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## ABSTRACT

Eltron Research Inc., and team members CoorsTek, McDermott Technology, Inc., Süd Chemie, Argonne National Laboratory, and Oak Ridge National Laboratory 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. This objective is being pursued using dense membranes based in part on Eltron-patented ceramic materials with a demonstrated ability for proton and electron conduction. The technical goals are being addressed by modifying single-phase and composite membrane composition and microstructure to maximize proton and electron conductivity without loss of material stability. 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.

During this quarter, ceramic, cermet (ceramic/metal), and thin film membranes were prepared, characterized, and evaluated for H<sub>2</sub> transport. For selected ceramic membrane compositions, an optimum range for transition metal doping was identified, and it was determined that highest proton conductivity occurred for two-phase ceramic materials. Furthermore, a relationship between transition metal dopant atomic number and conductivity was observed. Ambipolar conductivities of  $\sim 6 \times 10^{-3}$  S/cm were achieved for these materials, and  $\sim 1$ -mm thick membranes generated H<sub>2</sub> transport rates as high as 0.3 mL/min/cm<sup>2</sup>. Cermet membranes during this quarter were found to have a maximum conductivity of  $3 \times 10^{-3}$  S/cm, which occurred at a metal phase contact of 36 vol.%. Homogeneous dense thin films were successfully prepared by tape casting and spin coating; however, there remains an unacceptably high difference in shrinkage rates between the film and support, which led to membrane instability. Further improvements in high pressure membrane seals also were achieved during this quarter, and a maximum pressure of 100 psig was attained. CoorsTek optimized many of the processing variables relevant to manufacturing scale production of ceramic H<sub>2</sub> transport membranes, and SCI used their expertise to deposit a range of catalysts compositions onto ceramic membrane surfaces. Finally, MTI compiled relevant information regarding Vision 21 fossil fuel plant operation parameters, which will be used as a starting point for assessing the economics of incorporating a H<sub>2</sub> separation unit.

## 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. This objective will be accomplished by employing dense ceramic and composite membranes based in part on Eltron-patented materials (Patent No. US5821185) with a demonstrated ability for rapid proton and electron conduction. The primary technical challenge in achieving the goals of this project will be to optimize membrane composition and microstructure 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, McDermott Technology, Inc. (MTI), Süd Chemie, Inc. (SCI), Argonne National Laboratory (ANL), and Oak Ridge National Laboratory (ORNL).

During this quarter, ceramic, cermet (ceramic/metal), and thin film membranes were prepared, characterized, and evaluated for H<sub>2</sub> transport. Ceramic membranes were tested as a function of transition metal dopant quantity and identity, and cermet membranes were tested as a function of the volume percent of the metal phase. The focus for thin film development was on tape casting and spin coating processing conditions, however, a preliminary sample was evaluated for H<sub>2</sub> transport as well. Further improvements in high pressure membrane seals also were achieved during this quarter. CoorsTek optimized many of the processing variables relevant to manufacturing scale production of ceramic H<sub>2</sub> transport membranes, and SCI used their expertise to deposit a range of catalysts compositions onto ceramic membrane surfaces. Finally, MTI compiled relevant information regarding Vision 21 fossil fuel plant operation parameters, which will be used as a starting point for assessing the economics of incorporating a H<sub>2</sub> separation unit.

## 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 conductivity apparatus, (e) construction and operation of ambient-pressure hydrogen separation units, (f) construction and operation of high-pressure hydrogen separation units, (g) hydrogen transport and ambipolar conductivity measurements and calculations, and (h) 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.

