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ABSTRACT

During this quarter, work was focused on characterizing the stability of layered composite membranes in a one hundred percent permeate environment. Permeation data was also collected on cermets as a function of thickness. A thin film deposition procedure was used to deposit dense thin BCY / Ni onto a tubular porous support. Thin film tubes were then tested for permeation at ambient pressure. Process flow diagrams were prepared for inclusion of hydrogen separation membranes into IGCC power plants under varying conditions. Finally, membrane promoted alkane dehydrogenation experiments were performed.

EXECUTIVE SUMMARY

Eltron Research Inc. and team members CoorsTek, Süd Chemie, Argonne National Laboratory, and NORAM are developing an environmentally benign, inexpensive, and efficient method for separating hydrogen from gas mixtures produced during industrial processes, such as coal gasification. This project was motivated by the National Energy Technology Laboratory (NETL) Vision 21 initiative, which seeks to economically eliminate environmental concerns associated with the use of fossil fuels. Currently, this project is focusing on four basic categories of dense membranes: i) mixed conducting ceramic/ceramic composites, ii) mixed conducting ceramic/metal (cermet) composites, iii) cermets with hydrogen permeable metals, and iv) layered composites containing hydrogen permeable alloys. Ultimately, these materials must enable hydrogen separation at practical rates under ambient and high-pressure conditions, without deactivation in the presence of feedstream components such as carbon dioxide, water, and sulfur.

This report contains results for composite layered membrane stability experiments, membrane promoted alkane dehydrogenation, and cermet thin film fabrication and permeation testing. In addition progress is described on reactor engineering and catalyst development.

INTRODUCTION

The objective of this project is to develop an environmentally benign, inexpensive, and efficient method for separating hydrogen from gas mixtures produced during industrial processes, such as coal gasification. Currently, this project is focusing on four basic categories of dense membranes: i) mixed conducting ceramic/ceramic composites, ii) mixed conducting ceramic/metal (cermet) composites, iii) cermets with hydrogen permeable metals, and iv) layered composites with hydrogen permeable alloys. The primary technical challenge in achieving the goals of this project will be to optimize membrane composition to enable practical hydrogen separation rates and chemical stability. Other key aspects of this developing technology include catalysis, ceramic processing methods, and separation unit design operating under high pressure. To achieve these technical goals, Eltron Research Inc. has organized a consortium consisting of CoorsTek, Süd Chemie, Inc. (SCI), Argonne National Laboratory (ANL), and NORAM.

During this quarter composite layered membrane stability was characterized at intermediate temperature and high pressure. BCY / Ni cermets in the form of dense disks with varying thicknesses and thin film tubes were tested for permeation as a function of temperature. These results are presented in this report, in addition to progress with thin film cermet fabrication, catalyst development, and reactor engineering.

EXPERIMENTAL

The Experimental Section of the first quarterly report (January 1, 2001) contained detailed descriptions of equipment and procedures to be used over the duration of this program. The specific aspects presented were: (a) preparation of ceramic powders, (b) preparation of composite materials, (c) fabrication of tube and disk membranes, (d) construction and operation of ambient-pressure hydrogen separation units, (e) construction and operation of high-pressure hydrogen separation units, (f) hydrogen transport and ambipolar conductivity measurements and calculations, and (g) fabrication of thin film ceramics. For brevity, these general issues will not be repeated. However, modification of equipment or methods, as well as any other experimentally relevant issues, will be reported in the Results and Discussion section under their corresponding Tasks as outlined in the original proposal.

RESULTS AND DISCUSSION

Tasks 1 & 2 Preparation, Characterization, and Evaluation of Hydrogen Transport Membranes

Contributors: Eltron, CoorsTek, ANL

I. Composite Layered Membranes with High Hydrogen Permeability – Eltron

One of the key requirements for the eventual implementation of hydrogen separation membranes in coal gasification power plants is membrane stability when a pure hydrogen atmosphere exists on the permeate side of the membrane. Eltron tested several different layered composite membrane thickness under 100% permeate stream conditions. Figure 1 shows that at 435°C and a 300 psig feed stream the membrane permeation was stable for all three thicknesses. After three hours the experiments were voluntarily terminated for post reactor analysis. Membranes were also tested up to 100 hours. Results showed that the performance of membranes were equivalent when a 100% permeate was present compared to a partial hydrogen permeate stream. Eltron will continue to monitor membrane stability for longer time frames and under varying temperature and pressure conditions.

