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

MASTER

OAK RIDGE NATIONAL LABORATORY

OPERATED BY UNION CARBIDE CORPORATION FOR THE ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION

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PROGRESS REPORT FOR AUGUST 1976

Date Published: October 1976

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OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee 37830
operated by
UNION CARBIDE CORPORATION
for the
ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION

MASTER

PREVIOUS REPORTS IN THIS SERIES

ORNL/TM-5044, Progress Report for August 1974
ORNL/TM-5045, Progress Report for September 1974
ORNL/TM-5046, Progress Report for October 1974
ORNL/TM-4787, Progress Report for November 1974
ORNL/TM-4796, Progress Report for December 1974
ORNL/TM-4850, Progress Report for January 1975
ORNL/TM-4873, Progress Report for February 1975
ORNL/TM-4892, Progress Report for March 1975
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ORNL/TM-5010, Progress Report for June 1975
ORNL/TM-5037, Progress Report for July 1975
ORNL/TM-5092, Progress Report for August 1975
ORNL/TM-5124, Progress Report for September 1975
ORNL/TM-5186, Progress Report for October 1975
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ORNL/TM-5246, Progress Report for December 1975
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COAL TECHNOLOGY PROGRAM PROGRESS REPORT FOR AUGUST 1976

ABSTRACT

This report - the twenty-fifth 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 are those for hydrocarbonization research, solids-liquid separations, in situ gasification, analytical chemistry, materials engineering, engineering studies, process and program analysis, coal-fueled MIUS, and novel contactor development.

1. SUMMARY

J. P. Nichols

Highlights of our progress in August are summarized below:

- ° In our hydrocarbonization research project we successfully completed our first experiment utilizing the hydrocarbonization reactor in a recirculating fluidized bed mode.
- ° A residue carbonization experiment was completed in which there was no significant agglomeration in the fluidized bed reactor but which was terminated prematurely because of a plug in the off-gas line.
- ° In our solids-liquid separations work we continued characterization and settling tests of oils derived from the SRC process.
- ° In our high-temperature coal block pyrolysis studies we completed experiments to fill out a matrix of pyrolysis data as a function of heating rates at atmospheric pressure.
- ° In our engineering studies and technical support work we completed engineering evaluations of commercial-sized Synthoil and Hydrocarbonization plants. We also initiated a project to provide Process Research Digests for the ERDA Division of Fossil Energy Research.
- ° In our Coal-Fueled MIUS project we received bids for fabrication of the fluidized bed combustor and continued tests of system components.
- ° In our Materials Engineering work we continued experiments to develop inspection techniques for thin coatings and continued design studies of prestressed concrete pressure vessels. We also completed the assembly of our apparatus for measuring the formation rates of metal carbonyls.

- ° We initiated a new project to provide process and program analysis research studies for the ERDA Fossil Energy Office of Program Planning and Analysis.

- ° We also initiated a new project to develop better contactors for the noncatalytic hydroliquefaction of coal.

2. HYDROCARBONIZATION RESEARCH

H. D. Cochran, Jr.

2.1 Bench-Scale Hydrocarbonization System

H. D. Cochran, Jr., G. L. Yoder, P. R. Westmoreland,
C. H. Brown, and J. C. Rose

2.1.1 Work Accomplished

Design and Review. There was no effort to be reported in this area during August.

Fabrication and Installation. Modification of the bench-scale system for recirculating fluidized bed operation was completed early in August. An updated flowsheet is shown in Fig. 2.1. Changes from earlier flowsheets include the draft tube, coal feed location, and gas feed lines.

Coal for hydrocarbonization (and other solids sizing jobs at the Laboratory) can now be ground and sieved in a solids handling facility (Fig. 2.2); new and existing equipment from several other projects has been located in Bldg. 4501, Rm. 112. Drum quantities of solids can be dried, mixed, ground, and sized in a series of dust-free, inert gas blanketed operations. Equipment is also available to dry, mix, grind, and size small quantities of solids. Modification of the existing facility and installation of equipment is nearly completed.

Operation. The recirculating fluidized bed reactor was successfully tested during August in Experiment HC-7, hydrocarbonization of Wyodak subbituminous coal fed at 14.8 lb/hr for 6.9 hr, operating at 820°F and 300 psig. Minor difficulties with the electrical heaters and controllers limited operation to below design temperature.