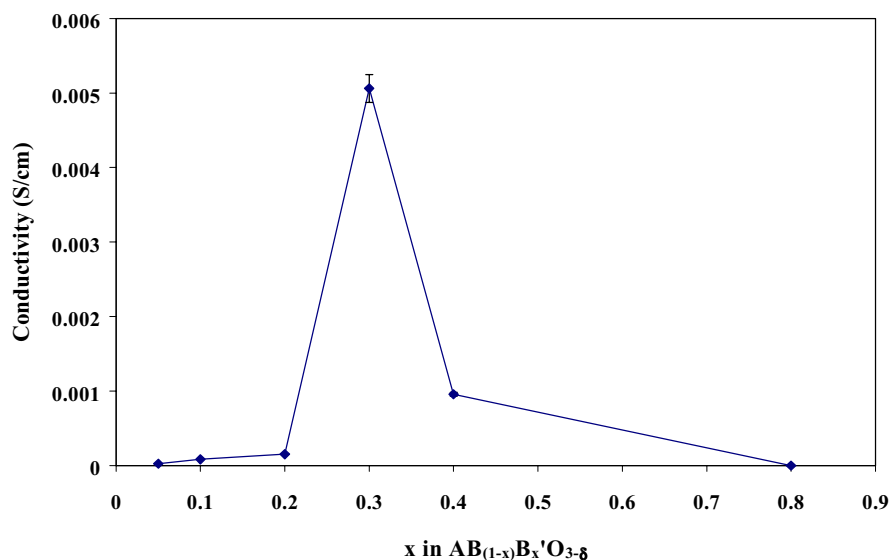
## RESULTS AND DISCUSSION

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

**Contributors:** Eltron, CoorsTek, MTI, SCI, ORNL

#### a. Model Perovskite Hydrogen Transport Membranes Doped with Transition Metals - Eltron

In the previous report, data was presented for a series of relatively thick tube membranes (~ 0.9 mm) possessing the general composition  $AB_{1-x}B'_xO_{3-\delta}$ , where  $B'$  represents a transition metal, and  $0.05 \leq x \leq 0.3$ . It was found that  $H_2$  transport increased significantly with dopant content. Accordingly, during this period the study was extended to compositions with  $x = 0.4$  and  $0.8$ . The results are presented in Figure 1 as conductivity versus the fraction of B-site transition metal doping. Conductivity is shown rather than  $H_2$  transport to normalize for differences in membrane thickness. As evident in the figure, conductivity was not increased at higher doping levels, and remained at a sharp maximum for  $x = 0.3$ . X-ray diffraction patterns indicated that at  $x = 0.05$ , the material was primarily a single-phase pseudo-cubic perovskite with a lattice parameter of  $4.393(1) \text{ \AA}$ , which was

