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## 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.

Over the past 12 months, this project has focused 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. The ceramic/ceramic composites demonstrate the lowest hydrogen permeation rates, with a maximum of approximately 0.1 mL/min/cm<sup>2</sup> for 0.5-mm thick membranes at 800 to 950°C. Under equivalent conditions, cermets achieve a hydrogen permeation rate near 1 mL/min/cm<sup>2</sup>, and the metal phase also improves structural stability and surface catalysis for hydrogen dissociation. Furthermore, if metals with high hydrogen permeability are used in cermets, permeation rates near 4 mL/min/cm<sup>2</sup> are achievable with relatively thick membranes. Layered composite membranes have by far the highest permeation rates with a maximum flux in excess of 200 mL/min/cm<sup>2</sup>. Moreover, these permeation rates were achieved at a total pressure differential across the membrane of 450 psi. Based on these results, effort during the next year will focus on this category of membranes.

This report contains long-term hydrogen permeation data over eight-months of continuous operation, and permeation results as a function of operating conditions at high pressure for layered composite membranes. Additional progress with cermet and thin film membranes also is presented.

## 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.

Layered composite membranes containing inexpensive metals with high H<sub>2</sub> permeability demonstrated good long-term stability. Permeation in excess of 6 mLAnin<sup>-1</sup>Am<sup>-2</sup> has been maintained at 320/C and ambient pressure for over eight months of continuous operation. Analogous membranes demonstrated stable permeation rates up to 66 mLAnin<sup>-1</sup>Am<sup>-2</sup> at a differential pressure of 250 psi. However, optimization of the test conditions yielded H<sub>2</sub> permeation greater than 200 mLAnin<sup>-1</sup>Am<sup>-2</sup> at a differential pressure near 450 psi. Additional compositions were identified that demonstrate very high resistance to embrittlement when exposed to H<sub>2</sub> at temperatures between 100 and 450/C. Also described in this report is progress with cermet and thin film membranes.

## 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.

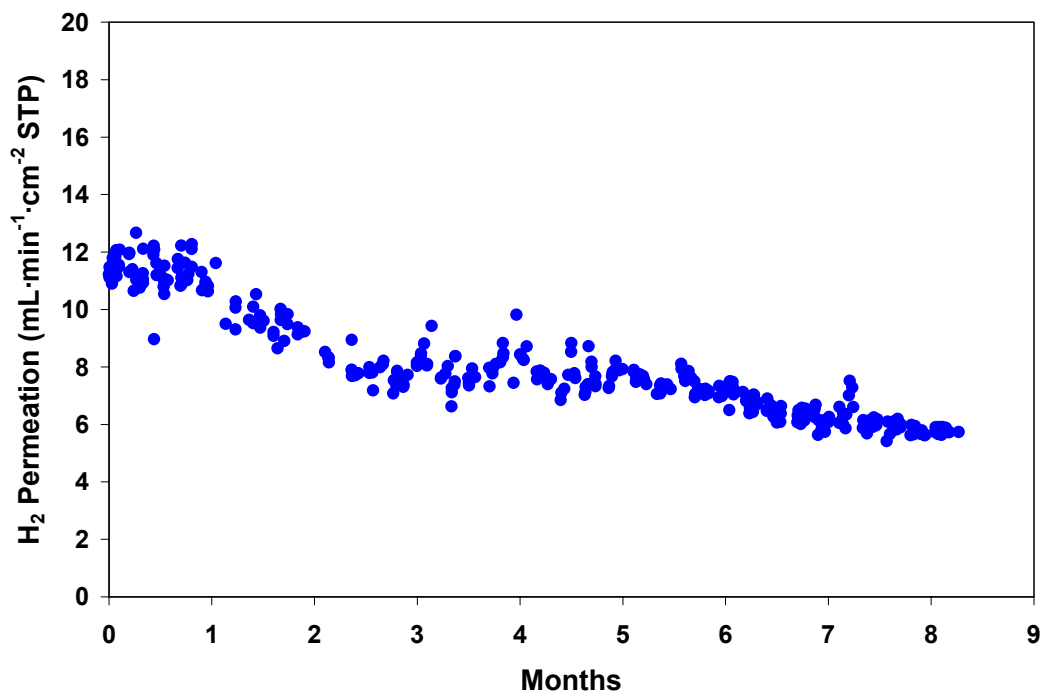
## RESULTS AND DISCUSSION

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

**Contributors:** Eltron, CoorsTek, SCI, ANL

#### I. **Composite Membranes with High Hydrogen Permeability** – Eltron

Figure 1 shows hydrogen permeation over more than eight months of continuous operation for a layered metal/ceramic composite membrane at 320°C. The total thickness of the membrane structure was approximately 2 mm, with only 0.13 mm from the metal phase. Over the first 500 hours of operation, permeation remained steady at approximately 11.5 mL<sub>A</sub>min<sup>-1</sup>cm<sup>-2</sup> with permeability greater than  $6 \times 10^{-8}$  mol<sub>A</sub>cm<sup>-2</sup>s<sup>-1</sup>Pa<sup>-0.5</sup>. There was a slow loss of performance between 500 and 1700 hours, which stabilized at roughly 8 mL<sub>A</sub>min<sup>-1</sup>cm<sup>-2</sup> until 4000 hours. Over the remaining 2000 hours, permeation decreased steadily to approximately 6 mL<sub>A</sub>min<sup>-1</sup>cm<sup>-2</sup>. The loss of permeation likely was due to build up of organic contaminants from the feed gas on the membrane surface. An attempt will be made to restore performance by applying an oxidation cycle using flowing air at 320°C.



**Figure 1.** Hydrogen permeation over time at 320°C for a layered metal/ceramic composite membrane. The feed gas was 80 mL/min 80/20 H<sub>2</sub>/He, and the sweep gas was 250 mL/min Ar.