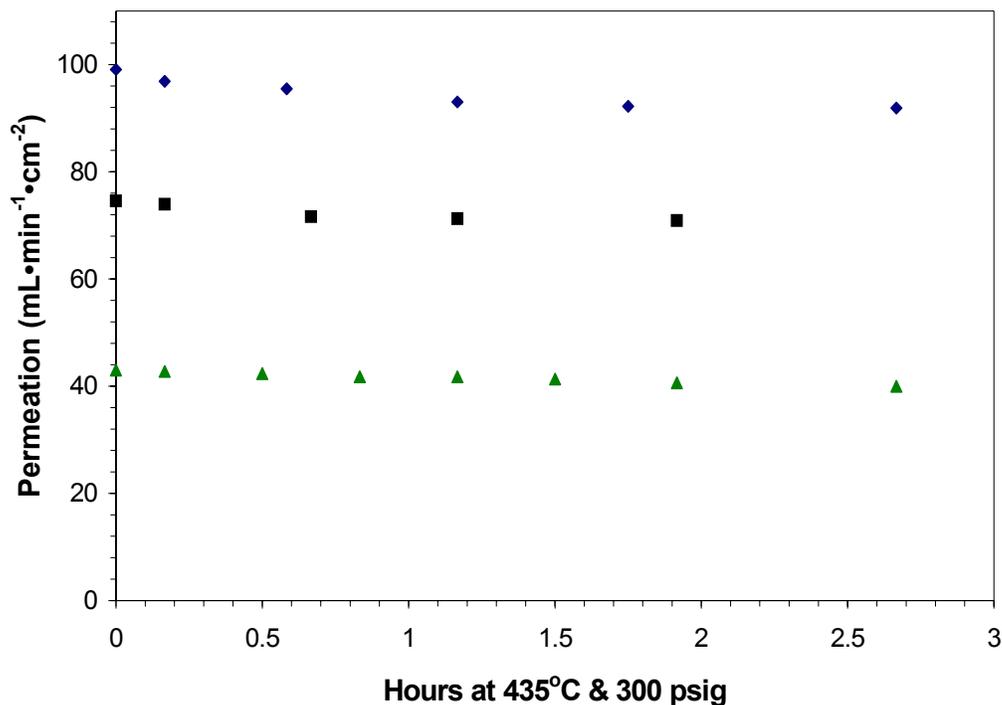


Figure 1. Hydrogen permeation for membranes with three different thicknesses tested at 435°C, 300 psig feed pressure, and a 100% hydrogen permeate stream.

II. Multi-Phase Ceramics and Cermets – ANL, CoorsTek

During this quarter, ANL investigated the thickness dependence of hydrogen flux for ANL-1a membranes in the thickness range 80-1160 μm . ANL-1a membranes contain a proton-conductor ($\text{BaCe}_{0.8}\text{Y}_{0.2}\text{O}_{3-a}$, i.e., BCY, purchased from Praxair) and an electron-conductor (Ni metal, purchased from Alfa Aesar). The metal phase boosts the ambipolar conductivity of the membrane by increasing its electronic conductivity. Because atomic hydrogen diffusion through the Ni phase is also detectable under some conditions, the total hydrogen flux through ANL-1a membranes, J_{H_2} , is expressed as the sum of ambipolar and atomic diffusion:

$$J_{\text{H}_2} = -\left\{ \frac{RT}{4F^2L} \int_{P_{\text{H}_2}^{\text{sweep}}}^{P_{\text{H}_2}^{\text{feed}}} \frac{\sigma_{\text{OH}^\bullet} \sigma_{e^-}}{\sigma_{\text{OH}^\bullet} + \sigma_{e^-}} d \ln P_{\text{H}_2} + \frac{P}{L} \nabla P_{\text{H}_2}^{1/2} \right\}$$

In this equation, σ_i is the partial conductivity of conducting species ($i = \text{OH}^\bullet, e^-$), R is the gas constant, F is Faraday's constant, T is temperature, P is the hydrogen permeability of Ni, and it is assumed that oxygen potential gradients do not influence the hydrogen flux. The hydrogen flux through the Ni phase was calculated using permeability data in the literature with the known applied chemical potential gradients and the measured membrane thickness. The ambipolar flux was calculated by subtracting the flux through Ni from the measured total flux. Figure 2 shows the hydrogen flux attributed to each diffusion mechanism as a function of temperature for feed gas of 4% H_2 /balance He with $p_{\text{H}_2\text{O}}$ of 0.03 atm. The flux due to ambipolar diffusion dominates over the entire temperature range investigated, but the flux through Ni increases with temperature due to the increase of hydrogen permeability.

The total hydrogen flux is shown versus temperature (Figure 3a) for ANL-1a membranes with thickness of 80-1160 μm using feed gas of 4% H_2 /balance He with $p_{\text{H}_2\text{O}}$ of 0.03 atm. The results show that the temperature dependence of hydrogen flux varies with the membrane thickness. For thicker ($>640 \mu\text{m}$) membranes, the flux decreases monotonically as temperature increases up to 900°C, but the flux for thinner ($<200 \mu\text{m}$) membranes increases as temperature increases up to $\approx 750^\circ\text{C}$ and then is nearly constant at higher temperatures. Membranes with intermediate thickness show a transition between these two types of behavior.

Figure 3b shows the flux due to ambipolar diffusion as a function of temperature. The ambipolar flux for thicker membranes, like the total flux, decreases monotonically as temperature increases. For thinner membranes, the flux due to ambipolar diffusion increases up to a temperature of $\approx 700^\circ\text{C}$ and then decreases as temperature increases further. This difference in the temperature dependence of flux might indicate that the kinetics of interfacial reactions dominate the properties of thin membranes at low temperature but are not dominant for thick membranes. In this case, the flux through thin membranes increases with temperature at low temperatures ($<700^\circ\text{C}$) due to thermal activation of rate-controlling surface reactions. At higher temperatures, where bulk processes dominate, the flux through thin membranes decreases due to the decrease in proton conductivity that results from a decreasing proton concentration. The decrease in proton concentration is indicated by the decrease in water solubility for perovskite-type proton conductors above 500°C, and is expected because the proton incorporation reaction is exothermic. In contrast to thin membranes, thick membranes do not exhibit a low-temperature regime in which the flux increases as temperature increases, because bulk properties dominate their behavior. As a result, the flux of thick membranes monotonically decreases as temperature

increases, because the decrease in proton concentration reduces the proton conductivity. Such a monotonic decrease in ambipolar flux for thick membranes is consistent with our previous findings.

In previous reports ANL results described the effect of metal and ceramic grain size on the conductivity of dense cermet. During the next quarter ANL will begin analysis of the effect of grain size and structure on cermet mechanical properties.

