COLLECTED WORK NO. 35

MATERIALS, NEEDS, OPPORTUNITIES AND PROBLEMS ACCORDING TO THE DESIGNER

W.C. Raisbeck

THE RALPH M. PARSONS COMPANY
PASADENA, CALIFORNIA

Presented at
ERDA, EPRI & AGA
CONFERENCE ON MATERIALS FOR
COAL CONVERSION AND UTILIZATION

GERMANTOWN, MD.

September 29

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W.C. Raisbeck
The Ralph M. Parsons Company
Pasadena, California

The Ralph M. Parsons Company is actively assisting the Energy Research and Development Administration (ERDA) in its program to develop viable commercial plants for the conversion of coal to clean fuels. There are two distinct parts involved in this role:

1. Preliminary design services in which Parsons develops conceptual designs and economic evaluations for commercial coal conversion plants. At present we are developing designs for a Fischer-Tropsch plant, an Oil/Gas plant, a Coal-Oil-Gas (COG) multiproduct facility, a commercial solvent refined coal (SRC) facility and a multi-unit demonstration plant. Each of these designs will include captive coal mines.
2. Parsons also supplies technical evaluation services to assist ERDA in monitoring certain liquefaction development programs.

Equipment

I have been asked to speak today on material needs from a designer's viewpoint. The treatment of materials problems for the designer is an extremely broad subject. In an effort to condense the subject into the available time, we will only be able to give detailed attention to limited areas of design. Each designer's point of view is dependent upon a specific process or area of work

All conversion processes will require a gasifier in producing a liquid or gaseous product from coal. Because of the primary role of the gasification program, we will concentrate on this program's material problems. Within the area of gasification, the material closest to the process is the refractory. We will place our greatest emphasis on the design problems of refractories today.

Various conversion plants have been proposed, including one which could consume up to 100,000 tons of coal per day. These quantities may seem at first a little overwhelming, but a closer analysis reduces many of the problems to those of logistics and scaling up of existing equipment which will not require extensive research into materials.

For a gasification system to be economically justifiable, a high degree of reliability must be developed. Failure to produce a plant with high reliability would result in system redundancy at very high cost. Usually 90% or higher onstream availability is set as a goal. To provide the required availability, we need to know the most probable failure point for each piece of equipment in the system. Knowing the probable failure points enables the designer to provide the plant operators with acceptable procedures for handling the equipment outage. We must either have equipment designed for replacment of parts "on-the-fly" or with a high operating factor built in. Let's look at the individual components of the system requiring materials development.

Coal Handling and Preparation Areas

With major steel and power companies currently processing up to about 33,000 tons of coal per day at one site, the proper equipment is available now from the designer's viewpoint. The coal handling and preparation systems will only require that slightly oversized equipment be provided to allow stockpiling during normal operations such that the plant can run on stockpiled coal during any outages.

Charging Systems

Two basic methods have been developed to introduce the coal in to high pressure systems (slurry and lock hoppers).

1. Slurry Systems

In slurry systems the coal is pumped to pressure with a carrier liquid, normally oil or water. Once pressurized, the coal slurry may be injected directly into the gasifier or may be dried prior to injection.

Slide 1 is a slurry with Typical Flash Dryer
Slide 2 is a slurry with Fluidized Bed Dryer
Slide 3 is a slurry with Lift Dryer

The problems of pumping abrasive coals up to the pressure are well recognized and considerable work is being done to solve these problems.

2. Lock Hoppers (Slide 4)

Lock hoppers, which pressurize the coals directly or in stages, will present unique materials problems. High differential pressures must be maintained across seals and valves that handle tons of abrasive coal while maintaining the required operating factor. Considering a commercial system which must handle over 30,000 tons of coal per day, the materials community cannot be asked to provide the sole means necessary to produce the 90% availability required. The equipment designer, working with the materials engineer, must develop equipment with the highest operating factor economically possible. With the lock hopper system it may be necessary to provide some redundancy within the system to allow quick replacement of high wear parts (valves, seals, etc.) without interrupting the process.

Of the two systems currently getting most of the attention, slurry systems may be more reliable because of the development work done on commercial coal slurry transport programs and the work underway at the pilot plants.

A third system, direct mechanical injection, may be favored when developed, due to possible energy savings over slurry systems which require energy for vaporization of the slurry medium and over lock hoppers due to flow monitoring problems.

Injection Systems (Slide 5)

A second, and possibly as great a problem as the charging system for some processes, may be the final injection of coal, steam and oxygen into the gasifier. The injection step may produce as many material problems as the pressurizing systems for a commercial unit. In the steel mill blast furnace all the air nozzles (blow pipes into tuyeres) are designed for quick replacement. Quick change injection nozzles on the gasifiers may be a major design requirement.

Gasifier

The gasifier, because of cost and size, should ultimately be the piece of equipment in the process train that limits the length of the run or campaign. Because of this, the gasifier must have a high operating factor designed into the unit. The gasifier vessels vary greatly depending upon which process is employed. The steel vessels being designed today follow ASME, Section VIII, Division 1 or Division 2. There are some questions concerning suitability of these two sections and this point will be reviewed in a later discussion.

The success of a commercial gasifier will depend upon the correct blending of vessel materials, cooling system, and refractory selection. Pilot plant experiences have been helpful in developing pumps, valves, and other equipment items, however, these experiences have not necessarily been meaningful in understanding a refractory system for a commercial gasifier.

Pilot plant vessels are small, castable lined, and have run only intermittently for short duration.

A number of factors must be considered in the gasifier design: metals to stand up to corrosive condensates, erosion at high temperatures, thermal efficiency of the wall design, and the residual lining thickness of refractory. Hopefully, we will be able to use materials which are commercially available at reasonable cost.

The designer does not have the freedom to consider materials and their problems independently, but must employ materials to build a unit which will meet all the needs of the total gasifier environment. The gasifier is a piece of equipment operating at high temperatures with a corrosive and erosive environment. Of all the materials in the gasifier, the refractory lining will be the component to come directly in contact with the severe environment. We would like to take a closer look at the refractory systems and their influence on wall design. One point of view presented by Koenig and represented by the "Residual Thickness of Refractory Lining of Blast Furnace Bosh" equation, tells use that we must consider the cooling as well as threshold temperature of reactions between the lining and some portion of the coal ash or slag. Research on slag refractory erosion traditionally has not incorporated the value of water cooling and may not be valid for those gasifier designs which will be water cooled. Laboratory experiments must not only reflect the environment but also must consider the wall design with the water cooling if used.

Slide 6

The equation, in its present form, is not completely correct for gasifiers. Each wall design requires a separate modification to the equation; however, Koenig's form of the equation is useful in examining the value of water cooling. The second half of the equation represents the resistance to heat transfer for the heat enclosure. The term $1/a_2$ represents the resistance of the cooling water (which may be vaporizing). As this term gets larger, as it would in a system which relied on the outside air for cooling, the equation gives progressively thinner lining at equilibrium until it predicts failure. The equation presents a strong case for water cooling of refractories even for temperatures which are normally considered to be less than the maximum service limits for the refractory. Most designers to date have only considered the original wall design and not the equilibrium lining which will represent the real life situation.

Slides 7 and 8

The steel mill blast furnace represents a piece of equipment that is water cooled even though much of the refractory system is below 2000°F on the hot face. If we assume a low T_C, the equation predicts poor performance for systems which are not water cooled. Also, as others have pointed out, the residual thickness is directly proportional to the K factor if all other values are constant.

If equations of this type are to be used, we need considerable additional information. The value of some parameters currently are based largely upon speculation. The designer must have accurate values for the thermal conductivities of slags and various refractories. These are available, but there is some question as to their accuracy. More important, the values for T_C and T_S must be accurately defined and these values must come from the materials community.

Like all equilibrium equations, what the Koenig equation does not do is recognize reaction rates or other outside effects such as erosion and abrasion. It may be that the rate of reaction is sufficiently slow to render the residual thickness analysis unimportant. Until empirical data has been developed from operating units or lab tests which will accurately duplicate the environment, many designers will follow the conservative water cooling approach to gasifier design.

There is a definite condensation problem in high pressure gasifiers, and it is necessary to use either some weld overlay at approximately \$100/sq. ft. or a water cooling system which will allow you to predetermine the wall temperature and maintain it above dew point by controlling the cooling water temperature. In the case of some low pressure gasifiers, weld overlays or hot shells may not be the only choices available. The Steel Mill Blast Furnace represents a piece of process equipment which has an operating environment similar to a low Btu gasifier with a carbon steel shell maintained at temperatures well below the dew point. The similarities of the two units should be looked at closely by the materials people to see if long term experience has already been generated.

Slide 9

As mentioned earlier, we may not be defining the optimum refractory design for commercial gasifiers in today's pilot plants. To date, we would estimate that 80% of the literautre on refractories for coal gasification deals with castables; we question the emphasis being placed on the use of castables at this time. Let us consider corrosive and erosive conditions and how each will influence the designer:

A. Hydrogen
B. Steam
C. CO Disintegration
D. Slag
E. Alkali
F. Erosion

Slide 10

Two of these, hydrogen and steam, were previously considered problems only in steam methane reforming for the production of H_2. The reforming process is considered clean with respect to the other forms of corrosion. With gasification all of these forms of corrosion must be considered simultaneously.

We may have reached a point of apparent contradiction. Up to now most of the work has concentrated on the selection of the proper castables. For a commercial gasifier, not only must the correct chemistry be identified for the refractory, but also the correct physical form (brick or castable). Consider each of the corrosive forms and the direction a designer will be led in handling the problem.

Hydrogen - will attach SiO_2. This has been studied in detail by Crowley and the system is fairly well understood. There is some question concerning the reasons for selective attack by hydrogen on bricks but not on castables. Fortunately, this does not present us with a design problem. Hydrogen attack is effectively stopped if water vapor is present in small quantities. The gasifiers usually will have a H_2/H_2O mole ratio of 1.2 to 1.5 in the main gasifier, and under thse conditions we would not expect any problems from hydrogen attack.

Steam - as mentioned above, has the positive effect of suppressing hydrogen attack; on the other hand, steam itself may present a most unique design problem. Researchers have established a number of problems associated with steam corrosion:

1. SiO_2 leaching
2. Loss of strength of high alumina brick
3. Possible rehydration of calcium aluminate cements

All the information on steam is inconclusive. There have been few failures of refractories attributed directly to steam. Steam may have been responsible for attack on refractories but not properly identified as the cause. The biggest question is, how important is steam corrosion relative to the other corrosive gases in the system? We need not only understand steam as a mechanism, but must also establish a quantative measurement of corrosive rates. Related research should also try and identify any possible retarding agents which may be in the gasifier raw materials or may be added to them. Steam may favor castable as a designers choice.

CO - will react in the presence of iron impurities in the refractories to form CO_2 and carbon. The carbon builds up on the refractories, producing disruptive forces in the refractory body. $CO-CO_2$ has been studied for years by both the refining and steel industries. The guidelines for the designer have been quite simple:

1. Use refractory systems which are low in iron. The iron which is present must be in a nonreactive form (Fe or FeO being reactive).

2. The CO reaction with iron acting as a catalyst is easily poisoned by sulfur; the sulfur present in the coal may be sufficient to block this reaction.

Refractories formulated to keep the iron in a nonreactive form are available. The designer can use low-iron fired brick where the iron present has been reacted with the glassy silica phase of the brick. Castables on the other hand are produced in a crushing, grinding, and blending operation which allows iron impurities to be picked up during manufacture. During installation, the premixing and pneumatic placement adds additional iron to the refractory castable.

The $2CO = CO_2 + C$ reaction is one which is favored by high pressure. Most of the work has been done at low pressures; therefore, additional information should now be developed relative to CO problems in a high pressure unit. Brick would be favored over castable for resistance to $CO - CO_2$ attack.

Slags - have only been considered from an equilibrium point of view in our earlier discussion of residual lining thicknesses. The very important effect of erosion rates on refractories must also be discussed. The designer's guidelines requiring refractories with low porosity and low permeability at operating temperatures lead us to a brick design. Where slag must be contained, the designer will either use a frozen slag layer or brick compatible with the specific coal to be handled. Considerable work is still required in the area of slag erosion of refractories for various coals available in the United States.

Alkalis - from various sources entering the gasifier will present definite problems if they tend to concentrate in the unit. The Steel Mill Blast Furnace has contended with alkali problems for years. The solutions have affected both the design and operation of the furnace. Alkali bursting and fluxing must be studied relative to their effects on the gasifiers and ancillary equipment downstream.

Slide 11

Each of the above corrosive and erosive conditions will have an influence, but the conditions are specific for each gasifier and coal. The designer must be provided with a means of selecting the proper refractory to provide the optimum service for each set of conditions. The data should consist of empirical guidelines that can be applied directly to a given process. If design curves similar to the Nelson curves for hydrogen attack on metals could be developed for each important corrosive gas, the application of refractories in coal gasification service could be greatly simplified.

Slide 12

Erosion of refractories by particles in the gas stream in the main gasifier should not be a major problem for processes with low gas velocities (25 to 50 feet/second). Processes which employ a heat transfer material (dolomite, refractory balls, ash) requiring dense phase conveying may have erosion problems even with low velocity. In general, erosion resistance of refractories increases with increasing modulus of rupture.

It is still necessary to produce good quantitive correlation for specific chars and ashes. Bricks, with their high modulus of rupture, generally have excellent erosion resistance properties. Suitable erosion resistant materials are readily available and at reasonable cost. Improvement in our materials selection skills may be all that is required for the gasifier proper. The limits of each material must be known if the most economical design is to be produced. We are trying and must continue to try to optimize our designs considering both cost and erosion resistance.

The materials community can make a major contribution for the design of equipment downstream of the gasifier. Currently velocity of gases leaving the gasifier are usually in the range of 25 to 50 feet/second. The design velocity is limited by erosion resistance of lining materials and allowable pressure drop. The designer wants the most economical erosion resistant refractories giving the required service while using pressure drop limitation to control line sizing.

Though there are questions concerning the best choice of refractories when considering a commercial gasifier, bricks are favored over castable. We must define the optimum combination of chemistry, physical form and cast levels for the refractory system designers. High Fired Super Duty Bricks may prove to be the optimum refractory except for a few special cases.

Considering materials cost, Slide 13 shows High Fired Super Duty Bricks are favored when considering materials cost only. Installation cost of various refractory forms (brick, castables and plastics) must also be considered. The results of a cost study showed comparable cost for insulation of Brick and Castables in a Steel Mill Blast Furnace. Using a complete reline as the base case, no cost or time advantages could be shown for either brick or castable lining. The study is now dated and we should again take a close look at this point of economics for the design of commercial plants.

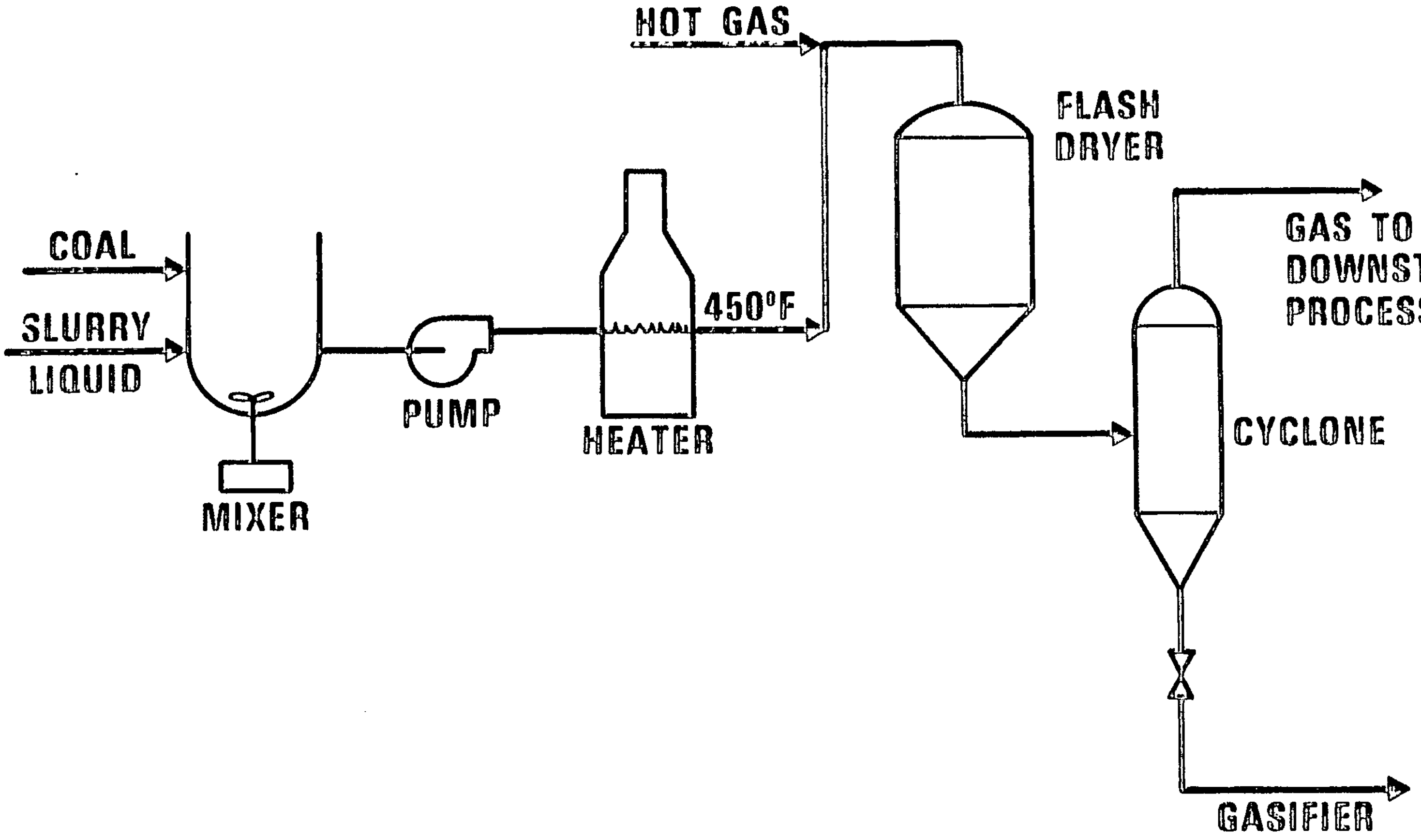
Ancillary Downstream Equipment

With some low Btu processes it would be advantageous to have a high temperature heat exchanger to maximize energy conservation. Ideally, all the raw materials will be preheated using the sensible heat from product gas.

