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**Session 9b**

***Membranes***

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**9b.1**

**High Temperature Size Selective Membranes**

**CONTRACT INFORMATION**

**Contract Number** DE-AC21-92MC29245

**Contractor** AlliedSignal, Inc.  
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Des Plaines, IL 60017  
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**Contractor Project Manager** Stephen F. Yates

**Principal Investigators** S. James Zhou  
David J. Anderson  
Alan E. van Til

**METC Project Manager** Venkat K. Venkataraman

**Period of Performance** September 19, 1992 to September 18, 1994

**FY94 Program Schedule**

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	FY92					FY93					FY94													
	O	N	D	J	F	M	A	M	J	J	A	S	O	N	D	J	F	M	A	M	J	J	A	S
NEPA	—																							
Test Plan	—																							
Membrane Development																								

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**OBJECTIVE**

The objective of this research is to develop a high temperature size selective membrane capable of separating gas mixture components from each other based on molecular size, using a molecular

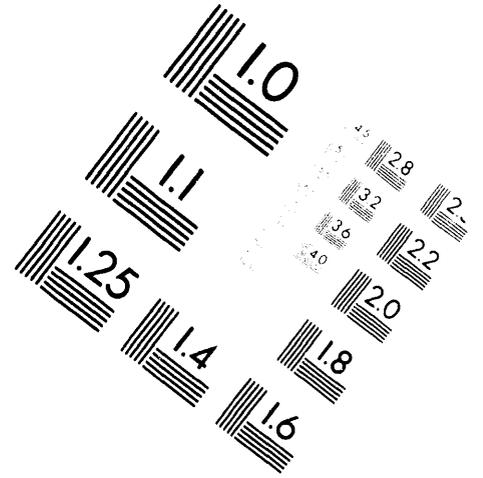
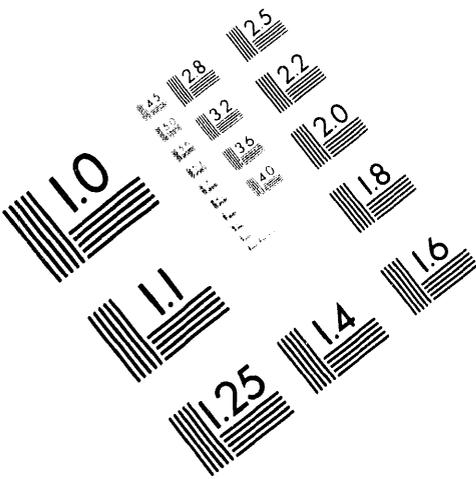
sieving mechanism. We are evaluating two concepts: a composite of a carbon molecular sieve (CMS) with a tightly defined pore size distribution between 3 and 4 Å, and a microporous supporting matrix which provides mechanical strength and resistance to thermal degradation, and a sandwich of a CMS film between the porous supports. Such a membrane would enable the separation of hydrogen



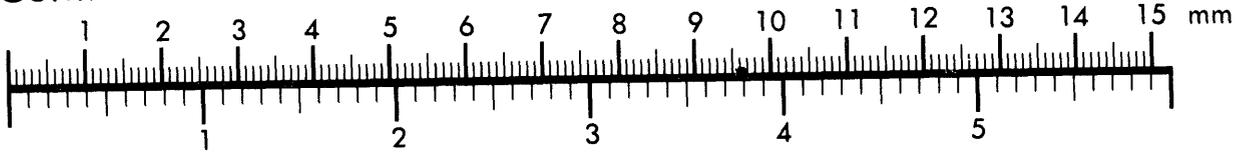
**AIM**

**Association for Information and Image Management**

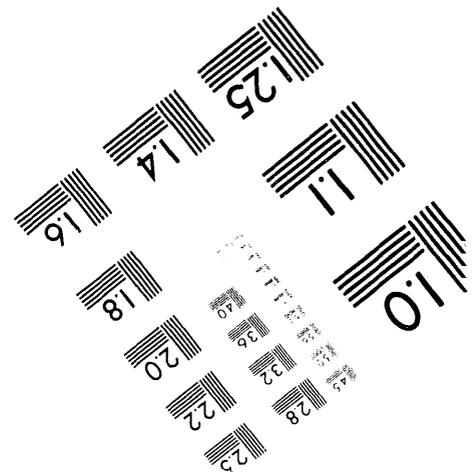
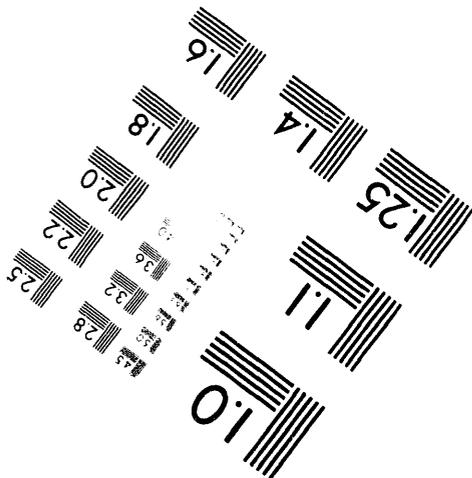
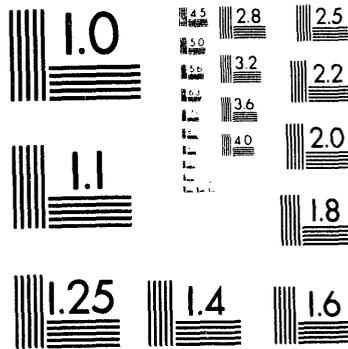
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from other gases.

## BACKGROUND INFORMATION

Membranes that operate at temperatures higher than 100°C and still possess suitable selectivity and last for a few hundred to a few thousand hours are rare. However, the benefit of performing the separation at reactor temperatures in terms of energy cost, capital cost, and process simplicity has led many to work on developing such membranes. The high temperature membranes we are developing can be used to replace the current low-temperature unit operations for separating gaseous mixtures, especially hydrogen, from the products of the water gas shift reaction at high temperatures. Membranes that have a high selectivity and have both thermal and chemical stability would improve substantially the economics of the coal gasification process. These membranes can also improve other industrial processes such as the ammonia production and oil reform processes where hydrogen separation is crucial.

## PROJECT DESCRIPTION

Our CMS membrane is composed of a thin layer of a size selective CMS supported by a microporous ceramic support. The separation of hydrogen from other gases is by a molecular sieving mechanism where the smaller hydrogen molecules (2.96Å) would pass through the pores while the larger molecules such as N<sub>2</sub>, CO, and CH<sub>4</sub> are prevented from passing through.

The ceramic support is a fiber reinforced ceramic composite prepared from AlliedSignal's proprietary Blackglas™ resin. The reinforcing fibers can be carbon, glass, ceramic, or metal depending on the end use temperature. The Blackglas material in its resin form can be processed using the conventional polymer processing techniques such as lamination, infiltration, hot molding, casting, and injection

molding. We have used hot molding, lamination, and infiltration techniques to make the fiber reinforced ceramic composite support. The Blackglas ceramic support has the potential for high temperature strength/stability with ease of fabrication because of the demonstrated chemical and thermal stability of Blackglas. The coefficient of thermal expansion of the support matches closely that of CMS. Such close match insures mechanical integrity and adhesion when the membrane is subjected to thermal cycles.

The CMS membrane is produced by controlled pyrolysis of polymeric precursors on the surface or in the pores of the support. Alternatively, a free standing CMS film can be made without the support and combined with a support structure in a later stage to form the membrane. The pore size of the CMS membrane can be modified by post-treatments.

The composite membrane will be tested for its permeation properties at 550°C or higher. Thermal, mechanical and chemical stability of the membrane will be assessed.

## RESULTS

### Supported Membrane

We have produced fiber reinforced ceramic microporous supports from Blackglas™ resin and carbon, glass, and ceramic fibers. The polymer composite is converted to a ceramic composite by pyrolysis under inert atmosphere (nitrogen). Repeated infiltration and pyrolysis steps are used to increase the density and decrease the porosity of the support. The resulting supports are tested for mechanical strength, pore size and pore size distribution at room temperature. The results are summarized in Tables 1 and 2.

Sample #	YIELD STRESS (psi)	YIELD STRAIN (%)	Modulus (psi)
1	1457	0.36	500500
2	3163	0.39	1031000
3	3709	0.29	1482000
4	5640	0.33	1984000

**Table 1. Flexural Test Results**

Sample #	MEDIAN PORE DIAMETER ( $\mu\text{m}$ )	POROSITY (%)
1	2.4077	23.8
2	1.1787	13.79
3	0.6216	7.02
4	0.2596	3.62

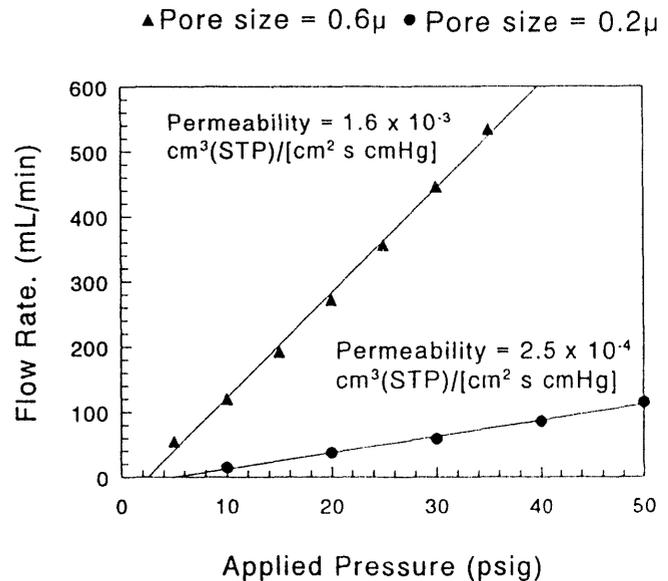
**Table 2. Mercury Intrusion Results**

The stress and strain results shown in Table 1 suggest that the supports are not only strong but also non-brittle as indicated by the yield strain of 0.3 to 0.4%. The pore size of the supports can also be changed to suit our needs as shown by the results presented in Table 2. The pore size distribution of the support is narrow.

SEM micrography of the porous ceramic support structure shows that the pores are formed by microcracks in the composite matrix. These microcracks come from the shrinkage associated with the pyrolysis of Blackglas resin. Thus, the pores generally have an elongated slit structure. Other pore size controlling techniques can also be used to modify the pore size and pore structure of

the support.

We have also measured the flow rate of nitrogen through the support at room temperature. The results are given in Figure 1. This figure shows that the resistance of the support to gas flow is small even at room temperatures and that the flow rate is a function of the support pore size.

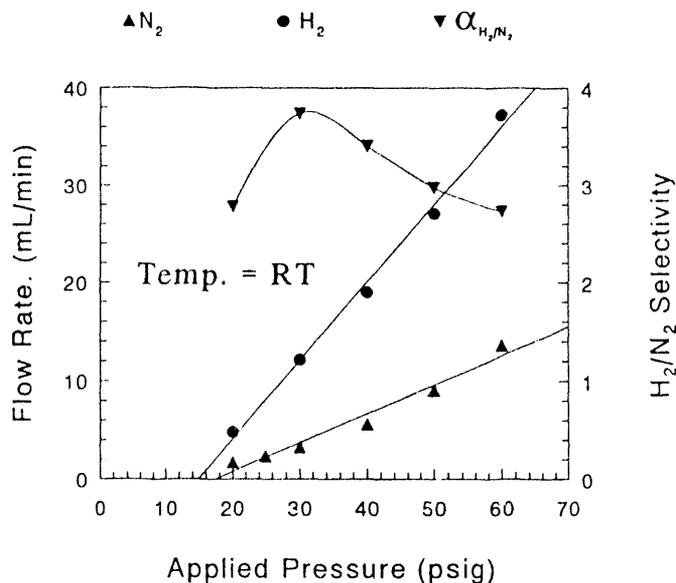


**Figure 1. Nitrogen Flow Rate Through Porous Blackglas Supports**

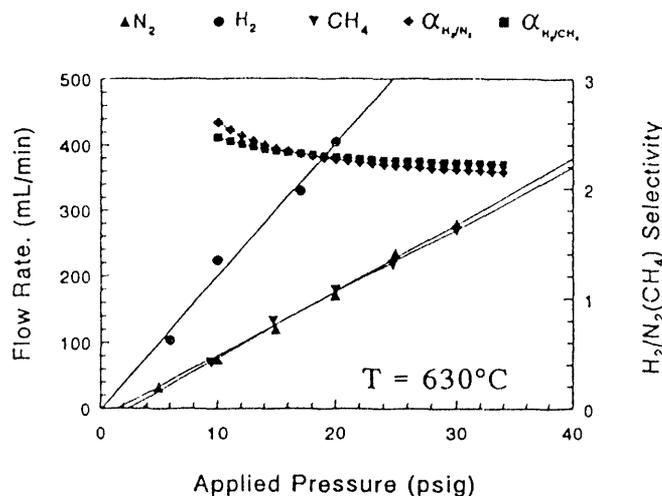
Our CMS membranes are prepared by a coating method where a CMS precursor polymer solution is coated on the microporous support and carbonized in nitrogen or helium. Repeated coating may be necessary to eliminate defects and cracks formed during carbonization because of the shrinkage of the polymer coating. The CMS is formed within the pores and on the surface of the porous support.

We have measured the flow rate and selectivity of nitrogen, hydrogen, and methane through the CMS membranes at both room temperature and at elevated temperatures up to  $700^{\circ}\text{C}$  and at pressures up to 60 psig. The results of these measurements are summarized in Figures 2

and 3. These figures show that the flow rate through the membrane (disk shape with effective diameter of 2.5 inches) increases with increasing temperature as expected. The ideal  $H_2/N_2$  and  $H_2/CH_4$  selectivities are not a strong function of temperature, and have values close to the Knudsen diffusion limit. These figures also show that the flux of all gases are high. The ideal selectivities obtained from the current study are comparable to those obtained by Koresh and Sofer [1,2] on unsupported CMS membranes prepared from cellulose material. The ideal selectivities reported are 20 and 8 for  $He/O_2$  and  $O_2/N_2$  before heat treatment and 2.8 and 0.92 after heat treatment, respectively. Hatori et al. [3] have also reported the following ideal selectivities for an unsupported carbon membrane made from Kapton polyimide:  $He/N_2 = 19.6$ ,  $He/O_2 = 4.3$ , and  $He/CO_2 = 0.4$ . These results may suggest that the pore size of the current CMS membrane is still too large or simply that there are still defects in the membrane. Recently, SEM has confirmed the presence of micro defects on the surface of the CMS membranes we made. Moreover, the permeabilities of our CMS membrane is about two orders magnitude higher than those reported.



**Figure 2. Nitrogen and Hydrogen Flow Rate and Selectivity at Room Temperature**



**Figure 3. Nitrogen, Methane and Hydrogen Flow Rate and Selectivity at Elevated Temperature**

#### Unsupported Carbon Film

We have developed methods for the carbonization of polymer precursor into continuous, defect-free, hydrogen impermeable, carbon films. During carbonization, the films lose 40% of their precursor weight and the isotropic shrinkage is 20%.

A carbon film prepared from the pyrolysis of 3 mil polymer precursor was sealed in epoxy and placed in a membrane test cell. A pressure of 320 psig of hydrogen was applied to one side of the membrane and a pressure of 290 psig of argon was applied to the other side. The static gas pressures were blocked off and allowed to remain for two hours. At the end of two hours the argon was collected and analyzed by gas chromatography for hydrogen. No hydrogen was observed and the limits of detection of the method used were 1000 ppm, which means that the maximum possible flux constant could not have exceeded about  $2 \times 10^{-10}$   $[cm^3(STP) cm]/[cm^2 s cmHg]$ . In a similar experiment, a 5 mil film was configured such that a differential pressure of 60 psig of hydrogen was

applied to one side and allowed to stand for several hours. Flux exceeding  $10^{-11}$   $[\text{cm}^3(\text{STP}) \text{ cm}]/[\text{cm}^2 \text{ s cmHg}]$  was not observed.

We have developed a method for determining the B.E.T. surface area of materials using hydrogen as a probe gas at 19.7 K. The development of the hydrogen B.E.T. method resulted in part from recent developments in temperature control at the condensation temperature of hydrogen using a Janis Research Super Varitemp cryostat. The recently developed hydrogen B.E.T. method can be used in conjunction with the existing and well-established carbon dioxide B.E.T. method. By using both methods, we can directly compare the properties of surface interaction such as surface adsorption between hydrogen and carbon dioxide, the gases of principal interest in this project. In particular, we can study the effects of varying carbonization and activation conditions on the pore structure and its influence on the adsorption of the gases onto the carbon material. The ability to make direct comparisons between the two gases is a tool we can use to tune the pore structure and adsorption properties through variations in our synthetic procedures. Figure 4 shows a pore-size distribution determined with carbon dioxide as probe gas.