CoorsTek has begun work developing the processing parameters necessary for the preparation of new hydrogen separation cermets containing a high permeability metal. A matrix of potential cermet compositions and processing parameters has been prepared. Initially, cermets will be prepared as dense disks. Eltron will perform permeation testing once dense disks have been prepared and characterized.

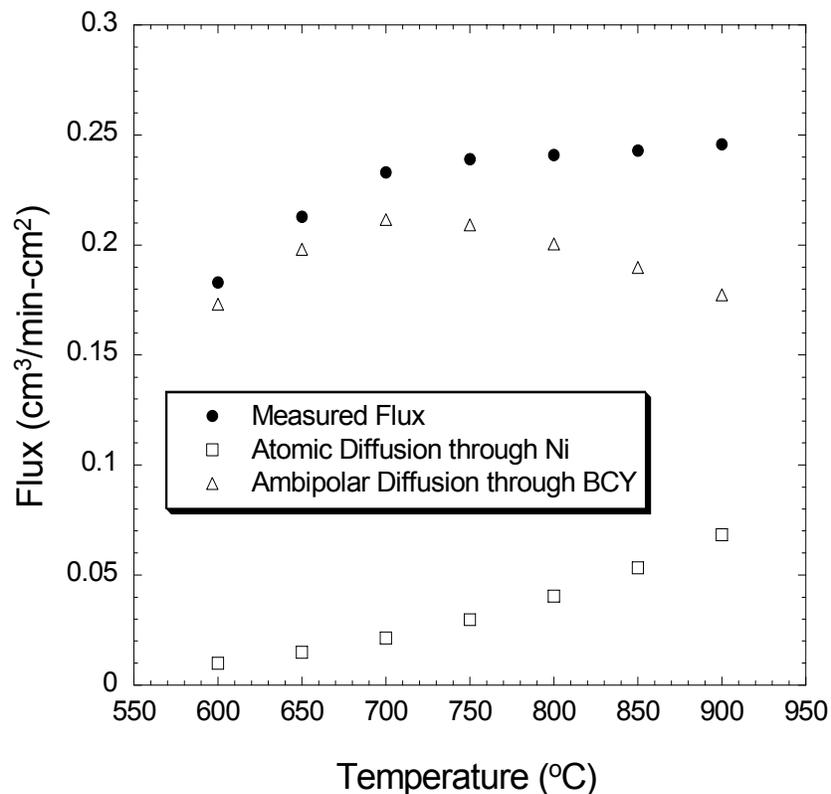


Figure 2. Measured hydrogen flux for 80- μm -thick ANL-1a membrane using feed gas of 4% H_2 /balance He ($p_{\text{H}_2\text{O}} = 0.03$ atm) with calculated contributions from ambipolar diffusion through BCY and atomic diffusion through Ni.

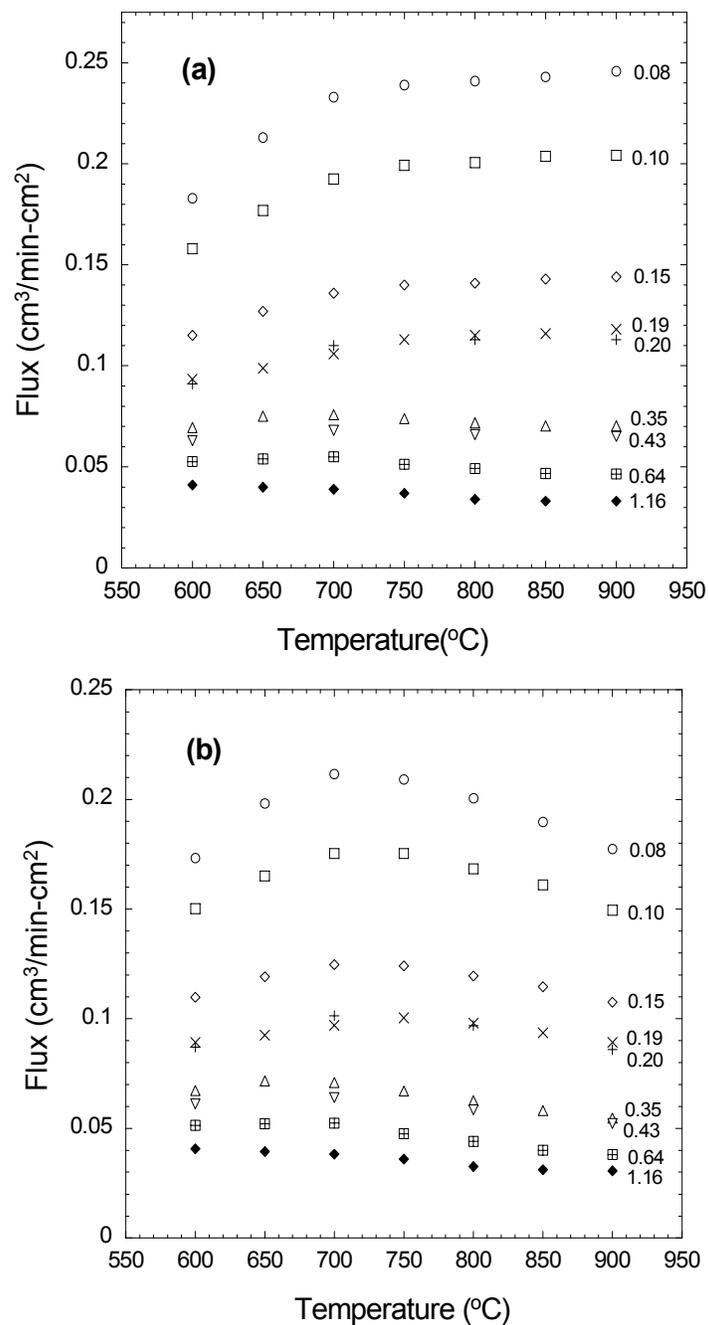


Figure 3. (a) Measured hydrogen flux, and (b) calculated flux due to ambipolar diffusion through BCY for ANL-1a membranes with various thickness (given in mm) using 4%H₂/balance He (pH₂O = 0.03 atm) feed gas.