Steady-state gas composition and preliminary material balances were determined for Experiment HC-7. Consistent deficiencies in overall mass, carbon, and ash balances are being investigated.

Aqueous samples from Experiments HC-5 and HC-6 were analyzed for carbon content, and carbon balances were corrected. Data from all experiments are being critically reviewed so that final material balances may be issued.

Fig. 2.1. Bench-scale hydrocarbonization flowsheet.

ORNL DWG 78-1422

LAB 112, BUILDING 4501

Fig. 2.2. Solids handling facility.

2.1.2 Work Forecast

Design and Review. No effort is planned in this area.

Fabrication and Installation. The solids handling facility will be sufficiently completed during September that coal preparation may begin. Repairs and minor modifications to the heating system on the hydro-carbonizer will be made.

Operation. Coal for the bench-scale system will be prepared during September. When sufficient coal is readied, a series of experiments investigating recirculating fluidized bed hydrocarbonization will begin.

2.2 Residue Carbonization

H. D. Cochran, Jr., J. B. Gibson, and R. L. Andrews

2.2.1 Work Accomplished

One residue carbonization run, RC-13, was performed in August. The temperature of the run was 1230°F, and the feed rate was 3 lb/hr at a 4:1 ratio of Wyodak char to residue. As with the previous two runs, run RC-13 lasted about 1 hr and was terminated when a plug developed in the 1/2-in. pipe leading from the reactor to the cyclone. Analysis of gas samples taken during the run showed small quantities of H₂, CH₄, CO, and CO₂.

Several runs have now been made in which there was little or no agglomeration in the reactor, but extensive solids carryover and plugging downstream of the reactor have terminated these runs. Modifications to the residue carbonizer are being designed which should minimize these problems. These modifications will include the addition of a 3-ft section with an 8-in.-diam expanded head to the top of the reactor and the addition of a high efficiency internal cyclone which will eliminate the 1/2-in. pipe connecting the reactor and the previous cyclone. Other modifications will include replacing the flat bottom of the scrubber with a conical bottom to make recovery of liquids easier and replacing the ball valve feeder with a specially designed plug valve to increase feeding rates.

2.2.2 Work Forecast

Modifications to the residue carbonizer can be completed in September and carbonization experiments with residue will resume near the end of the month.

3. SUPPORTING RESEARCH AND DEVELOPMENT IN SEPARATIONS TECHNOLOGY

B. R. Rodgers

3.1 Characterization Tests

B. R. Rodgers and D. A. McWhirter

Ageing

After ageing for 10 months under various storage conditions, analyses of Solvent Refined Coal (SRC)--Filtered Oil (FO) indicate continued stability of physical and chemical properties. The Unfiltered Oil (UFO), after ageing for 6 months, continued to show increases in oxygen content and a loss of carbon and hydrogen. However, the carbon/hydrogen ratio remained stable at ~ 12.3 . The one-year FO analyses scheduled next month will terminate the study of ageing characteristics of that material, but the UFO ageing analyses will continue for two additional months. As pointed out previously, mislabeled UFO samples from the SRC plant at Wilsonville, Alabama, caused a delay in placing the latter samples in storage.

Paraffinic analyses of settler overflows

Paraffinic analyses of the upper phase of settled UFO and UFO + 20% *n*-decane have provided some interesting information. Analysis of the neat UFO (UFO only) gave 2.5 wt % paraffins. Analysis of the UFO + 20% *n*-decane solution gave 1.3 wt % paraffins on an *n*-decane-free basis. The net loss to a process (assuming 70% overflow and total recovery of *n*-decane in overflow) would be 10.3 wt % of the *n*-decane originally added as precipitant. Process solvent losses at this level would not be tolerable using a pure compound. This result stresses the importance of studying the process recovery characteristics of paraffinic solvents used for de-ashing. For instance, a process similar to SRC using paraffinic process-derived precipitant would have to yield an amount of precipitant equal to 2.6 wt % of the reactor effluent in order to make a de-ashing solvent balance.

Using 20% toluene as agglomerator, the paraffin content of the overflow increased to 5.1 wt %. The aromatic content of this overflow, its recovery, and its effect on process recycle solvent needs to be studied further.