In an air blown system we would want to achieve the highest air preheat temperature possible. Slide 14 represents the type of design anticipated. Metal heat exchangers may not have sufficient long-term creep strength required for this service. Ceramics could easily handle the temperature but a gastight system is also a major requirement. Keep in mind the heat exchange will be occurring between a high temperature fuel and air being heated to a high temperature. This exchanger represents a design challenge in materials engineering.

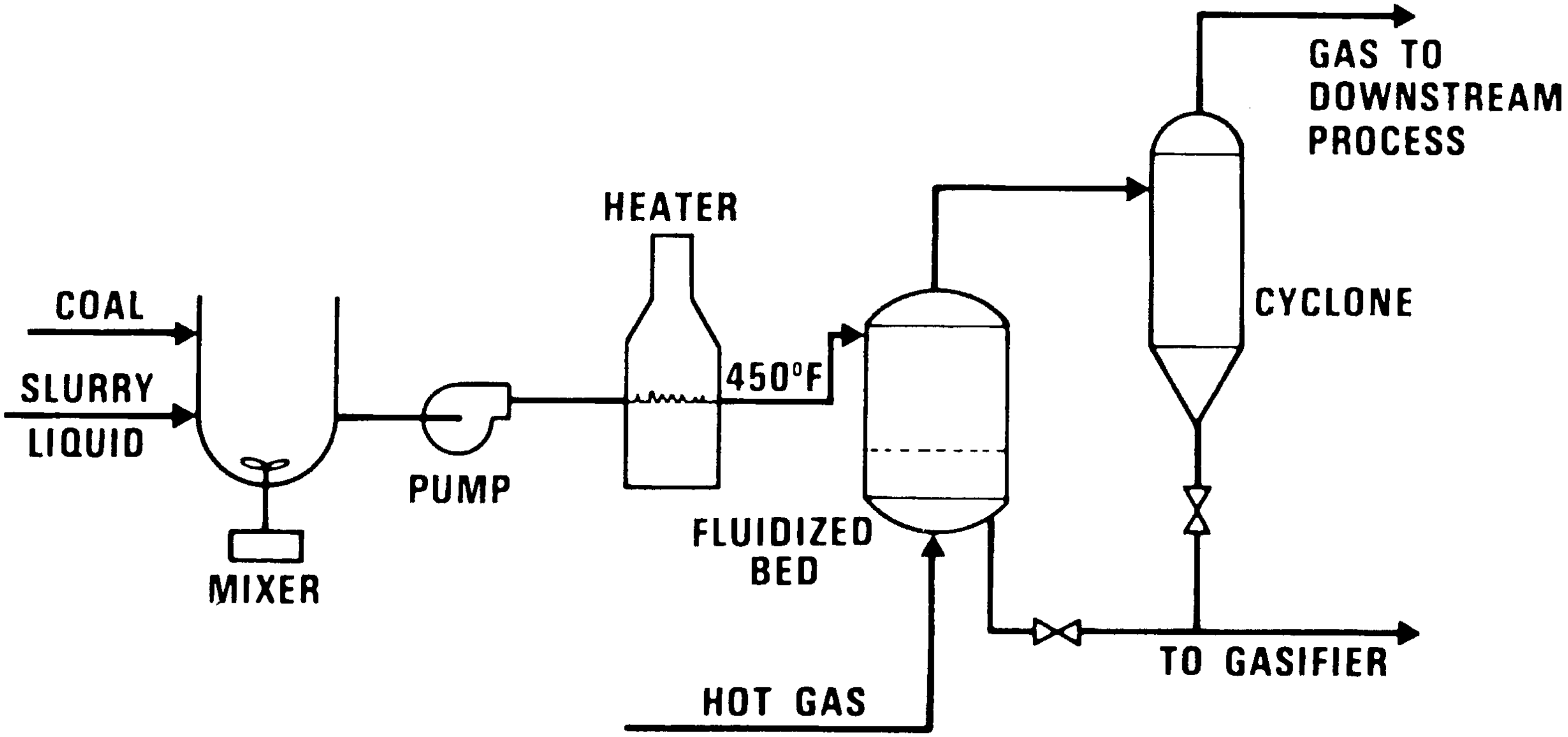
The gasifier and its ancillary equipment require ongoing major materials development. Other portions of the coal conversion system, gas purification, shift reaction and methanation, have already been research in petrochemical materials technology and will demonstrate the high reliability required with little need of further development.

SLURRY FEED WITH FLASH DRYER



SLIDE 1

SLURRY FEED WITH FLUIDIZED BED DRYER



COAL

SLURRY

LIQUID

MIXER

PUMP

HEATER

450°F

FLUIDIZED
BED

HOT GAS

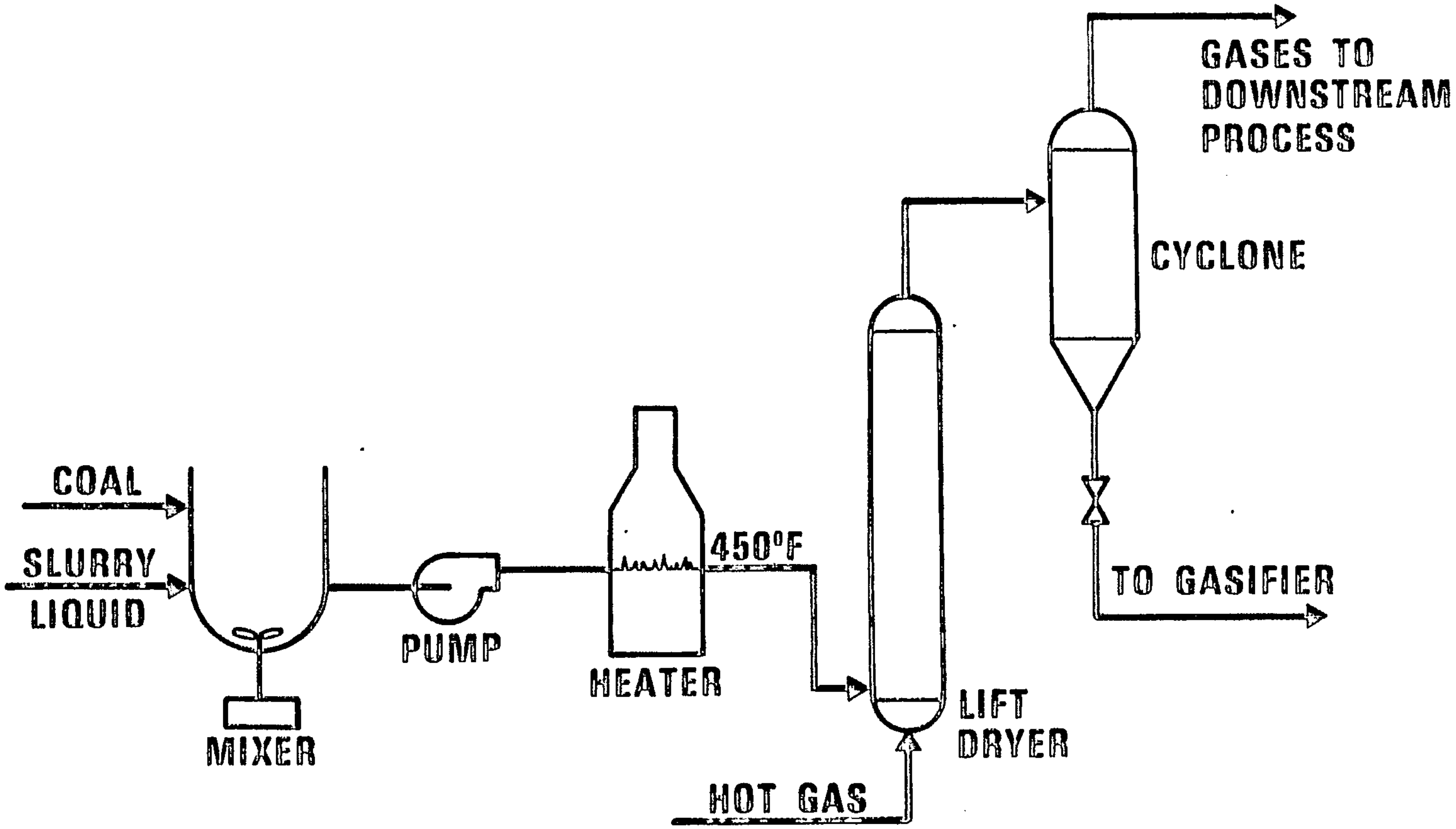
CYCLONE

GAS TO
DOWNSTREAM
PROCESS

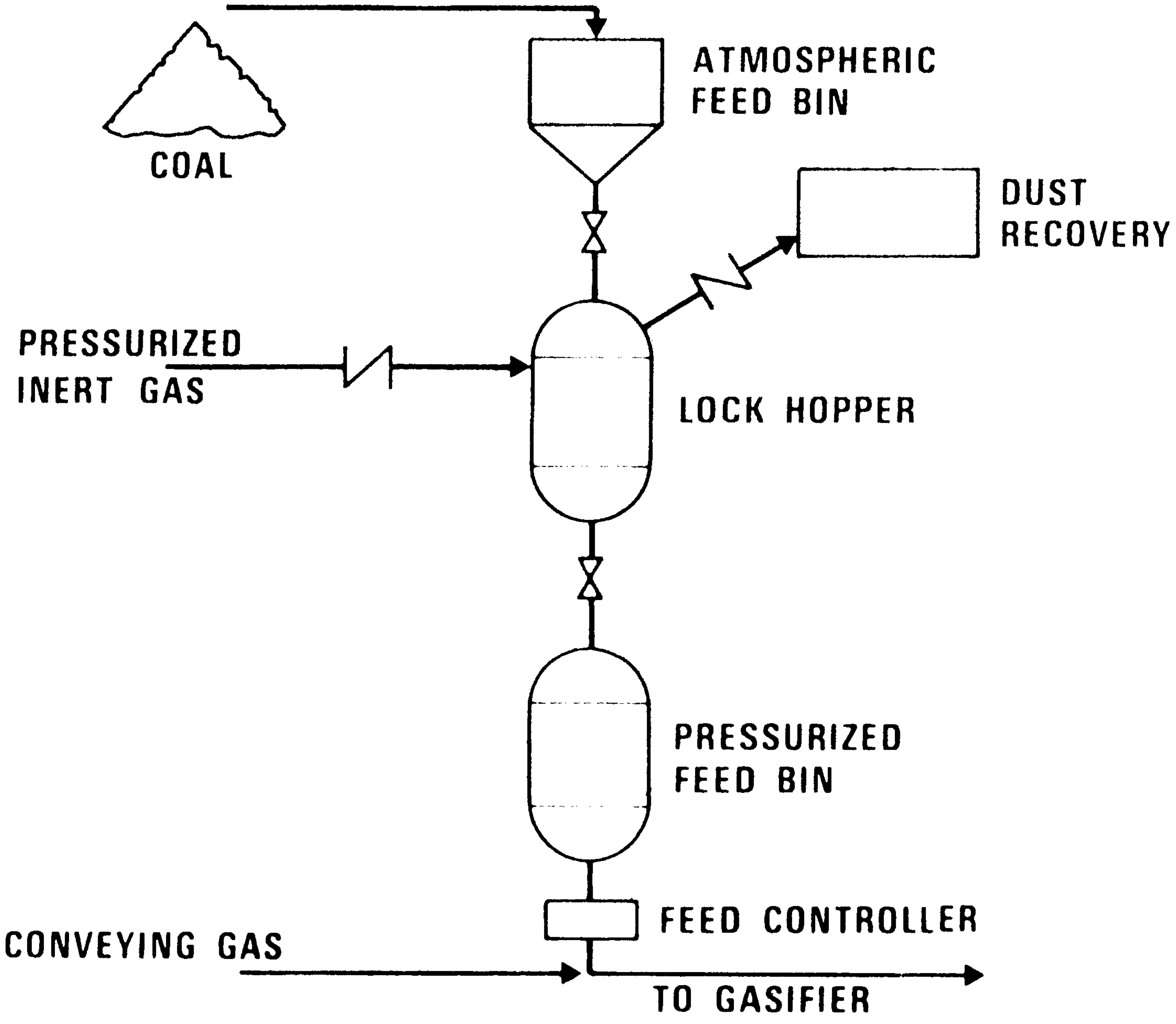
TO GASIFIER

SLIDE 2

SLURRY FEED WITH LIFT DRYER



LOCK HOPPER SYSTEM



COAL

ATMOSPHERIC
FEED BIN

DUST
RECOVERY

PRESSURIZED
INERT GAS

LOCK HOPPER

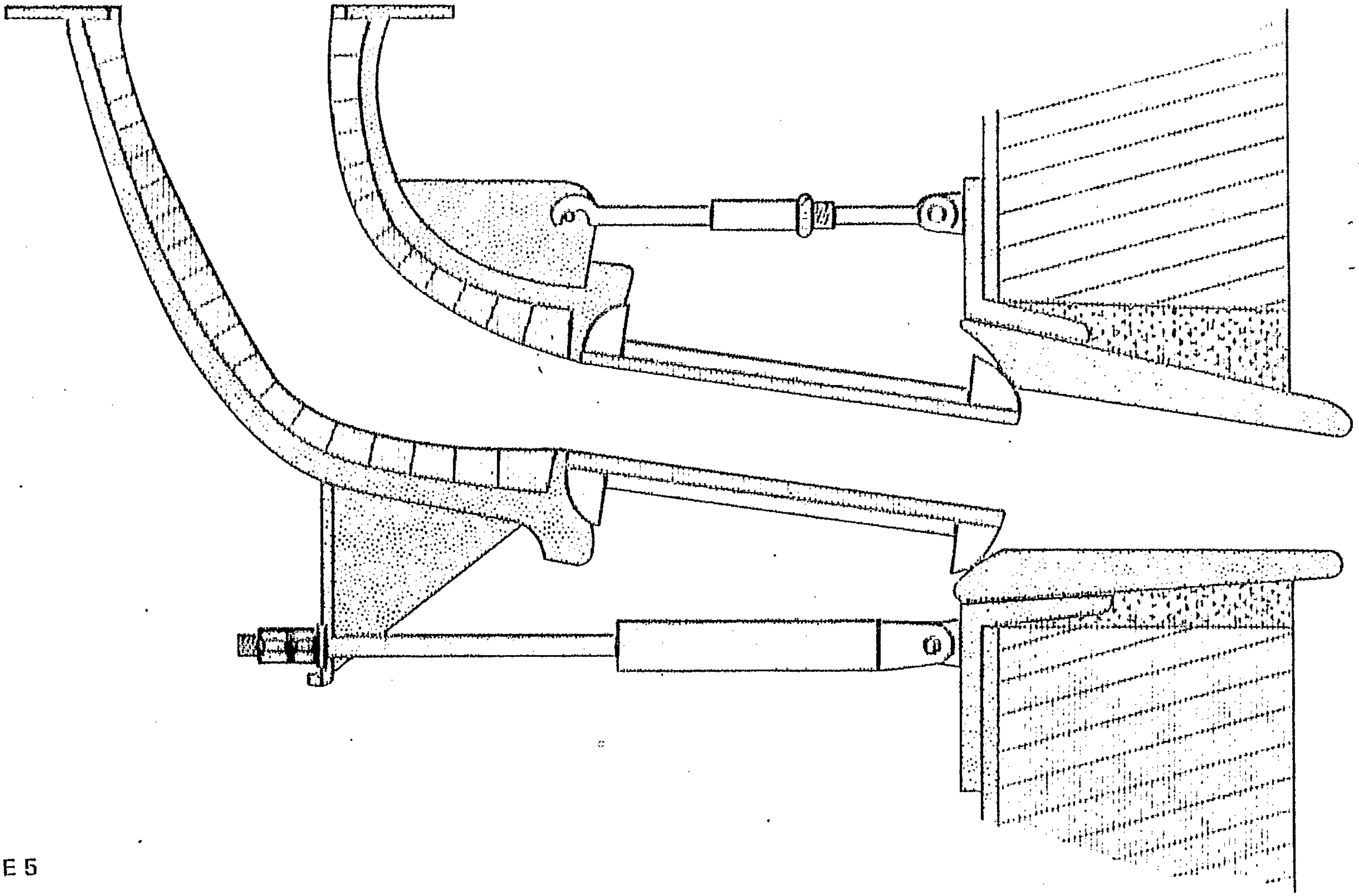
PRESSURIZED
FEED BIN

FEED CONTROLLER

CONVEYING GAS

TO GASIFIER

TYPICAL QUICK-CHANGE BLOW PIPE FOR BLAST FURNACE



SLIDE 5

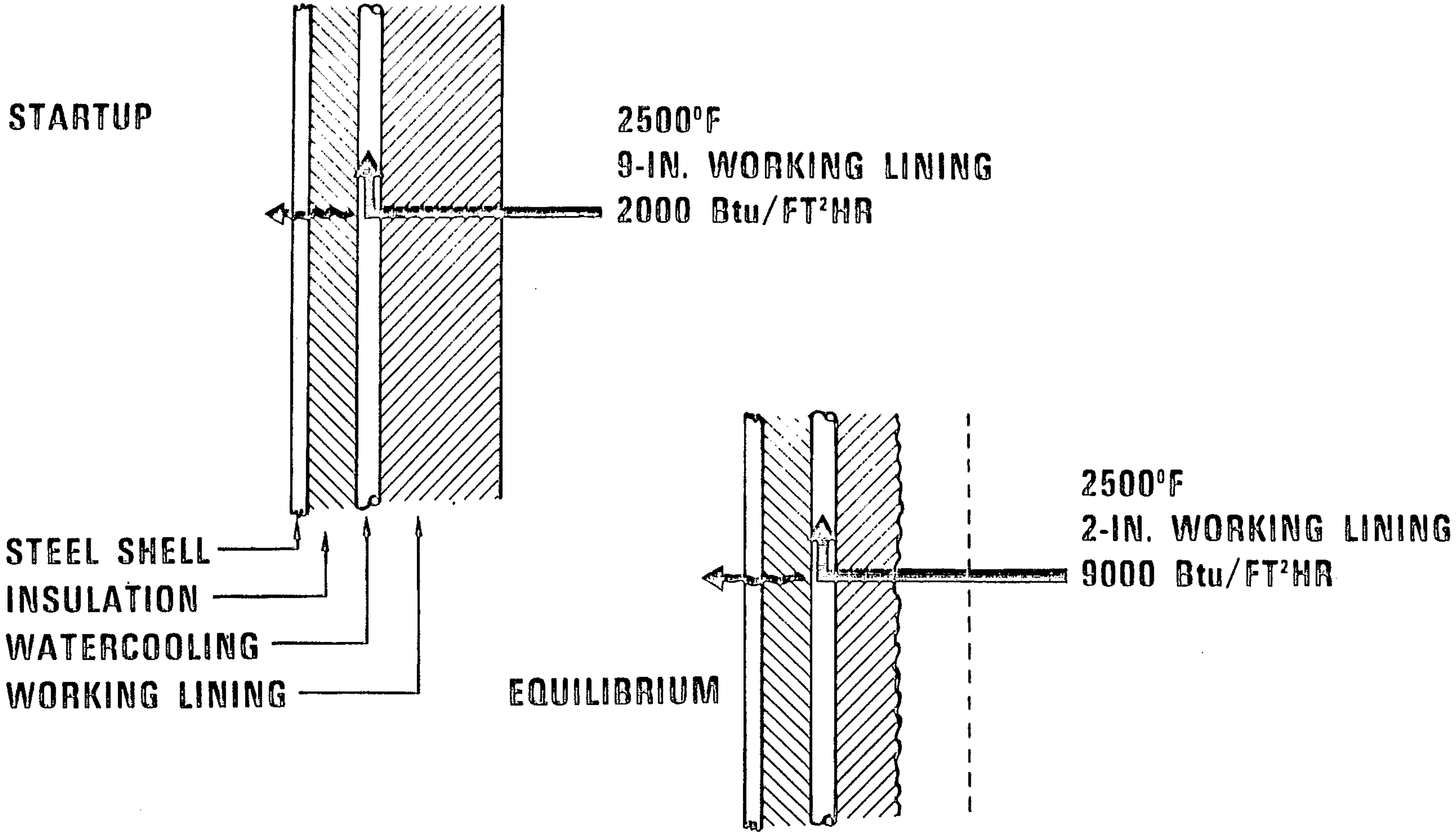
RESIDUAL THICKNESS OF REFRACTORY LINING IN BOSH
BLAST FURNACE

$$X = K \left[\left(\frac{T_c - T_w}{T_f - T_s} \; \frac{1}{a_1} \right) - \left(\frac{1}{a_2} + \frac{S_p}{K_p} + \frac{S_s}{K_s} \right) \right.$$