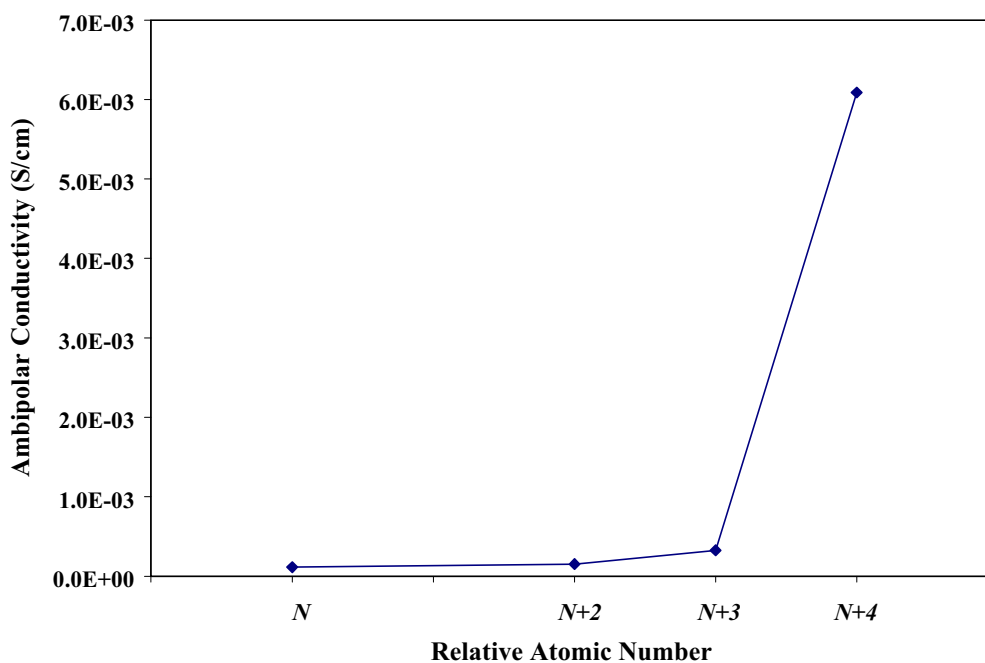


**Figure 1.** Plot showing ambipolar conductivity as a function of transition metal doping ( $B'$ ) for ceramic tube membranes with the general composition  $AB_{(1-x)}B'_xO_{3-\delta}$ . The analysis temperature was  $850^\circ\text{C}$ , and Ni was used as a catalyst. The sweep gas (Ar) flow rate for  $x = 0.05$  through  $0.3$  was  $\sim 65 \text{ mL/min}$ , and no humidity was added. However, for  $x = 0.4$  and  $0.8$ , the samples were tested under humid conditions with a sweep rate of  $120 \text{ mL/min}$ . The error bars represent  $\pm 1\sigma$ .

approximately equal to the undoped  $ABO_3$  analog. At  $x = 0.1$ , a second unidentified phase was evident, which became more prominent at  $x = 0.2$ . However, at  $x = 0.3$  the unidentified second phase was completely absent and a new phase appeared that was consistent with a rhombohedral perovskite with lattice parameters  $a = 5.797 \text{ \AA}$  and  $c = 28.595 \text{ \AA}$ . This new phase was even more apparent at  $x = 0.4$  along with the cubic perovskite phase; however, at  $x = 0.8$  the cubic perovskite phase was completely absent.

Tube membranes prepared from this material (*i.e.*,  $AB_{0.2}B'_{0.8}O_{3-\delta}$ ) had a tendency to crack after only about 1 day under  $H_2$  separation conditions. Coefficient of thermal expansion measurements were performed on a section of the material under air, and a normal expansion curve was observed giving a CTE value of  $\sim 6.5 \times 10^{-6} \text{ K}^{-1}$ . Under 10%  $H_2$  (bal. Ar), a gradual steady expansion was observed between room temperature and  $600^\circ\text{C}$ ; however, above  $600^\circ\text{C}$  a rapid expansion was observed followed by material failure. Post-mortem X-ray diffraction patterns indicated that the transition metal had been reduced from a 4+ to a 2+ oxidation state. Such material failures were not observed under  $H_2$  separation conditions for the other compositions, however, this data indicates that high levels of this particular transition metal dopant dramatically compromises stability.

Additional ceramic membrane samples with the general composition  $AB_{0.8}B'_{0.2}O_{3-\delta}$  were tested with a range of transition metal dopants ( $B'$ ). Figure 2 shows the ambipolar conductivity of



**Figure 2.** Plot showing ambipolar conductivity as a function of relative atomic number for four transition metal dopants ( $B'$ ) in the general composition  $AB_{0.8}B'_{0.2}O_{3-\delta}$ . The inlet gas was humidified 80%  $H_2$  (bal. He), and the sweep gas was Ar. Flow rates were 120 mL/min, and the temperature was  $850^\circ\text{C}$ . Data was taken for tube membranes, except for ( $N+4$ ) which was a disk. Ni was used as a catalyst.

these samples as function of relative atomic number of the transition metal additive. There was a gradual increase in conductivity from  $1.1 \times 10^{-4}$  to  $3.3 \times 10^{-4}$  S/cm as the transition metal dopant moved three positions from left to right across the periodic table (*i.e.*,  $N$  to  $N+3$ ). However, at  $N+4$  the conductivity increased almost 20-fold to  $6 \times 10^{-3}$  S/cm. A 1-mm thick membrane of this composition gave a  $H_2$  separation rate of  $\sim 0.3$  mL/min/cm<sup>2</sup>. X-ray diffraction measurements indicated that each sample contained an orthorhombic perovskite phase in addition to a second phase that was dependent on the transition metal dopant. The perovskite phase for each sample had the same lattice parameters, and the data indicated that very little transition metal was incorporated into the perovskite lattice.