Simulated distillations of aged samples

The SRC-FO samples which had been aged for 10 months generally showed higher boiling points for a given cut point (% distilled overhead). For example, the sample stored at 140°F required a temperature of 257°C for 50% distillation, compared to 210°F for a sample stored under the same conditions for 6 months. The UFO 50% cut point remained fairly steady at $\sim 270^\circ\text{C}$.

Viscosity-temperature dependence of SRC-UFO from the Ft. Lewis SRC plant

The dependence of viscosity of SRC-UFO from the PAMCO pilot plant on temperature was measured using a Cannon-Fenske viscometer. Generally, the viscosity was very similar to that of a sample from the Wilsonville, Alabama plant (reported in ORNL/TM-4968). For instance, at 150°C the PAMCO SRC-UFO viscosity was 53.5 cP compared to 56.3 cP for the Wilsonville material. For temperatures greater than 150°C, the PAMCO material had higher viscosities, and at process conditions (500°F) the viscosity was twice that of the Wilsonville material (6 cP compared to 3 cP).

3.2 Laboratory-Scale Tests

B. R. Rodgers and D. A. McWhirter

Solids content of agglomerated SRC-UFO

Last month the dry solids content of SRC-UFO, after the addition of 20% *n*-decane and 20% toluene at room temperature, was reported as 17.8 and 8.3 wt %, respectively. This should be compared to 6.8 wt % for neat UFO. Additional analyses using the same 1.2 μ Millipore filtration technique gave values of 8.8 and 6.8 wt % for SRC-process-derived light oil and wash solvent, respectively. Differences were more pronounced at high solvent-to-oil ratios. For S/O = 12/1, *n*-decane precipitate was 35 wt % of original UFO, light oil and toluene were 18%, and wash solvent remained at 6.8 wt %.

3.3 Bench-Scale Tests

B. R. Rodgers and D. A. McWhirter

Two additional settling runs at 530°F using a 200-300°F cut of light oil and a 400-500°F cut of wash solvent as 20% diluent in SRC-UFO gave results similar to those reported in the annual interim report (ORNL-5208). For instance, the time required to obtain 70% by volume clarified overflow was 1.4 and 2.2 hours, respectively, for the two cuts above, compared to 1.4 and 2.0 for light oil and wash solvent.

3.4 Dissemination of Information

B. R. Rodgers

A presentation and discussion of mutual results was made at Gulf Research and Development Company on August 16 and at Conoco Research and Development on August 17. The meeting at Conoco was particularly useful since representatives of Fluor, Inc., PAMCO, and ERDA were present.

A project review meeting and discussion of fiscal year 1977 plans was made at ERDA Headquarters and included members from DCCU. The interaction was very fruitful and it is anticipated that this type of meeting will become more frequent.

4. EXPERIMENTAL ENGINEERING SUPPORT OF AN IN SITU GASIFICATION PROCESS

R. C. Forrester III

4.1 Large-Block Pyrolysis Studies

R. C. Forrester III and G. D. Owen

Investigation of two-dimensional pyrolysis effects has continued this month with tests conducted at heating rates of 0.3 and 3.0°K/min to a temperature maximum of 1273°K. These tests complete the matrix of temperatures and heating rates to be studied at atmospheric pressure under inert atmospheres. A few repeat tests are planned in the coming months, as well as some "special-conditions" experiments designed to elucidate specific pyrolysis mechanisms which are thought to be taking place.

4.2 ERDA's UCG Program

R. C. Forrester III

The 2nd Annual Underground Coal Gasification Symposium, sponsored by the Morgantown Energy Research Center, ERDA, was held at the Lakeview Inn and Country Club, Morgantown, West Virginia, August 10-12. Every participant in UCG development in the United States was represented, as were a number of international organizations. Much current information was presented both formally and informally, and recent results of the ORNL experimental program were summarized in a paper entitled, "Two-Dimensional Studies of Coal Pyrolysis: Preliminary Results." The entire proceedings of the meeting will be published and are expected to be available by December. Reprints of the ORNL paper are already available.

