. of the draft tube was plugged. Since plugs have been found in the draft tube after every run, an attempt was made to operate the reactor as a spouted bed, without a draft tube in RC-6. An organic scrub liquid was used instead of water to alleviate plugging problems in the scrubber. The run was not successful due to feeding problems, but another attempt to operate under these conditions will be made the first week in June.

Severe agglomeration occurred in all four agglomeration tests. This indicates that agglomeration problems for H-Coal vacuum distillation bottoms is much more severe than normally found in caking coals.

2.2.2 Work Forecast

More runs are planned in both the residue carbonizer and the agglomeration test apparatus in order to determine conditions for which agglomeration problems are a minimum.

3. SUPPORTING RESEARCH AND DEVELOPMENT IN SEPARATIONS TECHNOLOGY

B. R. Rodgers

3.1 Bench-Scale Tests

B. R. Rodgers and D. A. McWhirter

The settler, as discussed last month, was subjected to modifications designed to eliminate internal convection currents. Three additional external and three internal thermocouples were installed to provide better temperature profiles. The external surface was sprayed with a 0.020-in. conductive layer of copper and covered with a 0.5-in. layer of Thermon, a heat transfer paste. The four calrods providing heat to the settler were connected to individual controllers to provide more control over the heat input rate. The settler is being reinstalled and will be in operation by June 9.

Three runs were made in the bench-scale filter using a 40- μ nominal pore opening sintered metal plate filter. At 390°F and 12 psi pressure drop, filtered oil (FO) from the PAMCO-SRC process gave an initial filtration rate of 464 lb/hr·ft² dropping to 100 lb/hr·ft² near the end of the filtration period and an overall average filtration rate of 168 lb/hr·ft². The rate reduction with time is surprising since the filtered oil contained only 0.06 wt % ash. Small organic particles may have plugged some of the filter pores. Increasing the pressure by a factor of 4 increased the filtration rate by a factor of 2.5.

Unfiltered oil (UFO) from the PAMCO-SRC process was filtered as described above at 390 and 500°F. At 390°F and 50 psig pressure drop, the initial filtration rate was 118 lb/hr·ft²; this decreased to 7.7 lb/hr·ft² after 1 1/3 hr. Doubling the pressure at this time only increased the rate by a factor of 1.7. At 500°F and 20 psig pressure drop, the initial rate was 165 lb/hr·ft², and the rate fell to 54 lb/hr·ft² after 7 1/2 min. There was considerable vapor loss in the atmospheric filtration receiver during this run due to the vapor pressure at 500°F (~20 psig), but the losses at 390°F (~2 psig) were minimal. Only filtrations at the lower temperature are planned until a closed filtration weighing system is built and installed.

4. EXPERIMENTAL ENGINEERING SUPPORT OF AN IN SITU GASIFICATION PROCESS

R. C. Forrester III

4.1 Large-Block Pyrolysis Studies

R. C. Forrester III, T. M. Somers, and G. D. Owen

Six-inch right circular cylinders of Wyodak subbituminous coal have been pyrolyzed at 800°C using heating rates of 3°C/min and 0.3°C/min. At the conclusion of all experiments, exposure of chars to air resulted in self-heating to temperatures as high as 300°C although ignition did not occur as with some of the chars produced at lower temperatures. Furnace controller malfunction delayed further work, but the problem is expected to be remedied by June 2.

Alteration of the data reduction code and detailed analysis of condensable pyrolysis products are continuing with results expected during the next report period.

4.2 ERDA's UCG Program

R. C. Forrester III

The third working group meeting for mathematical modeling in support of ERDA in situ coal gasification programs was held at the Lawrence Livermore Laboratory on May 18. Major efforts at LLL, MERC, and LERC were described and discussed informally. Attending the meeting as observers were representatives from ERDA, ORNL, ANL, and the University of Texas (Austin). The modeling work, although primarily concerned with describing the Underground Coal Gasification (UCG) reactions and associated pyrolysis and drying phenomena, has also addressed problems of permeability enhancement, surface subsidence, and horizontal ground movements which accompany these processes.