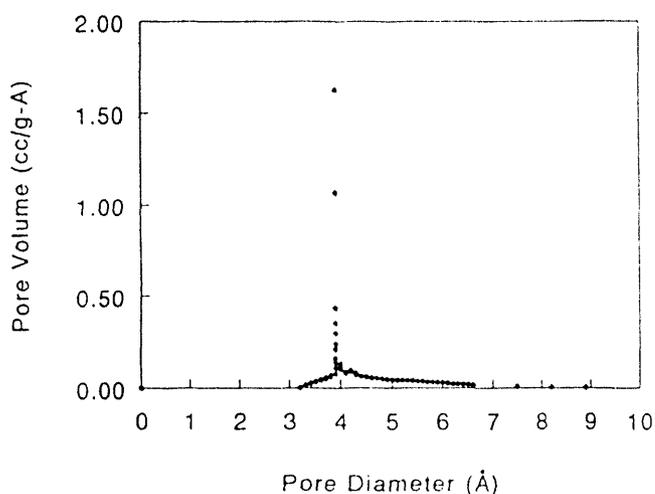


Figure 4. Pore-size Distribution of CMS Film Using  $\text{CO}_2$  as Probe Gas

Surface areas depend on carbonization and activation. Variations in carbonization conditions can be used to control pore size and median pore diameter from 4.2 to 5.1 Å and pore volumes up to  $0.20 \text{ cm}^3/\text{g}$ . Surface areas as high as  $400 \text{ m}^2/\text{g}$  have been measured. The thickness of the polymer film influences the surface area. In particular, the thicker the polymer film, the lower the overall surface area. These observations strongly suggest that an impermeable, non-porous "dead" layer exists within the film. The overall composition of the film may be such that porosity exists in a higher concentration near the surface of the film. We anticipate that development of thinner carbon films may produce a hydrogen permeable material since a thinner film would have less or no dead layer material. Figure 5 shows the measured surface area as a function of precursor film thickness for both hydrogen and carbon dioxide probe gases. Also, activation has been shown to increase the surface adsorption selectively in such a way that the hydrogen pore volume ( $\text{cc/g}$ ) increased from 0.10 to 0.19 but the carbon dioxide pore volume increased from 0.15 to 0.18.

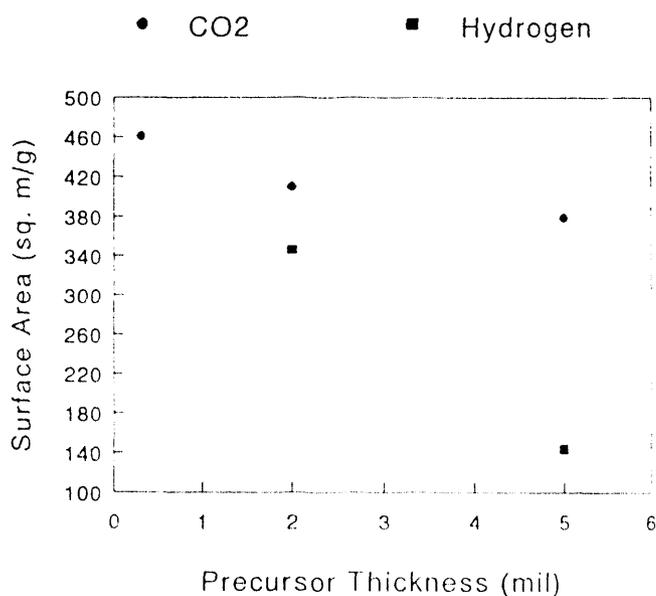


Figure 5. Surface Area vs. Precursor Thickness

## FUTURE WORK

We are working on eliminating the micro defects on the CMS membrane surface to improve selectivity using several techniques. We are also putting efforts into making the CMS membrane just inside the pore mouth of the support, such that the CMS is constrained within the pores, eliminating any possible defects from the fabrication process and from late thermal cycling. The membranes made will be tested at high temperatures for gas permeability and selectivity. The surfaces and the interior of the membrane will also be examined by an SEM method to better understand the development of defects and ways of eliminating them. In addition to obtaining membrane permeabilities for the various gases, we will obtain information on the stability of the membrane for extended hours of operation at elevated temperatures in the reducing environment of the gases.

## ACKNOWLEDGEMENT

The authors wish to thank Damyanti Patel for her efforts in performing the work discussed in this paper, Hayuta Zaffir for her help in preparing and studying the porous supports. We also wish to express our thanks to Dr. Stephen Yates for his support and Dr. Xavier Pillai for his work on setting up the test rig and initial work on the CMS. Thanks are also due to the authors' colleagues Drs. Brian Bedwell, Robert Sedath, and Li Wang for many of their invaluable suggestions and discussions.

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9b.2

## A Catalytic Membrane Reactor for Facilitating the Water-Gas Shift Reaction at High Temperature

### CONTRACT INFORMATION

**Contract Number** DE-FG03-91ER81229

**Contractor** Bend Research, Inc.  
64550 Research Road  
Bend, OR 97701-8599  
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**Contract Project Manager** David J. Edlund

**Principal Investigators** David J. Edlund

**METC Project Manager** Venkat K. Venkataraman

**Period of Performance** March 31, 1992 to March 30, 1995

### Schedule and Milestones

#### FY94 Program Schedule

J J A S O N D J F M A M

Conduct Parametric Tests

Conduct Slip-Stream Tests

Construct Computer Model of Process

Evaluate Process Economics

Prepare Final Report

### OBJECTIVES

This program is directed toward the development of a metal-membrane-based process for the economical production of hydrogen at elevated temperature by the reaction of carbon

monoxide with steam—i.e., the water-gas shift (WGS) reaction. Key to achieving this objective is the development of an inexpensive and durable metal-membrane module. The specific program objectives include the following:

- design, fabrication, and demonstration of prototype membrane modules;
- improving the membrane composition to increase the hydrogen flux;
- demonstrating that membrane lifetime  $\geq 2$  years is likely to be achieved; and
- conducting engineering and economic analyses of the process.

## BACKGROUND INFORMATION

Currently, the gasification of coal incorporates conventional high- and low-temperature WGS reactors for the production of hydrogen, valued as a chemical feedstock (Figure 1). The cost and complexity of the conventional gas-processing technology, with associated heat exchangers and acid-gas scrubbers, has given rise to research directed at developing a more-efficient and less-expensive process.

## PROJECT DESCRIPTION

### The Proposed Membrane-Based Process

The proposed membrane-reactor process for producing hydrogen via the WGS reaction at

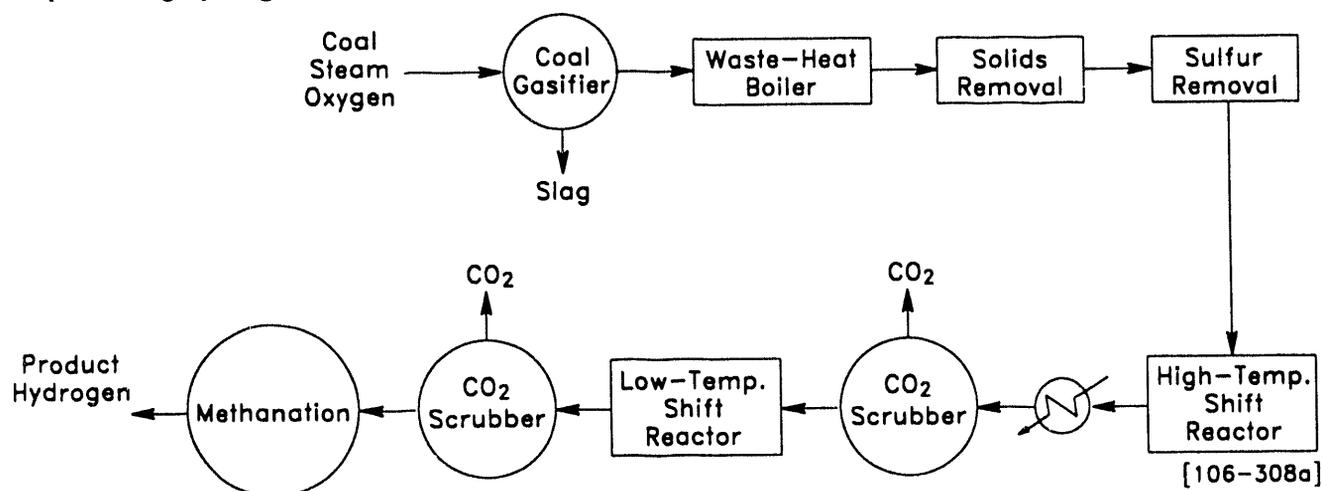
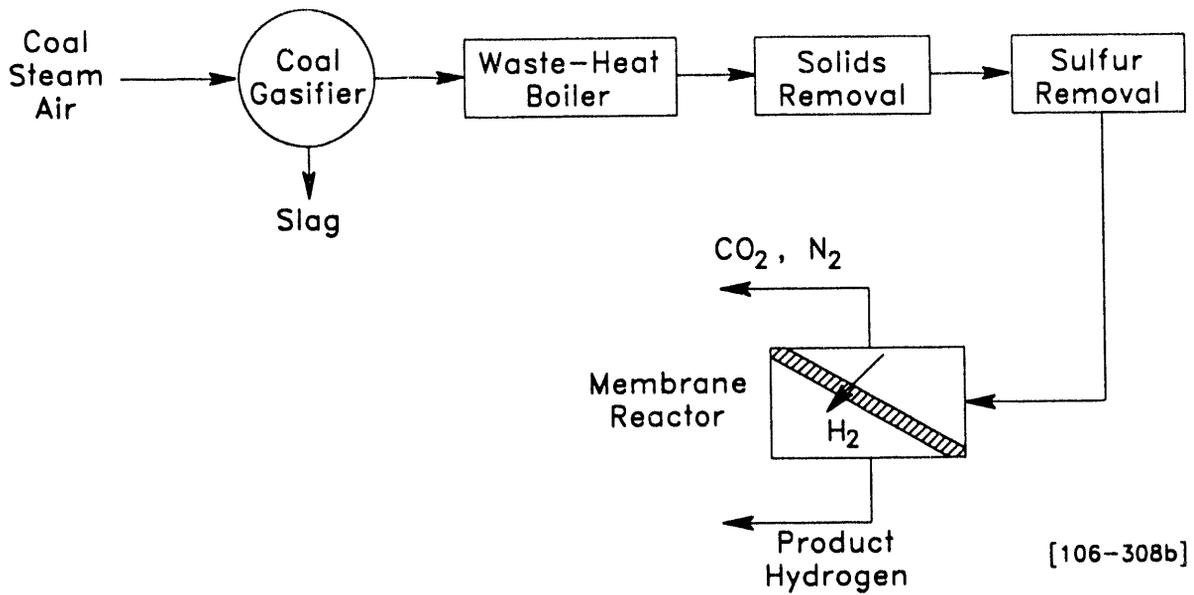


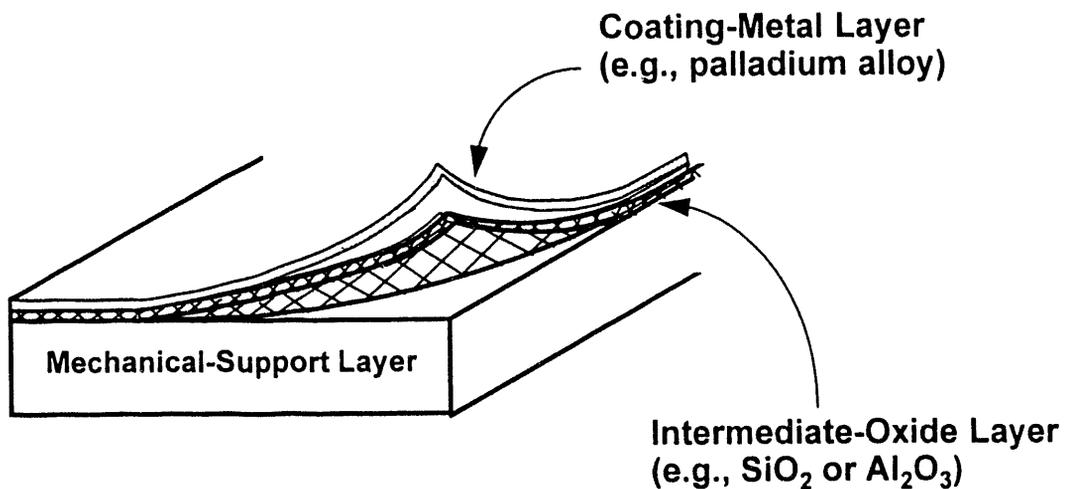
Figure 1. Conventional Process Scheme for the Production of Hydrogen

elevated temperature is shown in Figure 2. This process is based on the principle that an equilibrium-limited reaction can be driven toward complete conversion of reactants to products if one of the products is removed from the reactor as it is formed. This operating characteristic of membrane reactors has led to numerous investigations of membrane reactors for a wide range of applications, in addition to facilitating the WGS reaction (Hsieh, 1991; Shu et al., 1991; Armor, 1989)

Essential to the successful development of this process is an affordable, durable, highly-selective membrane. A composite-metal membrane under development at Bend Research promises to meet these requirements. The composite-metal membrane (Figure 3) utilizes a coating-metal layer that is permselective for hydrogen, rejecting all other feed-stream components. The mechanical-support layer prevents the membrane from rupturing under high transmembrane pressures, and the intermediate-oxide layer prevents intermetallic diffusion between the coating-metal layer and the mechanical-support layer. Intermetallic diffusion between these layers leads to rapid flux decline and shortened membrane lifetime.



**Figure 2. Proposed Membrane-Reactor Process For The Production Of Hydrogen**



**Figure 3. Key Elements Of The Bend Research Composite-Metal Membrane**

The membrane composition shown in Figure 3 is sensitive to poisoning by sulfur compounds. Therefore, an absorbent bed prior to the membrane reactor will be necessary to remove sulfur compounds from the feed stream. However, since the membrane only permeates hydrogen, the gasifier can be air-blown without adversely affecting the hydrogen purity.

**Hydrogen Production in a Membrane Reactor**

We have previously demonstrated that the WGS reaction is driven toward completion in a small, laboratory-scale membrane reactor incorporating the Bend Research composite-metal membrane (Edlund, 1991). Greater than 90% conversion to hydrogen was achieved in these

initial experiments using a 2:1 steam:carbon monoxide ratio at 235 psia and 700°C (Figure 4).

As expected, the overall rate of conversion decreases with decreasing pressure (Figure 4) since both the chemical-reaction rate and the rate of hydrogen transport across the membrane are decreased at lower feed pressures.

The reaction time required to achieve maximum conversion shown in Figure 4 are relatively long, as no shift catalyst was used in these experiments (the rate of the non-catalyzed gas-phase WGS reaction is relatively slow even at 700°C). Incorporating a commercial shift catalyst (e.g., Haldor Topsoe SK-201) at the feed side of the membrane is expected to increase the overall rate of conversion significantly. Additional improvements in the process design might include multiple membrane stages and/or a condensable sweep stream (such as steam) over the permeate side of the membrane to achieve  $\geq 95\%$  hydrogen

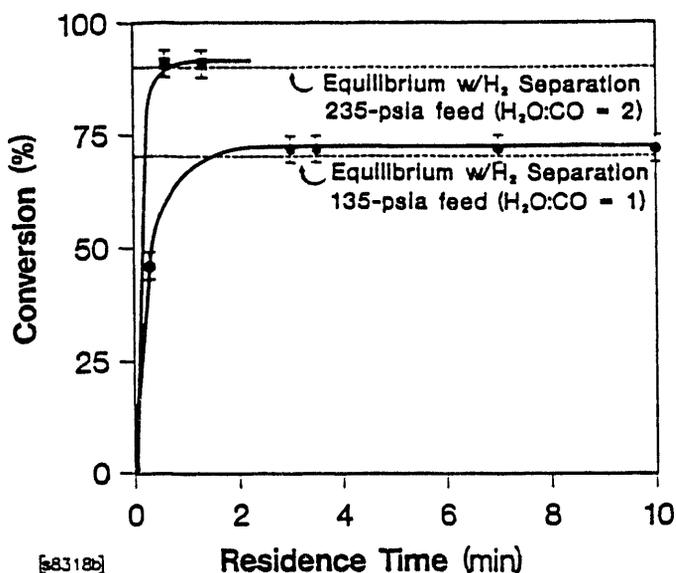


Figure 4. Conversion Versus Residence Time at 700°C in a Laboratory-Scale Membrane Reactor

recovery while maintaining moderate permeate pressure (several atmospheres).