5. ANALYTICAL CHEMISTRY

W. R. Laing

5.1 Analytical Services

L. J. Brady

During the month a total of 145 samples were submitted for analysis. A total of 97 samples from the solids-liquid separation program were tested the first week of this period. Since then no samples of this type have been received.

Two samples of coal-derived liquids were submitted for the determination of the simulated boiling range.

Three sets of samples comprising a total of 56 samples were tested for the concentration of CO, N, and CH₄. In addition, six of these samples were tested for CO₂ content.

A molecular sieve column is used to separate CO, N, and CH₄. A separate portion of the sample is separated on a silica gel column in order to determine CO₂.

6. ENGINEERING STUDIES AND TECHNICAL SUPPORT

J. R. McWherter and R. Salmon

6.1 Synthoil Process

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

The engineering evaluation of the Synthoil process was completed, and the draft report was issued on schedule. Copies of the report were submitted to ERDA/FE, Fossil Demonstration Plants Division. Preparation of a final report will proceed after receipt of comments from ERDA/FE.

The estimated overall capital investment for the 100,000 B/SD Synthoil facility is \$1897 million, consisting of \$1668 million depreciable and \$229 million nondepreciable capital. The cost of the coal mine is not included. Coal delivered to the plant is considered as an operating expense, and its cost is treated as a variable in the economic analysis. Interest during construction is not included in the capital investment, but is accounted for in the calculation of the product price. All costs are on a first-quarter 1976 basis.

Using the discounted cash flow procedure, the price of product oil was calculated as a function of coal cost and annual after-tax rate of return on equity capital. This was done for four capital structures ranging from 100% equity to 95% debt, using an annual interest rate on debt of 9%. By-product credits were included for sulfur at \$60/long ton and anhydrous ammonia at \$160/ton. Results were based on a federal income tax rate of 48%, a state income tax rate of 3%, and a federal investment tax credit of 7%. Project duration was 25 years, consisting of a 5-year construction period followed by a 20-year operation period.

6.2 Hydrocarbonization Process

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

A preliminary design and an economic evaluation of a hydrocarbonization facility based on U. S. Steel Corporation's Clean Coke process is completed and two reports have been issued. Volume I includes the conceptual design and cost estimate of a commercial facility for producing clean char, oil, and pipeline gas fuels having a heating value comparable to 100,000 bbl of fuel oil per day. The conceptual design includes a complete facility that will: (1) prepare the coal for hydrocarbonization in a beneficiation system; (2) hydrocarbonize a fraction of the beneficiated coal to solid, liquid, and gaseous fuel products; (3) provide energy to the facility by burning the remainder of the beneficiated coal in a fluidized-bed combustor; (4) hydrotreat the liquid products using hydrogen produced internally, and (5) treat the plant effluents to comply with regulations concerning disposal. The total capital investment, including initial working capital, initial raw materials, catalysts and chemicals, and land, is about \$1019 million. A discounted cash flow analysis assuming 100% equity financing, 12% return on equity, and coal priced at \$20/ton resulted in a product fuel price of \$3.15/million Btu. An alternative process where the coal liquids were marketed without hydrotreatment or filtration resulted in a product price of \$2.77/million Btu.

Volume II of a two-volume study concerning the preliminary design and economic evaluation of a hydrocarbonization facility includes: (1) a review of the current status of the major processing units, (2) an assessment of operating problems, (3) considerations of possible process alternatives, (4) an evaluation of the overall process feasibility, and (5) recommendations for future process development. Results of the study emphasize the need for testing the evaluated process, which is based on the Clean Coke process, in a continuous pilot plant using a wide variety of highly caking bituminous coals as feed material. A program suggested for the pilot plant would encompass: (1) development of improved methods for the prevention of agglomeration of highly caking coals during hydrocarbonization, (2) optimization of the yields of coal liquids, (3) investigation of a single-stage high-temperature hydrocarbonizer optimized for char production, and (4) optimization of beneficiation ratios employed during coal preparation.

6.3 Process Modeling Support

R. Salmon, D. S. Joy, J. K. Huffstetler

This project is a joint effort involving Purdue University, Lehigh University, and ORNL. The overall objective is to develop computer programs for the design and cost estimation of coal conversion processes and facilities. Purdue is concentrating on the development of steady-state mass and energy balance process design models; Lehigh on unsteady-state models. ORNL's computer facilities will be used for implementing the Purdue and Lehigh programs. ORNL will also help in developing, augmenting, and updating the programs, especially in the areas of equipment design and cost estimation.