During this reporting period, effort was focused on screening a range of compositions and identifying optimum testing conditions. Figure 2 shows H<sub>2</sub> permeation versus temperature for a 0.9-mm thick membrane at a differential pressure of 250 psi ( $P_{total}$ ). Under the flow conditions indicated in the figure, permeation increased to a maximum near 16 mLAnin<sup>-1</sup>Åm<sup>-2</sup> at approximately 400/C (permeability of  $1 \times 10^{-7}$  molAnAn<sup>-2</sup>Å<sup>-1</sup>Pa<sup>-0.5</sup>). Permeation stabilized beyond 400/C, and this trend generally was observed for most of the compositions tested. Permeation increased linearly with temperature between 130 and 380/C, yielding an apparent activation energy for hydrogen diffusion of 13 kJ/mol. Permeation also increased linearly according to Sieverts' Law (*i.e.*,  $p_f^{1/2} - p_s^{1/2}$ ) and peaked at over 20 mLAnin<sup>-1</sup>Åm<sup>-2</sup> at 360/C with the feed at 250 psi of 100% H<sub>2</sub>. The testing apparatus did not enable evaluation at greater differential partial pressures. The most significant result was that this sample did not suffer from hydrogen embrittlement until well below 100/C, which indicated an improvement in stability relative to other compositions tested previously.

To maximize permeation, the gas feed and sweep flow rates were increased from 500 to more than 1500 mL/min. Results for an analogous membrane are shown in Figure 3. Under these operating conditions, permeation was much higher and reached a maximum of 66 mLAnin<sup>-1</sup>Åm<sup>-2</sup> with a permeability greater than  $3 \times 10^{-7}$  molAnAn<sup>-2</sup>Å<sup>-1</sup>Pa<sup>-0.5</sup>. Deviation from Sieverts' Law occurred as the H<sub>2</sub> partial pressure differential increased beyond about 700 Pa<sup>1/2</sup>, and permeation remained constant as the feed pressure was raised to 200 psi. The deviation from Sieverts' could be either from a build up of surface contaminants over the duration of the experiment, or the sweep rate might not be sufficiently high to remove permeate H<sub>2</sub>.

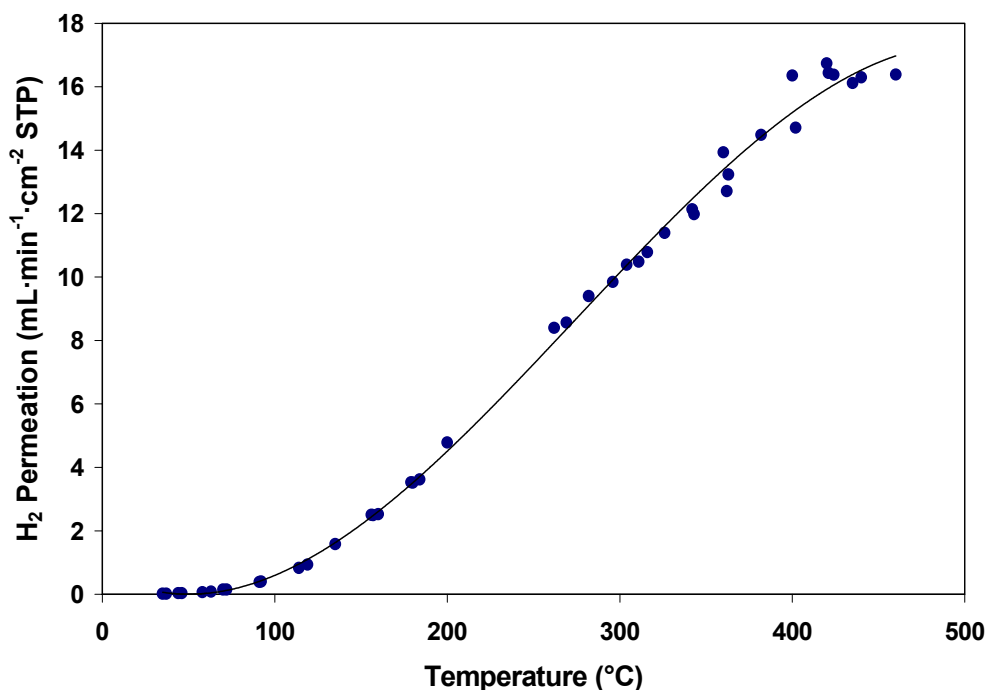
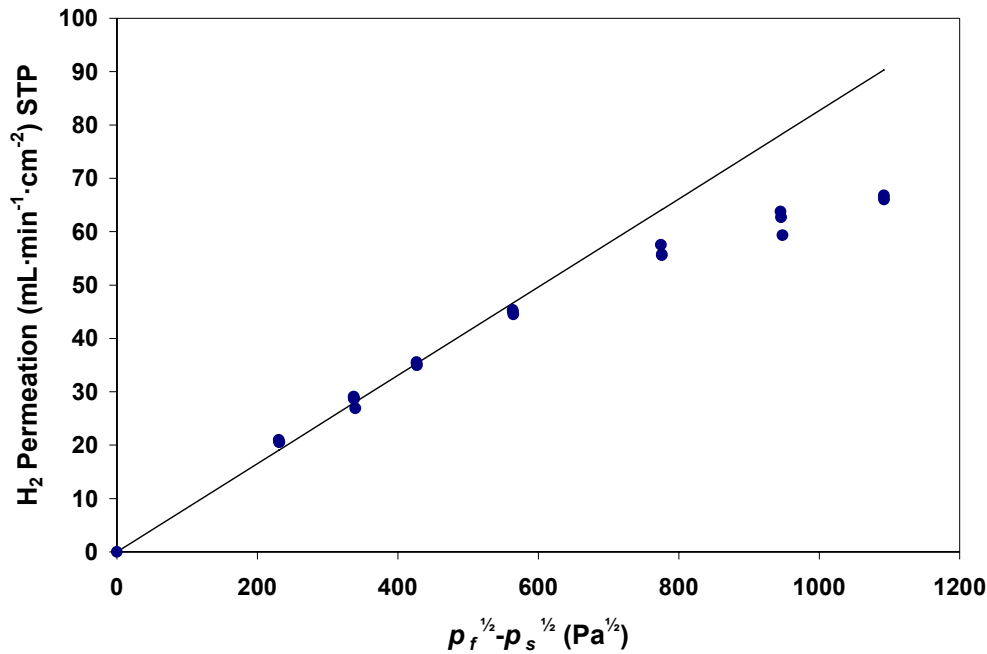