#### **b. Ceramic-Metal (Cermet) Composite Membranes - Eltron**

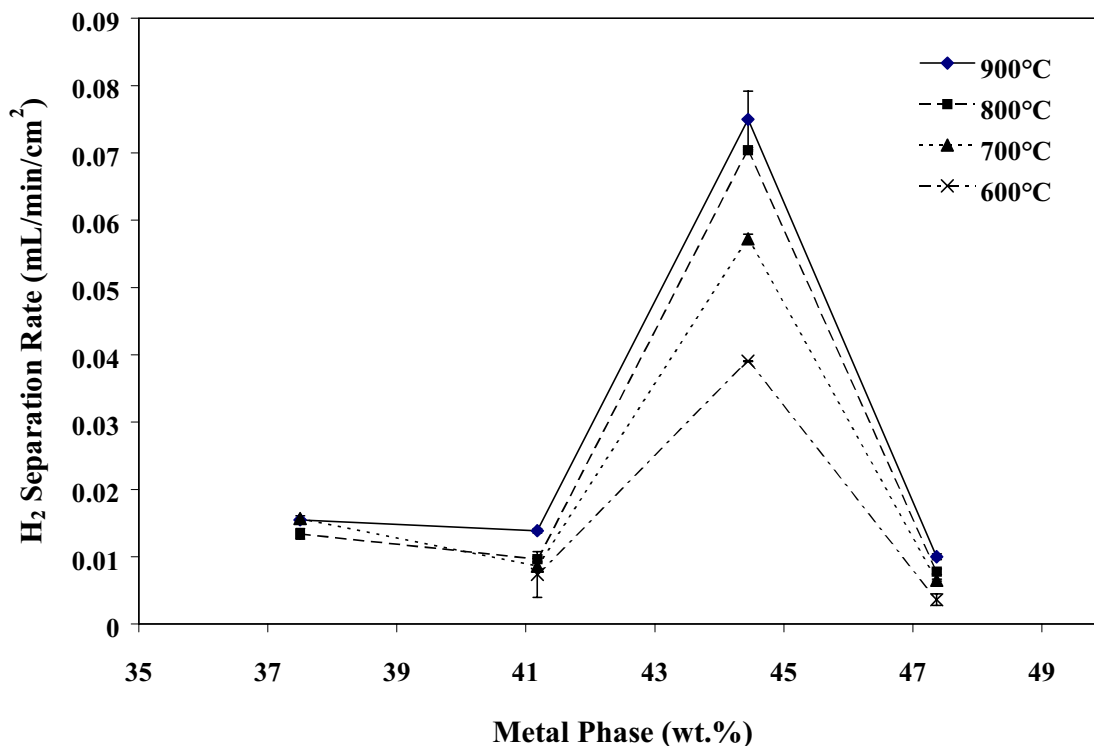
In the previous report, preliminary results were presented for cermet membranes using a mixed proton/electron conducting ceramic to demonstrate the increase in  $H_2$  separation upon addition of a metal. During this quarter, the same metal phase was used, however, the ceramic phase was replaced with a predominantly proton conducting perovskite. Samples were prepared with varying ceramic/metal ratios from powders with particle sizes between 0.4 and 1.5  $\mu\text{m}$ . The powders were mixed by ball milling for 1 hr, then pressed into pellets at 15 kpsi for 2 min. The pellets were sintered at 1425°C for 4 hrs under 10%  $H_2$  (bal. Ar), then sanded to the desired thickness and polished.

Membranes were prepared with 38, 41, 44, and 47 wt.% metal phase, which corresponded to 29, 33, 36, and 38 vol.%. Figure 3 shows an SEM image of the 44-wt.% metal sample, which demonstrated continuity between the phases. Despite differing quantities of metal phase in each sample, no obvious differences were observed in the SEM images. Continuity of the metal phase