A list was provided to ERDA/FE, Purdue and Lehigh Universities of all reports in the ORNL Coal Technology Information Center. Assistance will be provided by ORNL in obtaining information as necessary.

6.4 Process Research Digest

F. J. Endelman

Work on this project was initiated and details of the work statement confirmed in August.

Much of the organizational groundwork for publishing a triannual Process Research Digest (PRD) was accomplished in this reporting period. The Principal Investigator will serve as Editor of the Digest. He will supervise the work of an ORNL research staff member serving as a full time technical writer, and a production team from ORNL's Technical Publications staff.

The PRD staff is finalizing a publication schedule and is awaiting distribution by DFER of a letter of introduction to appropriate DFER contractors. This should greatly facilitate accumulation of the information to be reported upon in each issue of the Digest.

7. COAL-FUELED MIUS

A. P. Fraas and W. R. Nixon

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.

7.1 Furnace Procurement

Bids for fabrication of the furnace and tube bundles were received from two vendors. The bids are being evaluated for possible placement of the order upon receipt of additional funds. The present bids are valid until October 18, and the order will have to be placed by then to be assured of the bid price.

Consideration is being given to seeking bids on the basis of performance specifications with each vendor providing their own design. This has the potential advantage of providing a more economical and more nearly commercial design. Primary disadvantages would be delays in procurement and the need for additional design work.

7.2 Turbine-Generator Unit

Analysis of the existing electronic control package and design of instrumentation and controls for the turbine continued.

7.3 Site Preparation

The drawings for relocation of existing equipment were nearly completed. The decision was made to enlarge the control room and drawings for that change were near completion.

7.4 Cold Flow Tests of a Fluidized Bed

Fluidizing tests were completed and heat transfer tests are in progress using smaller sized limestone in the small cold flow model. The results indicate a considerably lower fluidizing velocity and a higher heat transfer coefficient than had been expected. Performance over a wide range of fluidizing velocity is yet to be determined.

7.5 Coal Metering and Feed System

Endurance running of the flow splitter feed system continued at full design power coal feed rate. The system has been operated for a total of 175 hr and no operating problems have been encountered.

Endurance testing on a prototype coal feed nozzle was continued. The nozzle has been tested for a total of 95 hr with no observed difficulties.

Drawings are in preparation for modifications that will allow continuous operation of the flow splitter feed system with the objective of running a 1000-hr endurance test.

7.6 Ash Handling System

Work is proceeding on ash handling system design based on the option of procuring individual components.

7.7 Supplemental Studies

About half of the corrosion test specimens are ready for shipment to FluidDyne. The balance of the specimens include Alonized sections which will be prepared early next month.

Heat transfer tests in the FluidDyne furnace were completed this month. The data will be analyzed and the report prepared next month.

The first draft of the report on system performance with alternate cycles has been completed.

8. 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/FE is included also.

Summary

Chapters of the Pressure Vessel and Piping Assessment have been reviewed by ERDA/FER, and we are including recommended changes to our manuscript. Revised chapters are being returned to FER for additional review.

We have prepared executive summaries for a forthcoming ERDA Materials Meeting, and they have been transmitted to ERDA/FER.

In our Inspection Techniques for Wear and Process-Resistant Coatings Program, our first coating thickness standards have been coated with a single layer ceramic (ZrO_2) and returned to the shop for final machining. Free-standing specimens of ZrO_2 (for properties determination) have been prepared by spraying on a smooth Ni substrate, from which the full coating spalls, almost in one piece, upon cooling.

We have initiated procurement of an x-ray fluorescence measurement system similar to one used at Y-12 for preliminary work. Thermal computer code calculations are continuing. We are now preparing to generate thermal profile curves to show the response to an unbond flaw. We are assembling experimental equipment to verify the computational results. We have made preliminary eddy current measurements on free-standing coating layers. Initial stressing of the crack specimen for penetrant testing has been done; stressing at higher levels will be required. A scanning device to control spacing has been built for high voltage probe experiments.