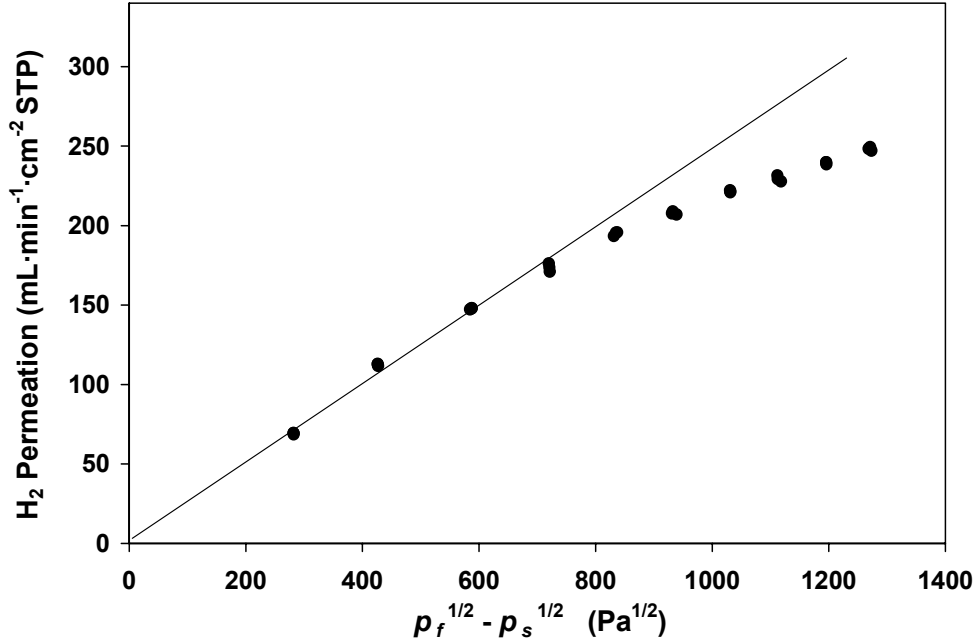
Figure 2. H<sub>2</sub> permeation versus temperature for a 0.9-mm thick membrane at a differential pressure of 250 psi. The feed gas was 500 mL/min of 40H<sub>2</sub>/10He (bal N<sub>2</sub>) and a the sweep gas was 450 mL/min N<sub>2</sub>.



**Figure 3.** H<sub>2</sub> permeability as a function of the H<sub>2</sub> partial pressure difference across a 0.6-mm thick membrane. The membrane temperature was 438/C. The feed gas was 80 vol.% H<sub>2</sub>/3 vol.% He (bal. Ar) at 1545 mL/min. The sweep gas was 1547 mL/min Ar. The line corresponds to the expected trend for Sieverts' Law.

Figure 4 shows permeation results for a different membrane analog using even higher flow rates. Permeation reached a maximum of almost 250 mL·min<sup>-1</sup>·cm<sup>-2</sup>, and the permeability was approximately  $2 \times 10^{-7}$  mol·m<sup>-2</sup>·s<sup>-1</sup>·Pa<sup>-0.5</sup>. Since the permeability was similar to the membrane of Figure 3, the dramatically higher permeation likely was due to the much lower membrane thickness coupled with a sweep rate of 4 L/min. Again, deviation from Sieverts' Law occurred above a H<sub>2</sub> partial pressure differential of 700 Pa<sup>1/2</sup>. Experiments are being performed to determine the cause of the deviation. However, the data suggest that the current membranes are capable of significantly higher permeation rates under appropriate operating conditions.





**Figure 4. Hydrogen permeation as a function of the differential hydrogen partial pressure across the membrane. The membrane was 0.12 mm thick and testing was performed at 440/C with a feed hydrogen concentration of 60 vol.%. The feed and sweep gas flow rates were 8 and 4 L/min, respectively. The total differential pressure across the membrane was at a maximum of 470 psi.**

## **II. Multi-Phase Ceramics and Cermets – CoorsTek**

Ceramic/ceramic composites consist of a proton conducting perovskite phase ( $AB_{0.8}B^c_{0.2}O_{3-x}$ ) and an electron conducting transition metal oxide. Previously it was shown that these materials perform equivalently to analogous cermets, yet have higher corrosion resistance in water. It also was shown that a second ceramic phase could be added to cermets to improve corrosion resistance without dramatically affecting hydrogen permeation. CoorsTek has begun constructing an apparatus to measure corrosion resistance to water vapor and other atmospheric conditions at high temperature.