III. Membrane Coatings and Catalysts – Eltron, SCI

Two different types of catalysts are needed for hydrogen separation membranes. The catalyst on the membrane surface exposed to the feed stream functions as a hydrogen dissociation catalyst. It must be stable to sulfur, steam, and CO₂. On the permeate side of the membrane, the catalyst coating promotes hydrogen desorption. SCI is currently developing sulfur tolerant catalysts for the retentate side of the membrane. Eltron is investigating catalysis on the permeate side of the membrane. Up to this point in the project the same catalyst has been used on both sides of the membrane for rapid screening. During the next quarter Eltron will test new permeate side catalysts.

Task 3 High Pressure Hydrogen Separation

Contributors: Eltron

Key results for high pressure hydrogen separation are discussed under Task 1. Eltron is currently upgrading a second high pressure reactor for performing long term permeation studies and for future evaluation of demonstration-scale hydrogen separation membranes.

Task 4 Thin-Film Hydrogen Separation Membranes

Contributors: CoorsTek, Eltron

As discussed in previous reports, CoorsTek has been developing a thin film deposition procedure for creating a dense thin film cermet membrane on a tubular support. In this process, Eltron prepared isopressed BCY / Ni closed one-end (COE) porous support tubes. Thin film slurry was then deposited on the inside diameter of the tubes and sintered by CoorsTek. Once sintered, tubes were brazed onto alumina tubing as shown in Figure 4. Ambient pressure permeation testing was then performed at Eltron Research Inc.

Dense thin film tubes were tested between 700 and 850°C and at ambient pressure with an 80% hydrogen feed stream. Permeation results for one of the tubes are shown in Figure 5. The data shows that permeation decreased as the temperature was decreased from 850°C to 800°C as expected, however, lowering the temperature to 700°C did not affect the permeation. This trend is consistent with ANL data which also showed a constant flux between 850 and 750°C. In addition to temperature the feed stream flow rates were varied, however, no effect on permeation was observed. Figure 6 shows an SEM image of the dense film on the porous support following permeation testing. The thin film is 20 µm thick. The measured permeation data of the thin film tubes was 0.012 mL/min/cm² which was lower than expected for a thin dense film of BCY / Ni. The low permeation data was attributed to a small leak which may have affected the data. SEM analysis following permeation testing indicated that the leak was not occurring through the thin film and was likely due to poor adhesion of the braze. The brazing process will be modified for future thin film tube testing. Several additional tubes will be tested in the next quarter which will wrap up thin film BCY / Ni testing. Once finished CoorsTek will focus on cermets containing a high permeability metal.



Figure 4. Dense BCY/Ni thin film tube brazed onto a 1/4" alumina tube.

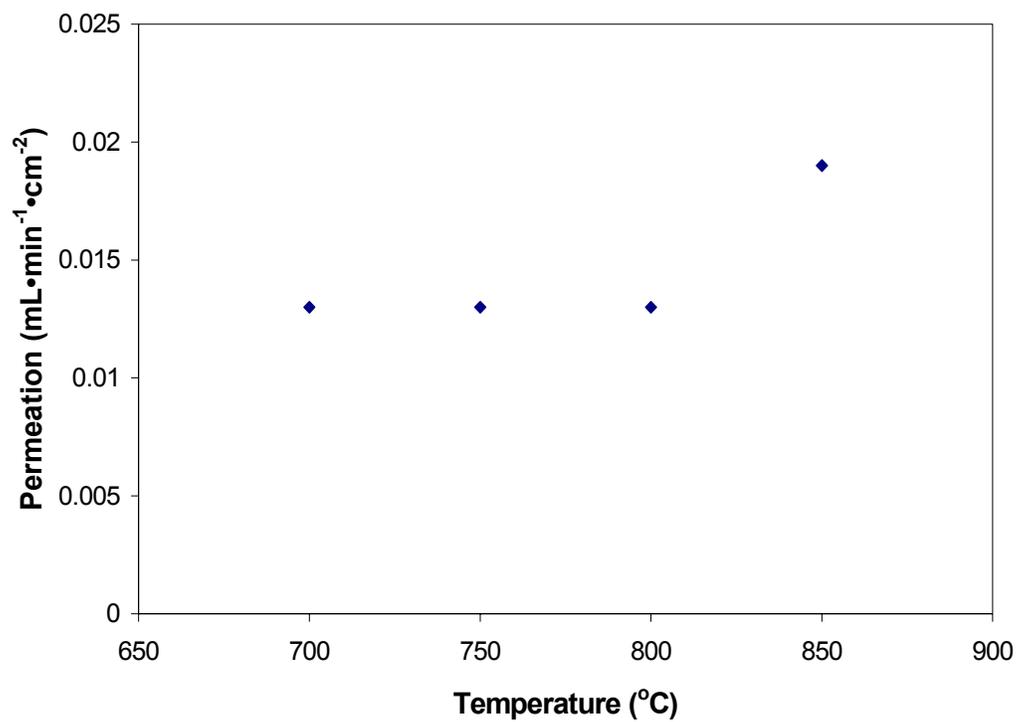


Figure 5. Permeation as a function of temperature for a thin BCY/Ni film on a tubular porous support.