In our Prestressed Concrete Vessel Studies, detailed analysis work is continuing in order to finalize the conceptual designs. Initial vessel drawings are for Synthane. Studies continued on the materials for a two-layer refractory liner system. Difficulties in conducting the thermal analyses for the liner-vessel interface are being overcome. Several methods were examined for mechanically holding the refractory liners in place and to transfer lever loads to the vessel. A double-anchoring system using stainless steel anchors appears to be the best choice.

8.1 Pressure Vessel and Piping Technology Assessment

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

This Assessment Program will identify those areas where additional material property data needs are required for pressure vessels and piping. The goal is to assure that the pressure vessels and piping in conversion systems can be designed, fabricated, and operated in a safe and reliable manner.

Fossil Energy Research (FER) has reviewed some chapters of the report, and their suggestions are being incorporated in the report. The Materials Selection chapter has been revised and will be transmitted to FER. Executive summaries for the forthcoming ERDA Materials Meeting were prepared and transmitted to ERDA/FER.

8.2 Inspection Techniques for Wear- and Process-Resistant Coatings

R. W. McClung and G. W. Scott

8.2.1 Review and Evaluation (G. W. Scott)

An executive summary of the current status of coating inspection work was prepared for the AGA-EPRI-ERDA Materials Meeting (Washington, D. C., September 30-October 1, 1976) and mailed on August 13. A set of posters (3) is being prepared for exhibition at the Annual ORNL Metals and Ceramics Division Advisory Committee Review and Information Meeting on September 15 and 16. The latter part of this meeting will include invited guests from other institutions and the technical press.

8.2.2 Specimen and Standard Fabrication (J. D. Hudson and D. P. Edmonds)

The Welding and Brazing Laboratory is preparing plasma sprayed coatings to be used for development of nondestructive examination (NDE) techniques. We have sprayed several ZrO_2 coatings on Incoloy 800 substrates, with no bond coat. The following samples have been prepared:

- (a) 0.254-mm (0.010-in.) thick coatings on 3.175-mm (0.125-in.) thick Incoloy 800;
- (b) 0.127-mm (0.005-in.), 0.254-mm (0.010-in.) and 0.381-mm (0.015-in.) thick coatings on thickness standards (Incoloy 800 substrates); and
- (c) 0.254-mm (0.010-in.) thick free-standing coatings for eddy current evaluation.

Uniform thickness standards are obtained by spraying slightly thicker than desired coatings on accurately machined substrates and surface grinding to the desired thickness. Free-standing samples (no substrate) are obtained by spraying directly onto a nickel substrate, from which the coating layer easily spalls away. Presently, we are preparing sprayed coatings using NiCr and NiCrAl bond layer materials.

Since surfaces to be plasma sprayed must first be sandblasted, we are investigating the effects of this sandblasting on the substrate. We found that when a minimum amount of sandblasting is performed, the apparent substrate thickness increases by 0.025-mm (0.001-in.) for Incoloy 800. We are performing a metallographic examination to observe the effects of sandblasting on the microstructure and to determine if a dimensional correction is required. Also, we are investigating the effects of different spraying techniques on the coating density.

8.2.3 Radiation Testing (B. E. Foster)

We have initiated procurement of an x-ray fluorescence measurement system similar to that which was used earlier at the Y-12 plant. Our system will have higher sensitivity and better resolution than the Y-12 system, and will include some additional features which will be useful in development of advanced techniques.

8.2.4 Thermal Testing (W. A. Simpson, Jr.)

We are continuing to generate computer data on the propagation of heat in the coating-substrate system for the evaluation of bond quality of resistant coatings. The results to date indicate that thermal testing should be a viable technique for evaluating the bond integrity of the coating. We are attempting to generate curves which will give the thermal contrast (defined as the temperature difference between bonded and unbonded regions under identical heat injection conditions) as a function of time and heat injection rate. This will allow us to optimize the heat input to the sample and to determine the appropriate time "window" in which to measure the surface temperature distribution.

In order to test these computer predictions, we are assembling the equipment necessary to experimentally evaluate the samples. Because of the very rapid development and dissipation of the non-bond thermal profile, as predicted by the computer, a fast response detector will be necessary to capture the transient. We have such a detector in the form of a liquid nitrogen cooled infrared camera that can be operated as a point source radiometer. We are also investigating the possibility of using a very low mass, fast response thermistor, which we have on hand. Suitable heat injection sources are also being investigated with top priority going to a plasma jet torch.