5. ANALYTICAL CHEMISTRY

B. R. Clark and M. R. Guerin

Work reported here is carried out within the Analytical Chemistry and Chemical Technology Divisions. Sponsoring agencies are ERDA, EPA, and NSF/RANN, the latter agency providing support through the Ecology and Analysis of Trace Contaminants Program for which R. I. Van Hook serves as Principal Investigator.

5.1 Fractionation of Fossil Fuel Derived Liquids

I. B. Rubin

The fractionation of a 6-liter batch of the shale oil product water has been completed. A set of samples has been delivered to J. L. Epler of the Biology Division for mutagenesis testing, and another set has been delivered to S. Herbes of the Environmental Sciences Division for toxicity testing. In addition, a 1-liter batch of shale oil product water was fractionated, using batch extractions rather than the continuous extraction procedure used for the larger sample. Despite the differences in handling, proportions of comparable fractions in the two samples agreed well. Soluble acid fractions accounted for about 87% of the total organics, soluble base fractions about 5%, and neutrals about 2.5%.

Two 20- to 25-g portions of shale oil were also fractionated by the Stedman procedure. Proportions of comparable fractions agreed well. Soluble acid fractions accounted for only about 2% of the total sample, soluble bases, 7%, and neutrals, 87%. Half of the neutral portion of the second sample was chromatographed on Florisil in the manner used previously for COED Syncrude and Synthoil oil. A complete set of fractions from sample 2 has been sent to J. L. Epler.

A sample of Louisiana-Mississippi sweet crude oil has been fractionated by the Stedman procedure for biological testing of the various fractions. These experiments should afford some comparison with data already obtained on synthetic crudes from coal and oil shale. Of special interest are the PAH containing fractions from the neutral portion developed in this separation scheme.

The crude petroleum fractions along with a portion of the Synthoil fractions have been delivered to Jim Epler of the Biology Division.

5.2 Analysis of PAHs in By-Product Waters

C. H. Ho and B. R. Clark

A reliable procedure, which is applied to the routine analysis of PAH materials in cigarette smoke, has also been used to determine the concentration of PAHs in by-product water from oil shale retorting. One liter of water was extracted four times with 200 ml of cyclohexane. The cyclohexane fractions were combined and reduced to 20 ml and extracted successively with four portions of DMSO (20 ml each). The combined DMSO extracts were diluted with water to 40% (volume) DMSO and inversely extracted with four 20-ml portions of cyclohexane. The combined cyclohexane extracts were reduced to 5 ml for chromatography on a column (50-ml burette) filled with 10 g of Florisil, using hexane/benzene (6/1) as eluting solvent. About 150 ml of eluate was pooled and reduced to 5 ml for another column separation [50-ml burette, 20 g of activity II neutral alumina, hexane/benzene (2/1) as eluting solvent]. Four hundred milliliters of eluate was collected and reduced to 1 ml for GLC analysis.

Five microliters of the PAH fraction were injected into a gas chromatograph equipped with a packed 3% Dexsil 400 column. The tentative identifications were made by co-chromatography and quantification was achieved using standard solutions. Structural confirmations are proceeding by GLC/MS analysis in W. T. Rainey's group. Estimations of PAH concentrations in shale oil by-product water are (ppb): naphthalene (86), 2-methylnaphthalene (330), 1-methylnaphthalene (110), biphenyl (33), 2,6-dimethylnaphthalene (46), 1,3- + 1,6-dimethylnaphthalene (360), 1,5- + 2,3-dimethylnaphthalene (100), 1,2-dimethylnaphthalene (38), fluorene (180), 9,10-dihydroanthracene (27), 1-methylfluorene (160), phenanthrene + anthracene (340), 2-methylanthracene (210), 1-methylphenanthrene (130), 9-methylanthracene (35), fluoranthene (18), pyrene (120), 1-methylpyrene (61), 1,2-benzoanthracene + chrysene (37), 1,2- + 3,4-benzopyrene (5), and 3-methylcholanthrene (6).