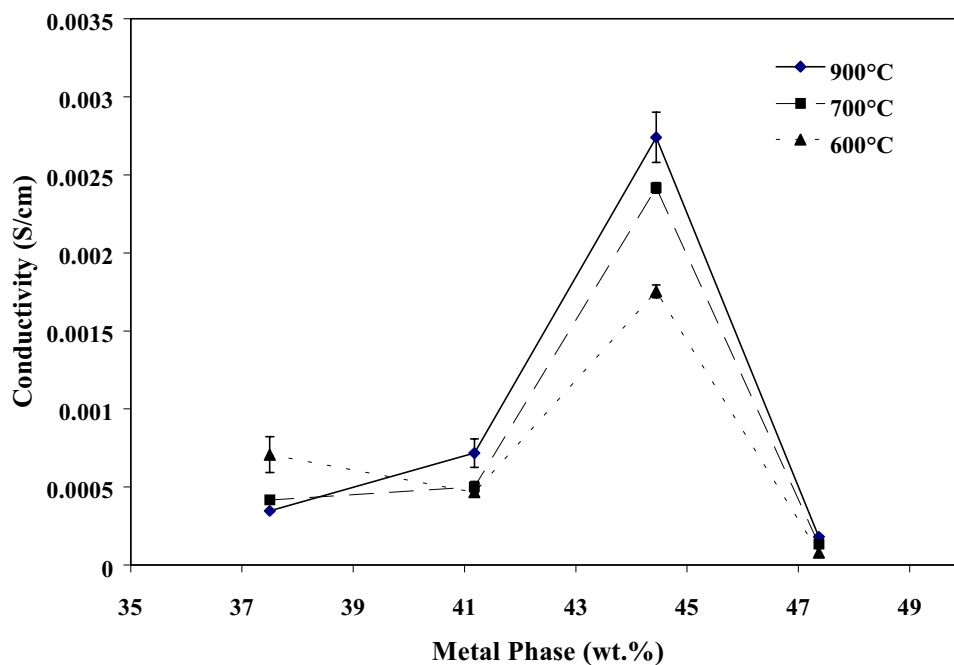
**Figure 3. SEM image of a 54/46 wt.% ceramic/metal membrane. The dark regions are the metal phase and the light regions are the proton conducting ceramic.**

was confirmed for each sample by measuring electrical resistance across the membrane using a voltmeter. Figures 4 and 5 show the H<sub>2</sub> transport rates and conductivity of these samples as a function of the metal phase content (under humid conditions). Since a range of membrane thicknesses between 0.75 and 2 mm were used for these tests, the conductivity data in Figure 5 provides a better comparison of the materials. However, both plots show a maximum for the 44-wt.% (36-vol.%) sample. A 1.2-mm thick membrane of this composition generated a H<sub>2</sub> transport rate of  $0.075 \pm 0.004$  mL/min/cm<sup>2</sup>, and a conductivity of 0.003 S/cm at 900°C. Conductivity and transport did not change much between 800 and 950°C for this sample, which indicated a plateau in the temperature dependence. These results represent approximately a four-fold improvement over the data in the previous report, and are essentially equivalent to those obtained by Balachandran *et al.* for similar compositions (Balachandran *et al.*; 26<sup>th</sup> Int. Tech. Conf. Coal Utilization and Fuel Sys., Clearwater, FL, March 5-8, 2001, pp's 751-761).



**Figure 4.** Plot showing H<sub>2</sub> separation rate as a function of metal phase wt.% at several temperatures for disk cermet membrane. The feed gas was ~ 80% H<sub>2</sub> (bal. He), and was bubbled through water prior to entering the cell. The sweep gas was Ar. Flow rates were maintained at ~100 mL/min. Membrane thicknesses varied between 0.75 and 2 mm, thus conductivities provide a more useful comparison between samples. No catalyst was used.