8.2.5 Eddy Current Testing (C. V. Dodd)

We have successfully made free-standing layers of sprayed ZrO_2 in the 0.010-0.020-in. range of thickness. Preliminary measurements indicate very low conductivity, just slightly above that of ambient air. By comparison, a high-purity Al_2O_3 circuit substrate had a conductivity slightly less than that of air.

8.2.6 Liquid Penetrant Testing (S. D. Snyder)

The first artificial-crack specimen coated with ZrO_2 for liquid penetrant experiments was stressed in a tensile machine to a load of 2850 lb. After the stress was removed, the specimen was inspected both visually and with penetrant for the formation of cracks. No indications of cracks were observed either visually or with the penetrant, so the specimen will be stressed to a higher load, 3500 lb, and inspected again.

Further experimentation was done during the penetrant inspection of this stressed specimen with a reduction in penetrant dwell time from 10 min. to 5 min. A comparison of the rate of removal of the excess penetrant by the solvent-removal method for the two dwell times indicates that 5 min. is too long for this extremely porous material. Future penetrant tests will be done with dwell times in the order of 1 min.

8.2.7 High Voltage Probe (G. W. Scott)

We have built a simple-scanning device for use with the high voltage probe. This scanner will provide precise spacing control between the probe and the substrate.

8.3 Iron and Nickel Carbonyl Formation and Prevention

J. Brynestad and J. H. DeVan

The valves for the experimental set-up were received, and the test apparatus for reaction rate studies is now fully assembled. Performance testing is underway. We were able to control temperature, pressure, and flow rate in the apparatus to the standards needed for our reaction rate determinations. Because of the safety hazards of metal carbonyls, we are being especially careful. An initial test is underway to measure the background rate of iron and nickel carbonyl formation relating to the final system itself, which is made of 316 stainless steel. Based on published data, we expect this background rate to be small but measurable. However, we don't expect the system to produce carbonyls at levels which would interfere with the testing of low alloy steels. If such should be the case, we will suppress the carbonyl formation by (1) oxidizing the heated surfaces of the apparatus, or (2) depositing copper electrolytically on the heated surfaces. The first test run will be conducted on 1/2% Mo

steel piping supplied by Pittsburgh Energy Research Center. The test will be operated at 260°C and 1000 psig, with the gas mixture being 30% CH₄, 51% H₂, 17% CO, and the balance CO₂.

8.4 Failure Analysis of Materials and Components

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

No work was performed this month. A topical report was written and is being edited covering prior work. The new procedures for conducting failure analysis were received from FER, reviewed, and our comments returned to ERDA within the ten days specified.

8.5 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.

Emphasis has continued on detailed analysis work necessary for conceptual design finalization. Intimately tied to these analyses are materials selections and geometrical details in the refractory-liner-vessel interface region.

Under Task 1, drawings of vessel conceptual designs incorporating details of the top closure, circumferential and vertical prestress systems, bonded reinforcement, penetrations, and vessel support structure are being prepared. The Synthane vessel drawings are being done first; completion of a number of layout details for the Synthane as well as the HYGAS vessel will depend on results from calculations now under way.

Thermal stresses in the liner are being examined along with the consequences of cyclic loading. Effects of local gradients in the anchor attachment regions are to be included in these investigations.

Under Task 4, materials for a two-layer refractory lining system selected for initial heat transfer investigations were given further study. Thermal conductivities for each of the materials (the dense, abrasion resistant inner material and the backup insulating material) were calculated assuming air in the pores is replaced by hydrogen. Extremes in porosity were used in calculating these conductivities. A double-anchoring system using stainless steel anchors appears to be the best choice for mechanically holding the refractory in place and transferring load to the vessel.

Difficulties in conducting thermal analyses for the refractory-liner-vessel interface region have impeded overall progress. These difficulties are being overcome, and improved progress is expected to result.

8.6 Other Related Work

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

8.6.1 Fluidized Bed Materials Support Activities

Nothing to report.