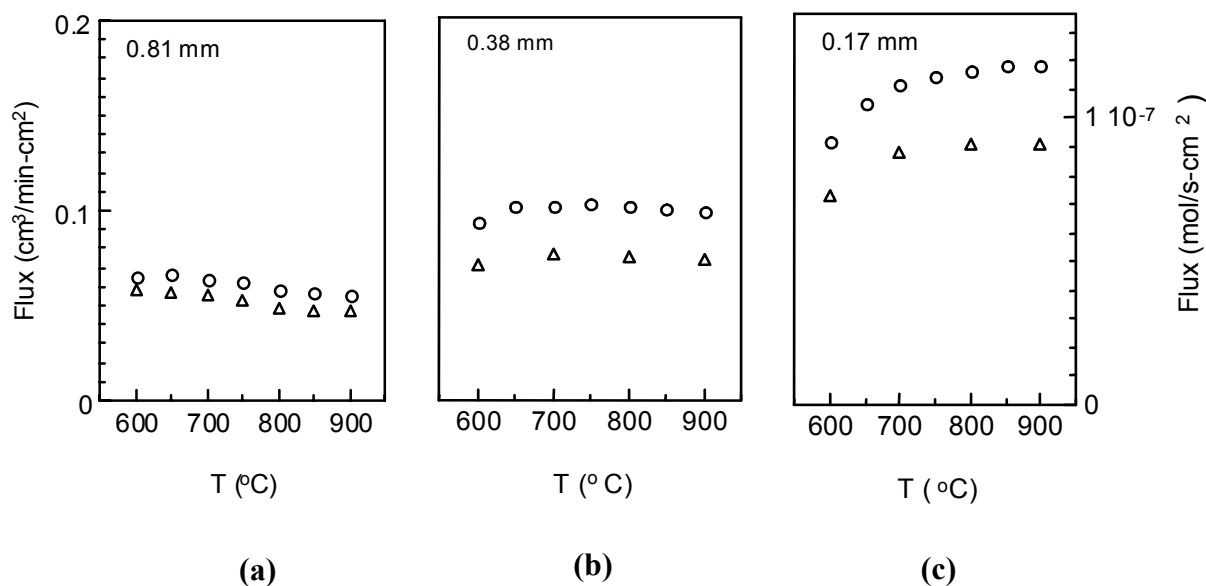
Attempts to optimize fabrication of these materials revealed unexpectedly high weight loss during firing. It was concluded that this extra weight loss was due to loss of oxygen from the perovskite structures and transfer of membrane material to the firing setter powder.

## **III. Dependence of Microstructure on Properties of H<sub>2</sub>-Permeable Cermets – ANL**

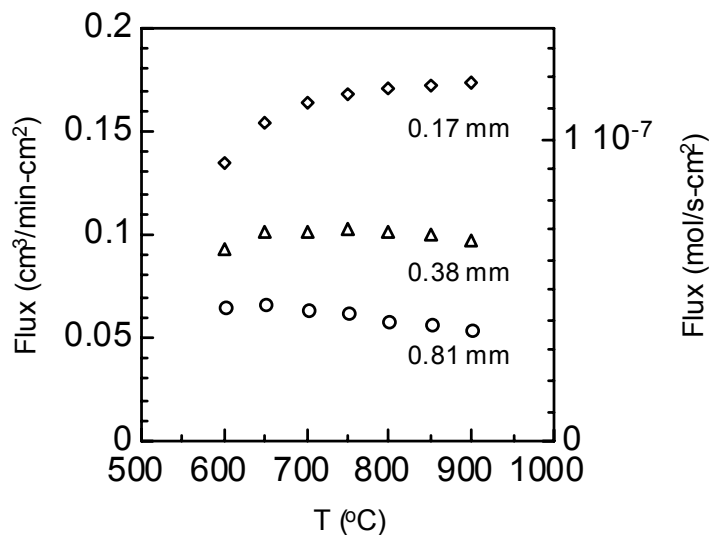
Previously it was reported that ANL-1a membranes containing metal phase with large grains (henceforth called large-grained membranes) gave a higher hydrogen flux than ANL-1a membranes containing metal phase with small grains (henceforth called small-grained membranes). In this quarter, these unexpected results were investigated.

Figure 5 shows the temperature dependence of the hydrogen flux of both large- and small-grained ANL-1a membranes. As expected, the flux increased for both types of membrane as the membrane thickness decreased. For membranes with thickness in the range 0.17-0.81 mm, the large-grained membranes gave higher flux than small-grained membranes. These results are in agreement with the previous report.

Hydrogen flux is re-plotted in Figure 6 as a function of temperature for large-grained membranes with thickness of 0.17, 0.38, and 0.81 mm. The hydrogen flux decreases as temperature increases for the thickest (0.81 mm) membrane. This result may be due to a decrease in proton conductivity with increasing temperature, which may result from a decrease in the proton charge-carrier concentration. The proton concentration may decrease because the water solubility for known perovskite-type proton conductors generally decreases with increasing temperature above about 500/C; below that temperature the water solubility is fairly constant. For the membrane with thickness of 0.38 mm, the hydrogen flux reached a maximum at approximately 650/C and then became flat. For the thinnest (0.17 mm) membrane, the hydrogen flux increased over the entire temperature range of study, although it was nearly flat above about 800/C.



**Figure 5.** Temperature dependence of the hydrogen flux for ANL-1a membranes containing metal with large-grains (circle) and small-grains (triangle). To compare the flux for small- and large-grained membranes, the flux for small-grained membranes was normalized to the same thickness as the large-grained membranes. The thickness for the large-grained membranes is given in each plot; the actual thickness for the small-grained membranes was (a) 1.16 mm, (b) 0.43 mm, and (c) 0.20 mm. Feed gas was 4%  $\text{H}_2/\text{He}$  (containing  $\sim 3\%$   $\text{H}_2\text{O}$ ).



**Figure 6. Temperature dependence of the hydrogen flux of ANL-1a membranes with large-grained metal phase. Feed gas was 4% H<sub>2</sub>/He (containing ~3% H<sub>2</sub>O).**

The results for the thinnest membrane may be related to hydrogen permeation through its metal phase. The metal phase in ANL-1a membranes is known to have a small, but measurable, hydrogen permeability at high temperatures; therefore, hydrogen permeation through the metal phase may become significant in thin membranes. For the thinnest membrane, hydrogen permeation through the oxide phase may decrease with increasing temperature, as was observed with the thick membrane, but the total hydrogen flux may still increase due to an increase in hydrogen permeation through the metal phase.