5.3 Analysis of Paraffins in Crude Shale Oil and By-Product Water

C. H. Ho and B. R. Clark

A sample of shale oil (1 g) and a sample of shale oil by-product water (1 liter) were dissolved in or extracted with hexane, respectively. The combined hexane extracts from the water were washed with NaOH solution (pH 11) and HCl solution (pH 4). The paraffin fractions were isolated by alumina column chromatography (neutral activity I), using n-hexane as eluting solvent. The column eluate was pooled and reduced to 1 ml for GLC analysis. A variety of liquid phases and supports were tried in GLC-columns of various lengths. The most satisfactory separation of the paraffin fraction from shale oil and shale oil by-product water was obtained on a 1/8 in. by 10 ft glass column packed with 3% Dexsil 400

on 100/120 Supelcoport. After an initial hold for 8 min at 100°C, the temperature was programmed to 320°C at a rate of 2°/min; detector, 320°C; injector, 320°C; helium carrier gas inlet pressure set at 60 psig. The tentatively identified compounds and their concentrations are summarized in the following table:

n-Alkanes in Shale Oil and Shale Oil By-Product Water

Compound	Oil (mg/g)	Water (µg/liter)	Compound	Oil (mg/g)	Water (µg/liter)
C ₁₁ H ₂₄	7.55	67	C ₂₄ H ₅₀	3.60	48
C ₁₂ H ₂₆	6.65	66	C ₂₅ H ₅₂	3.90	53
C ₁₃ H ₂₈	6.20	65	C ₂₆ H ₅₄	3.45	50
C ₁₄ H ₃₀	4.80	62	C ₂₇ H ₅₆	3.35	51
C ₁₅ H ₃₂	3.30	45	C ₂₈ H ₅₈	2.90	48
C ₁₆ H ₃₄	4.50	53	C ₂₉ H ₆₀	3.20	62
C ₁₇ H ₃₆	8.60	105	C ₃₀ H ₆₂	1.00	30
C ₁₈ H ₃₈	8.80	106	C ₃₁ H ₆₄	0.20	27
C ₁₉ H ₄₀	5.60	70	C ₃₂ H ₆₆	0.10	9
C ₂₀ H ₄₂	4.80	62	C ₃₃ H ₆₈	0.65	12
C ₂₁ H ₄₄	5.70	53	C ₃₄ H ₇₀	0.40	5
C ₂₂ H ₄₆	4.50	59	C ₃₅ H ₇₄	0.15	2
C ₂₃ H ₄₈	5.00	67			

5.4 Separations Using Sephadex LH-20

A. R. Jones

Depending upon the method of treatment, Sephadex LH-20 can be used to achieve a lyophilic-lipophilic separation or an aliphatic-aromatic separation. The latter separation is carried out using the resin after swelling with isopropanol.

Weighed portions (1.01, 1.01, 1.93, and 3.77 g) of dehydrated and filtered crude shale oil were injected onto a 2.5 x 50 cm column containing LH-20 resin swollen with isopropanol. Elution with isopropanol produced fractions (based on calibration with known materials) containing respectively: (1) aliphatic and alkyl-substituted monoaromatic substances: 73.0, 75.5, 73.2, and 75.2%, (2) heteroatom-containing monoaromatics and alkyl-substituted two-ring aromatics: 10.0, 14.9, 9.3, and 7.5%, (3) heteroatom four-ring aromatics: 5.0, 4.0, 3.4, and 7.4%.

Recoveries within the first 500 ml of eluate were 88 to 94%. GC analysis of the fractions confirmed the efficacy of the fractionation.

5.5 Analytical Services

W. R. Laing and L. J. Brady

A total of 294 samples were submitted for analysis during this month.

Ash determinations on SRC samples from solids-liquid separation tests continued to be the most common tests. In addition, four sets of gas samples were tested for CO, N, and CH₄ content, and 18 samples of coal-derived liquids were submitted for tests to help characterize these liquids. Solid coal and char samples, submitted for proximate and ultimate analyses, completed the list of samples tested.

Because of a strike of drivers for United Parcel, the coal samples which were prepared for testing in a commercial testing laboratory have not been delivered. It appears now that they will be delivered by May 21, 1976.

A new Perkin Elmer Model 3920-B gas chromatograph has just arrived and will be installed by a factory engineer within the next 10 days.