X = EQUILIBRIUM BRICK THICKNESS, IN.
K = THERMAL CONDUCTIVITY OF BRICK, Btu-IN./FT^{2}HR$^\circ$F
T_c = MINIMUM TEMPERATURE OF CHEMICAL REACTION, °F
T_w = COOLING WATER TEMPERATURE, °F
T_f = FURNACE TEMPERATURE, °F
T_s = MELTING POINT OF SLAG, °F
a_1 = HEAT TRANSFER NUMBER, FURNACE TO SLAG, Btu-IN./FT^{2}HR$^\circ$F
a_2 = HEAT TRANSFER NUMBER, SLAG TO COOLING WATER, Btu-IN./FT^{2}HR$^\circ$F
S_p = THICKNESS OF RAW MATERIAL BEHIND LINING, IN.
K_p = THERMAL CONDUCTIVITY OF RAW MATERIAL, Btu-IN./FT^{2}HR$^\circ$F
S_s = THICKNESS OF BOSH STEEL, IN.
K_s = THERMAL CONDUCTIVITY OF BOSH STEEL = Btu-IN./FT^{2}HR$^\circ$F

SLIDE 6

WALL DESIGN USING WATER COOLING



STARTUP

2500°F
9-IN. WORKING LINING
2000 Btu/FT²HR

STEEL SHELL
INSULATION
WATERCOOLING
WORKING LINING

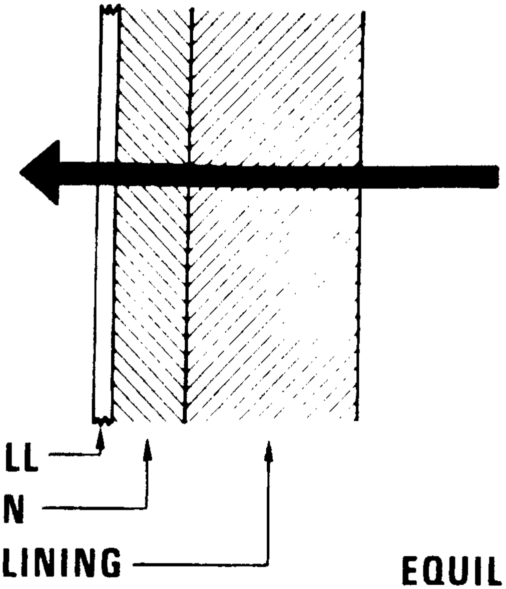
2500°F
2-IN. WORKING LINING
9000 Btu/FT²HR

EQUILIBRIUM

SLIDE 7

WALL DESIGN USING AIR COOLING

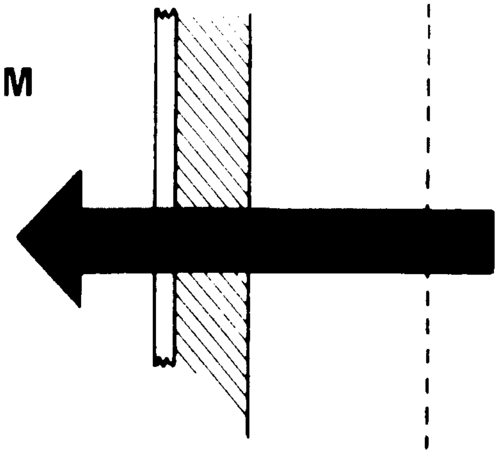
STARTUP



2500°F
9-IN. WORKING LINING
850°F Btu/FT^{2}HR

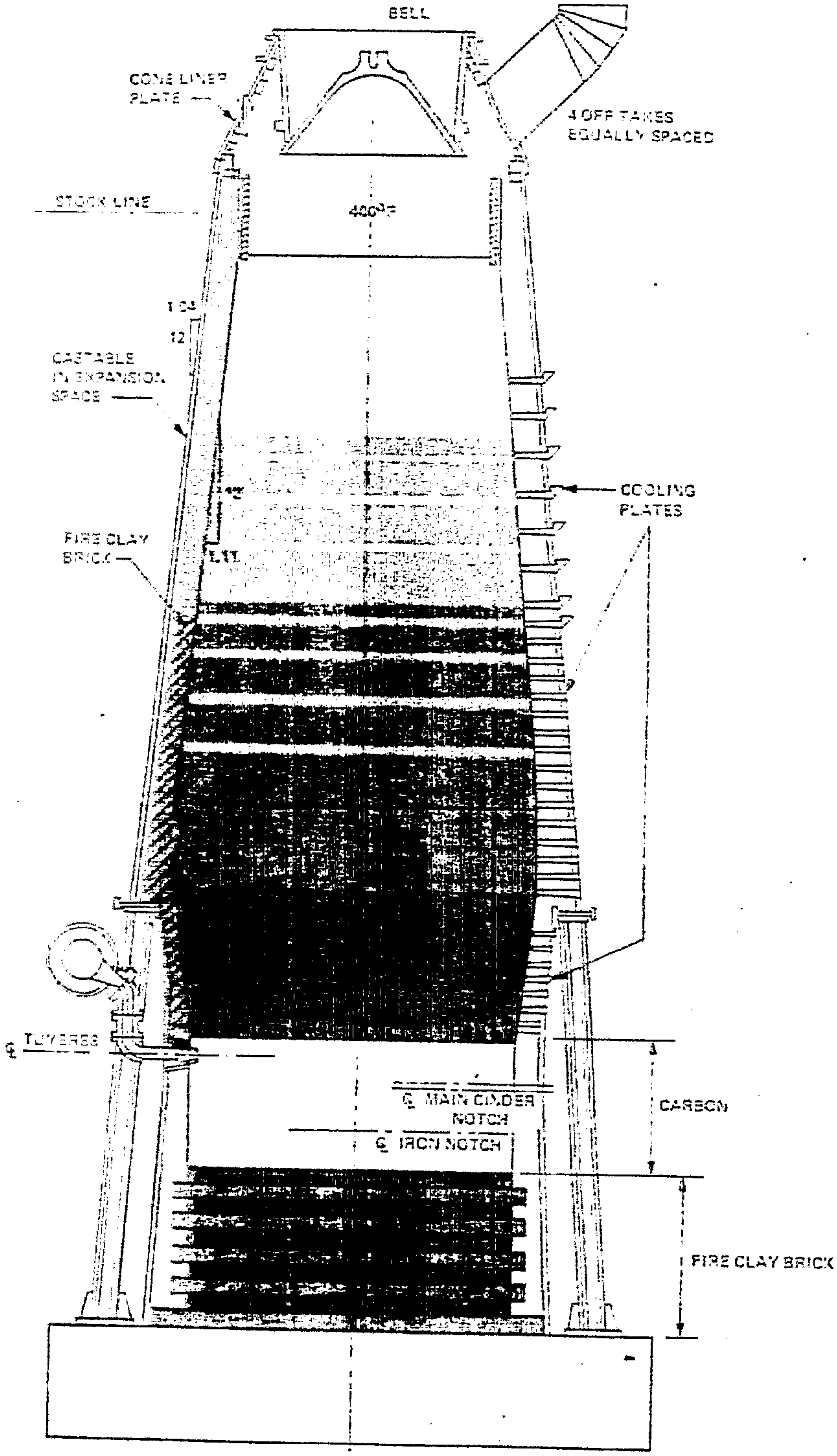
STEEL SHELL
INSULATION
WORKING LINING

**EQUILIBRIUM
(FAILURE)**



2500°F
0-IN. WORKING LINING
? Btu/FT^{2}HR

SECTION THROUGH A BLAST FURNACE SHOWING LINING REFRACTORIES AND WATER COOLING.



SLIDE 9

MAJOR REFRACTORY CONSIDERATIONS

A. HYDROGEN ATTACK

B. STEAM ATTACK

C. SLAG CORROSION

D. CO DISINTEGRATION

E. ALKALI ATTACK

F. PARTICLE EROSION

DESIGN CHOICE BASED ON CORROSION AND EROSION

ACID CONDENSATES — BRICKS, ANY TYPE

HYDROGEN — NO PROBLEM, H_2O DEPRESSED

STEAM — INCONCLUSIVE, MAY FAVOR FIRE-CLAY CASTABLE

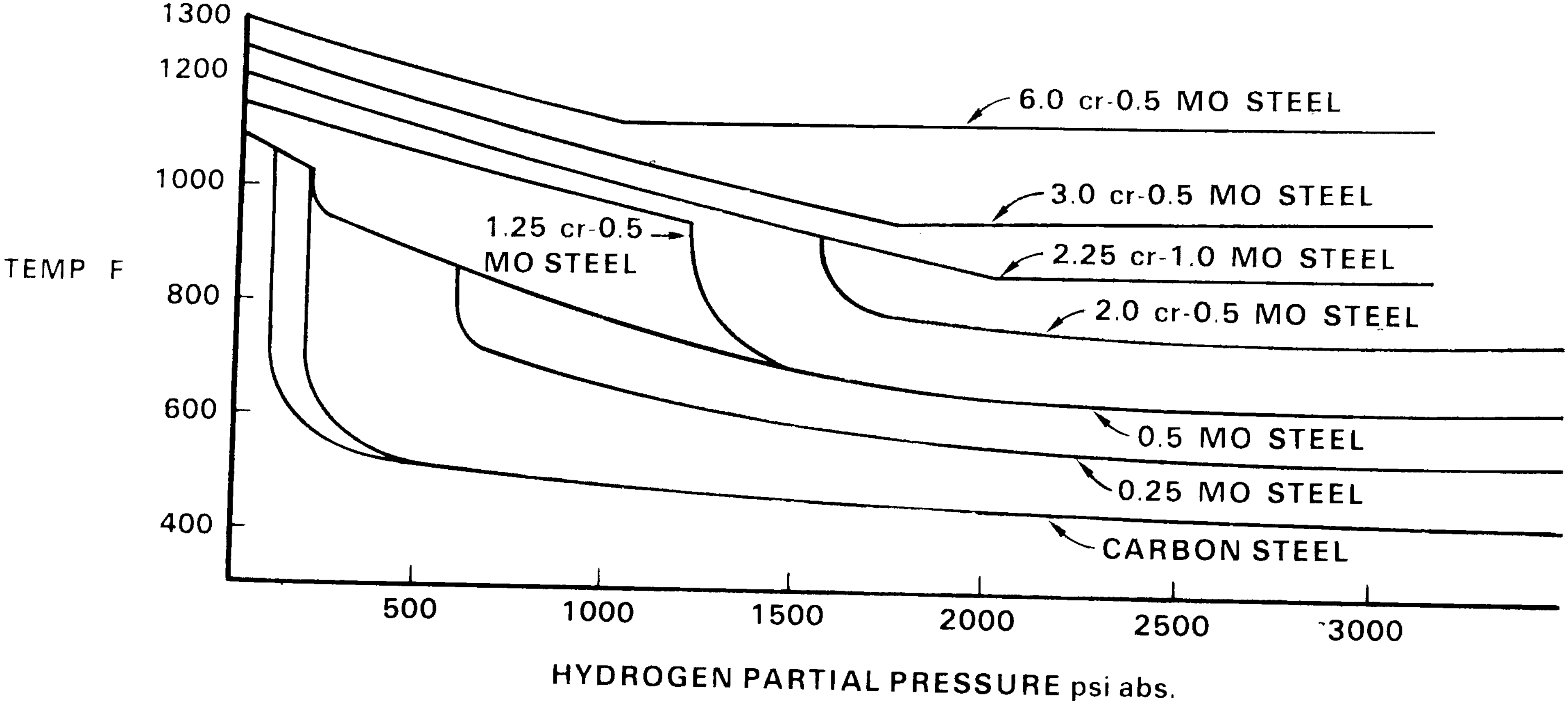
ALKALI SALTS — BRICK, MAY CAUSE DOWNSTREAM PROBLEMS

SLAG — BRICK, REFRACTORY MUST BE TAILORED TO WALL DESIGN

CO_2 — BRICK, LOW IRON, HIGH PRESSURE MAY ACCELERATE

EROSION — BRICK, HIGH MODULUS OF RUPTURE

Operating Limits, Metals In H_2 Atmosphere

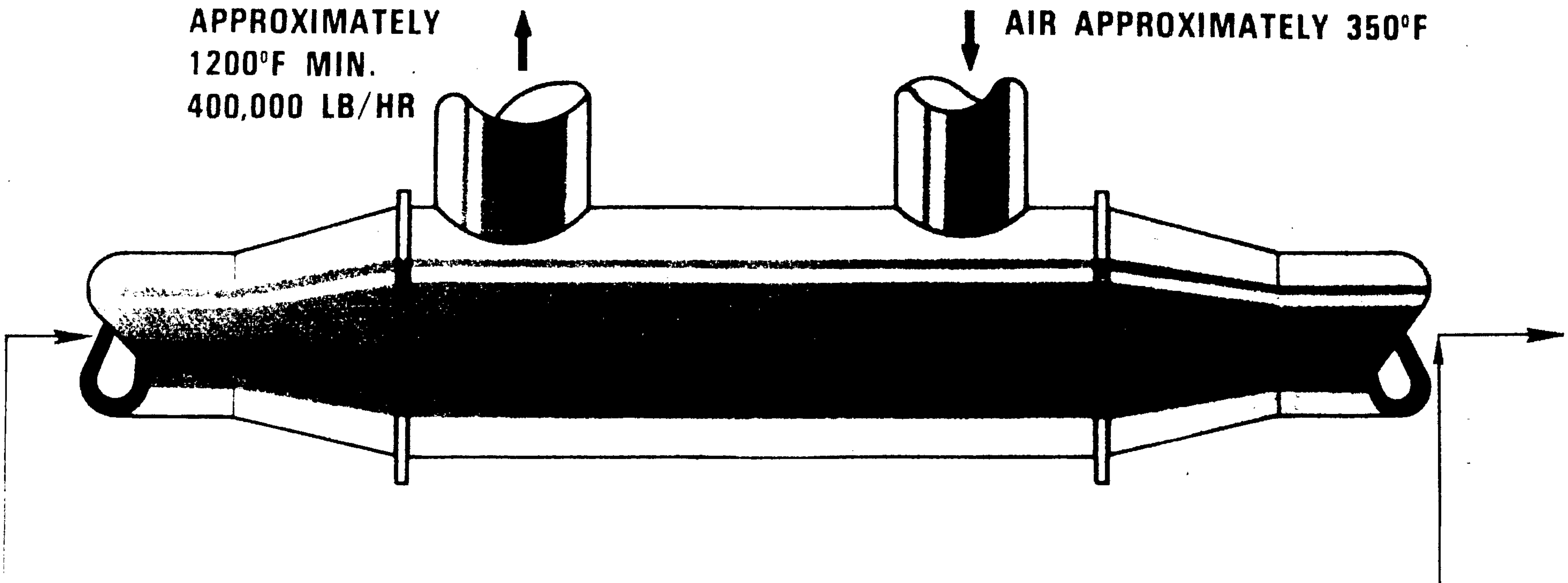


RELATIVE COST OF REFRACTORIES

REFRACTORIES	PRICE PER BRICK (18 X 4-1/2 X 3 IN.)	DENSITY	PRICE ($) PER 1000 FT^3
BRICK			
SILICON CARBIDE	$16.86/BRICK	175	119,893
CHROME ALUMINA	$15.67/BRICK	195	111,431
90% ALUMINA (MULLITE BOND)	$11.25/BRICK	183	80,000
HIGH-FIRED SUPER DUTY	$2.18/BRICK	145	15,502
CASTABLE			
94% ALUMINA	$720/TON	163	58,000 (89,500) [a]
FIRE-CLAY WITH HIGH-PURITY BOND	$280/TON	121	16,900 (27,850) [a]

[a] COST OF ANCHORS AND REBOUND CONSIDERED

AIR PREHEATER, USING PRODUCT GAS, LOW-BTU GASIFICATION



HEATED AIR
APPROXIMATELY
1200°F MIN.
400,000 LB/HR

AIR APPROXIMATELY 350°F

PRODUCT GAS
1500 TO 1700°F
CONTAINS CHAR
AND ASH

COOLED PRODUCT GAS
APPROXIMATELY
600,000 LB/HR

SLIDE 14

COLLECTED WORK NO. 36

MATERIAL CONSIDERATIONS IN COAL LIQUEFACTION

J. B. O'Hara, W. J. Lochmann & N. E. Jentz
The Ralph M. Parsons Company
Pasadena, California

From METAL PROGRESS - November 1976

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Material Considerations in Coal Liquefaction

By J. B. O'Hara, W. J. Lochmann, and N. E. Jentz

THE DEVELOPMENT of viable coal liquefaction methods is a national high-priority objective. ERDA-FE (Energy Research and Development Administration-Fossil Energy) has a prime responsibility in this area. It is being assisted in its program by Ralph M. Parsons Co., Pasadena, Calif. A key aspect of this work involves the selection and development of adequate construction materials for large commercial plants.