#### IV. Catalysts for H<sub>2</sub> Separation Membranes – SCI

SCI outlined new experimental procedures for testing catalysts materials with potential for hydrogen separation membranes. The objectives are to identify potentially stable catalysts with good activity for hydrogen dissociation, and determine the effect of surface roughness on catalyst activity. SCI will process catalysts onto membrane supports and analyze them using scanning electron microscopy (SEM), atomic force microscopy (AFM) and Wyko white-light interferometry. The goal will be to evaluate the size distribution, density and surface topography of the membrane.

Catalyst activity will be measured using a low-pressure environment (sub atmospheric pressures) in a closed chamber and monitor the partial pressures (and total pressure) changes over time using RGA and pressure gauges. In this method, SCI will use deuterium and hydrogen mixtures to monitor the D-H species. This approach appears to be a novel test procedure and should yield very relevant results.

### **Task 3**     *High Pressure Hydrogen Separation*

**Contributors:** Eltron

During this quarter, all testing was performed using flat samples and a compression flange assembly with annealed copper seal rings. This assembly only is good for somewhat malleable planar membranes; however, seals routinely are achieved at differential pressures between 250 and 450 psi (zero leak rate). Provided the membrane mechanical characteristics are adequate, this type of seal is very scalable.

### **Task 4**     *Thin-Film Hydrogen Separation Membranes*

**Contributors:** CoorsTek, Eltron

It was concluded that problems with tape casting thin films onto planar supports were the associated with uncontrolled variables during precursor preparation rather than the binder burnout and sintering cycles.

Based on results to date, it was decided to attempt coating thin membranes on thin-walled (0.5 mm) isopressed tubes. Green tubes were provided by Eltron and CoorsTek initiated a bisque firing study to maximize strength and minimizing shrinkage. Samples were bisque fired at 900/C, 1000/C, 1100/C and 1200/C. The only samples having reasonable strength were those fired at 1200/C. These samples exhibited uniform diametrical and longitudinal shrinkages of 19%. Membrane coating tests will be conducted on the 1200/C bisque fired parts as well as on green tubes and fired tubes. Depending on the test results, CoorsTek may need to reformulate the isopressing formulation with a lower amount of pore former. Presently, the emphasis is focused on combining the very slow air burnout cycle (Lindberg Furnace) with a Camco furnace bisquing cycle in Forming Gas. Although not conclusive at this point, early indications are that the pre-binder burnout cycle is a necessity to achieve a reasonable quality with handling strength bisque test. Trials are forthcoming to evaluate a green tube segment and selected bisque tubes with initial test slurries.

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

**Contributors:** Eltron

Engineering improvements were performed on the high-pressure reactors to maximize contact efficiency of the feed gas with the membrane surface, and to increase the sweep rates. These modifications definitely improved permeation rates, and will be incorporated into the demonstration-scale prototype design.

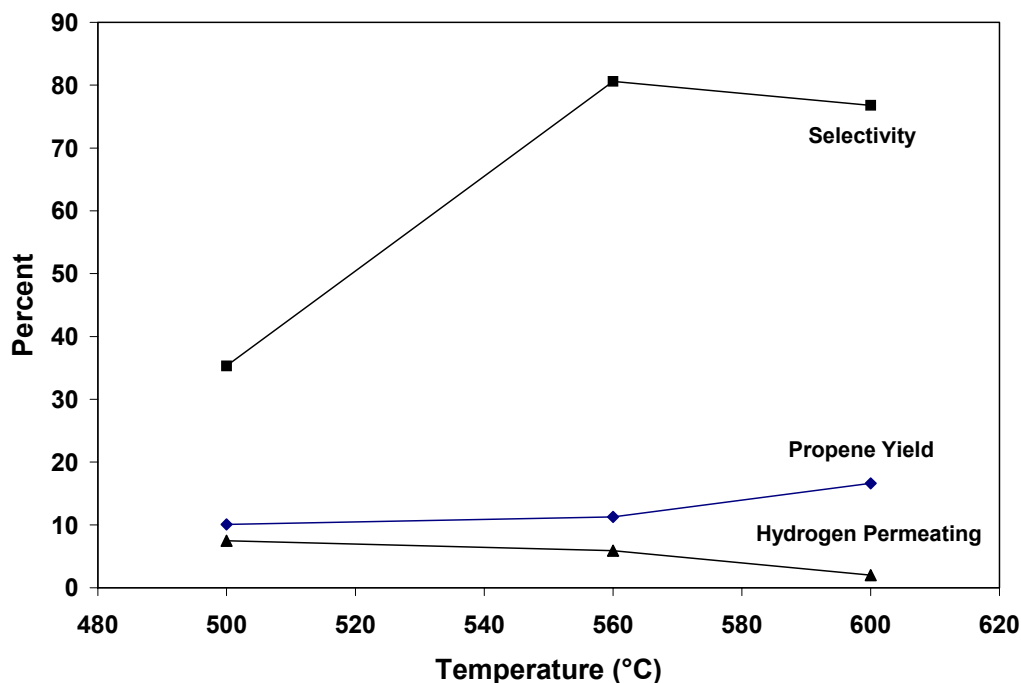
## Task 6 Membrane-Promoted Conversion of Alkanes to Olefins

**Contributors:** Eltron

Alkane dehydrogenation experiments were performed using a composite metal alloy membrane. The separation cell was packed with a Pt/Sn catalyst supported on mullite grains. Tests were performed at temperatures between 500 and 600°C using a feed gas comprising 60 mL/min of humidified 10 vol.% propane (bal. Ar) and 10 mL/min He for leak detection. The sweep gas was 120 mL/min Ar.

Results are presented in Figure 7 for propane to propene conversion, selectivity for propene formation, and percentage of hydrogen permeating the membrane at 500, 560, and 600°C. Propane yield increased from approximately 10 to 17% with increasing temperature, but still fell far short of the theoretical equilibrium value (~ 30%). Moreover, less than 1 mL/min of H<sub>2</sub> was generated during the dehydrogenation, and only a few percent were transported across the membrane surface. With these low values it is difficult to resolve any effect of the membrane on the target reaction. Selectivity showed a slight maximum at 560°C, but H<sub>2</sub> permeation decreased from 7.5% to only 2% over the test range. The decrease in permeation despite the increase in conversion likely is due to carbon formation on the membrane surface.