6. ENGINEERING EVALUATIONS OF THE SYNTHOIL AND HYDROCARBONIZATION PROCESSES

J. M. Holmes, R. Salmon, and E. G. St. Clair

6.1 Synthoil Process

R. Salmon, E. G. St. Clair, M. S. Edwards,
W. C. Ulrich, and D. A. Dyslin

6.1.1 Process Economics

Work continued on the overall cost estimate. Preliminary indications are that this will be in the neighborhood of \$2 billion.

Work also continued on the estimation of operating costs. Manpower and other operating requirements of the facility are in the process of being estimated.

In the April 1976 monthly report, a computer program was mentioned which calculates product price as a function of coal cost and rate of return on investment. In May this program was extended to produce parametric runs in which coal price is varied automatically over some desired range.

Cost estimates for a number of pressure vessels selected from various units making up the Synthoil facility were received from the Chicago Bridge and Iron Company. Both shop-fabricated and field-erected vessels were included in the list. This information will be used for calibrating our computer code which was developed for costing all the pressure vessels for the Synthoil plant.

The capital cost code, which develops an installed plant cost using factors applied to purchased equipment costs, was checked against several additional plants for which estimates of both purchased equipment costs and installed costs were available. Results indicate that further development of procedures to account for such factors as special materials of construction and high pressure operation is required before the code can be used as intended.

Pump costs were received from Pacific Pump and the Ingersoll-Rand Company.

6.1.2 Oxygen Plant Design

Contacts were made with three companies that manufacture oxygen plants to obtain answers to some basic questions that were raised concerning the design and cost of this unit of the Synthoil facility. Specifically, we wanted to find out (1) whether other suppliers would confirm the Linde Company's estimate of \$125 million for a 12,000 T/D unit consisting of six 2000-T/D modules, (2) whether a cost reduction could be made by increasing the size of the module and thus reducing the number of modules required, and (3) to affirm that the plant proposed by Linde was of the expansion type as opposed to the cascade type.

Air Products Company estimates \$120 million and gives the maximum module size as 2000 T/D, confirming Linde's figures. Airco gives the maximum module size as 1500 T/D and estimates the cost at \$17 million to \$20 million per module. This corresponds to a cost of \$136 million to \$160 million for a 12,000-T/D plant (consisting of eight 1500-T/D modules).

Linde's maximum module size is based on a maximum shipping diameter of 17 ft for the main rectifying column. They believe that compressor wheel size is also a limiting factor. Although Linde feel that there is little to be gained by going to larger field-fabricated vessels, we believe that this point ought to be investigated further. The Linde design is based on the expansion cycle. The expander turbines drive electric generators, generating about 3000 kW, of which about 1400 kW is used in the oxygen plant.

6.1.3 Flowsheets

The Sour Water Stripper and Ammonia Recovery Plant (Unit 28) was modified to include a dephenolization section, and a revised material balance for the unit was developed based on vendor-supplied information.

Material balances for all units are being checked to eliminate a few minor discrepancies and to ensure consistency from one flowsheet to another.

6.1.4 Final Report

Work continued on a preliminary draft of the final report. An outline of the report was written and was discussed with ERDA/FE representatives during a meeting at Oak Ridge National Laboratory May 25-26, 1976.

A preliminary overall plot plan of the facility was completed.

6.2 Hydrocarbonization Process

J. M. Holmes, D. A. Dyslin, M. S. Edwards,
D. S. Joy, G. R. Peterson, and C. B. Smith

The design and cost evaluation of a hydrocarbonization facility for the production of clean fuel equivalent to a nominal 100,000 bbl/day of fuel oil continued. Major effort was placed on capital and operating cost estimating. Revision of the equipment lists was continued to reflect changes in several of the process units. The utilities list was completed except for the utility requirements for the fluid bed combustor, which will be available upon completion of the Foster Wheeler study in mid-June:

(1) Hydrocarbonization: An evaluation of the overall thermal efficiency indicates that the value for the Hydrocarbonization process will be about 70.3%. The percentages of the total heat output for the various products are as follows:

<u>Product</u>	<u>% Total Heat Available in Products</u>
Char	68.6
Fuel oil	21.1
Pipeline gas	9.1
Sulfur	0.6
Ammonia	0.5
Phenolics	<u><0.1</u>
	100.0

The sulfur, ammonia, and phenolic products would not normally be sold for fuel but are included in the thermal efficiency because they represent finite heating values in the coal feed and therefore should be included in the analysis. Initial estimates of the overall process efficiency assumed that about 60% of the pipeline gas produced by the hydrocarbonizers would be consumed as fuel in the process. Later refined calculations indicate that this estimate was high which resulted in a higher net make of pipeline gas for the process. Therefore, the actual production from the plant will be equivalent to about 105,600 bbl of fuel oil per day for the plant rated at a nominal 100,000 bbl per day.