Coal liquefaction is not new; it has been and is being practiced industrially. Germany produced most of its aviation gasoline from coal during the later stages of World War II. The hydrogenation process accounted for most of this production. Germany's materials problems were severe, but the plants still managed to turn out about 80 000 bbl (12 720 m^3) of liquids per day. [1]

Another process now under consideration (Fischer-Tropsch indirect liquefaction technology) was also used in Germany. The SASOL plant in the Republic of South Africa, which has been in operation since 1955, is based on this process. [2]

However, such existing technology most probably will not be adopted in this country. It is felt that our synfuel plants must be large, efficient, simple, and reliable to be competitive with other fuels. Current thinking is that such a plant will be in the billion dollar class and process 10 000 to 40 000 tons (9000 to 36 000 Mg) of coal per day. Target dates for startup are in the 1980's.

Processes Dictate Materials

Liquefaction processes under development in the United States may be somewhat arbitrarily aligned into four classifications: hydroliquefaction, extraction, pyrolysis, and indirect liquefaction. Their operating conditions are spelled out in Table I.

In hydroliquefaction, a slurry of coal in a coal-derived liquid is contacted with a hydrogen-rich stream at elevated temperatures and pressures. As a result, the hydrogen to carbon ratio of the coal feed stock is increased, producing liquefaction.[3] Hydroliquefaction can take place with or without an added hydrogenation catalyst.

In extraction, a hydrogenated coal-derived liquid contacts the feed coal at elevated temperature and pressure, extracting a major portion of the carbonaceous coal constituents. [4] The hydrogenated coal-derived liquid serves as a hydrogen carrier to the extraction stage, which can be operated at a lower pressure than the hydroliquefaction process.

In pyrolysis, feed coal is heated to an elevated temperature, producing a gas, a tar, and a char. [5] The

Fig. 1—Simplified flow diagram for hydroliquefaction process.

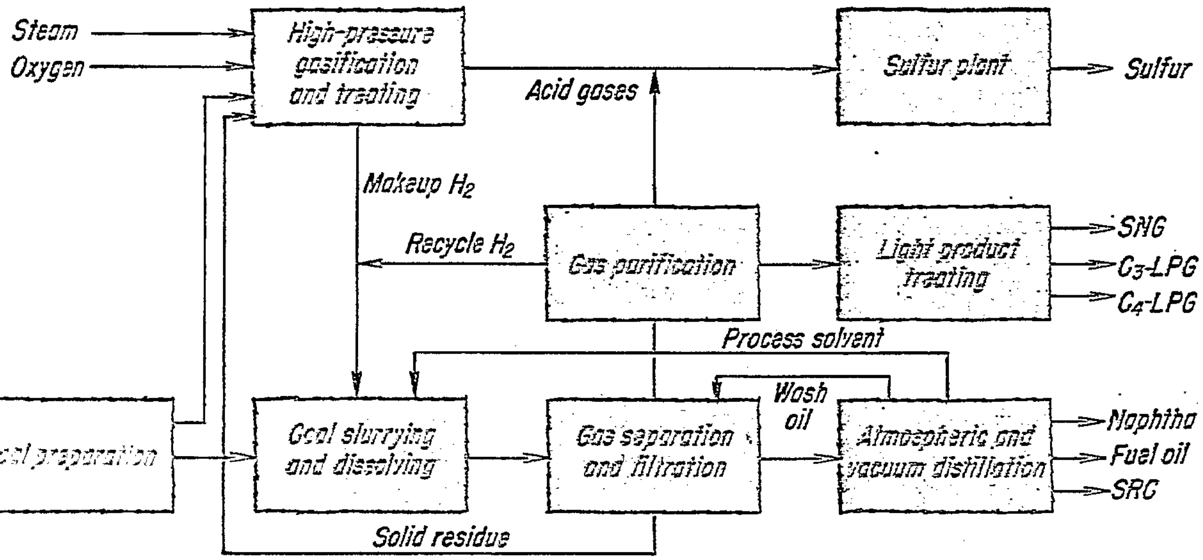


Fig. 2—Simplified flow diagram for extraction process.

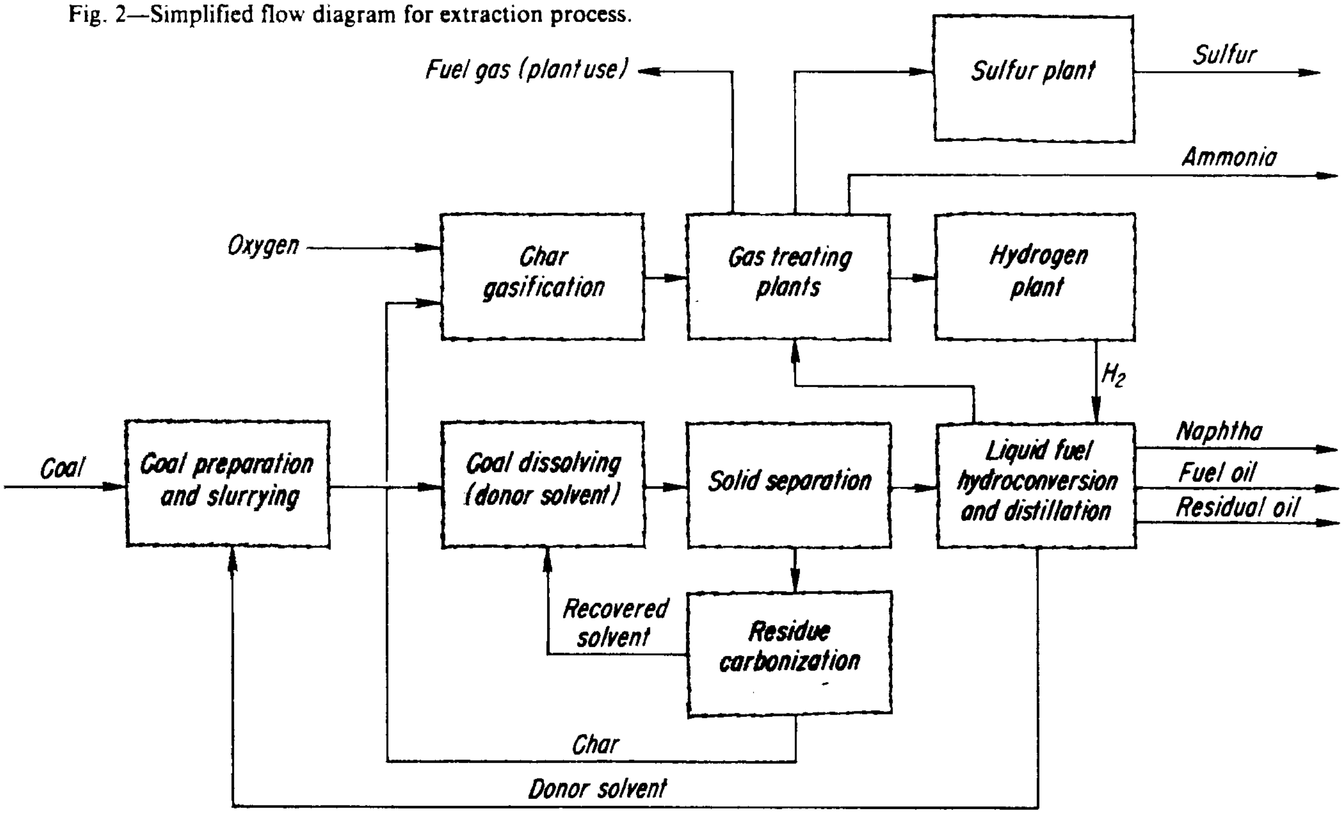
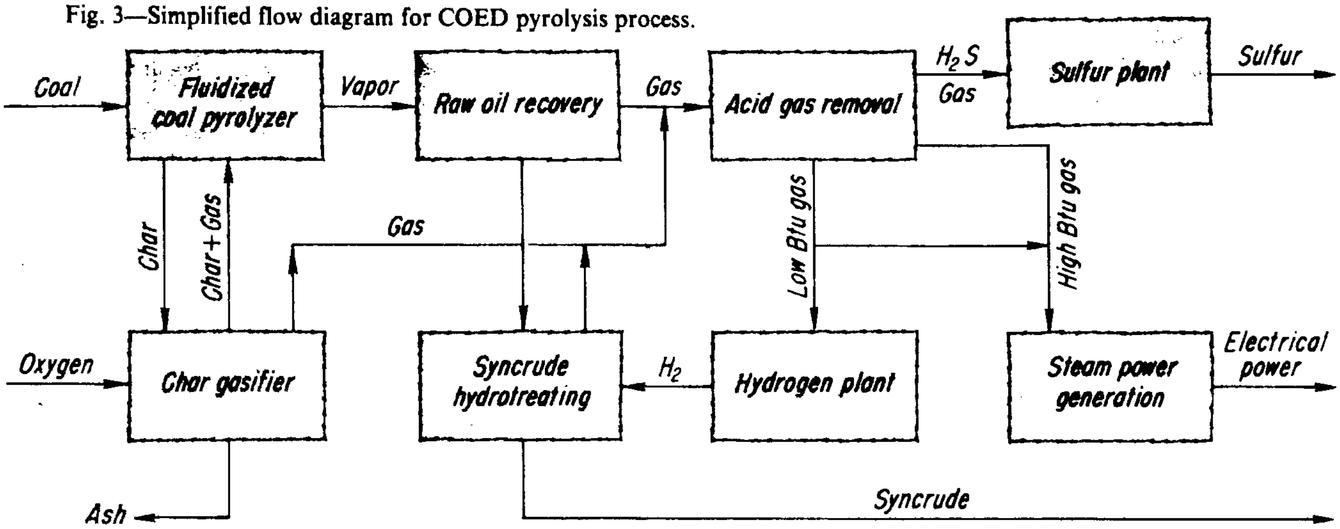


Fig. 3—Simplified flow diagram for COED pyrolysis process.



char may be gasified to produce a mixture of carbon monoxide and hydrogen—known as syngas—which in turn can be used as a feedstock for substitute natural gas (SNG), ammonia, methanol, or liquid hydrocarbon production; the gas can also be used as fuel for electric power generation. The tar can be hydrotreated to produce a low sulfur syncrude.

In indirect liquefaction, feed coal is gasified to produce a syngas, which is purified and reacted to produce liquid products. This is the Fischer-Tropsch technology.[6]

A key point: every coal liquefaction plant incorporates a gasification operation; but a plant producing SNG from coal does not necessarily include companion liquefaction operations. In a sense then, liquefaction technology development represents a broader challenge to scientific and engineering communities than gasification.

Materials for Hydroliquefaction Process

Key steps in one version of a hydroliquefaction process are shown in Fig. 1. Processing conditions are summarized in Table I. Typically, the dissolving or liquefaction coal

Table I — Summary of Operating Conditions for Liquefaction Processes

		Environment			
Process	Process steps	Temperature, F (C)	Pressure, psig (MPa)	Flow-phase conditions	Constituents
Hydroliquefaction Noncatalytic — SRC	Coal dissolving	800-900 (425-480)	1000-2000 (7-14)	Three-phase (liquid, gas, and solid) upflow; low linear velocity	Coal fines; coal-derived aromatic solvent of high sulfur and nitrogen content. Gases with H_2, CO, CO_2, H_2S, NH_3, HCN, and H_2O.
Catalytic	Coal dissolving Catalytic hydrogenation	800-900 (425-480)	3000-4000 (21-28)	Three-phase (liquid, gas, and solid) upflow through packed catalyst bed	Coal fines; coal-derived solvent with high aromatic, sulfur, and nitrogen content.
Extraction — CSF process	Donor solvent Coal dissolving	800-900 (425-480)	400 (2.7)	Oil-coal slurry-agitated	Ground coal; coal-derived solvent which has been enriched with H_2.
	Solvent hydrogenation	800+ (425+)	3000-4000 (21-28)	Three-phase (liquid, hydrogen, and catalyst particles) low velocity, upflow	Hydrogen-rich gas with H_2S, NH_3, and H_2O. Coal-derived aromatic liquid of high sulfur and nitrogen content.
Pyrolysis — COED	Pyrolysis	800-1150 (425-620)	10-15 (0.06-0.10)	Fluid bed; coal and/or char gas from pyrolysis	Ground coal and/or char. Unsaturated pyrolysis vapors composed of tar, H_2O, unsaturated gas. H_2S, CO_2, NH_3, COS, HCN, and char fines.
	Char conversion	1800 (980)	5-10 (0.03-0.06)	Fluid bed; char syngas product; steam and oxygen feed	Char fines; syngas composed of H_2, CO, CO_2, H_2O, H_2S, NH_3, COS, CS_2, HCN, and entrained char.
Indirect — Fischer-Tropsch	Liquefaction (synthesis)	600 (315)	400 (2.7)	Gas phase	Syngas of high purity plus products; paraffin hydrocarbons, oxygenated compounds, CO_2, and H_2O.

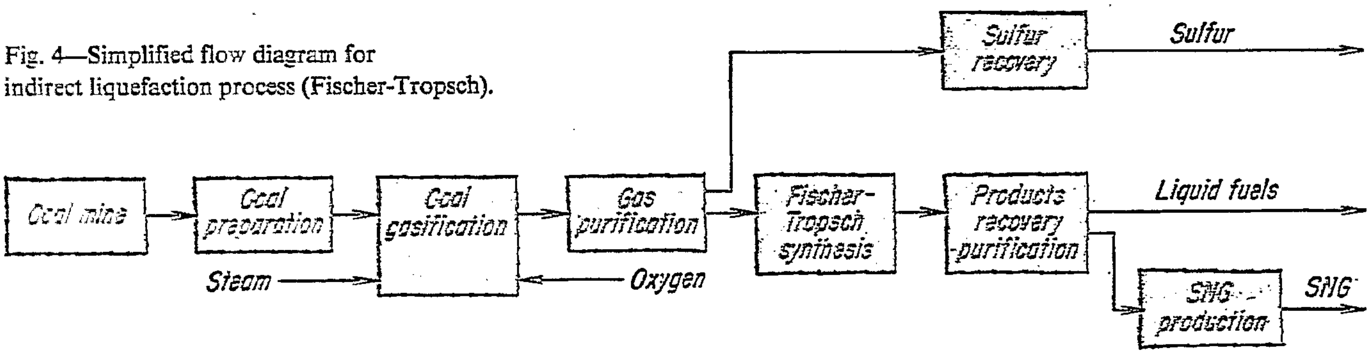


Fig. 4—Simplified flow diagram for indirect liquefaction process (Fischer-Tropsch).

conversion step takes place at about 800 to 850 F (425 to 455 C) and 1500 to 2000 psi (10 to 14 MPa).

Erosion-corrosion of equipment handling coal slurries is a general current problem which can be severe. Operators of coal conversion plants, it's reported, have yet to find a commercially available pump for slurries which performs satisfactorily for extended periods.

Another problem: service life of the pressure letdown valve for process slurries after the liquefaction step. In German plants, which were operated at higher pressures than U.S. plants, service life ranged from 500 to 1500 h. Similar results are being obtained in this country's pilot plants; but there is evidence that a life of 2000 h can be obtained with the proper use of tungsten carbide trim.

It's difficult to predict attack by erosion-corrosion. Relatively little is known about the mechanisms. Available data have been derived primarily from empirical test results and analysis of service experience.

In some noncorrosive environments, there appears to be a relationship between such factors as the size, speed, and hardness of a substance and its erosive effects on a particular material. But opinions on the subject differ.

Continued on p. 36

At this time, it is not possible to predict material loss from erosion or erosion-corrosion with reliability. Current designs are based on material exposure experience under actual or simulated operating conditions.

A number of materials have been tested in pilot plant pumps handling coal slurries. They include carbon steel, 300 series stainless steels, Ni-Hard, and coated carbon steel. Coatings range from chromium, ceramics, and plastics to elastomers and hard surfacing materials such as fused systems containing nickel, chromium, boron, and silicon. It's understood that service lives on the order of 700 to 2000 h have been obtained with coatings, but no pumps have been entirely satisfactory.

Proper mating of materials and design is necessary in centrifugal slurry pumps. Low speed (around 1500 rpm) and a pump with proper head-volume characteristics will tend to reduce erosion. Careful attention should be given to impeller and volute geometrical design. Long-term objectives include the following material characteristics: easy castability; weldability; small grain size; ability to maintain erosion-resistant smooth surfaces; resistance to high-temperature, high-pressure H_2S-H_2 environments; immunity to stress-corrosion cracking; inherent toughness and ductility.

ERDA has a Solvent Refined Coal (SRC) pilot plant at Ft. Lewis (Tacoma), Wash., which is operated by the Pittsburg & Midway Coal Mining Co., Div. of Gulf Oil. A smaller SRC unit in Wilsonville, Ala., is funded jointly by the Electric Power Research Institute and Southern Services. With units of this type, slurry preheaters were an original concern. Feed coal slurry and hydrogen are heated from about ambient temperature to 800 F (425 C) in helical coils. The Wilsonville coil is made of type 316 stainless steel, while Incoloy 800 was chosen for the Tacoma coil.

So far, performance has been satisfactory — operation of the Wilsonville coil exceeds 350 days, that of the Tacoma coil more than 300 days. However, new problems are anticipated at the commercial level. A design and flow pattern different from that for helical coils will be used.

Problems with erosion have been encountered in check valves for the coal slurry plunger feed pumps to the high-pressure dissolver section. A combination of material selection and valve design has solved the problem. It appears that a spherical, hardened chromium steel ball on a tungsten carbide seat provides adequate performance. Check valve life has been lengthened from as little as 4 h to as long as 1600 h.

Materials used in the product recovery section (fractionating columns and reboilers) have had to be replaced. Example: failure of carbon steel U-tubes in reboilers distilling intermediate coal-derived fractions at temperatures in the range of 400 to 700 F (200 to 370 C). Carbon steel has been replaced by type 304 stainless steel. Another example: type 410 stainless steel fractionating trays failed after about 150 days at temperatures of the order of 600 F (315 C). The replacement: type 304.