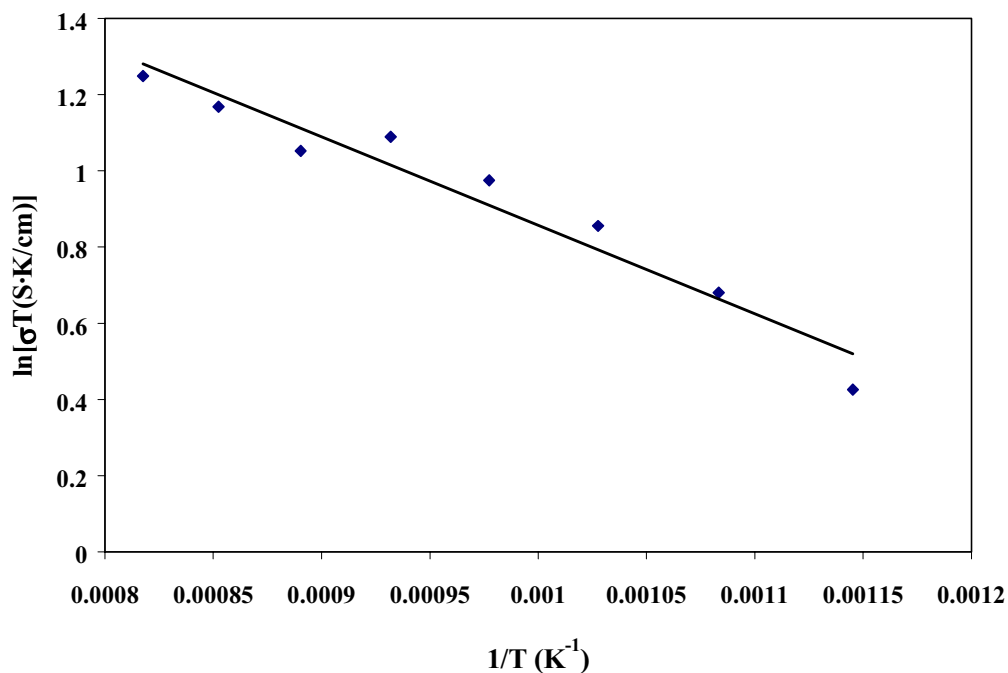
**Figure 5. Plot showing conductivity as a function of metal phase wt.% at several temperatures. Testing conditions were presented in the caption for Figure 4.**

Figure 6 shows the temperature dependence of the conductivity for the 44-wt.% metal phase cermet over the range of 600° to 950°C. There was a break in the data at ~800°C, which also was observed by Balachandran *et al.*, although at a lower temperature (700°C). The break in data was attributed to crystallographic changes at high temperatures that affect proton migration and/or proton concentration in the ceramic. Based on these results, the activation energy was calculated to be only 0.2 eV. Furthermore, although the other samples demonstrated much lower conductivity, activation energies also were between 0.2 and 0.3 eV. However, the pre-exponential factor was nearly ten-fold higher for the 44-wt.% metal phase sample.

### c. Effect of Catalyst on Hydrogen Transport Rates - Eltron, SCI

The membrane materials tested so far have been thick enough (~0.5 to 1 mm) that hydrogen flux likely is limited by transport through the membrane, rather than by surface kinetics. Furthermore, the highest conductivities typically observed at this stage are  $\sim 10^{-4}$  to  $10^{-2}$  S/cm, and it is expected that proton transport will limit H<sub>2</sub> flux until membrane thicknesses are reduced below about 0.1 mm. Accordingly, the time lines proposed for catalyst studies in this project have been extended as shown in the Time Lines section of this report. However, efforts have been continued toward testing various catalyst systems and deposition procedures.

This quarter, SCI concentrated on several techniques for applying catalytic metals to small disks of Eltron's proprietary proton conducting membrane materials. Ten disks of one composition



**Figure 6.** Plot showing the temperature dependence of conductivity for the 44-wt.% metal phase cermet sample. Testing conditions were presented in Figure 4.

were sent to SCI for coating. Five disks were polished and five were not. The catalytic metals of interest were Pt, Pd, and Ni. It was reported to SCI that the membrane material was unstable in water at room temperature, thus, it was necessary to employ organic solutions of these metals as precursors. The target loadings were 1% Pt and Pd and 3% Ni.

The precursor compounds chosen were Platinum (II) Acetyl acetonate (Ac-Ac), Palladium (II) Acetylacetonate (Ac-Ac), and Nickel (II) ethyl hexanoate. The Pt and Pd compounds were dissolved in tetra-hydrofuran and the Ni compound was dissolved in n-hexane. The original technique to be employed was to apply (via a pipette) enough solution to completely coat the disks, dry at 60°C, and then calcine at 500°C. This technique was acceptable for the disks to be coated with Ni since the concentration of the solution was quite high (~5% Ni) and therefore high loading was possible with only 3 applications. The solubility of Pt and Pd Ac-Ac was very low causing a low Pt and Pd concentration in solution (<1% Pt or Pd) and made impregnation to high loading impractical.

A solution to this obstacle was found by slurring the Pt and Pd Ac-Ac in acetone and then applying (via pipette) directly to the disks one side at a time. A thin, uniform, and controllable coating was possible after drying and calcination at 750°C. There seemed to be no difference in coating the polished from the unpolished disks. However, the coatings were very thin and must be treated gently. The disks did not break upon calcining. Another attempt was made to impregnate the disks with a Pd on activated carbon catalyst. This was unsuccessful, and the catalyst would not stick to the membrane disk.