(2) Fluid Bed Combustor: Foster Wheeler Corporation completed their third month of design effort on the fluid bed combustor unit. Current effort is directed towards the development of the final cost estimate and writing a draft of the final report. The final report will be completed by June 15. The overall efficiency estimated for the fluid bed combustor system is 86.6% based upon the combined raw coal--low-grade coal feed specified for the system. The area requirements for the six fluid bed modules and their auxiliary equipment will be approximately 100,800 ft² (240 ft x 420 ft).

7. MATERIALS

W. R. Martin and D. A. Canonico

The materials engineering and supporting technology reported herein are in support of activities directed by Materials and Power Generation, Division of Fossil Energy Research. Other related work not funded directly by this division of ERDA/Fossil Energy is included also.

7.1 Pressure Vessel and Piping Technology Assessment

D. A. Canonico, R. H. Cooper, R. K. Nanstad,
G. C. Robinson, and G. M. Slaughter

A review of the piping and pressure vessel needs for coal conversion systems is in progress. The program will identify those areas where additional material property data needs are required in order to assure that the pressure boundary components in conversion systems can be designed, fabricated, and operated in a safe and reliable manner.

Visits were made to Chicago Bridge and Iron (CB&I), Memphis, Tennessee, and Taylor Forge (T-F), Cicero, Illinois. Shop erection capabilities were discussed with CB&I. They said that any vessel equal to or less than 12 meters (40 ft) in diameter and weighs less than 1150 to 1250 tons can be built in Memphis and loaded on a barge.

Taylor-Forge is involved in pipe fitting and elbows. Currently T-F can manufacture elbows that are 1220 mm (48 in.) ID with 457 to 610 mm (18 to 24 in.) tangents with wall thicknesses of 37.5 mm (1 1/2 in.). They estimated that it would require about 2 years and \$1 to \$2 million to develop the manufacturing capability to produce 1.5 meter (5-ft) diameter elbows with 100 to 150 mm (4 to 6 in.) wall thicknesses.

7.2 Inspection Techniques for Wear- and Process-Resistant Coatings

R. W. McClung and G. W. Scott

Dr. Wate Bakker, ERDA/FE, visited ORNL on May 5. Computerized abstract searches are being used to search the literature for NDT methods and material-properties data. ANL Coal Technology reports (ANL-76-7 and -76-22) have been received and reviewed for information on coating specimens. Coating-thickness standards have been designed; fabrication is in progress. X-ray-fluorescence techniques were tested over a narrow range of coating thicknesses around 0.010 in.; some difficulty was experienced in producing suitable known thicknesses of coatings. Critical-angle ultrasonic studies show some potential for surface-wave methods on intermetallics, but ceramics do not appear to support surface waves. Penetrant techniques for both

intermetallic and ceramic coatings are being improved and made more efficient. Some preliminary work was done on electrolytic-probe and microwave techniques.

7.2.1 Review and Evaluation

Dr. Wate Bakker visited ORNL on May 5 to review Coal Technology Program Materials work. He was given a 30-min briefing on coating NDT work and a tour of the laboratory facilities.

We have employed a number of computerized abstract searches for data on coating NDT methods and material properties. Key-word lists were developed, and feedback from searches of Metals Abstracts have been received. Chemical and Physics Abstracts are still being checked. Checking of individual articles in retrieved listings is in progress.