9. PROCESS AND PROGRAM ANALYSIS

J. R. McWherter

Process and program analysis studies are being conducted for the ERDA Fossil Energy Office of Program Planning and Analysis. This effort includes research studies on most of the coal conversion and utilization processes. The program objective is to provide, on a consistent basis, technical and economic evaluations of competing processes and systems for coal conversion and utilization.

The subprograms under review are:

- (1) Low Btu Gas
- (2) High Btu Gas
- (3) Direct Combustion
- (4) Liquefaction
- (5) Advanced Power/Combined Cycle
- (6) In-Situ Coal Gasification

Also included are general subjects such as beneficiation and gas cleanup.

A survey will be conducted to identify the applicable processes, concepts and systems. The survey will cover not only activities in the United States but in other countries as well. The status of development shall be determined in sufficient detail to identify the promising processes, concepts, and systems.

A list of high potential, or candidate, processes, concepts and systems, including all of those currently being funded by ERDA, will be prepared from the above survey. These candidates will be considered for detailed study. The most favorable process for each subprogram will be determined on a regional basis. The potentially competitive processes will be identified with an evaluation of what needs to be done to make them the most favorable and what the chances of success are of such proposed actions.

9.1 Low Btu Coal Gas

J. P. Belk, B. L. Crynes,* H. F. Hartman, D. E. Reagan

ORNL personnel recently began a program to provide technical and economic evaluations of processes that convert coal to low and intermediate fuel gas. The following activities were completed: (1) project definition, (2) proposed method for economic comparison, and (3) outline of a Data Source Book. Work is in progress now to survey the gasification processes and select the more promising ones for detailed comparisons and evaluations. Program objectives and required information were reviewed with ERDA-FE-OPPA personnel at an August 30 meeting.

*Consultant, Oklahoma State University.

9.2 High Btu Gas

This project will be subcontracted.

9.3 Direct Combustion

H. I. Bowers, O. L. Culberson,* I. Spiewak

A preliminary work statement and discussion of this subject was submitted to ERDA for review.

9.4 Liquefaction

A subcontract for this project is being negotiated.

9.5 Advanced Power/Combined Cycle

A. P. Fraas, M. E. Lackey, G. Samuels, W. M. Wells

A large amount of information is being collected on the development of gas turbines. A more thorough look is being taken on the use of coal in gas turbines.

9.6 In-Situ Coal Gasification

R. C. Forrester III and W. C. Ulrich

A preliminary work statement on this project was submitted to ERDA for review. Work is in progress to determine the status of the processes.

9.7 Subsystems

O. L. Culberson*

9.7.1 Beneficiation (G. W. Peterson)

A work statement for this project is being developed.

9.7.2 Gas Cleanup (M. S. Edwards)

A work statement was prepared for this project. A review of the information available is in progress.

* Consultant, University of Tennessee.

10. ALTERNATE CONTACTORS FOR NONCATALYTIC HYDROLIQUEFACTION OF COAL

J. R. Hightower, Jr., and R. C. Lovelace

Improvements in hydrodesulfurization and hydroliquefaction of coal in hydrogen donor solvent systems can possibly be achieved by improving the design of the reactor in which the hydrogenation is carried out. Results obtained in the development of the Synthoil process^{1,2} at Pittsburgh Energy Research Center (PERC) suggest that under conditions of high turbulence and plug flow in a reactor, adequate desulfurization and liquefaction may be achieved utilizing only the catalytic activity of the ash minerals. The objective of the work reported here is to demonstrate that a highly turbulent plug flow reactor of novel design would allow adequate hydrodesulfurization and hydroliquefaction without the use of hydrotreating catalysts. The work is divided into two phases: (1) simulated contactor studies which have the objective of identifying suitable reactor configurations (we will investigate a reactor consisting of several venturis in series and one with vortex flow similar to a cyclone device) through tests with water/coal slurries and air, and (2) hydrogenation verification studies in which coal is hydrogenated in a suitable reactor whose design would be based upon results from the first phase studies.

Progress on the first phase (simulated contactor studies) since the start of the project (in July 1976) has consisted of a review of the literature for flow characteristics of venturis operating with gas-liquid or liquid-solid two-phase flow and the design and start of installation of a facility to circulate water/coal slurries through a three-phase contactor and to measure pressure drop across and residence time distributions in the contactor.

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