## RESULTS

Work in this program has focused on the first three program objectives listed above. Specifically, the following primary results have been obtained:

- Prototype plate-and-frame membrane modules containing up to 0.4 ft<sup>2</sup> of membrane have been designed, fabricated, and operated for up to 3 weeks. This represents a scale-up factor of about 100-fold relative to the laboratory-scale membrane samples that had been the focus of our testing. A patent application on the module design is being filed. The projected selling price of membrane modules is expected to be about \$275/ft<sup>2</sup>.
- The membrane composition has been improved, resulting in a several-fold increase in hydrogen flux versus that obtained using earlier-generation membranes, while the overall cost of membrane materials has decreased seven-fold. Thus, the current-generation membrane delivers a hydrogen flux of 150 SCFH/ft<sup>2</sup> at 400°C and 100-psig hydrogen feed pressure, and 210 SCFH/ft<sup>2</sup> at 400°C and 100-psig hydrogen feed pressure.
- Small, laboratory-scale membranes have been operated for 6 months at 500°C without showing any flux decline, indicating that operational lifetimes of  $\geq 2$  years are likely to be achieved.

In addition, we have begun a collaboration with Teledyne Wah Chang (Albany, OR) to develop and market metal-membrane modules for a wide range of applications. It is anticipated that Teledyne Wah Chang will become the exclusive manufacturer of the membrane modules.

## FUTURE WORK

We expect that the relatively high flux and low cost of the composite-metal membrane modules will lead to favorable process economics for producing hydrogen from coal. During the next year we will work toward completing an economic analysis of the membrane-reactor process. Parametric studies of conducting the WGS reaction within the membrane reactor will be completed. In these studies the prototype plate-and-frame modules will be used and a commercial shift catalyst will be placed within the reactor at the feed side of the membrane to ensure rapid chemical kinetics.

The parametric studies will be designed to determine the overall rate of conversion of reactants to hydrogen and carbon dioxide over a range of operating conditions. Furthermore, we will determine whether the catalyzed reaction kinetics or the rate of hydrogen removal from the feed side of the reactor is rate-limiting (the rate-limiting factor may vary with changing operating conditions). This information will serve as the basis for the design and optimization of the process.

Concurrent with the parametric studies, we plan to conduct a field test on a research-gasifier slip-stream in collaboration with the University of North Dakota Energy & Environmental Research Center.

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## 9b.3 New Developments in Hydrogen Permselective Membranes

### CONTRACT INFORMATION

<b>Contract Number</b>	DE-AC21-90MC26365
<b>Contractor</b>	California Institute of Technology 1201 E. California Blvd. Pasadena, CA 91125 (818) 395-6357
<b>Contractor Project Manager</b>	George R. Gavalas
<b>Principal Investigator</b>	George R. Gavalas
<b>METC Project Manager</b>	Richard A. Johnson Venkat K. Venkataraman
<b>Period of Performance</b>	January 9, 1990 to November 8, 1992

### OBJECTIVES

The objectives of the original project was to develop silica hydrogen permselective membranes and evaluate the economic feasibility of these membranes in hydrogen production from coal gas. The objectives of the work reported here were to increase the membrane permeance by developing new precursors or deposition conditions, and to carry out fundamental permeability measurements of the membrane at different stages of pore narrowing.

test their stability under conditions simulating the expected operating conditions in coal gas processing, the membrane tubes were heated under 3 atm of water vapor (and 7 atm N<sub>2</sub>) at 550°C for up to 21 days. During this hydrothermal treatment the hydrogen permeance declined and stabilized to a value about 0.1 cm<sup>3</sup>/cm<sup>2</sup>-min-atm at 500°C. The stable membrane permeance represented 80% of the total resistance to hydrogen permeation. The H<sub>2</sub>:N<sub>2</sub> selectivity after the hydrothermal treatment was in the range 500-1000.

### BACKGROUND INFORMATION

In the work performed under the above referenced METC contract<sup>1-3</sup>, the contractor developed hydrogen permselective membranes by chemical vapor deposition (CVD) of thin SiO<sub>2</sub> layers within the pores of Vycor tubes having mean pore diameter about 40 Å. The hydrogen permeance after CVD was about 0.35 cm<sup>3</sup>/cm<sup>2</sup>-min-atm versus about 0.5 for the original tube, both at 500°C, so that the resistance due to the deposit layer was 30% of the total resistance. To

To evaluate the economic feasibility of the silica membranes, KTI Inc. under subcontract to Caltech conducted a case study of an ammonia-from-coal process comparing a conventional process with a membrane-assisted process<sup>2</sup>. In the conventional process the coal gas was treated by catalytic water gas shift reaction followed by hydrogen separation by pressure swing adsorption (PSA). In the membrane-assisted process, hydrogen was separated simultaneously with the catalytic shift reaction, resulting in reduced consumption of steam and elimination of PSA. Although accurate capital costs for the hydrogen membrane were not available, approximate

estimates suggested that the membrane-assisted process would become competitive if the membrane permeance was increased from the then available  $0.1 \text{ cm}^3/\text{cm}^2\text{-min-atm}$  value to  $0.3 \text{ cm}^3/\text{cm}^2\text{-min-atm}$ .

In view of the results of the economic evaluation, a new project was undertaken to increase the hydrogen permeance of the silica membranes. This new project was supported by the DOE University Coal Research Program and by funds from internal Caltech sources (Gates-Grubstake Fund).

The obvious way to increase membrane permeance was to decrease the thickness of the silica deposit layer which represented about 80% of the resistance to permeation. The layer thickness depends on the penetration depth of the silica precursor within the pores of the tube wall. One way to decrease the penetration depth is to use silica precursors of higher reactivity. In our previous studies we used the silica precursors  $\text{SiCl}_4$ ,  $\text{Cl}_3\text{SiOSiCl}_3$ ,  $\text{Cl}_3\text{SiOSiCl}_2\text{OSiCl}_3$ . A literature survey revealed that one of the most reactive agents for liquid phase silylation is trimethylsilyl triflate ( $(\text{CH}_3)_3\text{SiOSO}_2\text{CF}_3$ ). To grow a  $\text{SiO}_2$  layer one would need to use the chloride analog  $\text{Cl}_3\text{SiOSO}_2\text{CF}_3$ . To this end we synthesized this analog and measured the reaction rate with Vycor glass in a thermogravimetric analyzer (TGA). It turned out that the reaction was too slow compared with the reactions of  $\text{SiCl}_4$  and the other silylating compounds used previously. Evidently, reaction of the gaseous reagent with the pore surface is sterically hindered and also lacks the stabilization of the transition state afforded by the solvent in liquid phase reaction.

In view of the negative results the emphasis on different silica precursors was abandoned in favor of exploring different deposition conditions. The first modification was to use alternating rather than simultaneous reaction with  $\text{SiCl}_4$  and  $\text{H}_2\text{O}$ . The second modification was to introduce carbon masks as means of decreasing the reactant penetration depth. These two techniques, and particularly the second one, resulted in dramatic improvements of membrane permeance as will be described in the following sections.

## PROJECT DESCRIPTION

### Alternating Reactants Deposition

Our previous membrane preparations<sup>1-3</sup> were carried out by one-sided CVD of  $\text{SiO}_2$  on porous Vycor tubes using  $\text{SiCl}_4$  (or some other related compound) and  $\text{H}_2\text{O}$  as the reactants. This standard deposition technique suffers from two disadvantages. The first is the development of nonuniform deposit layer thickness caused by depletion of  $\text{SiCl}_4$  in the direction of flow. The second is formation of small clusters or particles in the gas phase by the direct reaction between  $\text{SiCl}_4$  and  $\text{H}_2\text{O}$ , and subsequent deposition of these particles on the external surface of the support, causing additional thickening of the deposit layer. To avoid those two drawbacks of one-sided CVD we introduced the alternating reactants CVD. This new technique of membrane deposition entails two elements. The first element is the alternating rather than simultaneous contact of the support with the two reactants. The alternating contact completely eliminates formation of particles by gas phase reaction. The second element is the introduction of  $\text{SiCl}_4$  into the evacuated reactor volume in discrete dosages rather than in continuous flow. Introduction of  $\text{SiCl}_4$  into the evacuated volume eliminates or greatly reduces deposit layer nonuniformities. At the same time, limiting the dosage of  $\text{SiCl}_4$  introduced in each cycle, reduces the penetration depth into the support.

The deposition reactor has been described in earlier publications. Briefly, it consists of an external quartz tube (11 mm ID) surrounding a concentrically placed porous Vycor tube (7 mm OD, 4.8 mm ID,  $40 \text{ \AA}$  mean pore diameter) welded on both sections with nonporous quartz sections for convenient connection with inlet and outlet flows. The reactor is placed inside a split-tube electrical furnace. The reactant streams  $\text{SiCl}_4\text{-N}_2$  and  $\text{H}_2\text{O-N}_2$  were generated in bubblers at controlled temperatures. The  $\text{SiCl}_4\text{-N}_2$  stream was stored in a large storage flask from which it was admitted intermittently into the reactor.

A membrane deposition experiment consisted of several consecutive silylation-hydrolysis cycles at reaction temperature  $700\text{-}800^\circ\text{C}$ . Each cycle entailed evacuating the reactor, admitting a dosage of  $\text{SiCl}_4\text{-N}_2$  (the dosage being controlled by the

mol fraction of  $\text{SiCl}_4$  in the storage flask) and allowing it to react for 1 minute, evacuating the reactor, and finally passing continuously a stream of  $\text{H}_2\text{O}-\text{N}_2$  for 5 minutes. After each cycle, the permeance of  $\text{N}_2$  was measured and when that permeance dropped below a preassigned level (lower by a factor 30-100 than the initial permeance), the deposition was terminated and the permeance of  $\text{H}_2$  and  $\text{N}_2$  were measured at several temperatures.

Selected membrane tubes were annealed at  $500^\circ\text{C}$  under 3 atm of  $\text{H}_2\text{O}$  (and 7 atm  $\text{N}_2$ ) for several days to test their stability under expected operating conditions. After the hydrothermal treatment, the permeance of  $\text{H}_2$  and  $\text{N}_2$  were measured once more at several temperatures.

### CVD Assisted by Carbon Barriers

A new technique developed in this project is the use of temporary carbon barriers to reduce the thickness of the deposit layer. The technique of carbon barriers involves first forming a thermosetting polymer inside the pores of the support, carbonizing the polymer, conducting  $\text{SiO}_2$  deposition by one-sided or alternating CVD, and finally removing the carbon barrier by oxidation.

The polymer selected for these experiments was polyfurfuryl alcohol (PFA) which upon carbonization is known to undergo about 40% weight loss. The polymer was formed by polymerization of the furfuryl alcohol monomer (FA) using para-toluene sulfonic acid as the polymerization catalyst. After polymerization and cross-linking at  $100^\circ\text{C}$  for 24 hours, the support tube was heated slowly to  $600^\circ\text{C}$  to prepare it for CVD. Silica CVD was carried out by alternating deposition as described in the previous subsection. Finally, the carbon barrier was removed by oxidation with pure oxygen at  $600^\circ\text{C}$  for 18 hours. The permeance of  $\text{H}_2$  and  $\text{N}_2$  were measured after carbon deposition, after CVD and after the final oxidation step.

## RESULTS

### Alternating Deposition

Figure 1 shows the evolution of hydrogen and nitrogen permeance (based on the external diameter of the support tube) of two membranes formed by alternating CVD, one at  $700^\circ\text{C}$  and the other at  $800^\circ\text{C}$ . In each case the permeances were measured at the deposition temperature. Membrane 2 which was prepared at  $800^\circ\text{C}$  required a smaller number of cycles, had higher  $\text{H}_2:\text{N}_2$  selectivity but somewhat lower  $\text{H}_2$  permeance. These results can be attributed to a thinner but denser deposit layer at  $800^\circ\text{C}$ .

The two membranes shown in Figure 2 were heated at  $500^\circ\text{C}$  under 3 atm of  $\text{H}_2\text{O}$  (and 7 atm of  $\text{N}_2$ ) for two weeks. Table 1 shows the change in the hydrogen and nitrogen permeances during this treatment. Table 2 shows the results of the same hydrothermal treatment in terms of the net permeance of the deposit layer, i.e. after subtracting the resistance of the bare support tube. Hydrothermal treatment decreases the permeances of the deposit layer by about 10% at  $700^\circ\text{C}$  and 50% at  $450^\circ\text{C}$ . The dependence of the reduction factor on temperature is due to the fact that hydrothermal treatment increases the activation energy. It is also seen that the membrane prepared at  $800^\circ\text{C}$  undergoes a slightly smaller change during hydrothermal treatment. These differences become more clear by looking at the activation energies for hydrogen permeation shown in Table 3. After deposition, membrane 2 has activation energy of 20.1 kJ/mol versus 17.3 of membrane 1. During hydrothermal treatment, however, the activation energy of both membranes increases and reaches a common level of 26 kJ/mol.

Comparison of the permeances shown in Tables 1-3 with the permeances of membranes prepared in our previous work by one-sided deposition reveals the following differences. The layers deposited by alternating reactants CVD have higher activation energies (17-20 kJ/mol vs. 10-12 kJ/mol) but approximately equal hydrogen permeances implying that the layers are thinner and denser. Upon hydrothermal treatment all layers are densified to the same final state with activation energy about 26 kJ/mol. As a result of this

densification, the permeance (at 600 K) declines by a factor 1.6 to 1.9 for the layers prepared by alternating deposition. The decline factor for the layers prepared by one-sided deposition is much higher, about 15. These large differences refer to the permeance of the deposit layer. The differences are much smaller for the permeance of the whole membrane tube because of the significant resistance of the support.

### CVD Assisted by Carbon Barriers

Table 4 shows the H<sub>2</sub>-permeance of a membrane prepared with the help of carbon barriers. The permeance for the whole tube and the net permeance of the deposit layer are listed. The activation energy for the permeance of the deposit layer is about 26 kJ/mol, essentially the same as that of layers prepared by one-sided deposition. Table 5 compares the permeance of layers prepared with and without the help of carbon barriers. Using the carbon barriers increases the deposit layer permeance by a factor of about 5.

### FUTURE WORK

The practical result of using alternating deposition and carbon barriers is to increase the hydrogen permeance of the deposit layer by a factor of about 20 over the permeances obtained in our previous work. At 600 K the resistance to permeation due to the deposit layer is only 12% of the overall resistance, with 88% of the resistance residing on the support tube. To fully exploit the increased permeance of the deposit layer it is essential to use support tubes of lower resistance. One possibility is to use Vycor tubes of the same pore size as in the reported experiments but having smaller diameter and wall thickness. Reducing the wall thickness from 1.1 mm to 0.4 mm (corresponding to tubes with 0.2 mm ID) would

increase the overall hydrogen permeance at 500 K from 0.68 to 1.26 cm<sup>3</sup>/cm<sup>2</sup>/min-atm, based on the inside diameter of the tube. Using as supports composite mesoporous/macroporous tubes like the ones marketed by US Filter, the overall permeance at 500 K can be increased to about 3.8 cm<sup>3</sup>/cm<sup>2</sup>-min-atm. These higher permeances are well above the economic viability threshold identified in the background section.

A number of issues need to be addressed in future work to demonstrate the commercial feasibility of the silica membranes. A critical need is the development of technology for fabrication of multitube modules. It is also important to demonstrate the preparation techniques using as supports smaller diameter Vycor tubes or composite mesoporous/macroporous tubes. Finally, the membranes should be tested for stability over longer periods of time.