We have established a direct-exchange arrangement with Argonne National Laboratory to obtain current reports on ANL coating research. Our most recent acquisition had been ANL-76-7, Coal Technology Program Quarterly Progress Report for the Period Ending September 15, 1975, which was obtained through normal ORNL library channels. (On June 1, we obtained ANL-76-22, the quarterly report for the period ending December 31, 1975.) Based on our evaluation of the reports, we are initiating work on the following substrate-coating systems:

- (1) Type 304 stainless steel coated with zirconium oxide, aluminum oxide, magnesium-zirconium oxide, aluminum oxide over a bond coat, and cobalt-chromium-aluminum alloy.
- (2) Type 316 stainless steel coated with aluminum oxide and zirconium oxide over a bond coat.
- (3) Alloy 800 coated with chromium carbide in a nickel-aluminum matrix, zirconium oxide, magnesium-zirconium oxide, and aluminum oxide.

The bond coat, where used, is a nickel-chromium-aluminum alloy. Although a variety of combinations will be prepared for study, particular emphasis will be given to one ceramic coating and one coating with a ceramic-alloy mixture.

7.2.2 Specimen Fabrication

Development of spraying techniques continues. Initial efforts to spray prescribed thicknesses of ceramics were unsuccessful because of uncertainties in the manual technique used. Useful specimens are obtained from this process by sorting and measuring, then running tests in areas of specimens where dimensions are known.

For anticipated work requiring higher precision and known references, we are initiating production of standards. A design for a standard has been completed and a group of blanks ordered. Fabrication will include applying a coating thicker than desired and machining down to the desired thickness.

7.2.3 Radiation Techniques (B. E. Foster)

Attempts to produce complete specimens with uniform thicknesses between 0.009 and 0.011 in. were unsuccessful. We were able to produce specimens with thicknesses in that range at various points. We measured the specimen thicknesses by dimensional methods before and after coating at a number of points. To get x-ray-fluorescence (XRF) measurements, we used a lead mask with a 0.25-in. hole over each dimensionally measured point to provide collimation. Correlation between dimensional and XRF measurements is fair; there are some indications of inaccuracies in the dimensional measurements which are being investigated.

7.2.4 Ultrasonic Testing

Four specimens were taken to the University of Tennessee Acoustics Laboratory for testing on a combination schlieren/goniometer system. This system is equipped with a gaussian transducer which produces a narrow, although unfocused, sound beam. A narrow beam is useful for critical-angle investigations. A specimen of sandblasted Incoloy alloy 800 (IN-800) exhibited a Rayleigh angle near 30° with strong surface-wave propagation. A specimen of IN-800 coated with NiCr exhibited a Rayleigh angle near 34° with good surface-wave propagation. Two specimens of ZrO_2 on IN-800, with thicknesses of 0.010 and 0.020 in. failed to exhibit a Rayleigh angle and showed no evidence of surface-wave propagation.

7.2.5 Penetrant Testing (S. D. Snyder)

Work continues on techniques to reduce extraneous indications from porous materials. We have noted significant differences in the process steps required to obtain equivalent responses from intermetallic coatings (e.g., specimen ANL-C 5) and ceramic coatings. We have demonstrated the ability to achieve useful response from both types of coatings. Current efforts are directed toward determining the minimum number and duration of process steps.

Ceramic coatings require a rapid, light application of penetrant followed by stripping washes and emulsification. Some experiments have employed organic solvents applied directly to the surface for emulsification.

7.2.6 Miscellaneous Techniques (G. W. Scott)

A simple electrolytic probe has been designed and is now being fabricated.

Some microwave equipment has been located, and some experiments are being designed.

7.3 Iron and Nickel Carbonyl Formation and Prevention

J. Brynestad and J. H. DeVan

The atomic absorption spectrophotometer has been installed and tested, and an exhaust hood has been assembled over the instrument. Stainless steel autoclaves are being modified to serve as reaction vessels for studying the kinetics of iron carbonyl formation, and autoclave furnaces are under construction.

7.4 Failure Analysis of Materials and Components

D. A. Canonico, D. P. Edmonds, and T. K. Roche

This project is devoted to the posttest examination of components that have been removed from service in coal liquefaction processes. The goal is the avoidance of failures in commercial coal conversion systems.