**d. Neutron Diffraction Studies - Eltron, ORNL**

During this quarter, a series of experiments were outlined directed at elucidating aspects of the proton conduction mechanism in model perovskite ceramics. During the next quarter, the experiments will be refined, and arrangements will be made to conduct testing at the Oak Ridge National Laboratory facility.

**e. Manufacturing Issues and Relative Economics - CoorsTek, Eltron**

Part of Task 1 of this project involves outlining manufacturing issues and estimating the relative economics associated with large-scale production of the proposed ceramics and cermets. These goals are being pursued by CoorsTek, and involve i) reproducing Eltron's model materials and confirming equal performance, ii) reviewing membrane fabrication processes, iii) evaluating alternative processing methods, iv) assessing environmental aspects of processing methods, and v) investigating commercial sources and grades of starting materials to reduce production costs.

Variations of a model perovskite compound have been fabricated by CoorsTek and Eltron to test reproducibility and optimize processing variables. It is anticipated that most of the manufacturing issues associated with these preliminary samples will be somewhat general, and transferable to similar compositions, as well as the ceramic component of cermets. Studies described in the previous report were continued by CoorsTek, and the following conclusions regarding manufacturing of perovskite-based H<sub>2</sub> transport ceramics were compiled for this reporting period:

- Changing the binder composition from Ceracer 630 to Butvar B-79 resulted in roughly a 5% increase in membrane density.
- For the subject compositions, powders prepared from stoichiometric quantities of precursors resulted in multiphase materials that deviated from the target composition. This effect presumably was from volatility loss of the A-site oxide. Alternatively, an *excess* of A-site precursor resulted in single phase materials with the desired crystal structure.
- Soaking agglomerated calcined powder in alcohol broke up the agglomerates and facilitated further processing.
- In addition to the above conclusions, optimal calcining temperatures, milling times, screen mesh size, membrane pressing conditions, and sintering temperatures have been identified for selected compositions.

**f. Commercial Concepting - MTI**

During this reporting period, MTI completed gathering of background information to help define process conditions and to provide a starting point for assessing the economics for addition of a hydrogen separation system to a Vision 21 Plant. Relevant literature was reviewed, and appropriate contacts from Kellogg, Brown & Root were consulted regarding pertinent aspects of the Power Systems Development Facility (PSDF) in Wilsonville, Alabama. The information has been compiled in a memo to Eltron covering general background information, such as typical temperatures and pressures that can be expected for the separation membrane, the expected gas compositions, and trace species to which the membrane likely will be exposed. The memo begins to address these issues as inputs to the membrane testing. Results from the membrane testing then

will be used to help identify the acceptable range of process conditions for the reactor, so that viable process layouts can be confirmed.

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

**Contributors:** Eltron

Operation of dense ceramic hydrogen separation membranes at high temperatures and pressures requires a chemically resistant seal with similar mechanical and expansion characteristics as the membrane material. During this quarter, additional seal compositions were tested under highly reductive conditions. Seal formation was achieved by ramping the cell assembly up to 1000°C and holding for a period of time. The assembly then was cooled to 800°C for seal testing. The maximum pressure maintained under these conditions was 100 psig, which was over twice the pressure achieved in the previous quarter.

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

**Contributors:** Eltron

During this quarter, efforts were continued toward development of both tape casting and spin coating fabrication procedures for thin film ceramic membranes. A new thin film precursor slurry composition was identified for tape casting and spin coating that produced smoother, more homogeneous films, with greater mechanical strength. An example is shown in Figure 7 demonstrating a  $\sim 100 \mu\text{m}$  thin film membrane on a porous support of the same composition. This new slurry recipe required fewer applications to the porous support to achieve a defect-free membrane absent of “pin holes”. However, the shrinkage rate for this composition was much different than the support, which compromised adherence of the membrane, and made H<sub>2</sub> separation evaluation difficult. Despite this limitation, a relatively *thick* membrane (0.5 mm) prepared by the thin film tape casting technique demonstrated H<sub>2</sub> separation in excess of 0.01 mL/min/cm<sup>2</sup> and an ambipolar conductivity of  $\sim 10^{-4}$  S/cm. Although these values were well below the best results for thick ceramic membranes and cermets described above, it is encouraging that this procedure generated test-worthy samples.