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**Table 1. Permeance of Membranes Deposited on the Inner Surface Before and After Hydrothermal Treatment for 15 Days at 550°C under 3 atm H<sub>2</sub> and 7 atm N<sub>2</sub>. The Dosage of SiCl<sub>4</sub> per Cycle was 0.28 μmol/cm<sup>2</sup>.**

	Reaction T (°C)	Measurement T (°C)	Permeance (cm <sup>3</sup> (STP)/min atm cm <sup>2</sup> )			
			After Deposition		After Treatment	
			N <sub>2</sub>	H <sub>2</sub>	N <sub>2</sub>	H <sub>2</sub>
Membrane 1	700	700	0.0039	0.38	0.00042	0.33
		600	0.0036	0.37	0.00015	0.30
		450	0.0019	0.33	0.000094	0.21
Membrane 2	800	800	0.0013	0.37	0.00033	0.34
		600	0.00046	0.34	0.00018	0.28
		450	0.00020	0.28	0.00015	0.19

**Table 2. Permeance of Deposit Layers Excluding the Resistance of Vycor Tube. The Permeance is Given After Deposition and After 15 Days at 550°C under 3 atm H<sub>2</sub> and 7 atm N<sub>2</sub>.**

	Reaction T (°C)	Measurement T (°C)	Permeance (cm <sup>3</sup> (STP)/min atm cm <sup>2</sup> )			
			After Deposition		After Treatment	
			N <sub>2</sub>	H <sub>2</sub>	N <sub>2</sub>	H <sub>2</sub>
Membrane 1	700	700	0.0041	1.74	0.00042	1.04
		600	0.0037	1.33	0.00015	0.70
		450	0.0019	0.82	0.000094	0.34
Membrane 2	800	800	0.0013	1.65	0.00033	1.16
		600	0.00046	0.99	0.00018	0.63
		450	0.00020	0.56	0.00015	0.29

**Table 3. Activation Energy for H<sub>2</sub> Permeance of the Deposit Layers in Membranes 1 and 2 Before and After Hydrothermal Treatments for 15 Days at 500°C under 3 atm H<sub>2</sub>O and 7 atm N<sub>2</sub>**

	Activation Energy (kJ/mol)	
	After Deposition	After Treatment
Membrane 1	17.3	25.8
Membrane 2	20.1	25.8

**Table 4. Hydrogen Permeance of a Silica Membrane Prepared With Alternating CVD and Carbon Barrier With and Without the Resistance of the Support Tube**

Temperature, °C	H <sub>2</sub> Permeance, cm <sup>3</sup> (STP)/cm <sup>2</sup> -min-atm		
	Support Tube Plus Deposit Layer	Support Tube	Deposit Layer
450	0.667	0.867	2.90
523	0.679	0.824	3.85
600	0.692	0.787	5.75
700	0.687	0.745	8.56

**Table 5. Comparison of Deposit Layer Permeances of Membranes Prepared by Alternating CVD With and Without the Use of Carbon Barrier**

Measurement T °C	H <sub>2</sub> Permeance, cm <sup>3</sup> (STP)/cm <sup>2</sup> -min-atm	
	Membrane Prepared without Carbon Barrier	Membrane Prepared with Carbon Barrier
450	0.62	1.88
600	1.13	3.74
700	1.57	5.57

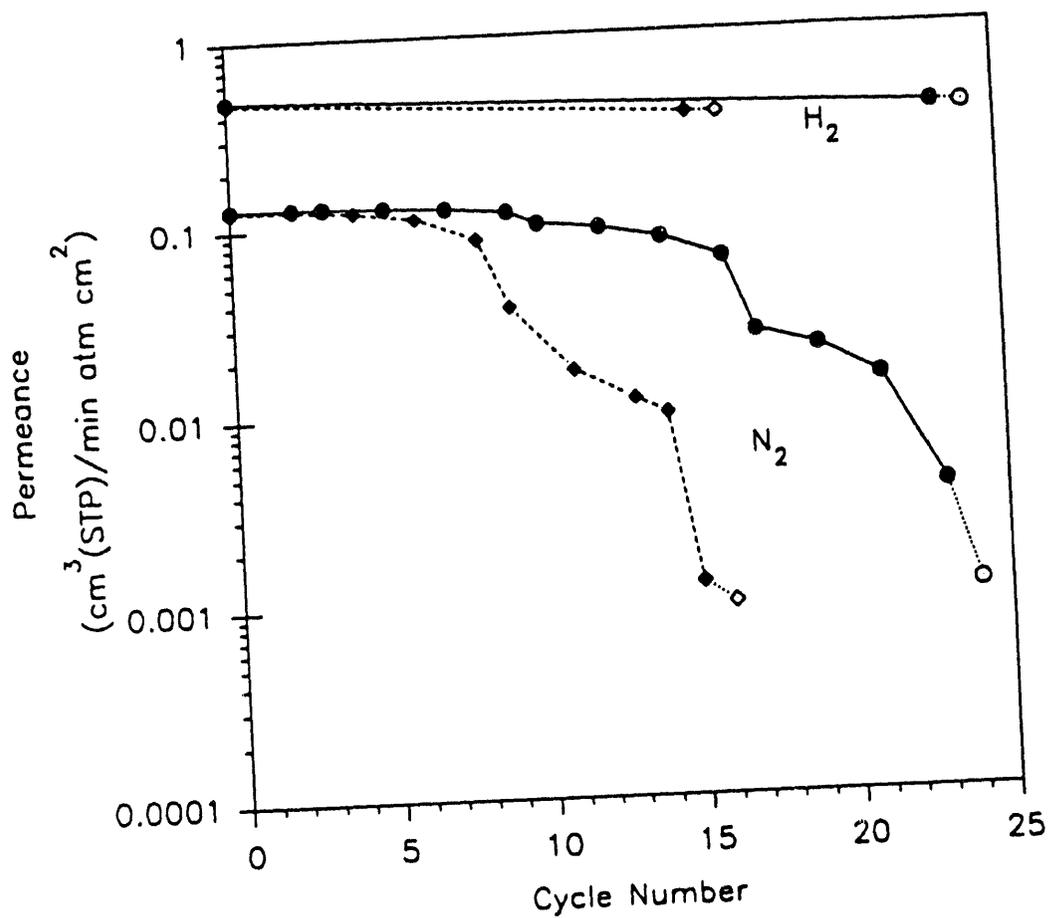


Figure 1. Permeances of H<sub>2</sub> and N<sub>2</sub> Versus Cycle Number for the Reaction at 700°C (Membrane 1. ●) and 800°C (Membrane 2. ◆). Using SiCl<sub>4</sub> Dosage of 0.28 μmol/cm<sup>2</sup> per cycle. (◊) and (○) Indicate the Permeance Changes After 5 Days of Hydrothermal Treatment at 500°C under 3 atm H<sub>2</sub>O and 7 atm of N<sub>2</sub>.



parameters like pH, temperatures, concentrations, type of reducing agent and sensitizing solution on the rate of deposition of the film.

## **ii. Characterization of Composite Membrane**

The work for this task will start with the fabrication of composite membranes. The membranes will be characterized by surface measurements and by evaluation of the physical properties of the composite (film composition by EDAX, film thickness by SEM). The measurements will also include hydrogen gas permeability, steady state diffusion and the effect of thermal cycling.

## **iii. Development of Theoretical Model For Hydrogen Gas Separation**

The work for this task will be to find or develop a suitable model to describe the permeation of hydrogen gas through a thin palladium film on a porous support. Models developed in the literature deal only with the permeation of gases through a metal membrane. These models may or may not be applicable to composite membranes. The applicability of the model will depend on the role of the porous support in permeation of gases. It is well known that the permeability of gases through porous ceramics increases at higher temperatures. However, the behavior of hydrogen permeation through a palladium film is much more complex process. It is believed that the hydrogen molecule dissociates to hydrogen atoms on one side of the membrane, diffuses through the film and reassociates on the other side. A theoretical model will be developed to facilitate prediction and interpretation of data obtained in the permeation experiments. Permeation through the porous substrate will be analyzed in terms of Knudsen and binary

gas diffusion as well as surface diffusion. In case of Pd/substrate composites, a model will be developed that accounts for the kinetics of hydrogen diffusion.

## **BACKGROUND INFORMATION**

The development of high temperature membranes to recover hydrogen is a topic of considerable scientific interest. Since coal gasification and several high-temperature industrial processes generate hydrogen as a byproduct, the recovery of hydrogen is of significant commercial importance. Recently, there has been increased interest in developing inorganic and composite membranes for in-situ separation of product hydrogen to achieve equilibrium shift in a catalytic reactor [1]. However, the productivity of these membrane reactors is severely limited by the poor permeability of currently available membranes. Commercially available non-porous membranes are either thick film or thick walled tubes. Since permeability is inversely proportional to film thickness, a thick film membrane acts as a poor perm-separator. Thus, the major challenge lies in developing a permselective thin film, without compromising the integrity of the film. The availability of such a membrane for high temperature applications could open new areas of research in membrane reactor technology and gas separation. The success of membranes in these applications will largely depend on the availability of membranes with acceptable permselectivity and thermal stability. The polymeric membranes currently available are not suitable for high temperature applications (>180 °C) because of their thermal instability. In addition, inorganic membranes (mostly ceramic), although thermally stable, are also unsatisfactory as they lack permselectivity because of their porous nature.

To develop a new class of permselective inorganic membranes, we have identified electroless plating as a potential route to deposit a thin metal film on microporous substrate. Electroless plating is a controlled autocatalytic deposition of continuous film on the surface of a substrate by the interaction of a metal salt and a chemical reducing agent. This method can give thin films of metals, alloys and composites on both conducting and nonconducting surfaces. In this research, we are using electroless plating to deposit thin palladium films on microporous ceramic and silver substrates.

## PROJECT DESCRIPTION

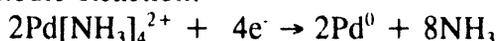
### Electroless Plating of Ceramic Substrate

Microporous ceramic alumina membranes (6 cm diameter, open porosity 10-15%, average pore size 5  $\mu\text{m}$  from Alfa/Johnson Mathey) were coated with a thin palladium film by electroless plating. Electroless plating is explained by a combination of the cathodic deposition of metal and the anodic oxidation of reductant at the immersion potential. Palladium deposition occurs as the result of the following simultaneous reactions:

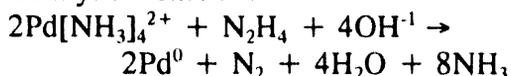
Anodic Reaction:



Cathodic Reaction:

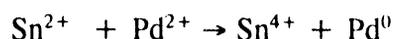


Autocatalytic Reaction:



Electroless plating is a three step process involving pretreatment of the substrate, sensitization and activation of the substrate surface, and electroless plating. Typical electroless plating bath compositions

given in Table 1. In electroless plating, pretreatment of the substrate is essential in order to deposit metals effectively. One of the common procedures is the two-step immersion sequence using an acidic stannous chloride solution followed by an acidic palladium salt solution. The first bath is referred to as the sensitizer (tin chloride solution), while the second bath is referred as the activator (palladium salt solution). The net result of the sequence is the formation of finely-divided palladium nuclei which initiate the autocatalytic plating process. The formation of the palladium metal nuclei is believed to be due to a redox reaction taking place between the adsorbed or absorbed stannous ions on the surface and the palladium ions in the activation solution. The sensitization and activation step can be described by the following reaction:



The sensitization process controls whether the final metallic film is uniform. The composition of the sensitization and activation solutions is given in Table 2.

### Membrane Characterization

The membranes will be characterized by measurements and evaluation of physical properties of the composite (film composition by EDAX, film thickness by SEM and a weight gain method).

The measurements will also include permeability and steady state diffusion, and the effect of thermal cycling. A steady-state counter diffusion method, using gas chromatographic analysis, will be used to evaluate the permeability and selectivity of the composite palladium membrane for hydrogen separation. Composite membranes will be characterized by conducting permeability experiments with hydrogen, argon, nitrogen and carbon dioxide. The

membrane characterization work is now in progress.

### Model for Hydrogen Permeation

The permeation of hydrogen through a palladium film is a complex process. The process begins with sorption of hydrogen molecules on the film surface and ends with hydrogen desorption from the ceramic substrate. It is believed that the hydrogen molecule dissociates into hydrogen atoms on one side of the film, diffuses through the film and reassociates on the other side [3]. Hydrogen permeation through palladium is thought to be taking place by the following mechanism:

*Sorption on the surface →  
Formation of hydrogen atoms →  
Diffusion of hydrogen atoms  
through the lattice → Formation of  
hydrogen molecules at the other  
surface → Desorption.*

The reaction kinetics of formation of hydrogen atoms from molecules and the reverse reaction are assumed to be very fast. The permeability can be considered as product of solubility and diffusivity. For isothermal, isobaric and plug flow conditions, the permeation rate of hydrogen can be given by [4]:

$$J_H = \frac{\bar{P}_H}{h} (p_1^n - p_2^n)$$

where  $J_H$  is the hydrogen flux,  $p_1$  and  $p_2$  are partial pressures of hydrogen on high and low pressure sides of the permeability cell.  $\bar{P}_H$  is the hydrogen permeability at a given temperature and  $h$  is the thickness of the palladium film.

If diffusion through the bulk metal is the rate limiting step and hydrogen atoms form

an ideal solution in the metal, then Sievert's law hydrogen solubility dependence holds and  $n$  is equal to 0.5. The hydrogen flux ( $J_H$ ) is inversely proportional to the palladium film thickness ( $h$ ) when the bulk diffusion is the rate limiting step. A value of  $n$  greater than 0.5 may result from several factors. For example, when the surface processes influence the permeation rate or when Sievert's law is not followed, a value of  $n$  greater than 0.5 may result. Leakage of hydrogen through defects in the metal film or through membrane seals may also increase the value of  $n$ .

With increasing temperature, the permeability coefficient increases and usually follows an Arrhenius type equation [5]:

$$\bar{P}_H = A_H \exp\left(-\frac{E}{RT}\right)$$

where  $A_H$  is the pre-exponential factor in the Arrhenius relationship for hydrogen permeability and  $E$  is the apparent activation energy of composite palladium-ceramic membrane. Here, it is assumed that the  $n$  in the hydrogen flux equation is essentially independent of temperature.

The above transport model will be used to facilitate the prediction and interpretation of data obtained in the permeability measurement experiments.

## RESULTS

This is a three-year research grant. Currently we are at the first year of the project. Although we have started only recently, several key results have been obtained, including the following:

- Developed a three step procedure for electroless deposition of palladium on

ceramic substrate. The three steps are: (i) preparation of the substrate; (ii) sensitization and activation of substrate; and (iii) electroless plating. Now, we are in a position to coat palladium thin-films on ceramic substrates by electroless deposition. Palladium (Pd) plating rate on a ceramic substrate at room temperature is shown in Figure 1.

- Designed and assembled a diffusion cell to measure diffusivity and permeability of hydrogen at high temperature and pressure.

## FUTURE WORK

During next two and half years, we will work on the following tasks:

- Design and build an experimental system to measure the permeability and diffusivity of hydrogen gas through palladium plated composite membrane. Also develop a method to measure the gas species composition by chromatographic method.
- Using SEM and EDAX, characterize composite ceramic-palladium membrane for film thickness and film composition.
- Evaluate the fabricated membrane for permeability and selectivity for hydrogen in presence of other gases, such as argon, nitrogen and carbon dioxide.
- Extend this work to investigate the silver-palladium composite membrane for separation of hydrogen.

## REFERENCES

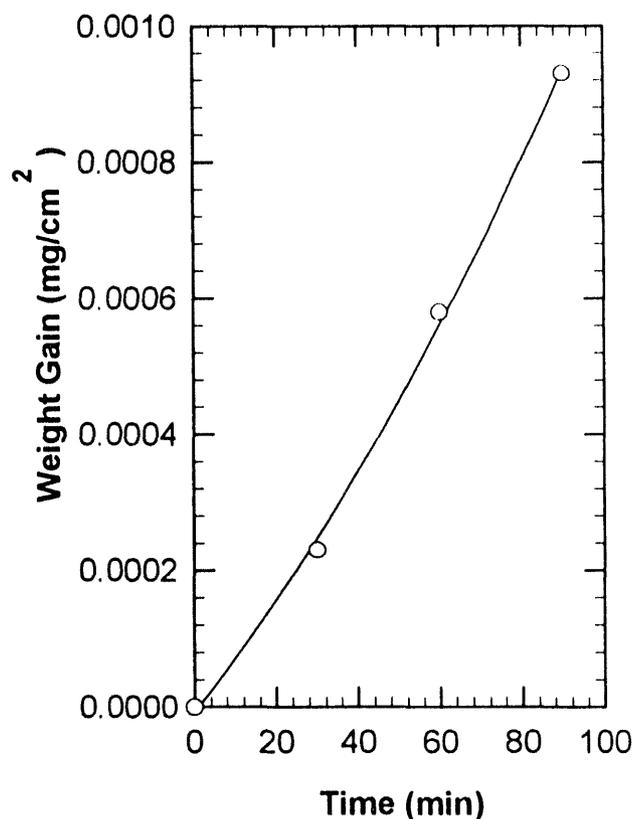
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4. Bohmholdt, G., and Wicke, E., "Diffusion of H<sub>2</sub> and D<sub>2</sub> in Pd and Pd-alloy," **Z. Physik. Chem., Neue Folge**, 56, 133 (1967).
5. Barrer, R.M., **Diffusion In and Through Solids**, Cambridge University Press, London, 1941.

**Table 1:** Typical Composition of Electroless Plating Bath.

Components/Variables	Concentration
PdCl <sub>2</sub>	5.4 g/l
Ammonium hydroxide (28 percent)	390 ml/l
EDTA	40 g/l
Hydrazine (1 molar solution)	10 ml/l
pH	11
Temperature	25 °C

**Table 2:** Composition of Sensitization and Activation Solutions.

Sensitization Solution	Concentration
SnCl <sub>2</sub>	1 g/l
HCl	0.2 N
Activation Solution	
PdCl <sub>2</sub>	0.9 g/l
HCl	0.2 N



**Figure 1:** Palladium plating rate on ceramic substrate by electroless deposition at room temperature. Ceramic substrate is 6.0 cm in diameter, 7 mm thick, 10-15 % open porosity and 5 μm pore size.