Limited examinations of the pressure let-down trim set from the Synthoil PDU at PERC are in progress. We are microscopically evaluating the Kennametal grade K701 valve seat insert and plug tip. This material is a sintered tungsten carbide with 8 to 10% cobalt-chromium binder. Cobalt is used for a binder here because of its low solid solubility (about 1%) of tungsten carbide and its superior ability to wet the carbides during sintering. Chromium is added to the binder to enhance the corrosion resistance of the material. The average carbide grain size was measured to be 2 μ in diameter. A carbon-deficient carbide phase known as eta phase ($\text{Co}_2\text{W}_3\text{C}$) was also identified in the microstructure. Areas close to the surface, especially corner and tip areas, were found to be deficient in eta phase. However, erosion occurred both in areas where eta phase was and was not present. We are presently performing microhardness traverses of these materials.

7.5 Prestressed Concrete Pressure Vessel Studies

W. L. Greenstreet

The objective is to investigate the potential use of prestressed concrete pressure vessels (PCPV) for coal conversion processes, to identify major problem areas, and to define and outline a test program (or programs) for feature and concept demonstration. Conceptual designs of pressure vessel and liner combinations for commercial size systems are to be developed and studied as vehicles for assessment and guidance.

Conceptual design work was continued as the main activity for both the HYGAS and Synthane gasifiers. To aid in collecting background information, a visit was made to IGT in Chicago to discuss gasifier design for a commercial HYGAS system. Needed information on manhole requirements and designs was obtained; additional information was also obtained on temperature requirements. The lower limit for vessel, or liner, temperature necessary to avoid condensation is about 450°F, which is less than the water vapor dew point. Further, it is not necessary for the ash quench pot at the bottom of the vessel to be an integral part of the vessel.

Under Task 1, the various aspects are being addressed in greater detail using more refined analysis methods. More thorough examinations are being made of vessel configurations, penetration designs, and prestressing requirements. Closure design and analysis continue to receive considerable attention.

A finite-element computer program is being implemented to rapidly and cheaply make design evaluations. This program treats axisymmetric and plane bodies subjected to pressure and to concentrated nodal loadings. General boundary conditions can be employed and a structure made up of five separate materials can be considered. The material model applies to isotropic, elastic behavior, and only small strains are considered.

This program will be used to address prestress forces required (Task 2) and the stresses and forces in discontinuity regions (Task 3) for both HYGAS and Synthane gasifier vessels. Analyses to date, in the main, have been based on analyses of thick-walled circular-cylinder geometries to examine internal pressure and tendon prestress influences.

Work is progressing on the design of hot liner concepts in which the temperature of the liner is above the dew point of the process gases. To be considered is each element of the composite structure consisting of, for example, refractory, liner, hot (or insulating) concrete, and structural concrete. An informal writeup has been prepared on the cold and hot liner concepts developed to date. This writeup is to provide a basis for detailed thermal-hydraulic studies as required for further establishing requirements and for determining overall feasibility.

Mr. J. J. Forst, ERDA/FE, visited ORNL on May 12, 1976, to discuss coal conversion process work. The progress to date under the feasibility study was reviewed in detail and follow-on tasks were discussed.

7.6 Other Related Work

W. W. Harris, R. H. Cooper, Jr., and J. H. DeVan

7.6.1 Fluidized Bed Material Support Activities

Fireside Corrosion in a Fluidized Bed Coal Combustor. Surveillance coupons of four candidate steels for MIUS exposed in the Combustion Power Company, Inc., Menlo Park, California, fluidized bed have been received and submitted for examination with scanning electron microscopy, metallography, electron microprobe, electron spectroscopy chemical analysis, and x-ray diffraction.

Coupons were removed at 500 hr and 1000 hr. The bed temperature was 1650°F and the test temperature from 1575 to 1590°F. The coal feed rate was of the order of 0.9 lb/min for a weight ratio of dolomite to coal of 0.23, and a weight ratio of inhibition (clay) to coal of 0.004.

Scanning electron microscope examination of exposed coupons consisted of micrographing represented areas of exposed scale and energy dispersive scans of scale surfaces and of metallographic cross sections of scales. Electron microprobe examination included both energy dispersive and wavelength dispersive scans of base metal, metal scale interface, scale interior, and scale outer surface interface. Results of these examinations as well as from optical metallography are being studied. Electron spectroscopy chemical microanalyses for two coupons are underway.