Spin coated samples also demonstrated the same shrinkage problem described above, and efforts to overcome this limitation resulted in highly porous films. During the next quarter, porous supports will be prepared from precursors with larger particles to minimize shrinkage while maintaining porosity.



**Figure 7.** SEM image of a dense thin ceramic film (bottom) on a porous support of the same composition (top). The white bar represents 100  $\mu\text{m}$ , which was also about the thickness of the film.

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

No actions were performed on this task during this reporting period.

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

No actions were performed on this task during this reporting period.

## SUMMARY AND CONCLUSIONS

Conclusions based on the work performed during this quarter, and other relevant actions are summarized as follows:

- For a selected perovskite mixed proton/electron conducting ceramic with the general composition  $\text{AB}_{(1-x)}\text{B}'_x\text{O}_{3-\delta}$ , maximum ambipolar conductivity of  $5 \times 10^{-3}$  S/cm was achieved for  $x = 0.3$ . A 0.9-mm thick membrane of this composition gave a  $\text{H}_2$  transport rate of

~0.13 mL/min/cm<sup>2</sup>. This composition contained both a pseudo-cubic and a rhombohedral perovskite phase. However, increasing the rhombohedral phase content resulted in a loss of conductivity, and the transition metal dopant was easily reduced under H<sub>2</sub> leading to material failure.

- For other ceramic compositions with the general formula AB<sub>0.8</sub>B'<sub>0.2</sub>O<sub>3-δ</sub>, conductivity increased with increasing atomic number of the transition metal dopant (B'). The maximum ambipolar conductivity for this series was 6 x 10<sup>-3</sup> S/cm. A 1-mm thick membrane of this composition gave a H<sub>2</sub> separation rate of ~0.3 mL/min/cm<sup>2</sup>.
- Processing model ceramic membranes was improved by changing the binder composition, soaking the calcined powder in alcohol to reduce agglomeration, and identifying optimal processing variables. Moreover, using an excess of the perovskite A-site cation precursor in the pre-calcined powder mixture resulted in a more crystallographically pure sample after calcining.
- Cermet membranes prepared with a primarily proton conducting ceramic and a metal showed electrical continuity for samples with 29, 33, 36, and 38 vol.% metal phase.
- A maximum conductivity of 3 x 10<sup>-3</sup> S/cm was achieved for cermet membranes with 36 vol.% metal phase. The activation energy for H<sub>2</sub> transport for this sample was only 0.2 eV.
- Additional improvements in membrane seal compositions enabled successful membrane seals at 100 psig.
- Homogeneous dense thin films were successfully prepared by tape casting and spin coating. However, differences in the film and support shrinkage rates during sintering led to membrane instability.
- Pt, Pd, and Ni based catalysts were applied to ceramic membranes using processes at SCI. These samples will be tested in the next quarter.
- Relevant information regarding Vision 21 fossil fuel operation parameters have been compiled by MTI. This information defines process conditions and provides a starting point for assessing the economics for addition of a hydrogen separation system to a Vision 21 Plant.

## OBJECTIVES FOR NEXT REPORTING PERIOD

Specific objectives for the next quarter is summarized as follows:

- Continue testing new ceramic mixed proton/electron conductors doped with higher atomic number transition metals. Achieve ambipolar conductivities near 10<sup>-2</sup> S/cm.
- Utilize recently identified ceramics with higher conductivity as the proton conducting component in future cermets. Increase ambipolar conductivity of cermets by a factor of four.
- Test a range of catalyst systems for promoting hydrogen transport. Determine if surface kinetics limit hydrogen flux.
- Continue evaluation of thin film membranes. Optimize thin film and porous support shrinkage rates.
- Increase pressure maintainable by high-pressure seals to 150 - 200 kpsig.
- Continue with stability, design, manufacturing, and commercial concepting issues.

- Arrange for neutron diffraction studies on model proton-conducting ceramics.

## TIME LINES

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