## 9b.5 Development of Hollow Fiber Catalytic Membrane Reactors for High Temperature Gas Cleanup

### CONTRACT INFORMATION

**Contract Number** DE-AC21-89MC26372

**Contractor** Chemical Engineering Department, Worcester Polytechnic Institute, 100 Institute Rd., Worcester, MA 01609

**Contractor Project Manager** Dr. Y. H. Ma

**Principal Investigators** Dr. Y. H. Ma  
Dr. W. R. Moser  
Dr. S. Pien  
Dr. A. B. Shelekhin

**METC Project Manager** Dr. V. Venkataraman

**Period of Performance** 4/93- 3/94

### FY94 Program Schedule

### OBJECTIVES

The *objective* of this project was to develop economically and technically viable catalytic membrane reactors for high temperature, high pressure gaseous contaminant control in IGCC systems. These catalytic membrane reactors were used to decompose H<sub>2</sub>S and separate the reaction products. The reactors were designed to operate in the hostile process environment of the IGCC systems, and at temperatures ranging from 500 to 1000 °C.

### BACKGROUND INFORMATION

The technology employed in the Integrated Gasification Combined Cycle (IGCC) permits burning coals with a wide range of sulfur concentrations. Emissions

from the process should be reduced by an order of magnitude below stringent federal air quality regulations for coal-fired plants. The maximum thermal efficiency of this type of process can be achieved by removing sulfur and particulates from the high temperature gas (e.g., 800 °C to 900 °C). High temperature sulfur removal can be accomplished with sorbents or by using membrane reactors. Catalytically active membrane reactors offer an inherent ability to combine reaction, product concentration and separation in a single unit operation. The conversion of H<sub>2</sub>S is limited by the thermodynamic equilibrium of the reaction but can, in principle, be improved by the removal of products (e.g., H<sub>2</sub>) during reaction. Here, selective removal of hydrogen could be provided by the membrane during reaction so that the reaction is continuously driven toward the product side. As the membrane is more

permeable to  $H_2$ , the reaction can be almost shifted to complete conversion.

## PROJECT DESCRIPTION

The membrane reactor is an integrated process which includes membrane and reactor processes in a single unit operation. Therefore, to ensure a proper design of the membrane reactor several components of the process should be analyzed including:

- \* membrane gas permselectivity and thermal stability;
- \* hydrodynamics of gas flow at elevated temperatures;
- \* thermodynamics of  $H_2S$  decomposition;
- \* reaction rates of homogeneous and heterogeneous reactions of  $H_2S$  decomposition;
- \* membrane reactor design;
- \*  $H_2S$  decomposition in the membrane reactor at different operational parameters.

Based on experimental information on the  $H_2S$  conversion in the membrane reactor, a mathematical model was developed providing an adequate (within 5%) description of the process. The model provided information on the best operation parameters of the membrane reactor process.

## RESULTS

Severe conditions encountered in the IGCC process (e.g., 900 °C,  $H_2S$ ,  $CO_2$  and  $H_2O$ ) make it impossible to use polymeric membranes in the process. Therefore, inorganic membranes were used in the design of the membrane reactor - molecular-sieve glass membrane and Vycor glass porous membrane.

### Membrane permselectivity

The molecular-sieve glass membrane

acquired from PPG Industries Inc. was found far superior to polymeric counterparts in terms of the selectivity, permeability and thermal stability. Temperature dependencies of gas permeability in the molecular-sieve membrane is presented in Figure 1. Separation of gases in the microporous membrane occurs according to configurational diffusion mechanism developed in [1-4]. Even though the selectivity coefficients of the molecular-sieve glass membrane are very attractive for separation of  $H_2$  at room temperature ( $\alpha[H_2/H_2S] \approx 200$ ) the selectivity decreases with the temperature and at 800 °C is approximately 10. This selectivity is approximately two times higher than the selectivity coefficient for Knudsen diffusion mechanism. In addition, the porous structure of the membrane collapses at temperatures higher than 300 °C and gas permeability irreversibly decreases (Figure 2).

On the other hand, the permeability of the Vycor glass membrane remains unchanged up to a temperature of 850 °C. The selectivity factor in the Vycor glass membrane is independent of the temperature and is equal to  $\alpha[H_2/H_2S]=4.1$ . Even though the selectivity coefficient is lower than for the microporous glass membrane, the Vycor glass membrane was used for the design of the high temperature membrane reactor because of its thermal stability.

### Experimental installation

A schematic diagram of the experimental installation used in this study is presented in Figure 3. Quartz tubes with  $OD/ID=16/13$  mm were used as the reactor shells. One end of the membrane was fixed to the quartz tube with epoxy resin Duralco 4525 (Cotronics) stable up to a temperature of 250 °C. The other end of the membrane was sealed with the same epoxy. The feed

gas mixture was supplied on the shell side of the membrane reactor. The furnace was 15 cm long. The porous glass membrane and the quartz tube were 30 cm long. The length of the permeable part of the membrane in the membrane reactor was equal to 22 cm. The temperature in the furnace was controlled by a thermocontroller. The gas composition on the shell and the tube sides of the membrane reactor was analyzed with a Hewlett Packard HP5890 GC. The pressure in the sampling loop was controlled with a specially designed vacuum gas sampling system to provide reliability and accuracy in a wide range of gas concentrations. The reactor ends were cooled by air flow to prevent overheating and decomposition of the epoxy resin. Flow rates on the feed and shell sides were measured with a bubble flowmeter. The pressure on the shell side of the membrane reactor was kept at either 2.36 atm or 7.8 atm while the pressure on the permeate side of the membrane was kept atmospheric. Before the experimental runs the system was kept at 800 °C in a feed gas flow for 24 hours.

### Hydrogen concentration

The stage cut for the membrane reactor is similar to the stage cut for the conventional membrane reactor and is defined as the ratio of the permeate flow to the sum of the permeate and reject flows according to

$$\text{Stage Cut} = \frac{\text{Permeate}}{\text{Permeate} + \text{Reject}}$$

During the membrane reactor operation hydrogen produced during the reaction of H<sub>2</sub>S decomposition was selectively removed through the Vycor glass membrane and concentrated on the permeate side (Figure 4). When the stage cut

increased the concentration of H<sub>2</sub> on both the permeate and reaction sides decreased. At the stage cut equal to one the concentration of H<sub>2</sub> on the permeate side is equal to the equilibrium concentration of H<sub>2</sub> for the reaction of H<sub>2</sub>S decomposition at 1 atm. Similarly, at stage cut equal to 0 the concentration of H<sub>2</sub> on the reaction side is equal to the equilibrium concentration of H<sub>2</sub> for the H<sub>2</sub>S decomposition reaction at 7.8 atm. Similar dependencies of the H<sub>2</sub> on the stage cut were observed when 100%, 1%, and 500 ppm H<sub>2</sub>S gas mixtures were used as a feed.

### H<sub>2</sub>S conversion

From the definition of the stage cut it follows that at the stage cut equal to 0 the effect of H<sub>2</sub> removal from the reaction gas mixture is negligible and cannot affect the total conversion in the membrane reactor. As the stage cut increased the effect of H<sub>2</sub> removal increased as well. At the stage cut equal to one, the total conversion in the membrane reactor is equal to the total conversion on the low pressure or permeate side of the membrane reactor (Figure 5).

Noteworthy, that even though the stripping of H<sub>2</sub> from the reaction side occurred in the membrane reactor the total conversion did not increase compared to the equilibrium conversion in the packed bed reactor. Therefore, the membrane with the Knudsen selectivity of gas separation appears to be not suitable for enhancement of the conversion in the membrane reactor.

### Mathematical model

A mathematical model was developed to simulate the performance of the membrane reactor. Comparisons between theoretical predictions and experimental data are shown in Figure 4. A good agreement

between the experimental data and simulations was also observed for feed mixtures with a different content of H<sub>2</sub>S.

The developed model was used to predict the optimum conditions at which the conversion of H<sub>2</sub>S can be conducted to the highest degree of the conversion. When the membrane with selectivity coefficients higher than those of the Knudsen selectivity is used in the membrane reactor the conversion may be considerably higher than the conversion in the conventional packed bed reactor (Figure 6). The best results were obtained when the selectivity coefficients are equal to 500-1000 with respect to H<sub>2</sub> separation. Such high selectivity coefficients are difficult to get at temperatures equal to 800 °C even in the molecular-sieve membranes.

Additionally the effect of downstream pressure on the total conversion in the membrane reactor was analyzed. It was demonstrated that only when the absolute pressure on the permeate side is lowered down to 0.01 atm the high conversions (up to 90%) of H<sub>2</sub>S can be achieved.

## FUTURE WORK

A comprehensive analysis of the membrane reactor demonstrated that under certain conditions the membrane reactor may provide high conversions at low stage cuts. These conditions include high membrane selectivity (100 times the Knudsen selectivity) and low pressure on the permeate side (0.01 atm). It is not recommended to use sweep gas in any part of the membrane reactor because hydrogen in the permeate will be diluted and can not be utilized. The conversion in the membrane reactor can increase only if highly selective membrane is used such as Pd or Pt and low pressures on the permeate side are kept. The

conversion can be further increased if simultaneously a higher pressure on the reaction side is applied to lower a pressure ratio (pressure ratio = downstream pressure/upstream pressure) and to improve the membrane separation performance.

The membrane reactor processes appears to be more appropriate for processes with high contents of the reactant material. It can be used in gas phase reactions whose components are difficult to separate. The membrane reactor unit operation in this case provides simultaneous reaction and separation in a single technological unit.

## CONCLUSIONS

- \* Feasibility of the membrane reactor process for decomposition of hydrogen sulfide was demonstrated.
- \* Permeability and selectivity of molecular-sieve and Vycor glass membranes were studied at temperatures up to 1000 °C.
- \* Experimental study of hydrogen sulfide in the membrane reactor was completed.
- \* A generalized mathematical model was developed for the simulation of the high temperature membrane reactor.
- \* The efficiency of the membrane reactor process is probably low for the high temperature clean-up of H<sub>2</sub>S from the IGCC gas mixture.
- \* Knudsen selectivity is insufficient to yield an increase of the H<sub>2</sub>S conversion in membrane reactor.

## REFERENCES

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2. A. B. Shelekhin, A. G. Dixon, Y. H. Ma, Adsorption, permeation and diffusion of gases in microporous membranes. II. Permeation of gases in microporous glass membranes, *J.Mem.Sci.*, 75 (1992) 233-244

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4. A. B. Shelekhin, A. G. Dixon, Y. H. Ma, Theory of gas diffusion and permeation in molecular-sieve membranes, *AIChE J.*, In print 1994

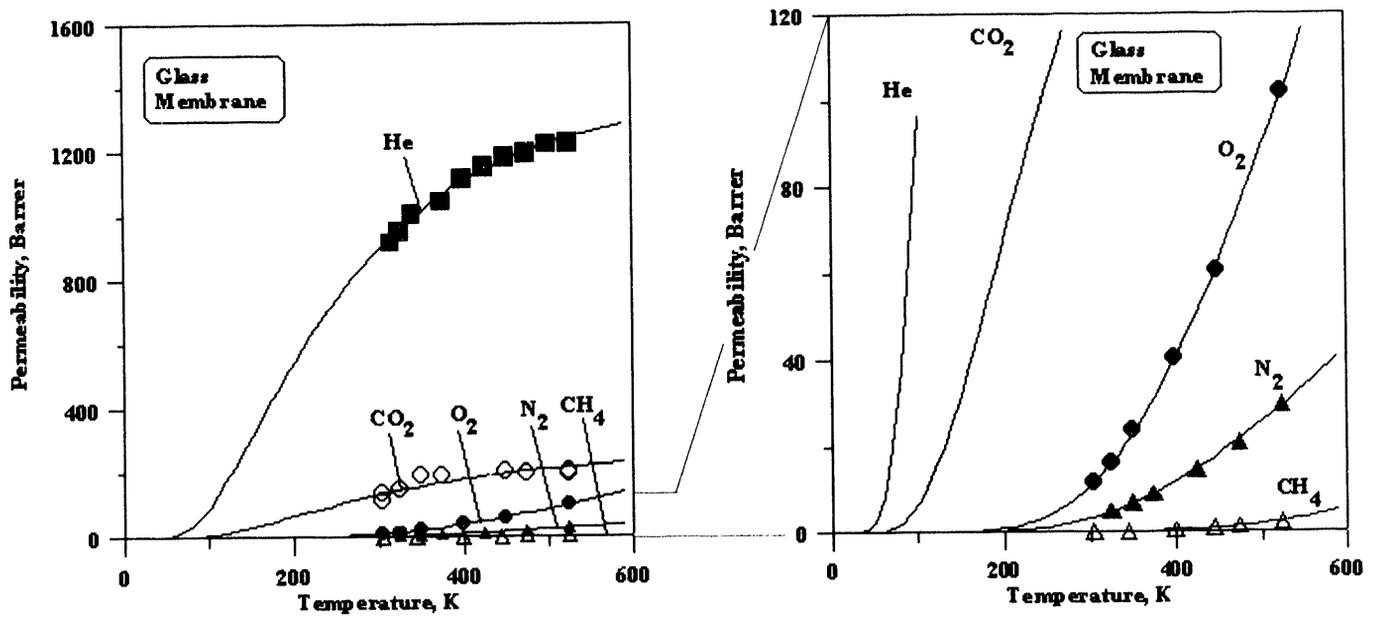


Figure 1 Permeability of molecular-sieve glass membrane.

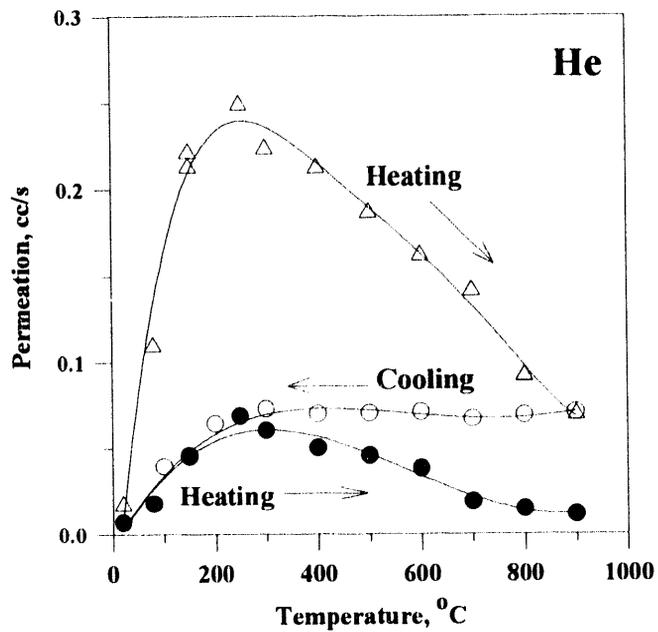
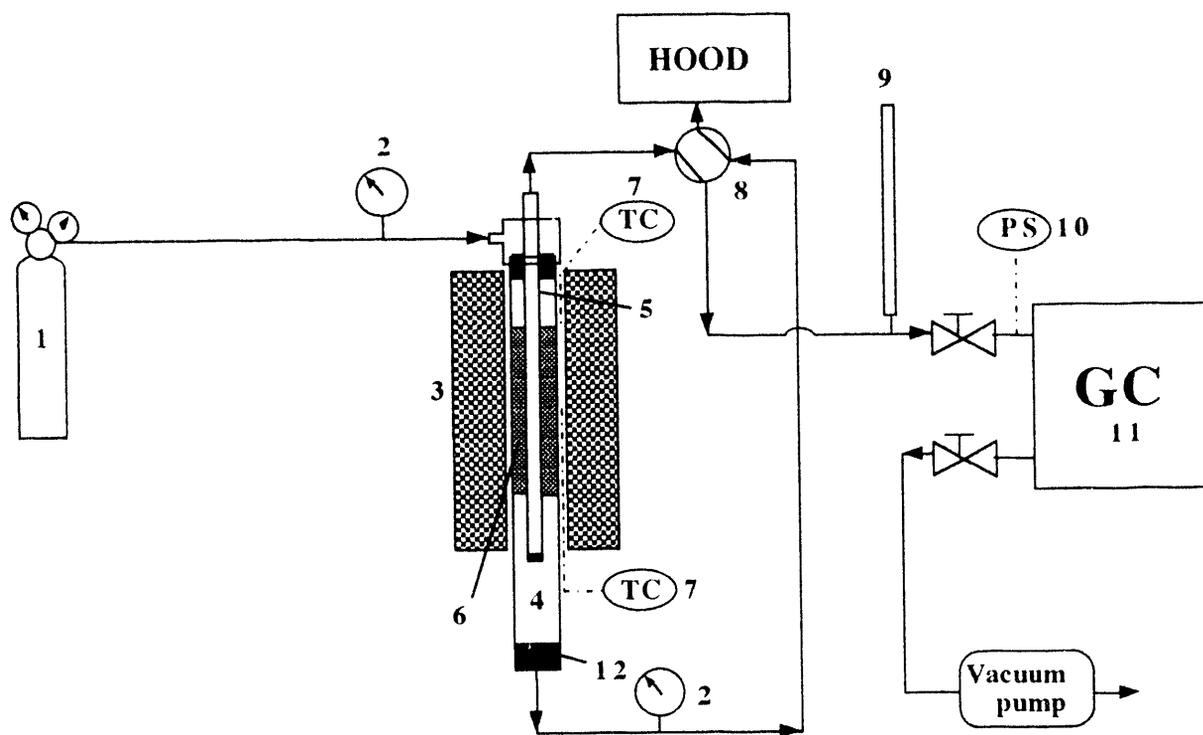


Figure 2 Temperature stability of molecular-sieve glass membrane.