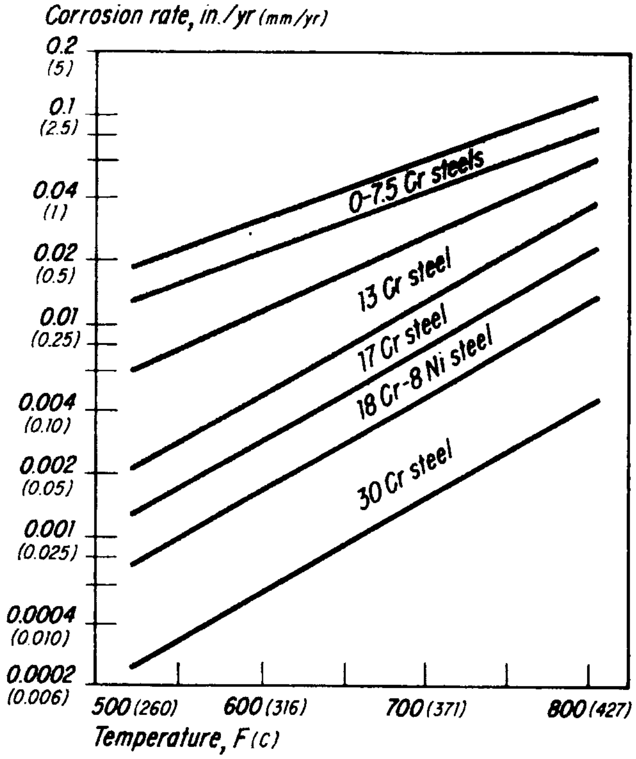


Fig. 5—Corrosion rates of various alloys in H_2S-H_2 mixtures. Source: National Assn. of Corrosion Engineers.

The dissolver, where the coal slurry is hydrogenated, is exposed to a corrosive environment. To date, the Tacoma pilot plant mentioned previously is getting satisfactory performance from a 2.25Cr-1Mo alloy overlaid with type 347 stainless steel. Potential troublemakers in dissolvers include sulfur compounds and hydrogen (see Table I).

Sulfur and Sulfur Compounds

Coal, especially that found in the eastern part of the United States, contains substantial sulfur. During processing, it is converted to hydrogen sulfide (H_2S) which becomes increasingly corrosive to carbon steel at temperatures greater than 450 to 550 F (230 to 290 C). Hydrogen (H_2) is also present. Here we can draw upon the experience of petroleum refineries with H_2S corrosion at temperatures from ambient to about 1000 F (540 C) and operating pressures to 3500 psi (24 MPa).

The combination of H_2S and H_2 is particularly troublesome. The 5Cr-0.5Mo and 9Cr-1Mo alloys commonly used to resist moderately high temperature sulfur and sulfur compound corrosion show little improvement over carbon steel in corrosion resistance to H_2S-H_2 atmospheres. A chromium content of at least 12% is required. The 300 series of austenitic stainless steels (minimum 18Cr-8Ni) have excellent resistance and are normally specified for the H_2S-H_2 environments found in petroleum refineries.

Extensive corrosion data and experience at temperatures to about 1000 F (540 C) have been accumulated (Fig. 5).[7]

At temperatures between 800 and 1550 F (425 to 845 C) the austenitic stainless steels tend to precipitate carbides (sensitization) along grain boundaries. In the case of process upsets, it is conceivable that the dissolver temperature could exceed 850 F (455 C).

This sensitization tendency may be mitigated by reducing the amount of carbon initially present, or by tying up the carbon with small amounts of strong carbide-forming elements (stabilization) such as columbium, tantalum, or titanium. Welding and fabricating problems are increased by the alloying additions; and reduced carbon content does not eliminate the possibility of polythionic and chloride cracking problems at low temperatures.

Petroleum refinery experience has demonstrated that austenitic stainless steels resist high-temperature H_2S during operation, but they may be susceptible to stress-corrosion cracking when the plant is shut down. Strict shutdown procedures, which include circulation of protective solutions, must be followed during downtime to prevent cracking.

In large equipment, such as pressure vessels, such procedures are impractical. The vessels are not made of solid stainless steel — because of cost and also to prevent catastrophic failure due to cracking. Clad or weld overlay construction is recommended because the polythionic and chloride cracking phenomena are not known to propagate into the commonly used ferritic alloy backing materials.

Attack by Hydrogen

Hydrogen at temperatures greater than 450 F (230 C) and at pressures greater than 200 psi (1.3 MPa) will cause carbon steel to decarburize and to form methane at internal interstices. The resulting internal pressure forms blisters, causing failure. Adding molybdenum and chromium suppresses this tendency because of their strong carbide-forming characteristics, allowing these alloys to be used at higher hydrogen pressures and temperatures. Molybdenum is also particularly beneficial in improving creep and stress-rupture strengths.

Carbon steel, carbon-molybdenum steel, and the chromium-molybdenum alloys are commonly used as the pressure retaining material for equipment experiencing metal temperatures to about 1000 F (540 C).[8] Data on these materials, based on more than 50 years of experience with failures and successes, are being maintained and updated by the American Petroleum Institute Committee on Refinery Equipment for Corrosion.

Chlorides: Stress-Corrosion Cracking

Chloride concentrations in the range of 100 ppm can be expected in the dissolver with a companion moisture content in the range of 1.5%. To date, there are no reported problems with stress-corrosion cracking in the SRC pilot plants.

However, the Project Lignite Process development unit at Grand Forks, N.D., suffered catastrophic failure of its 316 stainless steel preheater and general stress-corrosion cracking in the high pressure area when processing lignite in an SRC-type process. Chlorinated hydrocarbons had been used to clean some of the equipment prior to failure. Analysis indicated chlorides were the cause of failure.

The Extraction Process

The process is outlined in Fig. 2; conversion-extraction conditions are given in Table I.

In comparison with the hydroliquefaction process, initial extraction takes place at a lower pressure (about 400 psig [2.7 MPa]), reducing material requirements at that stage for resistance to high pressure-temperature H_2 attack. However, both processes have common shortcomings, including handling problems, pressure reducing valve erosion, sulfur compound corrosion, and a potential for chloride stress-corrosion cracking of austenitic steels.

A number of material and engineering problems were defined during operation of the Office of Coal Research (OCR) sponsored Consol Synthetic Fuel (CSF) pilot plant program during the 1963-1970 period. Some results of this program are summarized in the following paragraphs.

Centrifugal pumps handling slurries with an initial service life of 80 to 100 h were coated with an electrodeposited hard chromium plating, increasing their lives more than 400%. In another case, hard chromium plating was applied to the bottom of hydroclones used for solid liquid separations from a slurry containing approximately 50% solids. The objective was to eliminate a spiral wear pattern, which became noticeable after about 100 h of operation. After plating, 1800 h of service were obtained.

Ni-Hard pump components have been used with some success in slurry systems; but because of their brittle nature, they are suspect in case of fire and dousing with water.

No really satisfactory materials-designs were defined to give long maintenance-free life for valves in slurry service. Ball valves are used at lower temperatures, and open port construction is preferred. Tungsten carbide has been the most successful material for seating surfaces on control valves.

However, tungsten carbide in pressure letdown valves can be destroyed within 500 h of operation—the pressure drop is several hundred or even thousands of pounds.

Operating lives of up to 2000 h can be obtained with proper material procedures. The method of forming the tungsten carbide valve seat can account for considerable differences in performance. A needle type seat can be used with the flow reversed from normal practice.

Because of the critical nature of valve performance in coal conversion plants, a program to develop improved valves is underway at the ERDA facilities in Morgantown,

W. Va. Additional work to investigate materials for improving valve performance is underway at Bureau of Mines units in College Park, Md.; Rolla, Mo.; and Albany, Ore.; plus an EPRI program at Battelle-Columbus.

The Hydrogenation Step

Hydrogenation takes place in a reactor at pressures up to 4000 psig (28 MPa) and temperatures in the range of 775 to 850 F (410 to 455 C) in the presence of hydrogen and a catalyst. The catalyst and coal extract mixture is maintained in a fluidized or ebullient state.

Two equipment items are critical: the reactor and recirculating pumps. Type 347 stainless steel was used for the contact metal for the hydrogenation reactor in the CSF pilot plant. It performed satisfactorily, but the operating time was too short to provide a meaningful test. As with hydroliquefaction, such factors as performance in contact with H_2S-H_2 mixtures would be considered during design of commercial units. Currently, we would suggest 2.25Cr-1Mo reactor weld overlays with type 347 or 308L stainless.

The technology for large pressure-retaining reactors—about 10 ft (3 m) in diameter by 40 ft (12 m) long, a wall thickness of about 12 in. (305 mm), and operating temperatures of 850 F (455 C)—has been fairly well-established for hydrocracking process reactors.

Some success was experienced with canned pumps to provide the ebullating bed effect; but it appears that more experience is needed in material performance to assure reliable operation in commercial scale units at 3000 psi (21 MPa) and 850 F (455 C).

The Pyrolysis Process

A block flow diagram for a COED-based pyrolysis plant is shown in Fig. 3; reaction conditions are summarized in Table I. Results of materials testing in the pilot plant were reported by J.J. Jones and B.D. McMunn in a paper, "Materials Performance in Coal Pyrolysis and Coal Oil Hydrotreating," at the Corrosion/73 Forum, Anaheim, Calif., March 1973, sponsored by the NACE.

In COED pyrolysis, coal is heated in fluid bed reactors in successive stages over a temperature range of 600 to 1100 F (315 to 595 C) at a pressure of 10 psig (70 kPa). Char gasification can take place at temperatures up to 1800 F (980 C). No serious material problems were experienced at the COED Pilot Plant during the course of its operation (1970-74) at Princeton, N.J. For a commercial unit, the higher temperature char gasification vessels would be refractory-lined carbon steel.

The hydrotreating process used for the coal oil produced in pyrolysis is similar to that for first-stage hydrocracking of petroleum stocks in the petroleum industry. There is one exception: operating conditions in the former are more severe. Potential problems with sulfide corrosion and hydrogen attack would apply here; and again, a type 347 stainless steel weld overlay design should be satisfactory.

Indirect Liquefaction Process

A block flow diagram for a version of a Fischer-Tropsch indirect liquefaction plant is shown in Fig. 4. Synthesis reaction conditions are summarized in Table I. The coal gasification section, a major part of a Fischer-Tropsch plant, is outside of the scope of this discussion.

Material problems for the liquefaction section of a Fischer-Tropsch plant are not severe. Reason: the feed to this unit is a mixture consisting largely of carbon monoxide and hydrogen, which is produced by gasification and gas purification. The particulate matter and sulfur values have been removed, so conditions are similar to those encountered in petrochemical operations.

In summary, the material technology for the process conditions is considered adequate; greatest experience rests with SASOL in the Republic of South Africa. ●

For More Information: You are invited to contact the authors directly by telephone or letter. Dr. O'Hara is manager, Energy Dept.; Mr. Lochmann is chief, materials application engineer, Engineering Dept., and Mr. Jentz is chief process engineer, Petrochemical Div., Ralph M. Parsons Co., 100 W. Walnut St., Pasadena, Calif. 91124. Telephone: 213/440-2501. This article is based on a paper, "Coal Liquefaction: Materials Systems Design," presented at a recent conference, "Materials for Coal Conversion Systems Design," sponsored by the Power Activity, ASM Materials Systems and Design Division.

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COLLECTED WORK NO. 37

CORROSION ENGINEERING DESIGN INTERFACE FOR COAL CONVERSION

W.J. LOCHMANN
Chief Material Applications Engineer

R.D. HOWELL
Principal Project Engineer

THE RALPH M. PARSONS COMPANY
PASADENA, CALIFORNIA

Presented at the
NATIONAL ASSOCIATION OF CORROSION ENGINEERS
INTERNATIONAL CORROSION FORUM
PROTECTION AND FOR PERFORMANCE OF MATERIALS
SAN FRANCISCO, CALIFORNIA
March 15, 1977

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CORROSION/77

The International Corrosion Forum Devoted Exclusively to
the Protection and Performance of Materials/March 14-18,
1977, San Francisco Hilton Hotel, San Francisco, California

PAPER NO. 48

CORROSION ENGINEERING--DESIGN INTERFACE FOR COAL CONVERSION

W. J. Lochmann and R. D. Howell
The Ralph M. Parsons Company
Pasadena, California 91124

The design, fabrication, and operation of equipment for coal processing plants is presenting challenging problems to corrosion, materials and design engineers.[1] Coal conversion and its utilization imposes more severe requirements on process equipment than that generally experienced in the chemical, petroleum refining, steel and power generation industries. More than ever before, an interdisciplinary team effort is required between the materials community and designers to improve reliability and decrease costs of process equipment. Simple upgrading of materials for existing designs may no longer be adequate for desired equipment serviceability. Interdisciplinary action is needed to resolve material problem areas where operating conditions are so severe that they exceed satisfactory performance of materials currently available or economical.

BACKGROUND

Coal conversion and its utilization is emerging as a potential major industry. Although many of the basic technologies for converting coal into "clean" liquid, solid, and gaseous fuels have been around for half a century or more, they have been developed only to a limited extent in the United States because the availability of natural gas and the plentiful supply of petroleum have made it uneconomical to do so. For the past decade or so, the sources of energy in the U.S. have been predominantly oil and gas (44 and 31 percent respectively) with coal accounting for 21 percent and all other sources, including hydroelectric and nuclear plants, accounting for four percent. In contrast, coal accounts for 75-80 percent of the nations fossil fuel resources.

In respect to the total U.S. energy picture, it is important to note that a near-term improvement in the U.S. oil position will occur this Spring, when oil and gas production facilities on Alaska's North Slope will be completed ahead of schedule. Parsons serves as Managing Contractor for all production facilities in the east side of this production field at Prudhoe Bay.

Unlike oil, coal, because of its content of sulfur, ash and other pollutants, is considered to be a dirty fuel. The principal objective in coal conversion is to transform coal from its "contaminated" solid form to "clean" liquid, gaseous, or low-ash solid products which meet environmental standards. Conversion of "dirty" coal into "clean" fuels is expensive. In the U.S., a number of advanced processes are under development for more economical conversion of coal to clean burning gaseous, liquid, or solid fuels. Over the last decade, a number of processes have reached the pilot plant stage. The new developments are moving toward three main objectives. As shown in Figure 1, one plan is to replace much of the natural gas, fuel oil, and raw coal now burned in electric power plants with a cheap "power" gas that would have a rather low heating value [4-25 MJ/m^3 (100-600 BTU/CF)] but would be capable of attaining high generating efficiencies. As indicated in Figure 2, another development is to produce a gas of high heating value [30-40 MJ/m^3 (900-1000 BTU/CF)] to supplement natural gas in the nation's pipelines. Figure 3 shows a third objective which is to produce a range of synthetic-oil products, from crude to gasoline.

The Energy Research and Development Administration (ERDA) in its 1976 plan[2] for "Creating Energy Choices for the Future" estimated 20-140 coal gasification or liquefaction plants [8000 m^3/day (50,000 bbl/day oil equivalent each)] will be potentially required by the year 2000.

Because it is expensive to gasify and liquify coal, a key objective is the development of large, simple, minimum-cost plants. With such capital intensive plants, operating reliability is a primary concern.

In the anticipated large plants, loss of plant production can amount to a significant loss in revenue. Therefore, emphasis is made by designers for the highest possible on-stream time factors (90 percent or better). In other process industries, precautions such as the following have sometimes been incorporated to avoid emergency shutdowns and have the plant shut down only for planned maintenance.

- Sparing of critical equipment when necessary.
- Better quality materials such as higher alloys to obtain lower corrosion rates and longer service life.
- Additional quality control, including non-destructive examination, testing and inspection to assure proper material usage, and to avoid injurious defects.

Economic analysis, however, shows that required selling prices are highly sensitive to fixed capital investment. Therefore, it follows that reliability improvement should be obtained by efficacious design/materials interfacing to minimize overall plant costs.

CORROSION ENGINEER/DESIGN INTERFACE

Although coal conversion processes are in many ways similar to present coke-oven gas production, petroleum refinery operations and coal fired power boilers, there are many factors unique to coal conversion[3].

These conditions are summarized in Tables I and II. For viable coal conversion plants to be constructed and operated reliably, the corrosion engineer will be challenged as in few other industries. Simple substitution of more corrosion resistant materials in currently designed equipment is no longer adequate.

For improved reliability, new uses of materials, new design, and new fabricating techniques must be utilized.

For various equipment items, the following overview presents programs proposed or already underway in which corrosion engineers and designers interface for the construction of coal conversion plants.

Gasifiers, Liquefaction Reactors, and Other Large Pressure Vessels

These vessels are very large and may contain pressures to 35 MPa (5000 psi) and temperature over 1650°C (3000°F). Field construction may be required. Current thinking is that these vessels will be designed to Section VIII Division 1 or 2 of the ASME Code.

In the case of coal hydroliquefaction, the reaction step of adding hydrogen to the coal to form liquid products requires pressures from 7 MPa to 35 MPa (1000 to 5000 psig) and temperatures in the range of 425 to 650°C (800 to 1200°F). Because of residence time requirements, these reaction vessels are likely to have diameters in the order of 5 to 8 m (15 to 25 feet), lengths of 25 to 40 m (80 to 120 feet) and wall thicknesses of 250 to 300 mm (10 to 12 inches). These heavy wall vessels will:

- Increase the possibility of brittle fracture during vessel manufacture and during plant start-up and down time. Sufficient toughness must be incorporated into the pressure envelope and appropriate operating procedures adopted to avoid failures.

- Increase exposure times for radiographing welds. For field construction of vessels, more powerful radioactive sources will require larger isolated radiation areas causing work scheduling problems.

In gasifiers the temperatures range from about 800°C to over 1650°C (1500°F to over 3000°F), with pressures ranging from atmospheric to over 10 MPa (1500 psi). Because of the temperatures, most new processes have gasifier vessels that utilize an internal refractory lining. This lining must withstand erosive and chemical attack from gases, slags and coal and ash solids. Because the refractory forms a shield to protect the pressure envelope from disastrously high temperatures, it must have a very high degree of reliability.

Refractory anchors must be attached to the pressure,envelope and consideration must be given to anchors being lost during operation or refractory replacement. Welding of anchors directly to the pressure envelope may cause material degradation requiring postweld heat treatment. Because of the presence of hydrogen at high temperatures and pressures and potentially corrosive condensates in coal gasifiers, the corrosion engineer must give careful consideration to his selection of refractory anchor and shell materials.

In cold shell gasifiers, corrosive condensates may require protective cladding or weld overlay of the pressure envelope. Consideration must be given not only to corrosion prevention, but also to avoidance of material/design combinations[4,5] that could result in mechanical failure of the vessel. For example, Figure 4 shows a material/design combination that failed in a petroleum refinery application. Although Type 347 Stainless Steel has been used extensively as an overlay material, its greater propensity for cracking has resulted in the specification of a protective overlay composition equivalent to that of 304 L Stainless Steel for many vessels in petroleum refinery service.