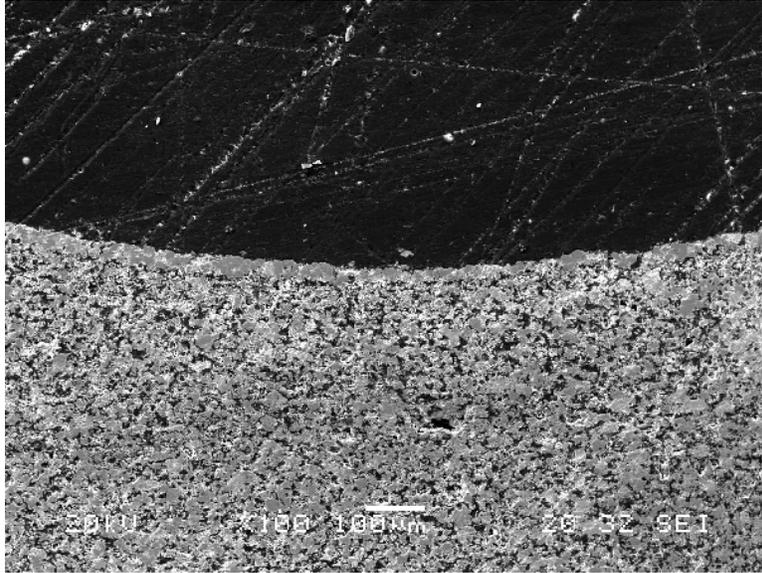


Figure 6. x100 SEM image of the cross-section of a dense BCY/Ni thin film on a tubular porous support.

Task 5 *Construction and Evaluation of Prototype Hydrogen Separation Unit*

Contributors: NORAM

NORAM's focus during this quarter was the preparation of five flowsheets that illustrate reasonable operating conditions for hydrogen separation membranes incorporated into an IGCC plant.

A review of IGCC and gas cleaning with comments on how Eltron's various membranes could be incorporated was completed earlier in the year and documented in NORAM's draft report dated January 22, 2004. Additional work has been performed during this quarter primarily aimed at incorporating the latest information available on warm gas cleaning. This is a rapidly developing area which offers considerable promise as a means to improve overall cycle efficiency and incorporate hydrogen membranes.

An extensive review of the latest published results pertaining to warm (500 to 1000°F) and hot gas cleaning (>1000°F), with an emphasis on desulfurization using regenerable metal oxide sorbents has been completed.¹⁻⁸ Through this review and discussions with one of the main developers (RTI), NORAM has prepared an estimate of the expected sulfur removal rates, temperatures and other considerations for a regenerable metal oxide (ZnO) based sorbent removal system.

Although no full scale commercial systems exist to date, based on recent smaller scale demonstration results it seems reasonable to assume that warm gas cleaning will be able to achieve total sulfur levels (the sum of COS and H₂S) of less than 10 ppmv in the near term. This information has been used to prepare several flowsheets incorporating warm gas cleaning.

Information on hot gas cleaning was reviewed previously; however cleaning systems at temperatures applicable to high temperature membranes (~1500°F) have thus far been unworkable. We are not aware of any hot gas desulfurization technology demonstrated successfully at temperatures suitable for high temperature membranes and it appears unlikely that suitable hot desulfurization will be commercially available in the near term. However, NORAM has prepared two flowsheets for high temperature membranes, one that utilizes warm gas cleaning and then partial oxidation of the syngas stream to reheat the syngas, and one that utilizes available hot gas cleaning but not hot desulfurization.

Information on established techniques for cold gas cleaning have been reviewed and incorporated into a cold gas cleaning flowsheet for use with intermediate temperature membranes.

The bulk of the mechanical considerations were investigated and reported on previously, however, with the completion of the flowsheets (Engineering Task 3) NORAM is now able to address some issues in more detail. Preliminary mechanical design criteria considered to date include:

- Tube sizing for internal / external pressures
- Multiple separator arrangement and manifolding
- Methods of dealing with thermal expansion (internal floating head vs. others)
- Sizing (based on flowsheets provided in Engineering Task 3).
- Fabrication considerations for different unit sizes
- Tube layouts for different unit sizes
- Pressure vessel calculations

NORAM has prepared several process simulations of a coal IGCC plant incorporating Eltron's hydrogen separation membranes using the commercially available process simulator, Hysys. These models simulate the plant unit operations affecting the conditions around the hydrogen separation membrane.

Elements such as the bottoming cycle, power generation, sulfur recovery plant, etc. have been considered when establishing the process conditions of the main units, but have not been simulated at this time as this would greatly increase the complexity of the model, without improving measurably the predictions of the conditions to which the hydrogen separation membranes will be exposed (temperature, pressure, bulk composition and contaminants).

While there are an almost infinite number of possible plant configurations which depend on the gasifier type selected, coal feed stock, plant size, percentage of hydrogen recovery, etc. it is possible to prepare flowsheets which describe reasonable conditions which would be appropriate for a hydrogen separation membrane.

NORAM has prepared five draft flowsheets describing conditions that hydrogen membranes could be expected to operate under when incorporated into a IGCC plant.

- Flowsheet 1: Warm Gas Cleaning for Intermediate Temp. Membranes
- Flowsheet 2: Warm Gas Cleaning for Intermediate Temp. Membranes & Sweep Steam
- Flowsheet 3: Cold Gas Cleaning for Intermediate Temperature Membranes
- Flowsheet 4: Warm Gas Cleaning & Reheat for High Temperature Membranes
- Flowsheet 5: Hot Gas Cleaning for High Temperature Membranes

All of the flowsheets are based on a 1000 psia slagging type, slurry fed gasifier with a radiant cooler (rather than quench). They are based on a plant size consuming approximately 3000 tpd of Pittsburg #8 coal. All of the flowsheets, except for Flowsheet 5, incorporate a water-gas-shift step to increase the amount of hydrogen available for separation.