Future effort will focus on optimization of test reactor engineering to improve conversion and permeation. This goal will require increasing catalyst content and feed gas retention time.



**Figure 7.** Plot showing percent selectivity for propane formation (■), propene yield (◆), and percent hydrogen transported across the membrane (▲).

## SUMMARY AND CONCLUSIONS

Conclusions based on the work performed during the last 12 months of this project are summarized as follows:

### *Cermets*

- Preliminary attempts to incorporate inexpensive H<sub>2</sub>-permeable metals into a cermet were successful, and a thick cermet membrane (0.83 mm) with 40 vol.% metal phase achieved a permeation rate of nearly 0.4 mL/min/cm<sup>2</sup>. Increasing the metal phase content and decreasing membrane thickness should significantly increase permeation for this category of cermets. However, these materials exhibited poor stability in the absence of a protective surface coating.
- Cermets incorporating 60 vol.% of inexpensive H<sub>2</sub>-permeable metals showed a maximum of 0.65 mLAnin<sup>-1</sup>Am<sup>-2</sup> (STP) at 800/C.
- Improvements were made in the fabrication process for cermets and dense, strong membranes with only the desired phases were prepared. A relatively thick membrane sample achieved a permeation rate of 3.6 mLAnin<sup>-1</sup>Am<sup>-2</sup> at only 320/C, corresponding to a very high permeability of 1.9 x 10<sup>-7</sup> molAnAn<sup>-2</sup>A<sup>-1</sup>Pa<sup>-0.5</sup>.
- Temperature-programmed reduction measurements indicated that model cermet materials absorbed 2.5 times as much H<sub>2</sub> than the pure ceramic analogs. This characteristic, in addition to higher electron conductivity, likely explains the relatively high permeation for these cermets. Incorporation of catalysts with these ceramics and cermets increased H<sub>2</sub> uptake by 800 to more than 900%.
- New high-pressure seals were developed for cermet membranes that maintained a pressure differential of 250 psi. This result indicated that the approach for high-pressure seal development could be adapted for a range of compositions.
- For cermets based on proton-conducting ceramics, it was determined that when the metal grain size was small, a higher metal content was necessary to achieve continuity. Additionally, despite an assumed decrease in surface resistance for H<sub>2</sub> transport with small-metal-grain samples, H<sub>2</sub> transport was higher for samples with larger metal grain size.
- New mixed proton/electron conducting cermets achieved a maximum permeation of approximately 0.17 mLAnin<sup>-1</sup>Am<sup>-2</sup>, corresponding to an apparent ambipolar conductivity of 0.003 S/cm. These results were slightly lower than previous analogs.
- Cermets prepared with smaller grain precursor powders sintered better and were more dense than the large grain analogs.

- Preliminary mechanical testing indicated much higher material strength and improved Weibull modulus for cermets relative to ceramics.

### ***Ceramic/Ceramic Composites***

- Two-phase ceramic/ceramic composite membranes had low H<sub>2</sub> permeability, likely due to interdiffusion of constituents between the phases. However, these materials did demonstrate high resistance to corrosion from water, and might be good candidates for other composite membranes.
- A range of titanate ceramics demonstrated either poor stability in H<sub>2</sub> or negligible proton conductivity.
- Addition of a stabilizing ceramic phase appears to be a viable approach for improving the performance of H<sub>2</sub> permeable materials based on proton-conducting ceramics.
- A sintering configuration for tube membranes was identified that enabled fabrication of very round samples with a wall thickness of only 0.5 mm.

### ***Thin Film Membranes***

- Supported thin film membranes were successfully fabricated. Only samples with three thin film layers were dense enough to permit testing, and these samples demonstrated permeation rates approximately three times lower than analogous membranes produced by conventional methods.
- For thin cermet membranes based on proton-conducting ceramics, H<sub>2</sub> permeation through the metal phase might be significant.
- Uptake of moisture from air was identified as a potential cause of defects in thin film membranes.
- Thin film membrane fabrication was shifted to a thin-walled tubular configuration.
- Layered composite membranes were not affected by high levels of CO<sub>2</sub>; however, 3 vol.% CO reduced permeation to 75% of its original value. Similarly, addition of 5 to 10 vol.% steam dropped permeability to roughly 50% of its original value.
- Layered metal membranes demonstrated H<sub>2</sub> permeation during alkane dehydrogenation equivalent to cermets, but at a temperature 100°C lower.
- High pressure seals easily were attained using a compression flange with annealed copper seal rings. Although this assembly only is good for planar membranes, a stacked configuration will allow high total membrane surface area.

### ***Layered Composite Membranes***

- A layered composite ceramic and metal membrane maintained H<sub>2</sub> permeation for over eight months of continuous operation.
- Layered membranes demonstrated good resistance to H<sub>2</sub> embrittlement, tolerance to pressure differentials as high as 470 psi, and H<sub>2</sub> permeation over 240 mL<sub>A</sub>min<sup>-1</sup>A<sub>m</sub><sup>-2</sup> under optimized operating conditions.
- Perfect seals were maintained at 450 psi differential pressure using a flange system with planar malleable samples.
- Improvements in permeation rates were attained by implementing engineering modifications to the high-pressure reactors.

### ***Alkane Dehydrogenation***

- Separation of pure hydrogen generated from the dehydrogenation of propane to propane was accomplished. Due to the relatively low propane conversion rate, it was not possible to determine if the hydrogen partitioning promoted the reaction.