Photomicrographs of some fields of the sectioned Incoloy 800 coupon showed possible submicron-sized metallic bits at the scale/base metal interface. Electron microprobe scans of these regions were inconclusive largely because the nearby base metal interface interfered. One side of both the Incoloy 800 and the Incoloy 825 was markedly rippled. This effect was not observed in the photomicrographs of 310 and 316 stainless steel. With respect to scale thickness estimates based on observations with the electron microprobe, the Incoloy 800 scale was thicker than that of the Incoloy 825 coupon.

8. COAL-FUELED MIUS

A. P. Fraas and W. R. Mixon

This project for analysis, design, and demonstration of a concept utilizing a fluidized-bed coal combustion system as a heat source for a gas turbine generator suitable for applications in Modular Integrated Utility Systems (MIUS) is carried out under the ORNL-HUD-MIUS Program within the Energy Division. Work is supported by the U.S. Department of Housing and Urban Development under HUD Interagency Agreement No. IAA-H-40-72 and by the Energy Research and Development Administration, Office of Fossil Energy (formerly Office of Coal Research, Department of the Interior), under ERDA Contract No. E(49-18)-1742. The project consists of four phases: I - Conceptual Preliminary Evaluation; II - Conceptual Design; III - Detailed Design and Construction; and IV - Shakedown, Performance, and Endurance Tests.

8.1 Furnace Procurement

Original plans were to prepare separate bid packages on the furnace housing and the tube bundles. However, the tube bundle drawings were proceeding so rapidly at the time the furnace housing package was ready, it was decided to combine both into a single bid package. The tube bundle drawings and specifications were completed during this period. The combined bid package is now scheduled to be sent to the vendors on June 10.

8.2 Turbine-Generator Units

Placement of the contract with AiResearch for modification of the gas turbine for closed cycle operation is awaiting transfer of additional funds.

8.3 Site Preparation

Design work required for the modifications of Building 9401-1 for the installation of the MIUS coal-fired gas turbine system was initiated in early May. Much of the effort to date has been devoted to design of the inside partition wall.

8.4 Cold Flow Tests of a Fluidized Bed

Tube vibration was successfully measured at various air velocities in the 4-ft square cold flow model. The frequency of vibration was found to vary from 10 to 20 Hz at a fluidizing velocity of 1.7 ft/sec up to 20 to 50 Hz at a fluidizing velocity of 3.5 ft/sec. A test to measure the natural frequency of a full-length furnace tube is being set up for comparison with the vibration data.

8.5 Coal Metering and Feed System

Endurance running of the flow splitter feed system continued at full design power coal feed rate. No operating problems have been encountered in the total of 110 hr of operation.

Performance tests have been run on a prototype furnace coal feed nozzle and satisfactory results were obtained. Endurance testing is in progress and no difficulties have been observed.

8.6 Coal Feed Preparation

A different approach to the preparation of coal for the plant is under study. The coal preparation process was originally based on use of run-of-mine coal with the expectation that this would minimize cost. Coal handling tests on wet or damp run-of-mine coal ground to 1/16 in. top size revealed that drying of the coal to 1% surface moisture would be required to assure reliable pneumatic transport. It was suggested by ERDA/FE that the need for drying might possibly be eliminated if stoker-sized coal were used as the starting material. The measured surface moisture of stoker coal was found to be about 2.5% after a heavy rain (or immersion in water) and a short drainage period. A sample of this wet coal was crushed for handling tests. The coal would not flow out of a 60 deg. conical vessel through a 2-in.-diam opening. The coal was placed in a small hopper feeding into an air-driven eductor, but it would not feed with twice the air pressure in the hopper normally used for feeding dry coal. Dry air was passed upward through the bin for 5 min and feeding with the eductor was again attempted. Feeding proceeded for a short time and then plugging occurred.

The conclusions from the handling tests are that some drying of wet stoker coal will be required. It appears to offer a considerable advantage over run-of-mine coal in that the surface moisture is only about 2.5% for the stoker coal as compared to 8% for run-of-mine coal. This would greatly reduce the heat requirements for drying in periods of rainy weather. The stoker coal will probably dry out more rapidly in the coal pile and permit operation with no drying over a greater fraction of the time between periods of rain.

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