1- Feed, 2- Pressure gage, 3- Furnace, 4- Membrane/packed-bed reactor, 5- Porous Vycor glass membrane, 6- Catalyst ( $\text{MoS}_2$ ), 7- Thermocouple, 8- 4-way-valve, 9- Bubble flow meter, 10- Pressure sensor, 11- Gas chromatograph, 12- Epoxy resin.

Figure 3. The schematic diagram of the experimental installation.

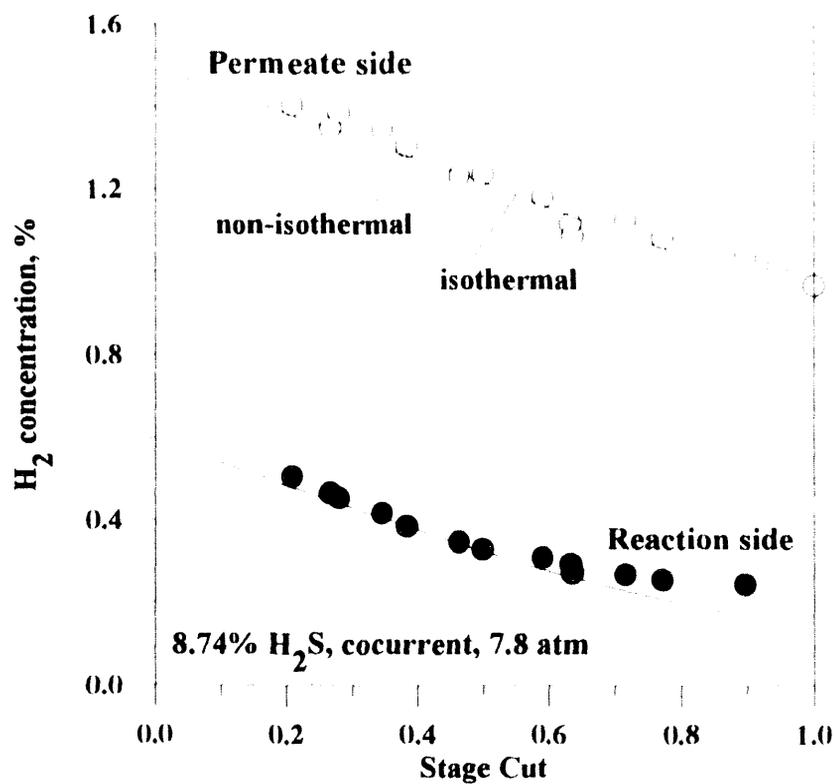


Figure 4 Concentration of hydrogen at the permeate and rejection side outlets in the membrane reactor.

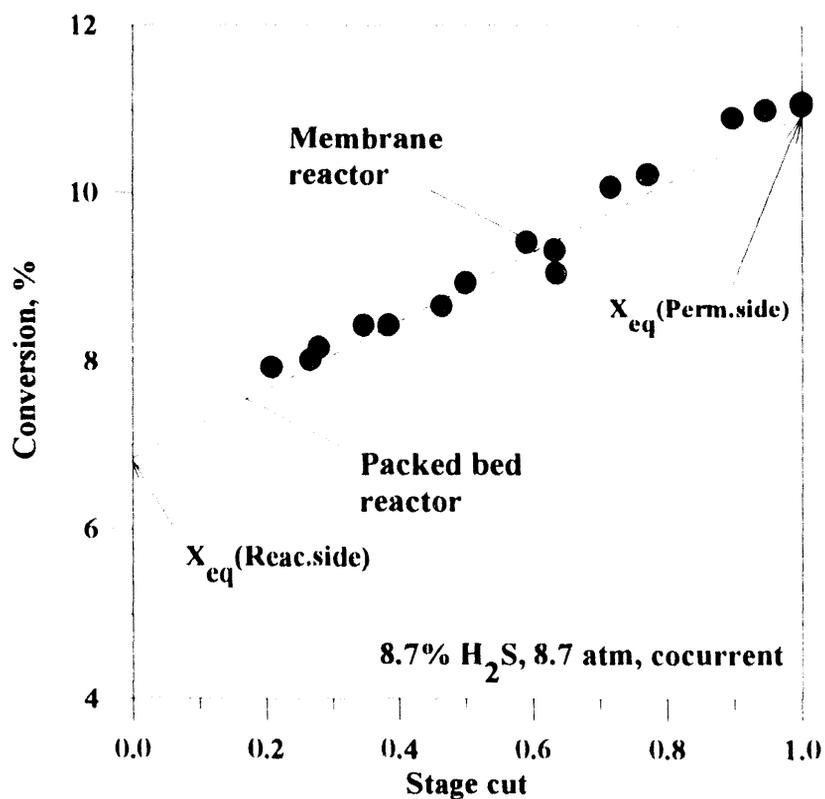


Figure 5 Total conversion of H<sub>2</sub>S in the membrane reactor.

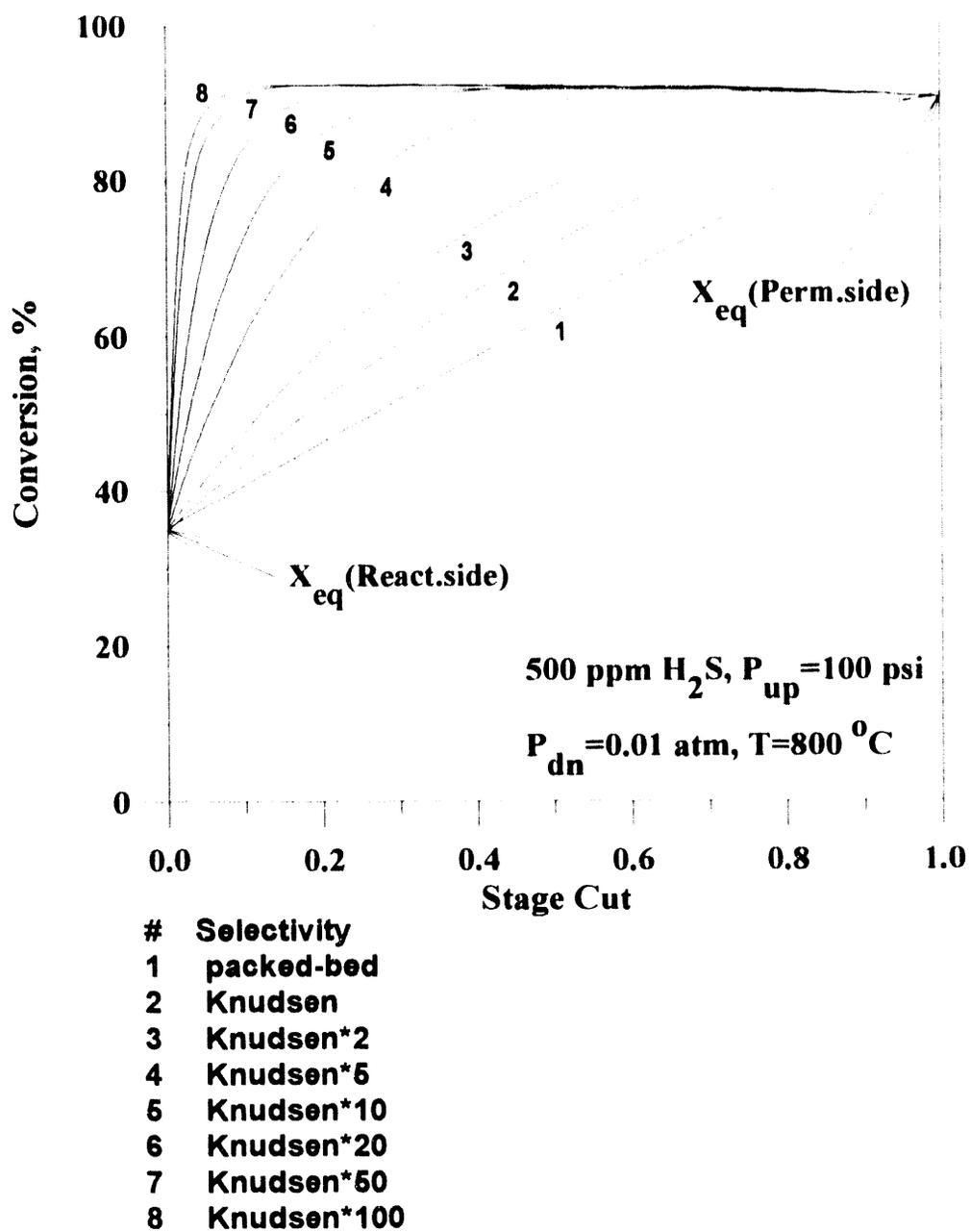


Figure 6. Effect of the membrane selectivity on the total conversion of H<sub>2</sub>S in the membrane reactor.

**9b.6**

**Thermal/Chemical Degradation of Inorganic Membrane Materials**

**CONTRACT INFORMATION**

**Contract Number** DE-AC21-92MC28053

**Contractor** SRI International  
333 Ravenswood Avenue  
Menlo Park, CA 94025  
(415) 326-6200

**Contractor Proj. Mgr.** Gopala N. Krishnan

**Principal Investigators** Gopala N. Krishnan  
Angel Sanjurjo  
Ashok S. Damle  
Bernard J. Wood  
Kai-Hung Lau

**METC Project Manager** Venkat K. Venkataraman

**Period of Performance** August 24, 1992 to December 19, 1994

**Schedule and Milestones**

**FY94 Program Schedule**

Task	S	O	N	D	J	F	M	A	M	J	J	A	S
Topical Report & Test Plan Development													
Experimental Testing													

**OBJECTIVES**

The overall objective of this program is to evaluate the long-term thermal and chemical degradation of inorganic membranes that are being developed to separate gaseous products produced by the gasification or combustion of coal in fixed-, fluidized-, and entrained-bed gasifiers, direct coal-fired turbines, and pressurized-fluidized-bed combustors. Specific objectives of this program are to (1) quantify the extent of the degradation process for the three most detrimental mechanisms by performing laboratory-scale experiments, and (2) develop a predictive model for membrane degradation under operating conditions.

**BACKGROUND INFORMATION**

Several impurities, such as H<sub>2</sub>S, NH<sub>3</sub>, SO<sub>2</sub>, NO<sub>x</sub>, and trace metal compounds are generated during the coal conversion process and they must be removed from the coal gas or the combustor flue gas to meet environmental standards. In an integrated-gasification combined-cycle plant, removal of such impurities from the gasifier product at high temperature increases the efficiency of electric power generation. Inorganic membranes are potentially attractive for such an application. They are also being considered as a means to separate from coal gas a high-value bulk component such as hydrogen. Hence, it is important to identify membrane materials that

possess the required characteristics of permeability, selectivity, and durability for use at high temperatures in a severe gaseous environment.

## PROJECT DESCRIPTION

To achieve the overall objective, the program is divided into the following tasks:

1. Development of evaluation methodology
2. Evaluation of potential long-term degradation mechanisms
3. Submission of a topical report and a plan for experimental testing
4. Experimental testing
5. Model development.

In Task 2 which has been completed, Krishnan, et. al. (1993) evaluated potential inorganic membrane degradation mechanisms based upon an extensive literature search and theoretical calculations. Thermal sintering, hydrothermal attack, reaction with ash components, attack by vapor and solid phase alkali compounds, and deposition of carbon from the gas phase were identified as the likely degradation mechanisms for ceramic and metallic membranes.

The topical report describing this work was submitted in March 1994. Experimental testing of candidate materials (Task 4) is currently in progress. Task 5 is a separate option that may be exercised by the U.S. Department of Energy at the conclusion of Task 4.

## CANDIDATE MEMBRANE MATERIALS

Micro-porous inorganic membranes, in which the separation process is based upon Knudsen diffusion, are available for commercial gas separation needs, but their selectivity is limited. A variety of developmental efforts are currently under way, many sponsored by U.S. Department of Energy (DOE), to improve the selectivity and flux characteristics of porous inorganic membranes (Liu, et. al., 1992; Goldsmith, et al., 1992; Gavalas, 1992; Yates, et. al., 1993). DOE is also sponsoring efforts to increase the applicability of metallic membranes (Edlund, 1993).

At present, no inorganic membranes are commercially available for application in the high-temperature, high-pressure (HTHP) gas environments encountered in integrated gasification combined cycle (IGCC), pressurized fluidized bed combustion (PFBC), and direct coal fired turbine (DCFT) applications. Most of the inorganic membrane developmental efforts have focused on hydrogen separation membranes which may be used in an IGCC system for maximizing hydrogen production from coal gas or to remove H<sub>2</sub>S and NH<sub>3</sub> contaminants via thermal or catalytic decomposition of these contaminants.

The candidate inorganic membranes may be grouped as follows:

1) Dense Metallic Membranes. Both palladium and platinum membranes have been investigated for their hydrogen separation ability. Developmental work is presently underway to increase their tolerance to high temperature as well as sulfur species by combining platinum and palladium metal layers with base metal and barrier oxide layers.

2) Silica Based Membranes. Micro-porous silica/glass membranes have been commercially available with pore sizes down to 40 Å (e.g., Vycor glass). Efforts are underway to reduce the pore size of such membranes by CVD techniques.

3) Alumina Based Membranes. Micro-porous gamma alumina/alpha alumina composite membranes are commercially available with pore sizes down to 40 Å (e.g., membralox membranes). Efforts are underway to reduce the pore size of the alumina membranes by depositing additional nanopore layers.

4) Carbon Based Membranes - Carbon molecular sieves (CMS) have been extensively used in the chemical industry for gas separation applications. Efforts are presently underway to develop carbon/carbon composite CMS membranes, where a top layer of CMS is deposited on a porous carbon support.

## EXPERIMENTAL TESTING

Three approaches are being used to evaluate the degradation of the membrane materials: (1) exposure of membrane materials to simulated coal gas streams under well-defined conditions, followed by characterization of the changes that

occur in their physical and chemical properties, (2) long-term testing of the permeation performance of membranes under representative conditions, and (3) exposure of the membrane materials to the hot coal gas stream of an operating coal gasifier, followed by examination and evaluation of durability.

## RESULTS

### Exposure Studies

The purpose of the exposure tests is to determine the changes in physical characteristics, chemical composition, and pore size distribution of the membrane materials as a result of exposure to simulated IGCC coal gas environments.

Figure 1 shows a schematic of the exposure test apparatus. For atmospheric pressure tests at temperatures up to 1000°C, a quartz exposure tube enclosed in a furnace is used. A movable quartz rack/platform, within the tube allows convenient placement of membrane samples. The desired gaseous environment is generated by mixing appropriate dry gases and adding steam and alkali vapors supplied from separate generation systems. The exhaust gas from the exposure tube is vented through a condenser into a fume hood, and a back pressure regulator which controls the exposure environment pressure to a desired level.

A similar apparatus has been constructed for experiments at elevated pressure. The exposure tube is made of an alloy steel and is equipped with flanged ends. Constant pressure is maintained in the tube by means of a back pressure regulator in the effluent stream.

To date, commercially available Vycor glass and  $\gamma$ -alumina membranes have been tested, in addition to platinum and palladium metal foils. The samples were exposed for various periods (24, 76, and 113 hours) to two coal gas compositions, at three temperatures (1000, 800, and 650°C), in separate, atmospheric-pressure experiments. The two gas compositions represent extremes in gasifier type. The gas simulating a fixed-bed, air-blown gasifier product contained 18% H<sub>2</sub>, 9% CO, 12% CO<sub>2</sub>, 1% H<sub>2</sub>S, 30% H<sub>2</sub>O and 30% N<sub>2</sub>, whereas that from an entrained-bed, oxygen-blown gasifier contains 30% H<sub>2</sub>, 10% CO<sub>2</sub>, 0.5% H<sub>2</sub>S, 15% H<sub>2</sub>O and 44.5% CO.

The exposed membrane samples were analyzed by various techniques to determine changes in the physical and chemical characteristics of the membrane materials. The results are described below for each of the membrane materials.