The effect of hydrogen separation rate on membrane area was investigated. Based on this work a hydrogen separation rate of 80% was established as being a reasonable balance between the hydrogen production and the relative membrane area required.

Given the assumptions of plant size, amount of water gas shift and hydrogen recovery, each plant flowsheet represents a production of just under 300 tpd of pure hydrogen, except for Flowsheet 5 that does not utilize water gas shift due to the high temperatures involved. This level of hydrogen production represents roughly half of the expected useful energy production of the plant.

The effect of sweep steam was also considered. This analysis found that for the expected plant conditions, sweep steam used at economic rates does not greatly affect the membrane area and will likely not be warranted. Sweep steam could, however, potentially be much more effective in cases where greater hydrogen recovery rates, lower syngas pressures, or higher hydrogen permeate pressures are required.

Engineering Task 3 is now substantially complete. A report documenting the assumptions, draft flowsheets and other results is in-progress and will be issued in draft form within the next month. Once these flowsheets have been reviewed by Eltron, NORAM will incorporate any design changes required and finalize the flowsheets in preparation for engineering of the full scale and pilot scale units (Engineering Task 4).

Engineering of the full scale and pilot scale units will begin once the draft flowsheets have been reviewed and approved by Eltron.

Task 6 *Membrane-Promoted Conversion of Alkanes to Olefins*

Contributors: Eltron

In the previous report membrane-promoted alkane dehydrogenation was tested with a Pt/Sn catalyst. During this reporting period alkane dehydrogenation and hydrogen permeation were tested as a function of catalyst surface area. The Pt/Sn catalyst was deposited onto mullite saddles. Three equivalent experiments were performed with an increasing amount of Pt/Sn dehydrogenation catalyst present in the feed stream of the reactor. The first experiment used 20 mullite saddles, 40 saddles were used in the second experiment, and 80 saddles used in the third experiment. Each experiment was performed at 500°C with a 70 mL/min flow rate of a 10% propane / balance Ar mixture fed to the reactor. A small amount of helium was included in the feed stream for leak detection. Propane conversion was calculated by measuring the amount of propane flowing into the reactor and the amounts of propane and propene exiting the reactor in the retentate stream. Figure 7 shows that the percent propane conversion and percent selectivity. Propane conversion varied between 1 and 2% and was not dependent on the surface area of the catalyst. The percent selectivity of propane to propene conversion was calculated to be 65% for each experiment and was also not dependant on the surface area of the catalyst. No hydrogen was detected by GC on the permeate side of the membrane. This suggests that the rate of hydrogen production from alkane dehydrogenation was not fast enough to provide the hydrogen partial pressure driving force necessary to effectively separate the hydrogen produced.

A catalyst with a higher rate of propane conversion would be necessary to quantify the advantages of membrane promoted alkane conversion.

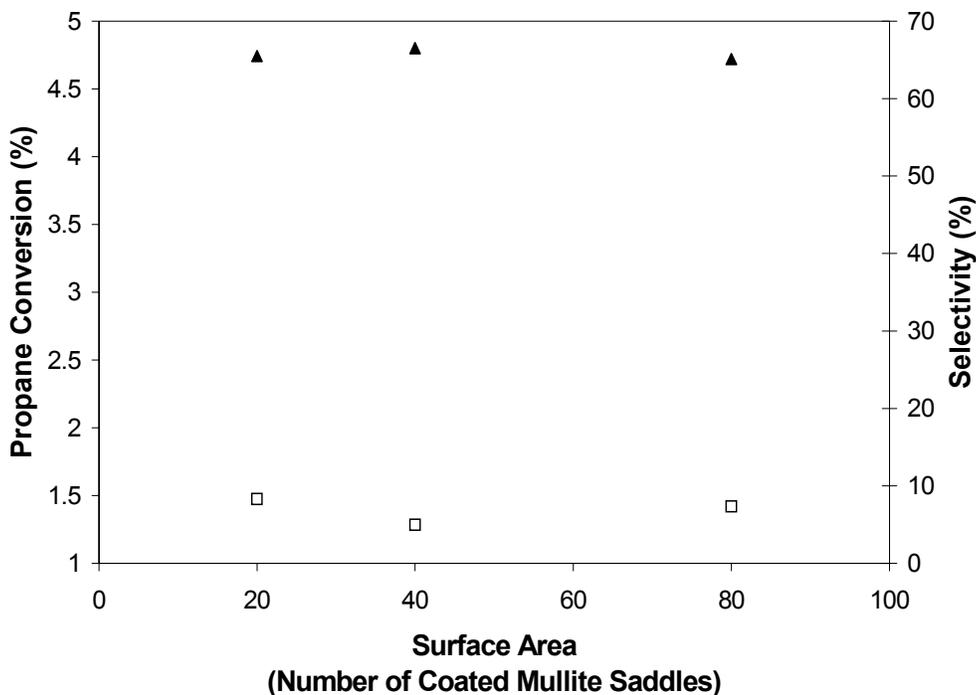


Figure 7. Propane conversion (open squares) and selectivity (filled triangles) of a Pt/Sn dehydrogenation catalyst used with a layered composite hydrogen separation membrane as a function of catalyst surface area.