## **PUBLICATIONS AND PRESENTATIONS**

S.E. Roark, R. Mackay, M.V. Mundschau, "Dense Layered Membranes for Hydrogen Separation," Patent Application No. 10/717,218, Submitted September 10, 2003

S.E. Roark, R. Mackay, A.F. Sammells, "Hydrogen Separation Membranes for Vision 21 Energy Plants," Proceedings of the 28th International Conference on Coal Utilization and Fuel Systems, Clearwater FL, March 10-14, 2003

Anthony F. Sammells *et al.*, "Oxygen and Hydrogen Transport Membrane Reactor Applications," 21<sup>st</sup> Annual Membrane/Separations Technology Planning Conference, Newton MA, December 3, 2003

## **OBJECTIVES FOR THE FOLLOWING YEAR**

Eltron will continue screening a variety of layered metal alloy membranes due to the very high flux, intermediate operating temperature, long-term permeation stability, high-pressure tolerance, apparent chemical resistance for selected species, and economics. CoorsTek will focus on development of thin film membranes, and ANL will continue testing the relationship between microstructure and H<sub>2</sub> permeation of cermets. SCI will proceed with catalyst testing outlined during this quarter. NORAM will focus on engineering of demonstration-scale and full-scale hydrogen separation units.

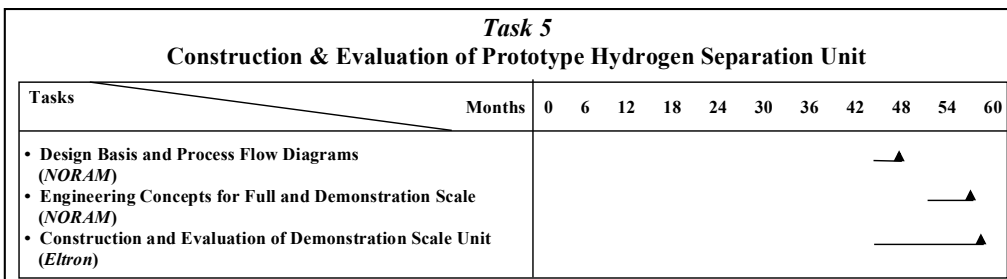
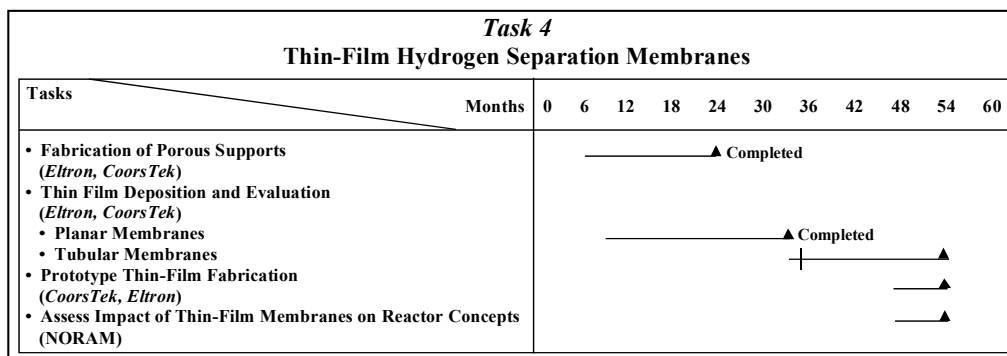
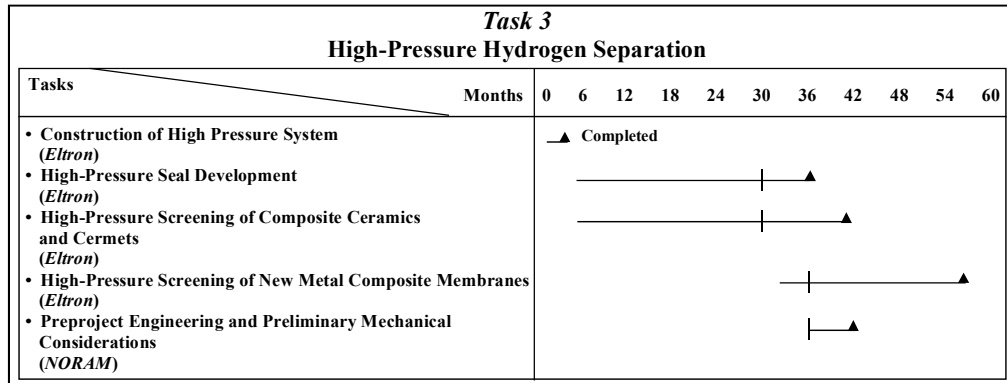
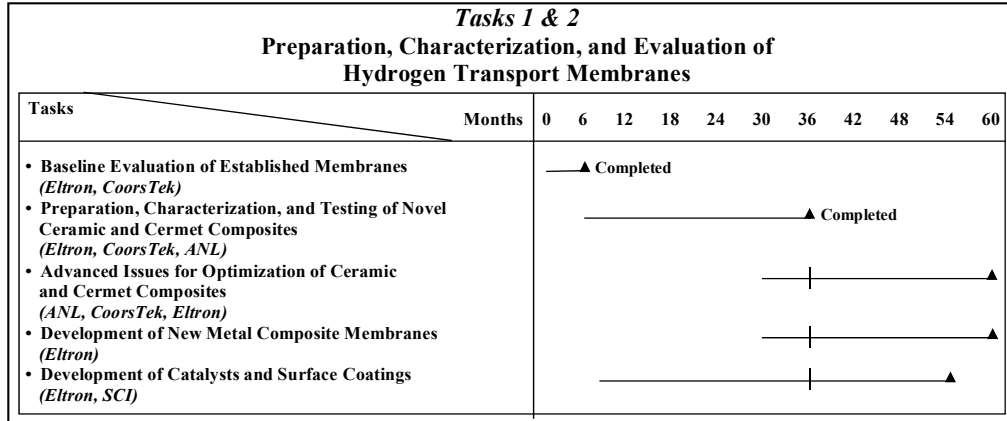