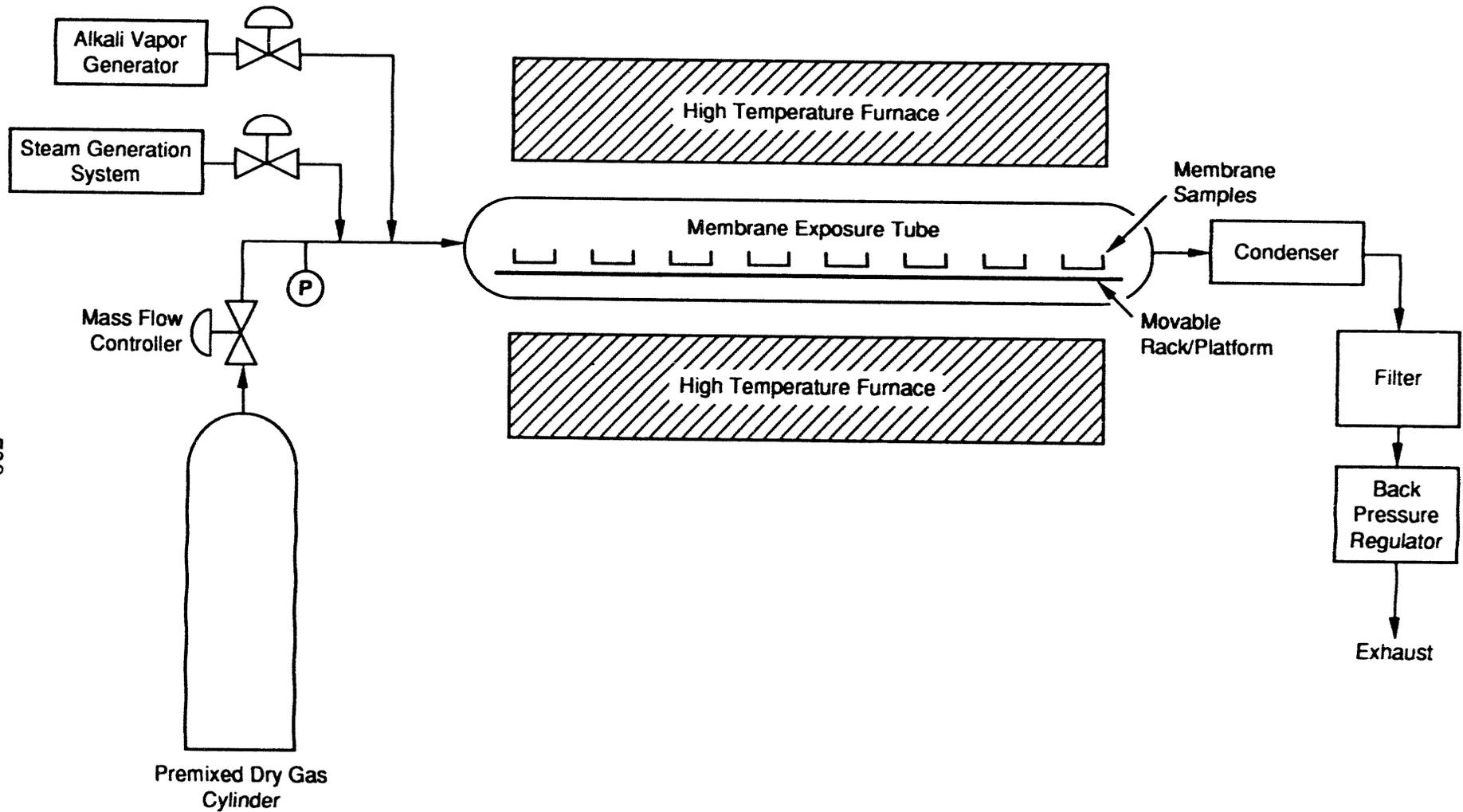
Surface areas and average pore diameters of Vycor membrane samples were determined by the B.E.T. gas adsorption method. The results are summarized in Table 1. The surface area of Vycor decreased from a high of 188 m<sup>2</sup>/g for an unexposed sample to a low of 1.9 m<sup>2</sup>/g for a sample exposed for 24 h at 1000°C. Mean pore size increased from 4.1 nm to 11.7 nm after 76 h exposure at 800°C, but after 24 h exposure at 1000°C, all microporosity was lost.

X-ray diffraction was used to determine changes in the crystallinity of the exposed membrane samples. The unexposed Vycor membrane sample, which was transparent, was found to be completely amorphous. After exposure at 800°C, the sample turned opaque and developed a crystalline cristobalite phase. The sample exposed to 1000°C appeared fused and completely crystallized. Thus, temperatures greater than 650°C in the coal gas environment are found to be detrimental to the silica based membranes. These results are significant also for modified membranes based on the addition of silica layers to commercial Vycor and alumina membranes.

**Table 1. Changes in Vycor Membrane Properties with Temperature**

Membrane Sample	BET Surface Area (m <sup>2</sup> /gm)	Mean Pore Diameter (nm)
Unexposed	188	4.1
Exposed at 650°C for 113 hrs	140	4.5
Exposed at 800°C for 76 hrs	33	11.7
Exposed at 1000°C for 24 hrs	1.9	---

-739-



CM-3856-24

Figure 1. Schematic Diagram of the Reactor for Membrane Exposure Tests

The gamma-alumina membrane samples were also analyzed by X-ray diffraction (XRD) as well as by B.E.T. XRD identified  $\alpha$ -alumina as the dominant bulk phase in the samples, but it failed to detect the  $\gamma$ -alumina phase present in the surface layers that is critical to the permselective character of the membrane. Refinement of this technique is necessary to determine the phase change in the thin  $\gamma$ -alumina layer due to high-temperature coal gas exposure. If phase changes in the surface layers occur, they would likely cause pore coarsening.

Nitrogen B.E.T. analysis of the alumina membranes indicated that a significant decrease in surface area occurred during exposure at all the temperatures studied (Table 2). The surface area of an unexposed sample was about 2.1 m<sup>2</sup>/g. Exposure for 114 h at 650°C decreased the surface area to 0.9 m<sup>2</sup>/g, and 76 h at 800°C reduced the surface area to 0.4 m<sup>2</sup>/g. Exposure for 24 h at 1000°C decreased the surface area to 1.3 m<sup>2</sup>/g. Because of these low values of surface area, these samples are being reanalyzed with an alternative adsorbent gas (krypton) to obtain greater accuracy. Both the exposure duration and temperature appear to affect the surface area reduction by mechanisms such as sintering and phase transformation.

**Table 2. Surface Area Changes of an Alumina Membrane as a function of Temperature.**

Test Conditions	BET Surface Area (m <sup>2</sup> /g)
Unexposed	2.1
Exposed at 650°C for 113 hrs	0.9
Exposed at 800°C for 76 hrs	0.4
Exposed at 1000°C for 24 hrs	1.3

During exposure of the  $\gamma$ -alumina membrane samples at 800 and 1000 °C, vaporization of trace contaminants in the material occurred. Such vapor species reacted with silica material present in the exposure reactor causing them to crystallize. This vaporization behavior was studied by high temperature mass spectrometry, and the vapors

were found to be CO<sub>2</sub>, H<sub>2</sub>O, Na (g), and Zn (g). Sodium vapor is detrimental to the operation of gas turbine components. Partial pressures of the N and Zn vapor species over a membrane sample are given in Table 3. In this experiment, the sample was heated slowly from room temperature. CO<sub>2</sub> evolution was observed at all temperatures whereas that of steam disappeared by 480°C. Significant evolution of elemental Na and Zn began at 480° and 1025°C respectively. The partial pressures of both species increased with temperatures. Sodium partial pressures were especially significant for coal gas exposure at 800 and 1000°C.

**Table 3. Partial Pressure of Gaseous Species Evolved from an Alumina Membrane**

Temperature (C)	Partial Pressure (atm)	
	Na Species	Zn Species
480	3.05E-10	< 1 E-10
545	1.15E-09	< 1 E-10
675	7.18E-09	< 1 E-10
780	3.25E-07	< 1 E-10
850	1.44E-06	4.71E-10
920	2.09E-06	5.52E-09
1025	1.20E-06	7.26E-09

Palladium foils exposed at 800° and 1000°C melted, presumably due to formation of PdS. Analysis of one of a resolidified sample by SEM/EDAX indicated bulk sulfidation of palladium. At 650°C the exposed Pd sample deformed extensively. This sample was also analyzed by Auger electron spectroscopy (AES) to determine the elemental sulfur content on its surface. Sulfur was not only a predominant component of the surface, but, upon depth profiling by argon bombardment, it was found at a high level within the bulk of the metal foil (Figure 2).

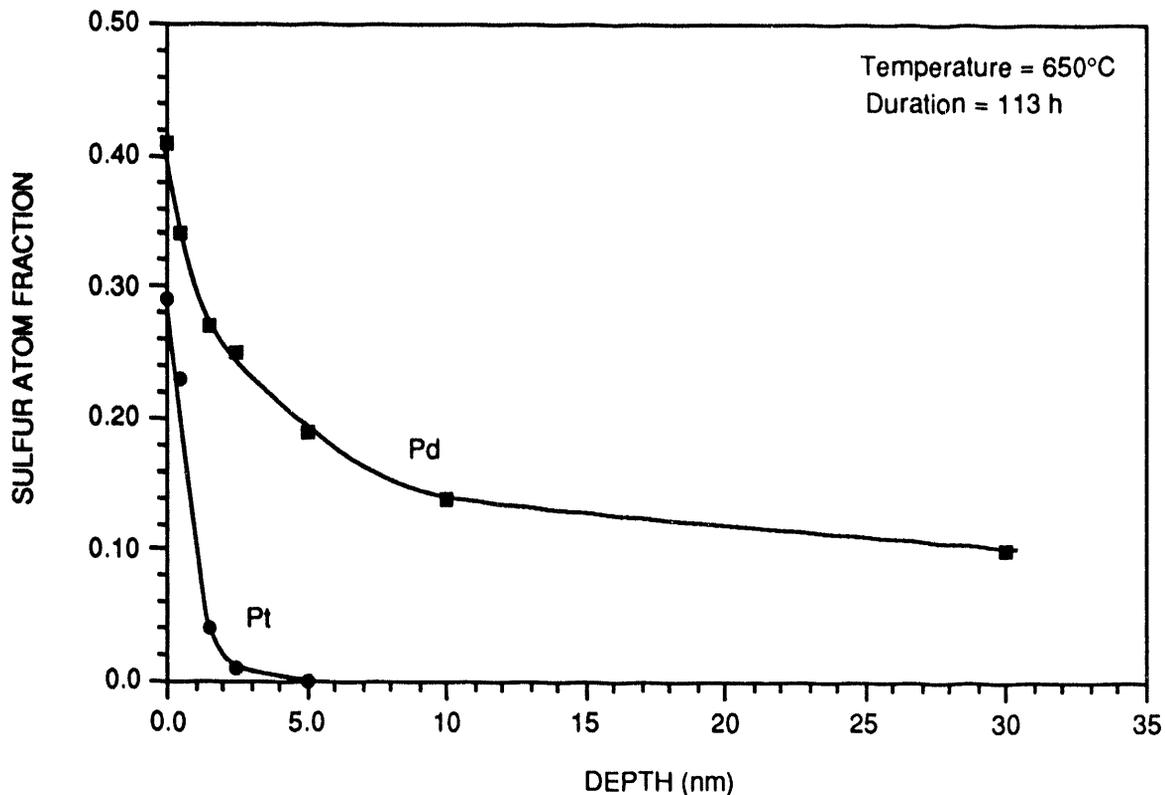


Figure 2. Incorporation of Sulfur on the Platinum and Palladium Membranes

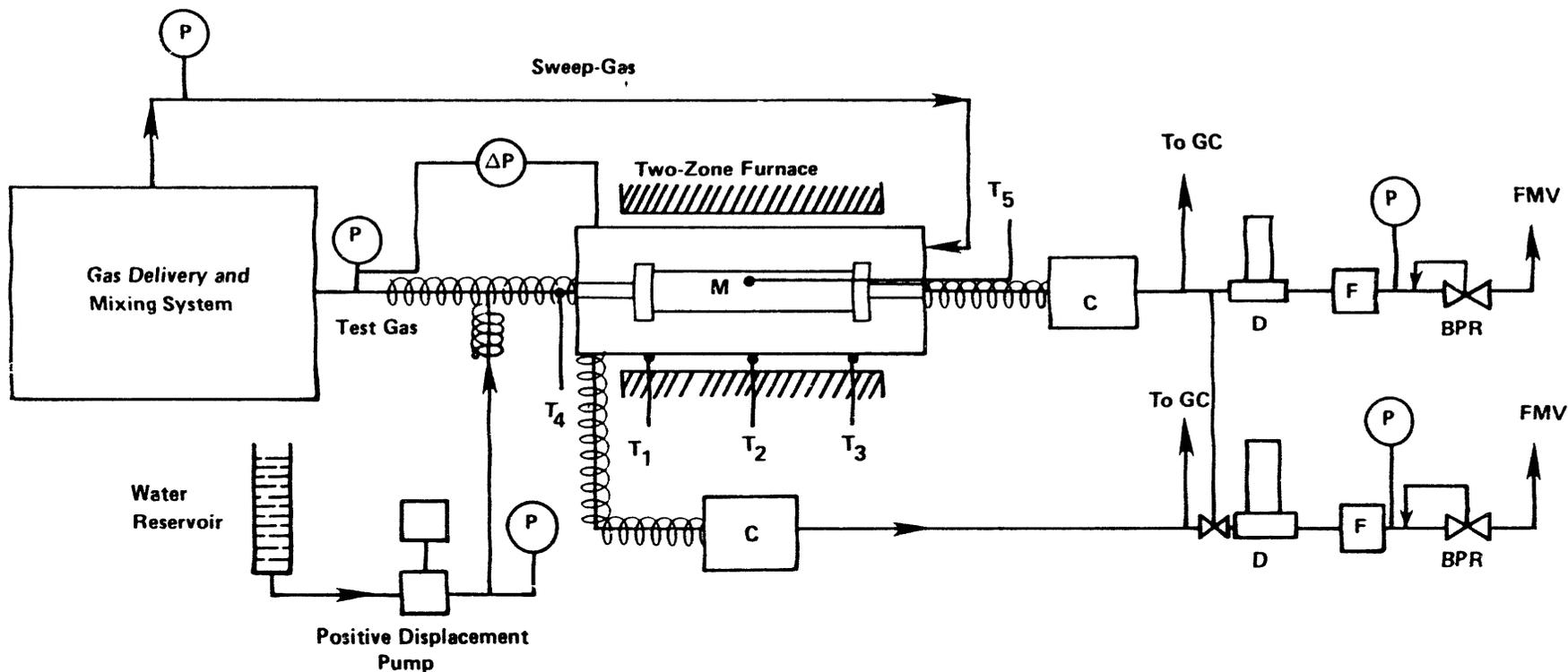
Platinum foil samples exposed under all experimental conditions were unaffected visually. AES revealed a sulfur adlayer on the surfaces of all the samples, but the concentration decreased rapidly with argon sputtering and no sulfur was detected at depths about 5 nm beneath the foil surface. Thus, platinum sulfidation appears to be confined to the surface regardless of the exposure duration, temperature, and gas phase  $H_2S$  concentration.

### Long-Term Membrane Permeation Testing

The purpose of long term testing of membrane permeation behavior is to quantify the changes in the membrane performance under controlled conditions. Specifically, changes in membrane permeation and selectivity characteristics will be determined as a function of time for a variety of operating conditions. The permeation

measurements will also identify the key variables leading to loss of performance, if any, and will allow correlation of the observed degradation characteristics of the membrane materials with actual membrane performance under specified conditions of exposure.

Figure 3 is a schematic diagram of the test apparatus being assembled for long-term permeation testing. The system consists of mass flow controllers for mixing gases of known composition, a high temperature furnace, a gas chromatograph (GC), and a controlling computer that commands the GC and logs GC data. Steam will be supplied by using a high pressure pump to meter water into a vaporizer situated in the gas line. Alkali vapors will be generated in a high pressure vapor generation system similar to the one used in the exposure studies.



FMV: Flow Meter and Vent  
M: Tubular Membrane Module  
P: Pressure Gauge  
 $\Delta P$ : Differential Pressure Gauge  
 $T_1$ - $T_6$ : Temperature Measurement and Control  
D: High Pressure Drier  
BPR: Back Pressure Regulator  
F: Filter

 Preheater/Heated Lines  
GC: Gas Chromatograph  
C: Condensation and Liquid Separation System

Figure 3. Schematic Diagram of the Membrane Test System

The candidate membranes will be placed in appropriate membrane holders. The membranes will be exposed to typical gasifier product compositions on the feed side at realistic conditions of temperature and pressure. The flow rate and composition of the membrane feed gas, permeate gas, and feed reject gas will be monitored for an extended period of time, up to 60 days.