Piping

The large size and high pressure of some coal conversion plants will require piping with diameters as large as 1830 mm (72 inches) and wall thickness of 75 to 125 mm (three to five inches). Standard bends, Tees, and other fittings may not be available, and mitered elbows may be required. Materials to resist erosive and corrosive flow conditions must be considered in their design.

Some common piping design practices may have to be avoided in coal conversion plant lines that contain solids in the form of coal, char or ash. These solids can be quite erosive in both gas and liquid streams. Any protuberances or gaps increase metal loss. For example,[3] Figure 5 shows the mushrooming erosion effect resulting from the gap in a carbon steel socket weld in a liquids/solids line from one of the pilot plants.

Valves

Erosion, corrosion, wear and abrasion are the damaging mechanisms to satisfactory valve operation. Valving is perhaps the most severely attacked equipment subsystem. Valves are required to perform pressure regulation, flow diversion, process mixing and disposal, as well as to provide positive shut off. Let-down valves in which pressures are dropped 7 MPa (1000 psi) or more have been a particularly troublesome area. A satisfactory valve to withstand the combination of a two or three phase gas-solid-liquid stream, high pressure drop and high temperature has yet to be developed. While valves have been designed for any one of the above conditions, valve life under combination of conditions may be only two weeks to 90 days. Material substitution alone, i.e., use of harder, more durable materials, has not been adequate. For example, increased use of tungsten and cobalt carbide materials has not achieved satisfactory valve life.

Programs are currently underway to develop suitable valves by closer interaction between valve designers and materials engineers. Also programs are underway to develop and evaluate single-phase material such as high-purity, dense, fine-grain oxides, carbides, borides, and nitrides prepared by sintering or hot-pressing, and two-phase cermet types of materials.

Pumps

Various water base slurries containing coal, ore, or other materials at ambient temperatures have been successfully pumped for years. However, there is relatively little experience with pumping hydrocarbon based slurries at elevated temperatures.

Erosion problems continue to occur in centrifugal slurry pumps with high suction pressures and low differential pressures. Mechanical seals continue to fail in centrifugal slurry pump service. There is a definite need for a reliable centrifugal pump to handle slurries at high temperatures and pressures.

Currently, positive displacement pumps are being used for high differential pressure slurry pumps. Both plunger and positive displacement reciprocating pumps have been used with success. However, these pumps are limited in size and require frequent maintenance. As plants increase in size, the number of reciprocating pumps required imposes severe space requirements. In one study by the Ralph M. Parsons Company, it was determined for one particular application that one centrifugal pump could replace five reciprocating pumps at 20 percent of the cost. Currently, studies are underway to develop multi-stage, moderate rpm, high pressure centrifugal pumps.

Centrifugal pumps for transporting slurries at low pressures have also experienced problems. To reduce wear, wetted parts have been coated or hard-faced. For abrasive slurries, severe erosion is experienced at 3600 rpm pump speeds. Proper mating of materials and design considerations is necessary in slurry centrifugal pump applications. A low speed, of the order of 1500 rpm, will tend to reduce erosion, as will use of a pump with proper head-volume characteristics. Careful attention should be given to impeller and volute geometric design.

Turbines

The limitations of current turbine materials are related to cleanliness of the inlet gases. Alkali metal impurities, vanadium, sulfur, char, and ash seriously impair the use of conventional gas turbines. Therefore, the cleaner the gas, the higher the allowable inlet temperature to the turbine. For clean gas, this temperature currently is about 650°C (1200°F) for power recovery turbines and 1040°C (1900°F) for power generation turbines.

Currently, there are no high temperature particulate removal devices in commercial service capable of removing small ($<2\mu$) particulates at the required efficiency. It has been postulated that cyclones, followed by granular bags of fabric filters made of high-temperature resistant materials would be capable of better than 99 percent removal of particulates to the 2μ level and 90% or more in the 2μ to submicron range. Indications are that low levels of small particulate matter may be tolerated in turbine inlet gas streams. As an alternate to "cleaning" hot inlet gases to turbines, the materials and design engineers are being challenged to develop equipment that will tolerate corrosive/erosive particulates. Excellent success with hot "dirty gases in turbine expanders has been achieved in petroleum refinery fluid catalytic cracking units. These units use single stage power recovery units that may be used at inlet pressure up to about 350 KPa (50 psi). Hot gas expanders have been used up to 850 KPa (125 psi) with clean gas. What is needed are advanced super alloys, cooling technology, and multi-stage turbine designs for high pressure, high temperature inlet gases. Less efficient design considerations utilizing heavier vanes, slower tip velocities, and lower blade path gas velocities may have to be developed to provide increased service life.

Heat Exchangers

Coal gasification and liquefaction plants will use heat exchange equipment such as shell and tube, double pipe, finned tube, air coolers, and direct water injection coolers. The principal heat exchange problem areas in coal conversion plants are:

- High pressure and high temperature closures
- Erosion in solid containing fluids
- Fouling and plugging
- Corrosion
- Prediction of heat transfer rate
- Uniform flow distribution

An example of material/design interfacing in heat exchanger design is the techque of extending tubes several inches beyond the tubesheet at the inlet end for erosive service. In one case, the life of tubing was practically doubled by increasing tube-length 100 mm (four inches). The protruding tube ends were attacked, but operation was not affected.

Fired Heaters

Fired heaters are used to heat feed streams in coal liquefaction plants. Heater coils for two and three phase flow containing solids are subject to erosion. Low stream velocities can result in plugging or coking. Pilot plants have used spiral coils with little erosion losses. Plants that have used return bends have experienced severe erosion. Some success has been obtained by providing wear resistant surfaces such as ceramic liners in the return bends. More materials experience is required with the use of hairpin tubes.

Instrumentation

In addition to the standard instrumentation requirements for a process plant, coal conversion plants present the following challenges; on-line composition monitoring of process streams, and measurement of mass flow rates of mixed phase streams, levels of fluidized beds, and temperatures in reaction vessels and on vessel walls.

Flowmeters such as venturi meters which rely on differential pressure measurements for determining mixed phase mass flow are unsatisfactory because of plugging of the pressure taps. Flowmeters such as orifice and turbine meters which have obstructions in the flow stream are subject to excessive erosion. However, ultrasonic techniques for flow measurements appear promising.

Measurement of very small particulate matter in gas streams is extremely important for protection of turbines in pressurized fluidized-bed combustion systems. Particle sizes are generally less than a few tenths of a micron; the loading is usually less than 23 g/m^3 (10 grains per standard cubic foot) of gas; temperatures are in the range of 900 to 1100°C (1600 to 2000°F) or greater; and pressures may be as high as 31 MPa (4500 psi).

Design/materials considerations are in obtaining samples and reclosing the system in the presence of high temperature, high pressure, and the abrasive properties of the process material.

Measurement of temperatures up to as 1650°C (3000°F) or greater is a problem in gasifier and combustion systems. The higher temperature systems tend to be those for which temperature fluctuations must be detected and responded to rapidly and a heavy thermowell introduces a delay in response. Thermowells must be capable of surviving severe temperature, pressures, corrosive and erosive conditions.

Hydroclones and Cyclones

Hydroclones are simple separators in which there are no moving parts. They have been used in coal liquefaction plants for solids/liquids separation. Because of high tangential velocities, erosion resistant materials are required. However, at higher temperatures, high thermal stresses in the nozzle area can cause failure of erosion resistant materials because of their brittleness. In multicone units, differential expansion between the metal housing and the ceramic hydroclone bodies has occured. Designs must allow for thermal growth, or suitable materials must be developed. Hard chrome plating of stainless steel feed and body sections has had some success, but has provided only mediocre service.

Cyclones are similar except that separation is to remove solids from gas or vapor streams. The environmental conditions are similar to those found in gasifiers, except velocities may be 20 to 30 m/s (70 to 100 feet per second) in the internals.

SUMMARY AND CONCLUSIONS

The technology for more extensive utilization of the nations coal resources is available now. Many of today's advanced processes for coal conversion are viable with existing state-of-the art materials and design concepts. However, there is a fertile field awaiting the corrosion engineer and equipment designer; by combining their talents, greater strides can be made to improving plant reliability and overall hardwear costs than can be made by individual discipline efforts alone.

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Table 1 - Gasification Processes
Examples of Environmental Conditions

Area	Methods	Constituents	Problems
I Coal Preparation	A. Dry Grinding	Coal & Low O_2 Air	Moisture & SO_2 in stream of flue gas, sulfur in coal, erosion Temperatures for dust removal are limited by bag materials (can use Venturis for wet scrubbing)
	B. Wet Grinding	Coal & Water	Corrosion - Sulfur in coal attacking metals Erosion - Solids
II Coal Feed Inspection	A. Lock Hoppers Lurgi CO_2 Acceptor	Coal, Pressurizing Gas	Valve wear due to erosion
	B. Slurry Feeding & Vaporizing IGT Hy-Gas BCR Bi-Gas	Coal with either Oil or Water Heating medium is process Gas Containing CO, H_2, H_2S, CO_2, H_2O, CH_4, and possibly Oils & Tars	Pump wear due to erosion Piping wear due to erosion Flow metering - erosion Gas velocity - 35 to 100 ft/sec Liquid velocity - 6 to 10 ft/sec Erosion within injector
	C. Mechanical Inspection	Pulverized Coal	
III Gasification	A. Moving Bed Lurgi Wellman General Electric GE Gas	Coal, Char, CH_4, CO, CO_2, H_2S, COS, Tars, Oil, H_2O, NH_3, Ash	Erosion Corrosion Temperatures - 1700 to 2600°F Pressures - Atmos to 450 psig
	B. Fluidized Bed IGT Hy-Gas Agglomerating Ash CO_2 Acceptor Synthane Winkler Exxon	Same as Above Plus Inert Solids for CO_2 Acceptor (Calcined Dolomite)	Erosion Corrosion Temperatures - 1500 to 2100°F Pressures - Atmos to 1200 psig
	C. Entrained Flow BCR Bi-Gas Koppers-Totzek Texaco B&W-DuPont Combustion Engineering Foster-Wheeler	Same as Above	Erosion due to char & ash particles Corrosion Also possible slag attack Temperatures 2500 to over 3000°F Pressures - Atmos to 1200 psig

Table 1 - Gasification Processes
Examples of Environmental Conditions (Contd)

Area	Methods	Constituents	Problems
IV Ash Removal	A. Lock Hopper (Dry)	Ash, Inert Gas	Valve wear due to erosion Temperatures to 1200°F Pressures to 450 psig
	B. Lock Hopper (Slurry)	Ash, Cl, Water, H_2S, CO_2, CO	Valve wear Temperatures to 500°F Pressures to 1200 psig
	C. Slurry with Letdown Valves	Same as B, Above	Valve wear - Flashing across valves Temperatures to 500°F Pressures to 1200 psig
V Dust Removal	A. Dry - Electrostatic Dust Bag Cyclones Sand Filters	High Solids Loadings Same Gas as in Gasifier	Erosion Inlet temperatures to 1700°F Pressures to 1200 psig
	B. Wet - Scrubbing Columns Venturi	Same as A	Corrosion - CO_2 in H_2O, H_2S, H_2, chlorides High velocity through Venturi throats
VI Sulfur Removal	A. Absorption Physical Chemical At Low to Moderate Temperatures	CH_4, CO, CO_2, H_2S, COS, H_2O, HCN, Plus Solvent	Corrosion - These processes are in commercial use, satisfactory materials of construction have been established
	B. High Temperature Sulfur Removal Processes (1200 to 1800°F)	Same Gases as above with a strong alkali-based reactive agent either as a solid or a liquid	Corrosion - Caused by alkalis and sulfur compounds Erosion - By solids Temperature

Table 2 - Liquefaction Processes

Examples of Environmental Conditions

Process	Process Steps	Environment			
		Temp (°F)	Pressure (psig)	Flow/Phase Conditions	Constituents
(1) Hydroliquefaction (a) Noncatalytic - SRC	Coal dissolving	800-900	1000-2000	Three-phase (liquid, gas, and solid) upflow - low linear velocity	Coal fines, coal-derived aromatic solvent of high sulfur and nitrogen content. Gases with H_2, CO, CO_2, H_2S, NH_3, HCN, and H_2O
(b) Catalytic	Coal dissolving Catalytic hydrogenation	800-900	3000-4000	Three-phase (liquid, gas, and solid) upflow through packed catalyst bed	Coal fines, coal-derived solvent with high aromatic, sulfur, and nitrogen content
(2) Extraction - CSF process	Donor solvent Coal dissolving	800-900	400	Oil-coal slurry-agitated	Ground coal, coal-derived solvent which has been enriched with H_2
	Solvent hydrogenation	800+	3000-4000	Three-phase (liquid, hydrogen, and catalyst particles) low velocity, upflow	Hydrogen-rich gas with H_2S, NH_3 and H_2O. Coal-derived aromatic liquid of high sulfur and nitrogen content
(3) Pyrolysis - COED	Pyrolysis	800-1150	10-15	Fluid bed; coal and/or char gas from pyrolysis	Ground coal and/or char. Unsaturated pyrolysis vapors composed of tar, H_2O, unsaturated gas, H_2S, CO_2, NH_3, COS, HCN, and char fines
	Char conversion	1800	5-10	Fluid bed - char Syngas product Steam and oxygen feed	Char fines Syngas composed of H_2, CO, CO_2, H_2O, H_2S, NH_3, COS, CS_2, HCN, and entrained char
(4) Indirect - Fischer-Tropsch	Liquefaction (synthesis)	600	400	Gas phase	Syngas of high purity plus products: paraffin hydrocarbons, oxygenated compounds, CO_2, and H_2O

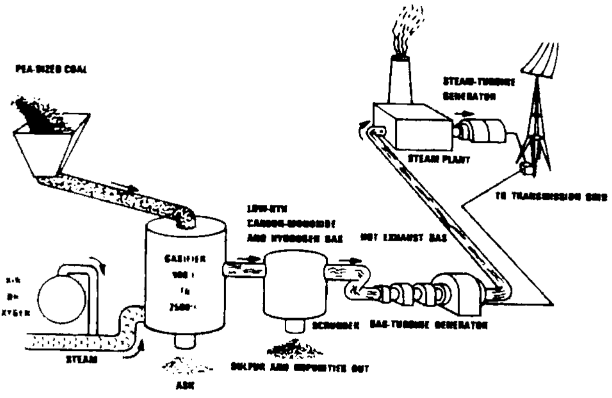

FIGURE 1 - Low-Btu gas for electric power generation.

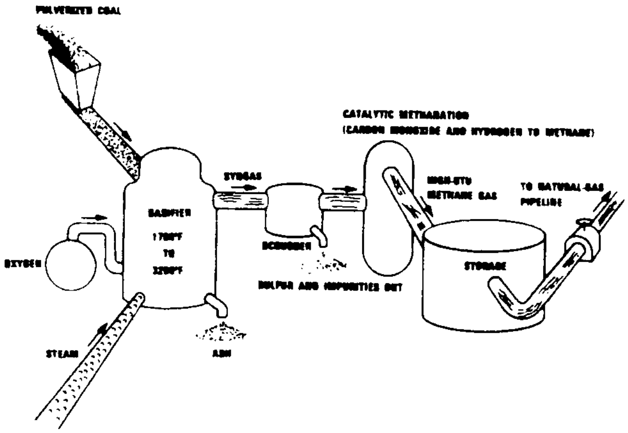

FIGURE 2 - High-Btu gas.

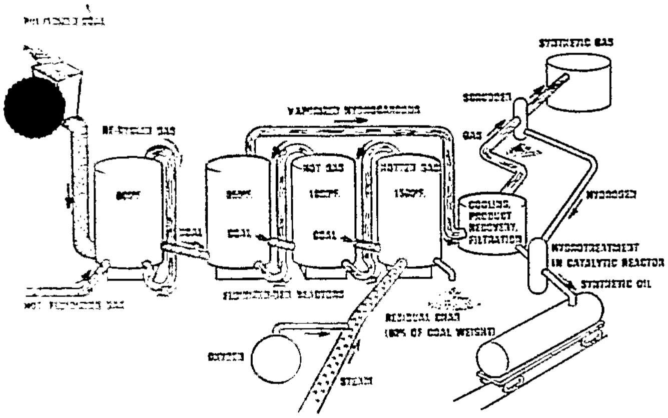

FIGURE 3 - Synthetic oil and gas.

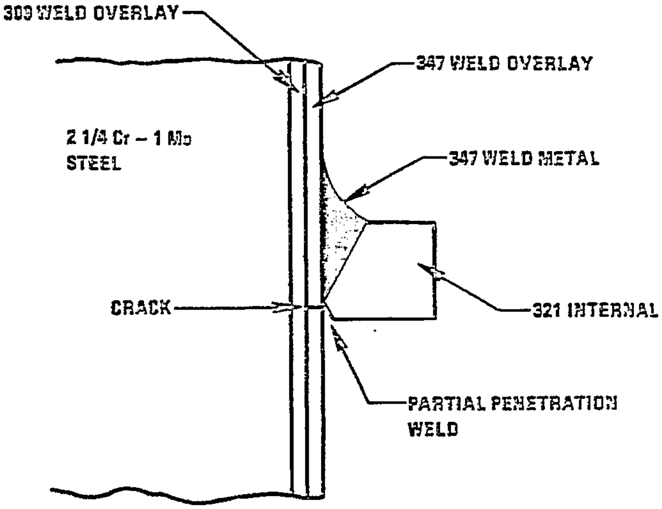

FIGURE 4 - Schematic of pressure vessel internal attachment.

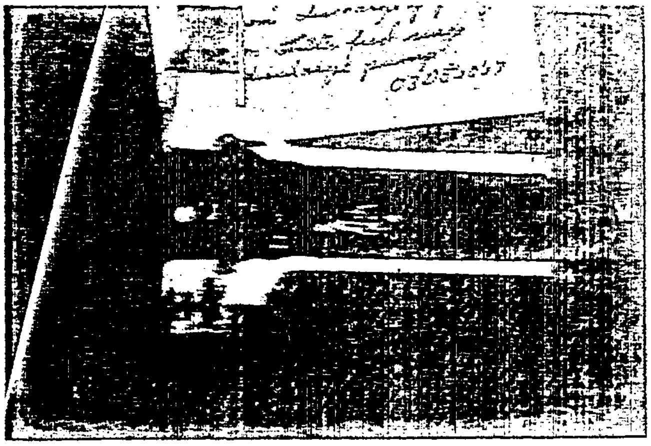

FIGURE 5 - Illustration of pipe erosion (SRC pilot plant).

