Title: Advanced Hydrogen Transport Membranes for Vision 21 Fossil Fuel Plants

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ABSTRACT

During this quarter of the no cost extension a cermet composition referred to as EC101 containing a high permeability metal and a ceramic phase was prepared for sealing and permeability testing. Several different types of seals were developed and tested. In addition membrane surface stability was characterized.

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 developing and testing seals for a cermet membrane referred to as EC101, and characterization of surface stability of EC101.

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.

A no-cost extension was obtained to finalize cermet development at CoorsTek. Eltron Research, Inc. monitored cermet development progress and characterized appropriate materials. All other milestones and tasks were completed during the last quarter. During this quarter of the no cost extension a cermet composition referred to as EC101 containing a high permeability metal and a ceramic phase was prepared for sealing and permeability testing. Several different types of seals were developed and tested. In addition membrane surface stability was characterized.

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

Multi-Phase Ceramics and Cermets

CoorsTek continued developing the processing conditions necessary to sinter a new cermet referred to as EC101 containing a high permeability metal and a ceramic phase. Temperatures, ramp rates, and atmospheres were fine tuned to achieve planar membranes. Results showed that sintering under the appropriate conditions led to cermets with less than 1% open porosity and distinct metal and ceramic phases as characterized by X-ray diffraction. Effort during this quarter was also focused on developing appropriate seals for characterizing these materials. Several different sealing techniques were investigated. Key issues included ability to form a leak free seal while preventing contamination of the membrane surface. Two different types of brazed seals were leak tested at Eltron Research, Inc on membranes 13-15 mils thick. Both samples could not be sealed 100% leak tight and therefore accurate permeability values were not obtained.

The stability of the as prepared membrane surface was also characterized. The surface of one of the prepared cermet disks was characterized with X-ray diffraction before and after a wet-polish. No surface degradation or undesired oxide phases were observed. In addition, the sample was allowed to sit exposed to air for one month following the wet polish. After one month the disk was once again characterized with X-ray diffraction and no undesired phases were observed. These results are encouraging as they demonstrate that the surface of these cermets is stable to exposure to air and water.

Task 3 High Pressure Hydrogen Separation

No actions were performed on this task during this quarter.

Task 4 Thin-Film Hydrogen Separation Membranes

No actions were performed on this task during this quarter.

Task 5 Construction and Evaluation of Prototype Hydrogen Separation Unit

No actions were performed on this task during this quarter.

Task 6 Membrane-Promoted Conversion of Alkanes to Olefins

No actions were performed on this task during this quarter.

Task 7 Catalyst Membrane Compositions for Scale Up

No actions were performed on this task during this quarter.

Task 8 Manufacturing Processes for Demonstration-Scale Hydrogen Separation Membranes

No actions were performed on this task during this quarter.

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. Cermet EC101 was prepared with less than 1% porosity.
2. Cermet chemical stability was demonstrated by exposure to water.

OBJECTIVES FOR NEXT REPORTING PERIOD

CoorsTek will finish work on EC101 during the last quarter of the project extension. Sintering conditions will be finalized and if gas tight seals are obtained permeability of EC101 cermets will be determined.

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.

<div>Task 6</div> <div>Membrane-Promoted Conversion of Alkanes to Olefins</div>												
Tasks	Months	0	6	12	18	24	30	36	42	48	54	60
<ul style="list-style-type: none"> Lab-Scale Reactor Construction (Eltron) Catalyst Development (Eltron, SCI) Membrane Reactor Evaluation (Eltron) 	<div> <div> <div>—▲ Completed</div> </div> <div> <div></div> <div>Completed</div> </div> <div> <div></div> <div>Completed</div> </div> </div>											

Task 7 Catalyst Membrane Compositions for Scale Up													
Tasks	Months	0	6	12	18	24	30	36	42	48	54	60	
<ul style="list-style-type: none"> • Compile Performance Data (Eltron, CoorsTek) • Select Candidate Compositions (Eltron, CoorsTek) • Select Material Suppliers (CoorsTek, Eltron) 													<div> <div></div> <div>Completed</div> </div> <div> <div></div> <div>Completed</div> </div> <div> <div></div> <div>Completed</div> </div>

[illegible]

<div> <div>Task 9</div> <div>Fabrication and Evaluation of Demonstration-Scale Hydrogen Separation Unit</div> </div>												
Tasks	Months	0	6	12	18	24	30	36	42	48	54	60
<ul style="list-style-type: none"> Modification of High-Pressure Unit (Eltron) Refinement and Application of High-Pressure Seals (Eltron, CoorsTek) Evaluation of Demonstration-Scale Unit (Eltron) 												<div>Completed</div> <div></div>

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