## FUTURE WORK

Exposure studies will continue to determine changes in membrane characteristics in presence of fly ash and alkalis at high pressures, and for much longer durations. Modified Vycor and  $\gamma$ -alumina membrane samples will be included in these tests. Membrane permeation behavior will be determined in typical coal gas environments during long term tests. Also, selected membrane materials will be exposed to an actual hot coal gas stream at a pressurized fixed-bed gasifier facility, and the changes in the physical, chemical, and mechanical properties of the membrane that occur will be determined.

In Task 5, at the option of U.S. Department of Energy, the results from Task 4 and the data available in the literature will be used to develop a predictive model that can be used to estimate the long-term degradation of three selected inorganic membrane materials. Both theoretical and empirical approaches will be used in developing this model.

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Edlund, D. J., 1993. Catalytic Membranes for Facilitating the Water- Gas Shift Reaction. *Proceedings of the Coal-Fired Power Systems 93 -- Advances in IGCC and PFBC Review Meeting*, 233-237. DOE/METC-93/6131. NTIS/DE93000289. Springfield, Va.: National Technical Information Service.

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## **Appendices**

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## Appendix A

### Coal-Fired Power Systems 94 -- Advances in IGCC and PFBC Review Meeting

June 21- 23, 1994

#### AGENDA

##### TUESDAY, JUNE 21, 1994

- 7:30 a.m. Registration/Coffee and Refreshments
- 8:15 a.m. METC Site Tour
- 9:30 a.m. *Opening Remarks*  
Harvey M. Ness  
Morgantown Energy Technology Center

##### SESSION 1 -- OPENING COMMENTARIES

Chairperson: Harvey M. Ness

- 9:40 a.m. 1.1 *The Future for Advanced Power Systems*  
Thomas F. Bechtel, Director  
Morgantown Energy Technology Center
- 10:10 a.m. 1.2 *Evolving Utility Business Structure and Impact on New  
Technology Application*  
J. Wayne Leonard  
Senior Vice President and Chief Financial Officer  
PSI Energy, Inc.
- 10:55 a.m. BREAK

##### SESSION 2 -- CHANGES IN THE MARKET AND TECHNOLOGY DRIVERS

Chairperson: Rita A. Bajura

- 11:15 a.m. 2.1 *Overview of Global Utility Market for Advanced Coal-Fired Systems*  
Ronald Wolk  
Electric Power Research Institute

- 11:40 a.m. 2.2 *Overview of the Independent Power Producer (IPP) Market  
for Advanced Coal-Fired Systems*  
Dale Simbeck  
SFA Pacific, Inc.
- 12:05 p.m. 2.3 *The Search for Innovative Technology*  
C. Lowell Miller  
DOE Headquarters
- 12:30 p.m. LUNCH -- METC ENERGIZER

### SESSION 3 -- ADVANCED IGCC SYSTEMS

Co-Chairpersons: Justin L. Beeson and Dale K. Schmidt

- 1:30 p.m. 3.1 *IGCC System Analysis*  
Larry K. Rath  
Morgantown Energy Technology Center
- 1:55 p.m. 3.2 *Transport Reactor Development Status*  
William M. Campbell  
The M.W. Kellogg Company
- 2:20 p.m. 3.3 *Gasification Product Improvement Facility Status*  
Vijay B. Dixit  
CRS Serrine Engineers, Inc.
- 2:45 p.m. BREAK

### SESSION 4 -- ADVANCED PFBC SYSTEMS

Co-Chairpersons: Donald L. Bonk and Randall J. Dellefield

- 3:10 p.m. 4.1 *PFBC System Studies*  
Michael E. Reed  
Morgantown Energy Technology Center
- 3:35 p.m. 4.2 *Second-Generation PFBC Systems Research and Development -  
Circulating Pressurized Fluidized Bed Combustor Test Results*  
Rich Conn  
Foster Wheeler Development Corporation

4:00 p.m. 4.3 *Status of the Advanced PFBC at the Power Systems  
Development Facility*  
Darrell L. Moore  
Southern Company Services, Inc.

4:25 p.m. ADJOURN

6:30 p.m. BARBECUE  
Lakeview Resort and Conference Center

**WEDNESDAY, JUNE 22, 1994**

7:30 a.m. Coffee and Refreshments

8:00 a.m. *Opening Remarks*  
Venkat K. Venkataraman  
Morgantown Energy Technology Center

**SESSION 5 -- ADVANCED FILTER SYSTEMS**

Chairperson: Richard A. Dennis

8:10 a.m. 5.1 *Status of the Morgantown Energy Technology Center's Particulate  
Cleanup Program -- Enabling Technology for Advanced Coal  
Based Power Systems*  
Richard A. Dennis  
Morgantown Energy Technology Center

8:20 a.m. 5.2 *Potential Industry Use of Continuous Fiber Ceramic Composites*  
Jill Jonkouski  
U.S. Department of Energy, DOE Chicago Operations Office  
Office of Energy Efficiency and Renewable Energy

8:45 a.m. 5.3 *Ceramic Fiber Ceramic Matrix Filter Development*  
Roddie R. Judkins  
Oak Ridge National Laboratory

9:10 a.m. 5.4 *Westinghouse Advanced Particulate Filter System*  
Thomas E. Lippert  
Westinghouse Electric Corporation

9:35 a.m. BREAK

- 9:55 a.m. 5.5 *IF&P Fibrosic™ Filters*  
Paul Eggerstedt  
Industrial Filter & Pump Mfg. Co., Inc.
- 10:20 a.m. 5.6 *Multi-Contaminant Control Granular Bed Filter*  
John C. Haas  
Combustion Power Company
- 10:45 a.m. 5.7 *Westinghouse Standleg Moving Granular Bed Filter Status*  
Richard A. Newby  
Westinghouse Electric Corporation
- 11:10 a.m. BREAK
- 11:30 a.m. 5.8 *MTCI Acoustic Agglomeration Particulate Control*  
Ravi R. Chandran  
Manufacturing and Technology Conversion International, Inc.

## SESSION 6 -- DESULFURIZATION SYSTEMS

Chairperson: Ronald K. Staubly

- 11:55 a.m. 6.1 *METC Hot Gas Desulfurization Program Overview*  
Daniel C. Cicero  
Morgantown Energy Technology Center
- 12:05 p.m. 6.2 *Integrated Operation of a Pressurized Fixed Bed Gasifier,  
Hot Gas Desulfurization and Turbine Simulator System*  
David Najewicz  
GE Corporate R&D
- 12:30 p.m. LUNCH -- METC ENERGIZER
- 1:30 p.m. 6.3 *Enviropower Hot Gas Desulfurization PDU*  
Jukka T. Kontinen  
Enviropower, Inc.
- 1:55 p.m. 6.4 *Slipstream Testing of the Direct Sulfur Recovery Process*  
Jeffrey W. Portzer  
Research Triangle Institute
- 2:20 p.m. 6.5 *METC Fluid-Bed Hot-Gas Desulfurization PDU*  
Larry A. Bissett  
Morgantown Energy Technology Center

2:45 p.m.

BREAK

### SESSION 7 -- TURBINE SYSTEMS

Chairperson: Darren J. Mollot

- 3:05 p.m.    7.1    *Development of Topping Combustor for Advanced Concept  
Pressurized Fluidized Bed Combustion*  
Thomas E. Lippert  
Westinghouse Electric Corporation
- 3:30 p.m.    7.2    *Development and Testing of Low Btu Fuel Gas Turbine  
Combustors*  
Alan S. Feitelberg  
GE Environmental Services, Inc.
- 3:55 p.m.    7.3    *Advanced Turbine Systems Program Overview*  
Holmes A. Webb  
Morgantown Energy Technology Center
- 4:10 p.m.                    POSTER SESSION

### POSTER SESSION

- P1    *High Temperature Electrochemical Separation of H<sub>2</sub>S from Coal  
Gasification Streams*  
Jack Winnick  
Georgia Institute of Technology
- P2    *Characterization and Fixed-Bed Testing of a Nickel-Based Hot Gas  
Desulfurization Sorbent*  
Lee Gasper-Galvin  
Morgantown Energy Technology Center  
Kurt Hammerbeck  
Oak Ridge Associated Universities
- P3    *Pilot Gasification and Hot Gas Cleanup Operations*  
John M. Rockey, Edwin Galloway, and Teresa A. Thomson  
Morgantown Energy Technology Center  
Jay Rutten and Alain P. Lui  
EG&G Technical Services of West Virginia

- P4 *Coal Ash Behavior in Reducing Environments*  
Thomas A. Erickson  
Energy and Environmental Research Center  
University of North Dakota
- P5 *Hot Coal Gas Desulfurization With Manganese-Based Sorbents*  
Rachid Ben Slimane  
University of Minnesota
- P6 *A Calcium Oxide Sorbent Process for Bulk Separation of Carbon Dioxide*  
Douglas P. Harrison  
Louisiana State University
- P7 *COMPCOAL™ -- A Stable, High-Btu Fuel from Western Coals*  
Norman Merriam  
Western Research Institute
- P8 *Development of Biological Coal Gasification (MicGas Process)*  
Radosvet J. Manolov  
ARCTECH, Inc.
- P9 *Simultaneous Removal of H<sub>2</sub>S and NH<sub>3</sub> in Coal Gasification Processes*  
K. Jothimurugesan and Adeyinka A. Adeyiga  
Hampton University  
Santosh K. Gangwal  
Research Triangle Institute
- P10 *Granular Filtration in a Fluidized Bed*  
J. S. Mei, P.C. Yue, and J. S. Halow  
Morgantown Energy Technology Center
- P11 *Dust Cake Behavior in Filters with High Surface Area to Volume Ratios*  
Douglas Straub, Ta-Kuan Chiang, and Richard Dennis  
Morgantown Energy Technology Center
- P12 *Computer Simulation of IGCC/PFBC Components*  
Thomas O'Brien, Edward J. Boyle, and Lawrence J. Shadle  
Morgantown Energy Technology Center  
Madhava Syamlal and Suresh Venkatesan  
EG&G Technical Services of West Virginia
- P13 *Scaling of Pressurized Fluidized Beds*  
Leon R. Glicksman  
Massachusetts Institute of Technology

- P14 *Evaluation of Options for CO<sub>2</sub> Capture/Utilization/Disposal*  
Richard D. Doctor  
Argonne National Laboratory
- P15 *Absorption of Hydrogen Sulfide by Zinc Ferrite in the Temperature Range 315 to 538 °C (600 to 1000 °F)*  
Thomas Grindley  
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C. Elaine Everitt  
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- P16 *High Temperature Hydrogen Sulfide Removal with Stannic Oxide*  
Robert J. Copeland  
TDA Research, Inc.
- P17 *Fossil Fuel Conversion - Measurement and Modeling*  
Peter R. Solomon  
Advanced Fuel Research, Inc.
- P18 *CeraMem Filters for Removal of Particles from Hot Gas Streams*  
Najib Khali  
CeraMem Corporation
- P19 *Development of Disposable Sorbents for Chloride Removal from High-Temperature Coal-Derived Gases*  
Gopala Krishnan  
SRI International
- P20 *PyGas Design Support*  
Lawrence J. Shadle  
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- P21 *Chloride and Mercury Monitors for Air Toxics Measurements*  
Glenn A. Norton  
Ames Laboratory
- P22 *Cooperative Research and Development Agreements at METC*  
J. Christopher Ludlow  
Morgantown Energy Technology Center
- P23 *Optimal Design and Synthesis of Advanced Power Systems Under Uncertainty*  
U.M. Diwekar and E.S. Rubin  
Carnegie Mellon University

P24 *Development of a Radio Frequency Surface Contour Mapping System*  
Warren E. Straszheim  
Ames Laboratory

6:00 p.m. ADJOURN

**THURSDAY, JUNE 23, 1994**

7:30 a.m. Coffee and Refreshments

**SESSION 8A -- FILTER TECHNICAL ISSUES**

Chairperson: Theodore J. McMahon

8:15 a.m. *Opening Remarks*  
Daniel C. Cicero  
Morgantown Energy Technology Center

8:25 a.m. 8a.1 *Tidd PFBC Hot Gas Filter Operating Experience:  
July 1993 - April 1994*  
John D. Hoffman  
American Electric Power Service Corporation

8:50 a.m. 8a.2 *Karhula Hot Gas Cleanup Test Results*  
Thomas E. Lippert  
Westinghouse Electric Corporation

9:15 a.m. 8a.3 *Durability of Ceramic Filters*  
Mary Anne Alvin  
Westinghouse Electric Corporation

9:40 a.m. BREAK

10:00 a.m. 8a.4 *Properties of Ceramic Candle Filters*  
Stuart Starrett  
Southern Research Institute

10:25 a.m. 8a.5 *Results of Patch Tests*  
Duane H. Pontius  
Southern Research Institute

10:50 a.m. 8a.6 *PFBC Dust Cake Studies*  
Richard A. Newby  
Westinghouse Electric Corporation

11:15 a.m. BREAK

## SESSION 8B -- HAZARDOUS AIR POLLUTANTS

Chairperson: Peter E. Botros

8:15 a.m. *Opening Remarks*  
Heather M. McDaniel  
Morgantown Energy Technology Center

8:25 a.m. 8b.1 *Comprehensive Assessment of Toxic Emissions From  
Coal-Fired Power Plants*  
Thomas D. Brown  
Pittsburgh Energy Technology Center

8:50 a.m. 8b.2 *Trace Element Emissions*  
Steven A. Benson  
Energy & Environmental Research Center  
University of North Dakota

9:15 a.m. 8b.3 *HAP Sampling at Tidd PFBC*  
Patrick A. Dal Porto  
American Electric Power

9:40 a.m. BREAK

10:00 a.m. 8b.4 *Trace Species Emissions for IGFC*  
A. Ed Pigeaud  
Energy Research Corporation

10:25 a.m. 8b.5 *HAPs Sampling for Advanced Power Systems*  
Robert M. Mann  
Radian Corporation

10:50 a.m. 8b.6 *Instrumentation for Trace Emission Measurements*  
William P. Chisholm  
Morgantown Energy Technology Center

11:15 a.m. BREAK

## SESSION 9A -- SORBENTS AND SOLID WASTES

Chairperson: Lee D. Gasper-Galvin

- 11:35 a.m. 9a.1 *Moving-Bed Sorbents*  
Raul E. Ayala  
GE Corporate Research and Development
- 11:55 a.m. 9a.2 *Fluid-Bed Sorbents*  
Raghubir P. Gupta  
Research Triangle Institute
- 12:15 p.m. 9a.3 *Fluidized Bed Desulfurizer Using Phillips Z-Sorb Sorbent*  
Gil J. Greenwood  
Phillips Petroleum Corp.
- 12:30 p.m. LUNCH -- METC ENERGIZER
- 1:30 p.m. 9a.4 *Desulfurization Sorbent Development at the Morgantown Energy  
Technology Center*  
Ranjani V. Siriwardane  
Morgantown Energy Technology Center
- 1:55 p.m. 9a.5 *Stabilization of Spent Calcium-Based Sorbent*  
Satyan Katta  
The M.W. Kellogg Company
- 2:20 p.m. 9a.6 *Market Assessment and Technical Feasibility Study  
of PFBC Ash Use*  
Alan E. Bland  
Western Research Institute
- 2:45 p.m. 9a.7 *Co-Firing Waste Materials in an Advanced Pressurized  
Fluidized-Bed Combustor*  
Michael R. DeLallo  
Gilbert/Commonwealth, Inc.
- 3:10 p.m. *Closing Remarks*  
William T. Langan  
Morgantown Energy Technology Center

## SESSION 9B -- MEMBRANES

Chairperson: Susan K. Joines

- 11:35 a.m. 9b.1 *High Temperature Size Selective Membranes*  
S. James Zhou  
AlliedSignal, Inc.
- 12:00 p.m. 9b.2 *A Catalytic Membrane Reactor for Facilitating Water-Gas Shift  
Reaction at High Temperature*  
Dwayne Friesen  
Bend Research, Inc.
- 12:25 p.m. LUNCH -- METC ENERGIZER
- 1:30 p.m. 9b.3 *New Developments in Hydrogen Permselective Membranes*  
George R. Gavalas  
California Institute of Technology
- 1:55 p.m. 9b.4 *Preparation and Characterization of Composite Membrane for High  
Temperature Gas Separation*  
Shamsuddin Ilias  
North Carolina A&T State University
- 2:20 p.m. 9b.5 *Development of Hollow Fiber Catalytic Membrane Reactors for  
High Temperature Gas Cleanup*  
Yi Hua Ma  
Worcester Polytechnic Institute
- 2:45 p.m. 9b.6 *Thermal/Chemical Degradation of Inorganic Membrane Materials*  
Gopala Krishnan  
SRI International
- 3:10 p.m. *Closing Remarks*  
Harvey M. Ness  
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## Appendix B

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