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COLLECTED WORK NO. 38

MATERIALS CHALLENGES OF COAL LIQUEFACTION

J. B. O'Hara, W. J. Lochmann & N. E. Jentz
The Ralph M. Parsons Company
Pasadena, California

From CHEMICAL ENGINEERING - April 11, 1977

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MATERIALS ENGINEERING FORUM

Materials challenges of coal liquefaction

Planned units face such adversities as erosion, high-temperature degradation, sulfur and hydrogen attack, and stress-corrosion cracking.

J. B. O'Hara, W. J. Lochmann and N. E. Jentz,
The Ralph M. Parsons Co.

☐ To compete with alternative fuel sources, coal liquefaction plants must be reliable, available and easily maintained. A key effort needed to reach this goal is the development and selection of materials for commercial plants that will be large, efficient, and highly capital intensive. (Plants processing 20,000 to 40,000 tons of coal per day will likely be in the billion-dollar class.)

In magnitude, the materials problems facing coal-process designers compare with those of Alaska's North Slope project, for which modular oil and gas production units had to be transported by long ocean voyage from the mainland U.S. to regions where temperatures drop to -50°F.

Previews of the size and layout of commercial coal-conversion plants that we might see within the next 10-15 years are revealed in conceptual designs and economic studies being developed and published under ERDA (Fossil Energy) sponsorship [*1,2,3,4*]. These designs indicate that the equipment will be large and subject to hostile services where most, but not all, problems have been solved. Equipment size and materials may well be restricted by the manufacturers' ability to fabricate and erect it—in addition to operational constraints.

Liquefaction process types

Germany produced most of its aviation gasoline from coal during the latter part of World War II, with the major portion of this production being done by hydrogenation. Though materials problems were severe, these plants still produced approximately 80,000 bbl of liquids/d [*5*].

There is renewed interest in the U.S. in Fischer-Tropsch indirect liquefaction technology, also used in Germany, and still used in the Republic of South Africa's SASOL plant. The latter is reported to have an 85% operational availability record [*6*].

Liquefaction processes under development in the U.S. may be somewhat arbitrarily grouped into four classifications: hydroliquefaction, extraction, pyrolysis, and indirect liquefaction. Operating conditions and stream descriptions are summarized in Table I.

In *hydroliquefaction* units, coal slurried in a coal-derived liquid is contacted with a hydrogen-rich stream at elevated temperatures and pressures to increase the hydrogen-to-carbon ratio of the coal feedstock, and yield a liquid product [*7*].

In *extraction* processes, a hydrogenated coal-derived liquid contacts the feed coal at elevated temperature and pressure to extract a major portion of the carbonaceous coal constituents [*8*]. The hydrogenated, coal-derived liquid serves as a hydrogen carrier to the extraction stage, which can be operated at a lower pressure than the hydroliquefaction process.

In *pyrolysis*, feed coal is heated to an elevated temperature to produce a gas, a tar, and a char [*9*]. The char may be gasified to produce a mixture of carbon monoxide and hydrogen known as syngas, which in turn can be used as a feedstock for SNG, ammonia, methanol, or liquid hydrocarbon production. The gas can also be used as fuel for electric power generation. The tar can be hydrotreated to produce a low-sulfur syncrude.

In *indirect liquefaction*, feed coal is gasified to produce a syngas, which is purified and then reacted to produce liquid products. Examples include Fischer-Tropsch techniques [*10*], and methyl-fuels production.

Hydroliquefaction materials needs

Process conditions for one version of a hydroliquefaction conversion process are described in Table I. Typically, the dissolving (or liquefaction) step takes place at about 800–850°F and 1,500–2,000 psig.

A prevalent problem that can be quite severe is erosion/corrosion of equipment during the handling of coal slurries. Operating plants continue to seek a commercially available centrifugal pump to perform satisfactorily for extended periods while pumping coal slurries.

Another problem is pressure-letdown valve service

Operating conditions encountered in four coal-liquefaction services

Table I

Process	Process steps	Environment Temperature, °F	Pressure, psig	Flow/phase conditions	Constituents
Hydroliquefaction (a) Noncatalytic (SRC)	Coal dissolving	800-900	1,000-2,000	Three-phase (liquid, gas, solid). Reactor upflow at low linear velocity	Coal fines; coal-derived aromatic solvent of high sulfur and nitrogen content. Gases with H_2, CO, CO_2, H_2S, NH_3, HCN, and H_2O
(b) Catalytic	Coal dissolving Catalytic hydrogenation	800-900	3,000-4,000	Three-phase. Upflow through packed catalyst bed	Coal fines; coal-derived solvent with high aromatic, sulfur, and nitrogen content
Extraction (CSF)	Donor solvent Coal dissolving	800-900	400	Oil-coal slurry Agitated	Ground coal; coal-derived solvent enriched with H_2
	Solvent hydrogenation	800+	3,000-4,000	Three-phase (liquid, hydrogen, and catalyst particles). Low-velocity upflow	Hydrogen-rich gas with H_2S, NH_3 and H_2O. Coal-derived aromatic liquid of high sulfur and nitrogen content
Pyrolysis (COED)	Pyrolysis	800-1,150	10-15	Fluid bed; coal and/or char gas from pyrolysis	Ground coal and/or char. Unsaturated pyrolysis vapors composed of tar, H_2O, unsaturated gas, H_2S, CO_2, NH_3, COS, HCN, and char fines
	Char conversion	1,800	5-10	Fluid bed handles char. Syngas product. Steam and oxygen feed	Char fines Syngas composed of H_2, CO, CO_2, H_2O, H_2S, NH_3, COS, CS_2, HCN, and entrained char
Indirect (Fischer-Tropsch)	Liquefaction (synthesis)	600	400	Gas phase	Syngas of high purity plus products: paraffins, oxygenated compounds, CO_2, H_2O

for process slurries after the liquefaction step. In the older German coal-hydrogenation plants (which operated at higher pressures than do U.S. units), pressure letdown valves had an operating life of 500 to 1,500 h [*11*]. Current U.S. pilot-plant experience has demonstrated a similarly modest life span but indicates that 3,000 h may be reached with improved designs and materials—for example, the use of multiple letdown valves in series, and tungsten carbide trim.

Severe metal loss tends to occur in high-velocity regions, with erosion concentrated at the seat, plug, and the exit downstream of the seat. Sintered tungsten carbide applied to seats and plugs has met with some success. However, in many instances extreme wire-drawing has resulted in loss of positive shutoff functions in a matter of weeks.

Little is known about the mechanisms of erosion/corrosion attack. Existing data have been derived primarily from empirical test results, and from analysis of operating experience.

For some noncorrosive services, there appears to be a relationship between a slurry particle's size, speed and hardness and its erosive effect on a particular material. But opinions differ. It is not now possible to reliably predict material loss due to erosion or erosion/corrosion. Current designs depend heavily upon data gathered on materials exposed to actual or simulated operating conditions.

A common design guideline for transport of slurries is to use a velocity of 7–10 ft/s. Little erosion may occur where flow is laminar. However, substantial loss may occur at elbows and tees where flow is turbulent.

A number of materials have been tested in pilot-plant centrifugal pumps handling coal slurries. These materials include carbon steel, 300-series stainless steels, Ni-hard, and coated carbon steel. Coatings that have been tried include chromium, ceramics, plastics, elastomers, and hard surfacing materials such as fused coatings that contain nickel, cadmium, boron and silicon.

The pumps have performed moderately well in many instances, with erosion occuring most often at the impeller periphery, balancing holes, and other areas in the casing. Metal losses tend to be localized, and have not been quantified yet on a mils-per-year basis. Service lives in the range of 700–2,000 h have been attained, but not without maintenance difficulties.

(As an illustration of the type of attack that can occur in centrifugal pumps, Fig. 1 shows the effects of erosion on the discharge port of a pump that handled a slurry bearing 10–15% solids. This pump, constructed of 12% Cr steel and used to transport a slurry to the filter at the Tacoma SRC plant, is of the high-head, low-flow type which operates near shutoff on the pump curve. Evidence indicates that considerable internal recirculation enhanced the erosive action of the slurry.)

Proper mating of materials and design is essential in centrifugal pump slurry applications. A reduced impeller speed of about 1,500 rpm, with adequate head-volume characteristics, will tend to lessen erosion. Adjustments can also be made to impeller pitch and volute geometry. For long-term service, materials should be ductile; easily cast and welded; small grained; able to retain an erosion-resistant smooth surface; resistant to high-temperature, high-pressure H_2S/H_2 attack; and immune to stress-corrosion cracking. Most likely candidates are chrome-moly alloys with and without protective coatings.

The slurry preheater proved a concern in the two U.S. SRC pilot plants located in Tacoma, Wash., and Wilsonville, Ala. In these plants feed-coal slurry and hydrogen are heated from approximately ambient temperature to 800°F in helical coils. The Wilsonville coil is made of Type 316 stainless steel, the Tacoma coil of Incoloy 800. Both are still in operation—the Wilsonville coil having passed 350 days of operation and the Tacoma one 300 days. Commercial-scale plants are expected to use different coil geometry, which may pose new obstacles for the multiphase slurry-preheating step. If U-bend tubes are employed, erosion will be enhanced.

Problems have also cropped up with check valves in the plunger-type feed pumps preceding the high-pressure dissolver section. A combination of materials selection and valve design has extended the life of these components. Use of a spherical, hardened chromium-steel ball on a tungsten carbide seat has raised check valve life from 4 h to as long as 1,600 h.

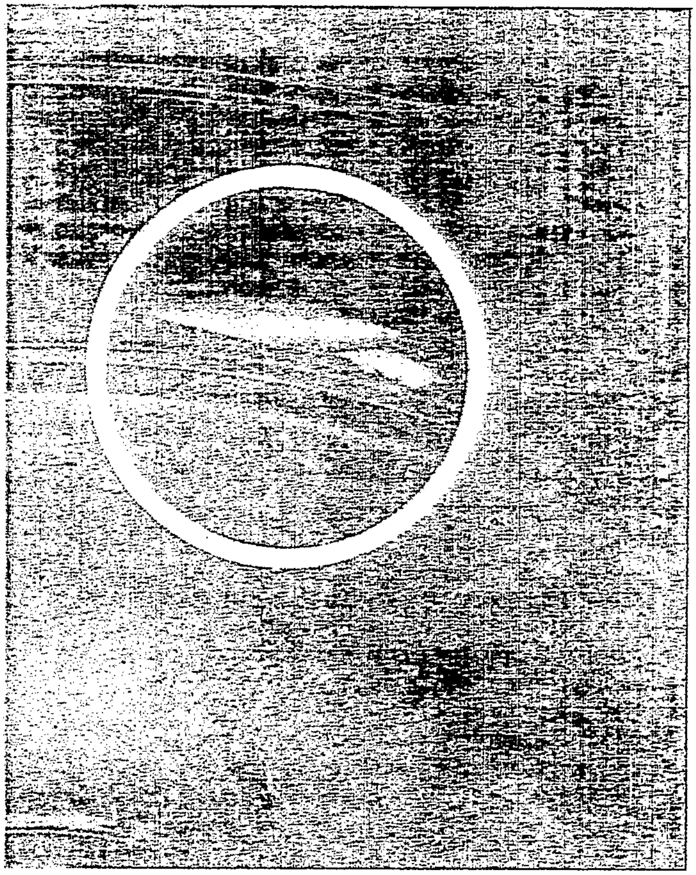
Currently, positive-displacement pumps are being employed for high-differential-pressure slurry feed pumps. Both plunger and positive-displacement reciprocating pumps have been used with success. However, these pumps are limited in size and require frequent maintenance. As plants increase in size, the number of reciprocating pumps required imposes extreme space requirements. In a Parsons study, it was found that in one particular application, a single centrifugal pump could replace five reciprocating pumps at 20% of the cost. Currently, studies are underway to develop multistage, moderate-rpm, high-pressure centrifugal pumps for this application.

Other materials difficulties occur in fractionating columns and reboilers in the product recovery section. Carbon-steel U-tubes in reboilers that distill intermediate coal-derived fractions at temperatures in the range of 400–700°F have been replaced with Type 304 stainless steel. In another unit, Type 410 stainless steel failed after about 150 d at temperatures near 600°F and were replaced with Type 304 stainless construction. The latter has since been replaced with Type 316, which has likewise proved marginal. Sample tubes of Incoloy 800 have so far incurred negligible corrosion.

The dissolver, where the coal slurry is hydrogenated, is exposed to a corrosive environment. Potential troublemakers are sulfur compounds, hydrogen, and such other compounds as are shown in Table I.

Sulfur and sulfur compounds

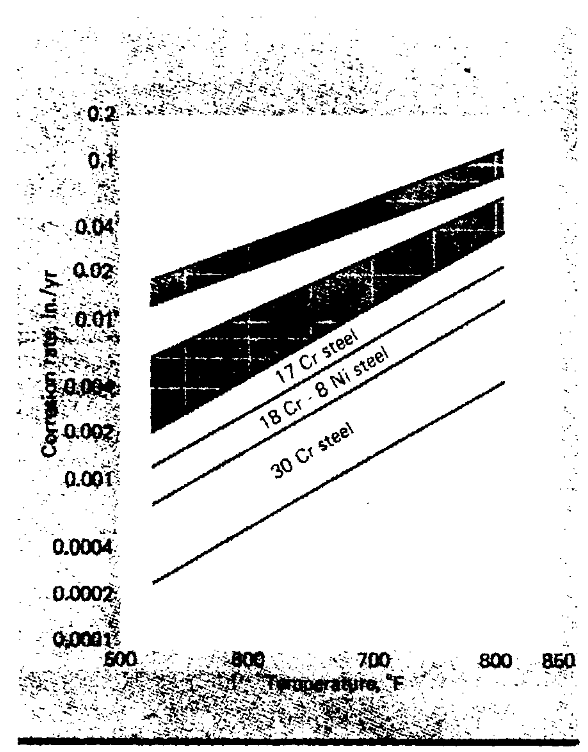
Coal, especially that found in the Eastern part of the U.S., contains substantial sulfur. During processing, much of this is converted to hydrogen sulfide (H_2S), which becomes increasingly corrosive to carbon steel at temperatures greater than 450–550°F. Hydrogen is also present. Here we can draw upon the experience of petroleum refineries, which have collected extensive data on H_2S corrosion at temperatures from ambient to about 1,000°F, and at operating pressures to 3,500 psi.



Centrifugal pump handling coal slurry suffered erosion damage in discharge section Fig. 1

The combination of H_2S and H_2 is particularly troublesome, since the 5 Cr-½ Mo and 9 Cr-1 Mo steels commonly used to resist sulfur corrosion at moderately high temperature show little improvement over carbon steel in their resistance to H_2S/H_2 atmospheres. A chromium content of at least 12% is required. The 300 series of austenitic stainless steels (minimum 18 Cr-8 Ni) have excellent resistance and are normally specified for the H_2S/H_2 environments found in petroleum refineries. Extensive corrosion data [*12*] have been developed for various stainless steel materials in H_2S/H_2 atmospheres at temperatures to about 1,000°F.

At temperatures between 800°F and 1,550°F, the austenitic stainless steels tend to sensitize and precipitate carbides along grain boundaries. In the event of process upsets, it is conceivable that the dissolver temperature could exceed 850°F. The tendency toward sensitization may be diminished by reducing the alloy's carbon assay, or by tying up the carbon by adding small amounts of stabilizing elements such as columbium, tantalum or titanium. Welding and fabricating problems are compounded by the alloying additions; and a reduced carbon content, though beneficial, does not eliminate the possibility of polythionic and chloride cracking problems at low temperatures.



Corrosion rate of stainless steels when exposed to mixtures of H_2 and H_2S **Fig. 2**

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Petroleum refinery experience shows that although the austenitic stainless steels resist high-temperature H_2S during operation, they may be susceptible to stress-corrosion cracking when the plant is shut down. Therefore, strict precautionary shutdown procedures, which include circulation of protective solutions, must be followed during downtime to prevent cracking.

In large equipment, such as pressure vessels, where these procedures are impractical, the vessels are made of other than solid stainless steel; such construction not only prevents catastrophic failure of the vessel due to cracking, but also saves money. Clad or weld overlay construction is recommended because polythionic and chloride cracking are not known to propagate into the commonly used ferritic-alloy backing materials.

Hydrogen attack

Hydrogen at temperatures and pressures greater than 450°F and 200 psi will cause carbon steel to decarburize, forming methane at the interstices and building up internal pressure to induce blistering and failure. Adding molybdenum and chromium as alloying additives suppresses this tendency because of their strong carbide-forming characteristics, and permits the use of these alloys at higher hydrogen pressures and temperatures. Molybdenum also improves the creep and stress-rupture strengths.

Performance of carbon steel, carbon-molybdenum steel and the chromium-molybdenum alloys in hydrogen atmospheres has been well documented in empirical curves prepared by the API's Committee on Refinery Equipment for Corrosion [*13*]. These metals are commonly used as the pressure-retaining materials for equipment in which metal temperatures approach 1,000°F.

Chlorides and stress-corrosion cracking

Chloride concentrations in the range of 100 ppm, with a companion moisture content in the range of 1.5%, can be expected in the hydroliquefaction dissolver. The potential for stress-corrosion cracking in the SRC pilot plants has recently received attention.

The Project Lignite process development unit at Grand Forks, N.D., has suffered failure of its Type 316 stainless-steel preheater, and general stress-corrosion cracking in the high-pressure area, when processing lignite by an SRC-type technique. Chlorinated hydrocarbons had been used to clean some of the equipment prior to failure. Analysis indicated the cause of failure was chlorides. A conclusive explanation of the mode of chloride failure is being sought in view of the successful materials performance record of the two SRC pilot plants.

Extraction processes

Extraction step—Processes using extraction, where hydrogen is transferred to the coal by a donor solvent, encounter most of the problems previously described for hydroliquefaction. The initial extraction takes place at a lower pressure than in hydroliquefaction (about 400 psig), which reduces the material requirements at that stage for resistance to high-pressure, high-temperature H_2 attack. However, both processes have a number of problems in common, including slurry handling, letdown valve erosion, sulfur-compound corrosion, and the potential for chloride stress-corrosion cracking of austenitic stainless steels.

A number of materials and engineering problems were pinpointed during the operation of the OCR-sponsored Consol Synthetic Fuel pilot-plant program at Cresap, W. Va., over the period 1966 to 1970. Some conclusions drawn from this program are summarized here:

Centrifugal pumps handling slurries had an initial service life of 80 to 100 h before they were coated with an electrodeposited, hard-chrome plating. This action increased the service lives of these pumps by more than 400 percent.

In another instance, hard chrome plating was applied to the bottom of hydroclones used for separating solids from a slurry containing approximately 50% solids. The objective of this treatment was to eliminate a spiral wear pattern, which showed up after approximately 100 h of operation. After plating, 1,800 h of service were obtained.