Task 7 Catalyst Membrane Compositions for Scale Up

Testing during the past year focused on layered composite membranes. Results for these materials were compiled and compared to all the categories of membranes developed under this program. Based on hydrogen permeation rates, mechanical stability, and economics, the results to date clearly indicated that the layered composites have the greatest potential for scale up and commercial viability. In terms of ceramics and cermets, effort now is being focused on new cermets based on high permeability metals that have acceptably high permeation rates. In addition, cermets may provide chemical and mechanical stability advantages and have potential as protective/catalytic layers in the layered composite membranes.

Task 8 Manufacturing Processes for Demonstration-Scale Hydrogen Separation Membranes

CoorsTek has developed a thin film deposition procedure for the application of BCY / Ni thin films ranging from 10 to 100 μm onto the inner surface of a tubular support. This process has been discussed on a research and development scale under Task 4. These procedures are easily adaptable to manufacturing scale, however, the permeability of BCY / Ni materials will limit its commercial potential. As mentioned under Task 7, composite layered membranes have the greatest potential for scale up. New cermets based on high permeability metals also have potential, however, these cermets are in an early stage of development. Therefore, current work in Task 8 will focus on identification of materials and manufacturing processes for composite layered membranes.

Task 9 Fabrication and Evaluation of Demonstration-Scale Hydrogen Separation Unit

No actions were performed on this task during this quarter.

SUMMARY AND CONCLUSIONS

Conclusions based on the work performed during this quarter are summarized as follows:

1. High pressure, intermediate temperature permeation experiments performed with a 100% permeate stream demonstrated the stability of composite layered membranes.
2. A study of the permeation dependence of BCY / Ni ANL-1a membranes as a function of thickness showed that the temperature dependence of hydrogen flux varies with the membrane thickness.
3. Tubular cermet thin film membranes on porous supports were successfully fabricated and tested for permeation. Results showed a temperature independent permeation below 800°C.
4. Process flow diagrams were developed for five different scenarios for inclusion of Eltron's hydrogen separation membranes in a coal IGCC plant.
5. Membrane promoted alkane dehydrogenation was tested as a function of catalyst surface area. No difference in propane conversion was observed as the surface area of the catalyst was increased.

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OBJECTIVES FOR NEXT REPORTING PERIOD

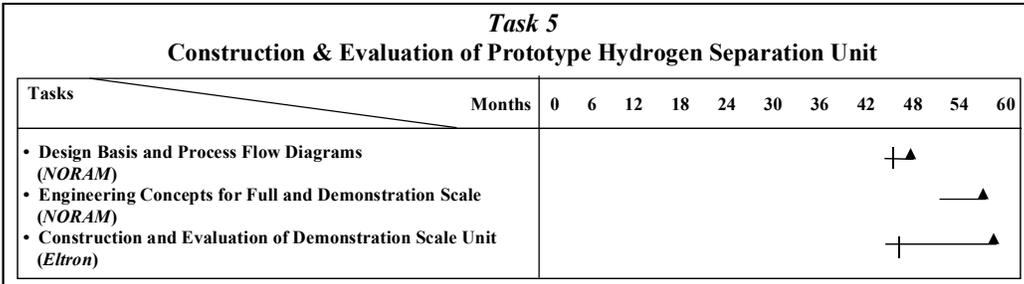
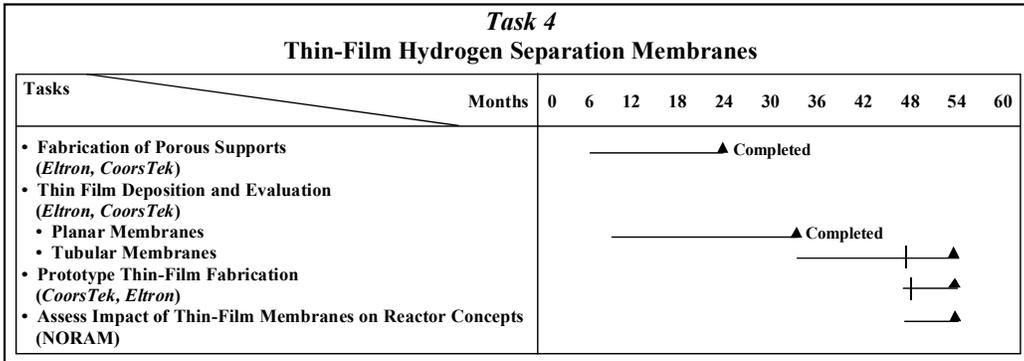
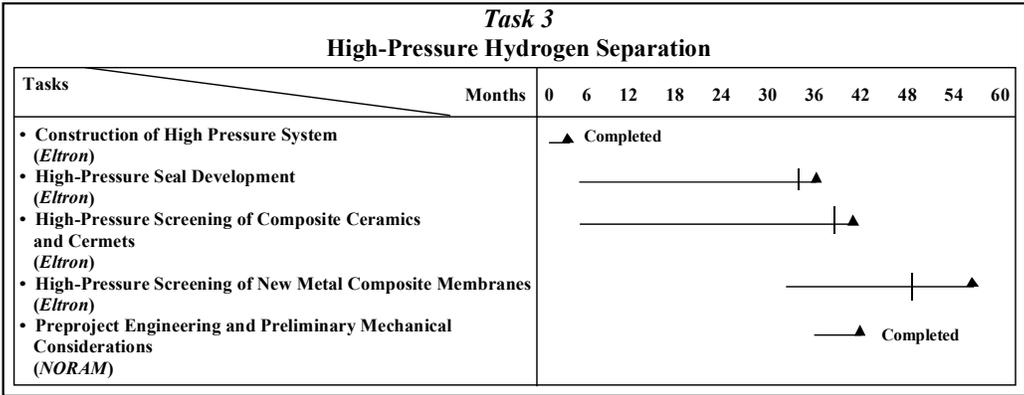
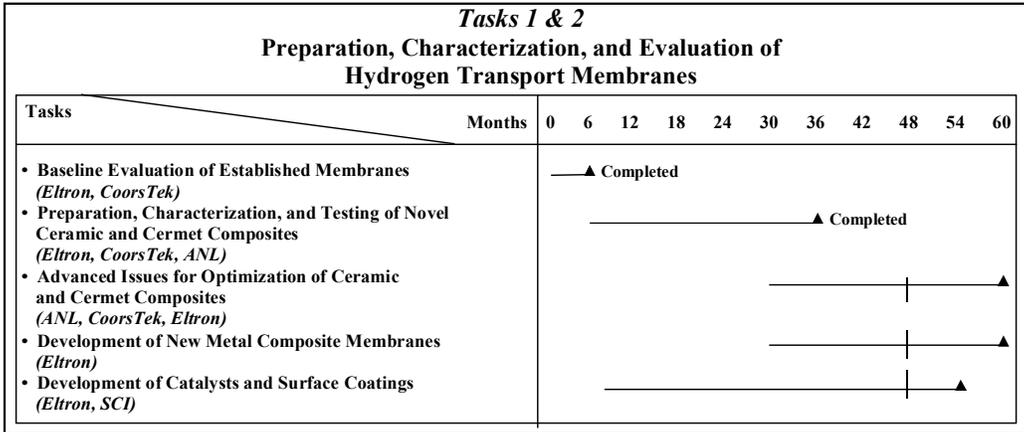
During the next reporting period efforts will be continued on characterizing membrane stability. Additional thin film BCY / Ni tubes will be tested, and new desorption catalysts analyzed. NORAM and Eltron will review the draft flow sheets created. Once approved, NORAM will begin engineering of a full scale unit. ANL will focus on mechanical properties of ANL-1a membranes. CoorsTek will continue development of new cermet compositions. Finally SCI will continue development of sulfur tolerant catalysts.