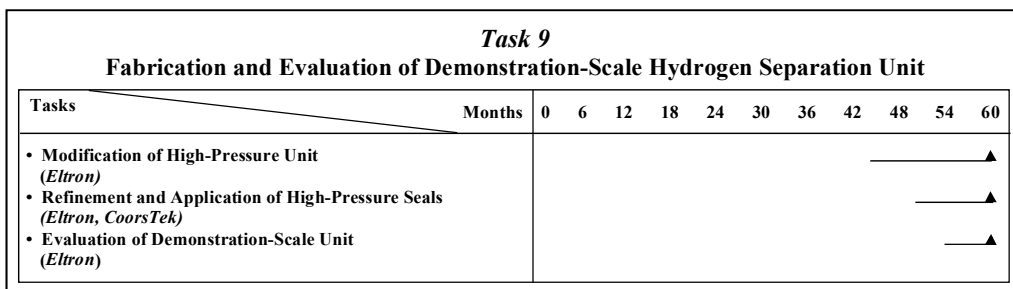
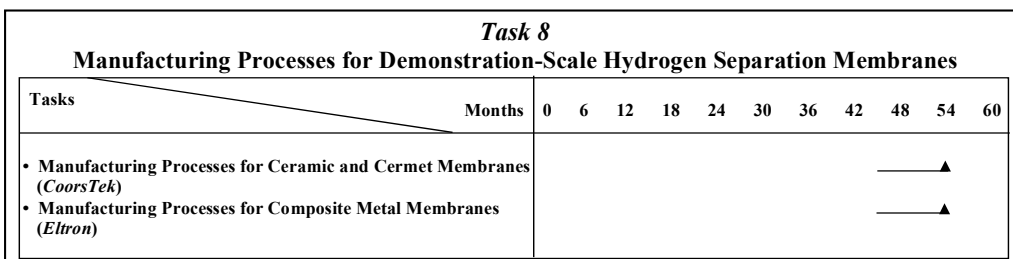
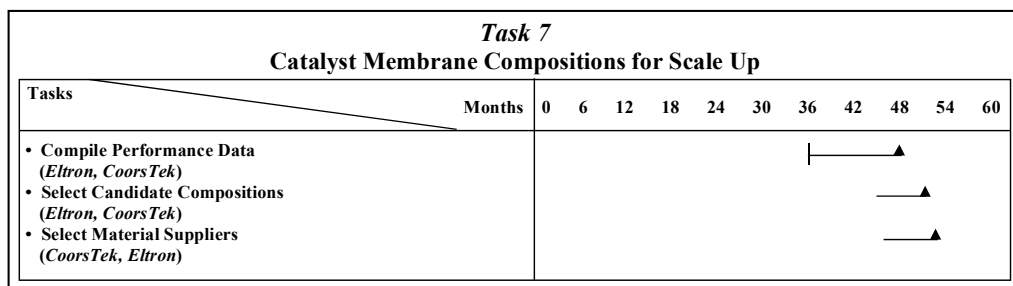
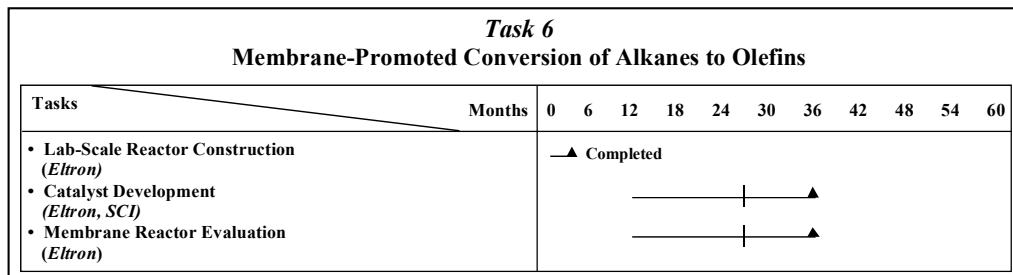
## OPEN ITEMS OR COOPERATIVE AGREEMENT CHANGES

NORAM has joined the consortium for this Vision 21 project. NORAM's role will be to facilitate engineering scale up of the H<sub>2</sub> separation technology. The time line below was changed to reflect the addition of NORAM and an extension of the contract until September 30, 2005.

## TIME LINES

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





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## A. AWARDEE ACTION (AWARDEE COMPLETES PART A. 1-5)

1. Document Title: Advanced Hydrogen Transport Membranes for Vision 21 Fossil Fuel Plants
  2. Type of Document:     : Technical Progress Report     9 Topical Report     9 Final Technical Report  
9 Abstract     9 Technical Paper     9 Journal Article     9 Conference Presentation  
9 Other (please specify) \_\_\_\_\_
  3. Date Clearance Needed: \_\_\_\_\_
  - 4. Results of Review for Possible Inventive Subject Matter:
    - a. : No Subject Invention is believed to be disclosed therein.
    - b. 9 Describes a possible Subject Invention relating to \_\_\_\_\_
      - i. Awardee Docket No.: \_\_\_\_\_
      - ii. A disclosure of the invention was submitted on \_\_\_\_\_
      - iii. A disclosure of the invention will be submitted by the following date: \_\_\_\_\_
      - iv. A waiver of DOE's patent rights to the awardee: 9 has been granted, 9 has been applied for, or 9 will be applied for by the following date: \_\_\_\_\_
  - 5. Signed \_\_\_\_\_ Date October 30, 2003  
(Awardee)
- Name & Phone No. Eltron Research Inc. 303-530-0263
- Address 4600 Nautilus Court South, Boulder, CO 80301-3241

## B. DOE PATENT COUNSEL ACTION

9 Patent clearance for release of the above-identified document is granted.

9 Other: \_\_\_\_\_

Signed \_\_\_\_\_ Date \_\_\_\_\_  
(Patent Attorney)

— **Must be** completed by the awardee.