Ni-hard pump components have been used with some success in slurry systems, but because of their brittle nature are vulnerable to thermal shock if they are doused with water during a fire.

No really satisfactory answers were found for lengthening maintenance-free life of valves in slurry service. Ball valves are used at lower temperatures and open-port construction is recommended. Tungsten carbide has proved the most successful material for pressure-letdown valve seating surfaces. However, even tungsten carbide can be destroyed within 500 h of operation, although as much as 2,000-3,000-h service has been reached. The considerable differences in performance are influenced by the method used to form the tungsten-carbide valve seat.

Because of the critical nature of valve performance in coal-conversion plants, a program to develop improved valves is underway at the ERDA facilities in Morgantown, W. Va. Additional work to investigate materials for improving valve performance is underway at Bureau of Mines units located in College Park, Md., Rolla, Mo., and Albany, Ore.; and also at Battelle Laboratories in Columbus, Ohio, under EPRI sponsorship.

Hydrogenation step—Hydrogenation of coal extract is done at pressures up to 4,000 psig and at temperatures in the range of 775–850°F in the presence of hydrogen and a catalyst. Two equipment items are critical: the reactor and the recirculating pumps. Type 347 stainless steel was used as the contact metal for the hydrogenation reactor in the Cresap pilot plant. It performed satisfactorily during the operating time it had, which was too short to provide a meaningful test of material performance.

For hydrogenation, factors such as performance in contact with H_2S/H_2 mixtures must be considered during design of commercial units. At present, we recommend 2¼ Cr-1 Mo reactor weld overlaid with Type 347 or 308L stainless steel.

The technology of large pressure-retaining reactors—approximately 10 ft dia. and 40 ft long, with a wall thickness of about 12 in., and operating at temperatures of 850°F—has been fairly well established for hydrocracking process reactors in the petroleum industry.

Some success was attained with canned pumps used to provide an ebullating bed effect at the Cresap plant, but it appears that more experience is needed in material performance to assure reliable operation in commercial-scale units at 3,000 psi and 850°F.

Pyrolysis

Reaction conditions in a COED (Char-Oil-Energy-Development) pyrolysis plant are summarized in Table I. The results of materials testing in the pilot plant [14], and recommendations for materials in a commercial plant [4], have already been published.

In COED pyrolysis, coal is heated in fluidized-bed reactors in successive stages over a temperature range of 600 to 1,100°F at a pressure of 10 psig. Char gasification takes place at temperatures up to 1,800°F. No serious material problems were experienced at the COED pilot plant during the course of its operation in 1970–74 at Princeton, N.J. In a commercial unit, however, the higher-temperature char-gasification vessels should be of refractory-lined carbon steel.

The hydrotreating process used for making coal oil by pyrolysis is similar to that used for first-stage hydrocracking of petroleum stocks in the petroleum industry, with the exception that operating conditions are more severe. Previous comments regarding sulfide corrosion and hydrogen attack are applicable, and again, designs using a Type 347 stainless steel weld overlay are recommended.

Indirect liquefaction

As indicated by the experience of South Africa's SASOL, material problems for the liquefaction section of a Fischer-Tropsch plant are not severe. This is because the feed to this unit is a mixture consisting largely of carbon monoxide and hydrogen, which is produced by gasification and gas purification. (The gasifying section, a major part of the plant, is beyond the scope of this discussion.) The particulate matter and sulfur values have been reduced, so the feed presents characteristics similar to those encountered in petrochemical operations. It is, however, necessary to design carefully to handle the organic acids produced during the synthesis step. These should be neutralized as formed.

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The authors

J. B. O'Hara is Manager of the Energy Dept. of The Ralph M. Parsons Co., Pasadena, CA 91124, and was previously Manager of Process Licensing and Development, as well as Chief Process Engineer. He holds degrees in chemical engineering from the University of Illinois (B.S.), the University of Michigan (M.S.), and Case Institute of Technology (Ph.D.). He belongs to the AIChE, Alpha Chi Sigma, The New York Academy of Science, and the Los Angeles World Affairs Council.

W. J. Lochmann is Chief Materials Application Engineer, Engineering Dept., of The Ralph M. Parsons Co., where he is involved in materials selection, fabrication, and quality control. He received a degree in metallurgical engineering from the Colorado School of Mines and is a member of the American Soc. for Metals, American Welding Soc., Natl. Assn. of Corrosion Engineers, and the Materials Advisory Committee to ERDA.

N.E. Jentz is Chief Process Engineer, Petroleum and Chemical Div. of The Ralph M. Parsons Co., where his responsibilities include design of coal processing units and process evaluation. He received a B.S. in chemical engineering from the University of Wisconsin and an M.S. in chemical engineering from Case Institute of Technology.

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COLLECTED WORK NO. 39

AN OVERVIEW OF . . .
EQUIPMENT FOR COAL CONVERSION

W.J. Lochman and R.D. Howell

THE RALPH M. PARSONS COMPANY
PASADENA, CALIFORNIA

Published in
HYDROCARBON PROCESSING

May 1977

An overview of . . .
Equipment for coal conversion

Simple substitution of more corrosion resistant materials won't solve problems in coal conversion plants. Here is an overview of what's facing designers

W. J. Lochmann and **R. D. Howell**, The Ralph M. Parsons Co., Pasadena, Calif.

FOR VIABLE coal conversion plants to be constructed and operated reliably, the corrosion engineer will be challenged as in few other industries. Simple substitution of more corrosion resistant materials in currently designed equipment is no longer adequate.

For various equipment items, the following overview [illegible]ents programs proposed or already underway in which corrosion engineers and designers interface for the construction of coal conversion plants.

Pressure vessels will be very large and may contain pressures to 35 MPa (5,000 psi) and temperatures over 1,650° C (3,000° F). Field construction may be required. Current thinking is that these vessels will be designed to Section VIII Division 1 or 2 of the ASME Code.

In coal liquefaction, the reaction step (adding hydrogen to form liquid products) requires pressures from 7 MPa to 35 MPa (1,000 to 5,000 psig) and temperatures in the range of 425 to 650° C (800 to 1,200° F). Because of residence time requirements, these reaction vessels are likely to have diameters in the order of 5 to 8 m (15 to 25 feet), lengths of 25 to 40 m (80 to 120 feet), wall thicknesses of 250 to 300 mm (10 to 12 inches) and will weigh up to 2,500 tons. These heavy wall vessels will:

- Increase the possibility of brittle fracture during vessel fabrication, plant startup and downtime. Sufficient toughness must be incorporated into the vessel shell and appropriate operating procedures adopted to avoid failures.
- Increase exposure times for radiographing welds. For field construction of vessels, more powerful radioactive [illegible]rces will require larger isolated radiation areas causing work scheduling problems.

In gasifiers the temperatures range from about 800° C to over 1,650° C (1,500° F to over 3,000° F), with pressures ranging from atmospheric to over 10 MPa (1,500 psi). Because of the temperatures, most new processes have gasifier vessels that include an internal refractory lining. This lining must withstand erosive and chemical attack from gases, slags, coal and ash solids. Because the refractory forms a shield to protect the pressure vessel from disastrously high temperatures, it must have a very high degree of reliability.

Refractory anchors must be attached to the pressure vessel and consideration must be given to anchors being lost during operation or refractory replacement. Welding anchors directly to the pressure vessel may cause material degradation requiring postweld heat treatment. Because of the presence of hydrogen at high temperatures and pressures and potentially corrosive condensates in coal gasifiers, the corrosion engineer must give careful consideration to his selection of refractory anchor and shell materials.

In cold shell gasifiers, corrosive condensates may require protective cladding or weld overlay. Although Type 347 Stainless Steel has been used extensively as an overlay material, its greater propensity for cracking has resulted in the specification of a protective overlay composition equivalent to that of 304 L Stainless Steel for many vessels in petroleum refinery service.

Piping. The large size and high pressure of some coal conversion plants will require piping with diameters as large as 1,830 mm (72 inches) and wall thickness of 75 to 125 mm (3 to 5 inches). Standard bends, Tees and other fittings may not be available, and mitered elbows may be required. Materials to resist erosive and corrosive flow conditions must be considered in their design.

Some common piping design practices will have to be avoided in coal conversion plant lines that contain solids in the form of coal, char or ash. These solids can be quite erosive in both gas and liquid streams. Any protuberances or gaps increase metal loss.

Valves. Erosion, corrosion, wear and abrasion are the damaging mechanisms to satisfactory valve operation. Valving is perhaps the most severely attacked equipment subsystem. Valves are required to perform pressure regulation, flow diversion, process mixing and disposal, as well as to provide positive shut off. Let-down valves in which pressures are dropped 7 MPa (1,000 psi) or more have been a particularly troublesome area. A satisfactory valve to withstand the combination of a two or three phase gas-solid-liquid stream, high pressure drop and high temperature has yet to be developed. While valves have been designed for any one of the above conditions, valve life under combination of conditions may be only two weeks to 90 days. Material substitution alone, i.e., use of harder, more durable materials, has not been adequate. For example, increased use of tungsten and cobalt carbide materials has not achieved satisfactory valve life.

Programs are currently under way to develop suitable valves by closer interaction between valve designers and materials engineers. Also programs are underway to develop and evaluate single-phase material such as high-purity, dense, fine-grain oxides, carbides, borides and nitrides prepared by sintering or hot-pressing, and two-phase cermet types of materials.

Pumps. Various water base slurries containing coal, ore or other materials at ambient temperatures have been successfully pumped for years. However, there is relatively little experience with pumping hydrocarbon based slurries at elevated temperatures.

Erosion problems continue to occur in centrifugal slurry pumps with high suction pressures and low differential pressures. Mechanical seals continue to fail in centrifugal slurry pump service. There is a definite need for a reliable centrifugal pump to handle slurries at high temperatures and pressures.

BACKGROUND

Coal conversion and its use is emerging as a potential major industry. Although many of the basic technologies for converting coal into "clean" liquid, solid, and gaseous fuels have been around for half a century or more, they have been developed only to a limited extent in the United States because the availability of natural gas and the plentiful supply of petroleum have made it uneconomical to do so. For the past decade or so, the sources of energy in the U.S. have been predominantly oil and gas (44 and 31 percent respectively) with coal accounting for 21 percent and all other sources, including hydroelectric and nuclear plants, accounting for four percent. In contrast, coal accounts for 75-80 percent of the nations fossil fuel resources.

Unlike oil, coal, because of its sulfur content, ash and other pollutants, is considered to be a dirty fuel. The principal objective in coal conversion is to transform coal from its "contaminated" solid form to "clean" liquid, gaseous, or low-ash solid products which meet environmental standards. Conversion of "dirty" coal into "clean" fuels is expensive. In the U.S., a number of advanced processes are under development for more economical conversion of coal to clean burning gaseous, liquid, or solid fuels. Over the last decade, a number of processes have reached the pilot plant stage. The new developments are moving toward three main objectives. One plan is to replace much of the natural gas, fuel oil, and raw coal now burned in electric power plants with a cheap "power" gas that would have a rather low heating value [4-25 MJ/m^3 (100-600 Btu/cf)] but would be capable of attaining high generating efficiencies. Another development is to produce a gas of high heating value [30-40 MJ/m^3 (900-1000 Btu/cf)] to supplement natural gas in the nation's pipelines. A third objective is to produce a range of synthetic-oil products, from crude to gasoline.

Currently, positive displacement pumps are being used for high differential pressure slurry pumps. Both plunger and positive displacement reciprocating pumps have been used with success. However, these pumps are limited in size and require frequent maintenance. As plants increase in size, the number of reciprocating pumps required imposes severe space requirements. In one study by the Ralph M. Parsons Co., it was determined for one particular application that one centrifugal pump could replace five reciprocating pumps at 20 percent of the cost. Currently, studies are underway to develop multi-stage, moderate speed, high differential pressure centrifugal pumps.

Centrifugal pumps for transporting slurries at low pressures have also experienced problems. To reduce wear, wetted parts have been coated or hard-faced. For abrasive slurries, severe erosion is experienced at 3,600 rpm pump speeds. Proper mating of materials and design considerations is necessary in centrifugal slurry pump applications. A low speed, of the order of 1,500 rpm, will tend to reduce erosion, as will use of a pump with proper head-volume characteristics. Careful attention should be given to impeller and volute geometric design.

Turbines. The limitations of current turbine materials are related to cleanliness of the inlet gases. Alkali metal impurities, vanadium, sulfur, char and ash seriously impair the use of conventional gas turbines. Therefore, the cleaner the gas, the higher the allowable inlet temperature to the turbine. For clean gas, this temperature currently is about 650° M (1,200° F) for power recovery turbines and 1,040° C (1,900° F) for power generation turbines.

Currently, there are no high temperature particulate removal devices in commercial service capable of removing small (less than two microns) particulates at the required efficiency. It has been postulated that cyclones, followed by granular bags of fabric filters made of high-temperature resistant materials would be capable of better than 99% removal of particulates to the 2 micron level and 90% or more in the 2 micron to submicron range. Indications are that low levels of small particulate matter may be tolerated in turbine inlet gas streams. As an alternate to "cleaning" hot inlet gases to turbines, the materials and design engineers are being challenged to develop equipment that will tolerate corrosive/erosive particulates. Excellent success with hot "dirty" gases in turbine expanders has been achieved in petroleum refinery fluid catalytic cracking units. These units use single stage power recovery units that may be used at inlet pressure up to about 350 KPa (50 psi). Hot gas expanders have been used up to 850 KPa (125 psi) with clean gas. What is needed are advanced super alloys, cooling technology and multi-stage turbine designs for high pressure, high temperature inlet gases. Less efficient design considerations using heavier vanes, slower tip velocities and lower blade path gas velocities may have to be developed to provide increased service life.

Heat Exchangers. Coal gasification and liquefaction plants will use heat exchange equipment such as shell and tube, double pipe, finned tube, air coolers and direct water injection coolers. The principal heat exchange problem areas in coal conversion plants are:

- High pressure and high temperature closures
- Erosion caused by solid containing fluids
- Fouling and plugging
- Corrosion
- Prediction of heat transfer rate
- Uniform flow distribution.

An example of material/design interfacing in heat exchanger design is the techque of extending tubes several inches beyond the tubesheet at the inlet end for erosive service. In one case, the life of tubing was practically doubled by increasing tube-length 100 mm (4 in.). The protruding tube ends were attacked, but operation was not affected.

Fired heaters are used to heat feed streams in coal liquefaction plants. Heater coils for two and three phase flow containing solids are subject to erosion. Low stream velocities can result in plugging or coking. Pilot plants have used spiral coils with little erosion losses. Plants that have used return bends have experienced severe erosion. Some success has been obtained by providing wear resistant surfaces such as ceramic liners in the return bends. More materials experience is required with the use of hairpin tubes.

Instrumentation. In addition to the standard instrumentation requirements for a process plant, coal conversion plants present the following challenges: on-line composition monitoring of process streams and measurement of mass flow rates of mixed phase streams, levels of fluidized beds and temperatures in reaction vessels and on vessel walls.

Flowmeters such as venturi meters which rely on differential pressure measurements for determining mixed phase mass flow are unsatisfactory because of plugging of the pressure taps. Flowmeters such as orifice and turbine meters which have obstructions in the flow stream are subject to excessive erosion. However, ultrasonic techniques for flow measurements appear promising.

Measurement of very small particulate matter in gas streams is extremely important for protection of turbines in pressurized fluidized-bed combustion systems. Particle sizes are generally less than a few tenths of a micron; the loading is usually less than 23 g/m^3 (10 grains per standard cubic foot) of gas; temperatures are in the range of 900 to 1,100° C (1,600 to 1,900° F) or greater; and pressures may be as high as 31 MPa (4,500 psi).

Design/materials considerations are in obtaining samples and reclosing the system in the presence of high temperature, high pressure and the abrasive properties of the process material.

Measurement of temperatures up to as high as 1,650° C (3,000° F) or greater is a problem in gasifier and com[illegible]stion systems. The higher temperature systems tend to be those for which temperature fluctuations must be detected and responded to rapidly and a heavy thermowell introduces a delay in response. Thermowells must be capable of surviving severe temperature, pressures, corrosive and erosive conditions.

About the authors

WALT J. LOCHMANN is chief materials application engineer with The Ralph M. Parsons Co., Pasadena, Calif. He supervises the Materials Application Group which is responsible for resolving material and material related problems associated with metallurgy, corrosion, welding, fabrication and quality control. Mr. Lochmann holds a degree in metallurgical engineering from the Colorado School of Mines. He has previously worked as a corrosion, metallugical and materials research engineer with Shell Oil Co. and as a welding metallurgist on the U.S. Navy Nuclear Program for General Electric Co. He is a member of NACE, ASM, AWS, MPC, ERDA-Fossil Energy Materials Advisory Group and is chairman of the NACE Committee on Fossil Energy, TPC T 12.

RICHARD D. HOWELL is a principal project engineer with The Ralph M. Parsons Co., Pasadena, California. He is responsible for various study projects in coal liquefaction and gasification. Mr. Howell holds a B.S. degree in chemical engineering from the University of Utah. He previously worked with Koppers Co. on coal carbonization and gasification, with U.S. Steel to set up a coal carbonization research lab, with Callery Chemical Co. on high energy fuels, with Aerojet-General Corp. on high energy fuels, with Swift, CB&I and with C F Braun on coal gasification.

Hydroclones and cyclones. Hydroclones are simple separators in which there are no moving parts. They have been used in coal liquefaction plants for solids/liquids separation. Because of high tangential velocities, erosion resistant materials are required. However, at higher temperatures, high thermal stresses in the nozzle area can cause failure of erosion resistant materials because of their brittleness. In multiclone units, differential expansion between the metal housing and the ceramic hydroclone bodies has occured. Designs must allow for thermal growth, or suitable materials must be developed. Hard-chrome plating of stainless steel feed and body sections has had some success, but has provided only mediocre service.

Cyclones are similar except that separation is to remove solids from gas or vapor streams. The environmental conditions are similar to those found in gasifiers, except velocities may be 20 to 30 m/s (70 to 100 feet per second) in the internals.

Conclusion. Many of today's advanced processes for coal conversion are technically feasible with existing state-of-the art materials and design concepts. However, there is a fertile field awaiting the corrosion engineer and equipment designer; by combining their talents, greater strides can be made to improving plant reliability and over-all hard-wear costs than can be made by individual discipline efforts alone.

ACKNOWLEDGMENT

Abstract from the NACE paper "Corrosion Engineering—Design Interface for Coal Conversion" presented in San Francisco, March 16, 1977. ■

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