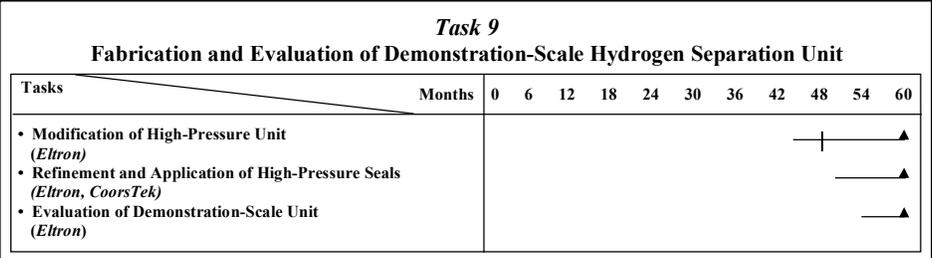
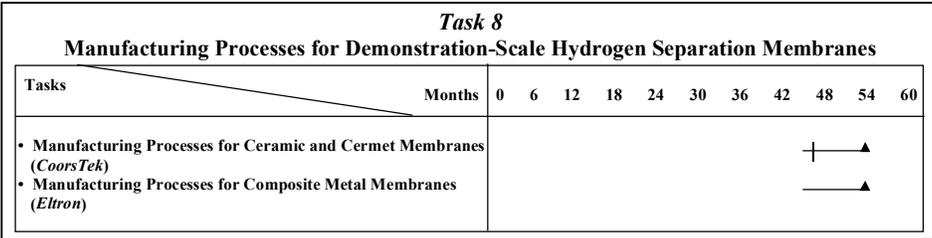
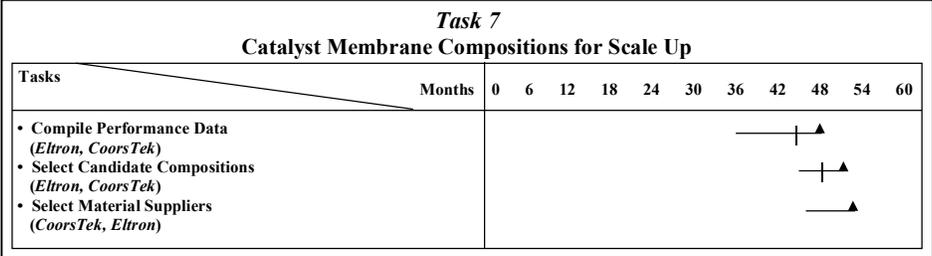
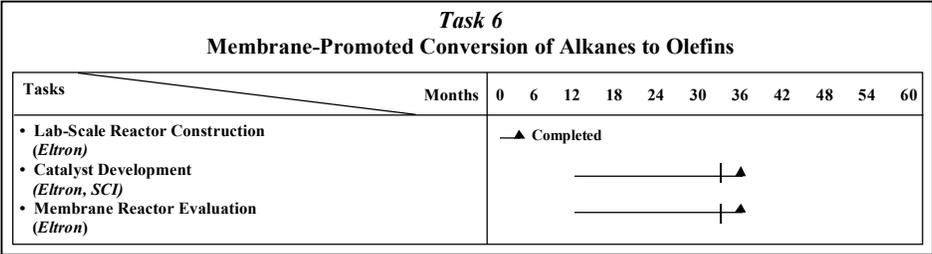
OPEN ITEMS OR COOPERATIVE AGREEMENT CHANGES

None.

TIME LINES

The time lines separated into each task are presented below, with markers indicating overall progress for each subtask.





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