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QUARTERLY TECHNICAL REPORT

September 1, 1994 to November 30, 1994

1. **Project Title:**

Preparation and Characterization of Composite Membrane for High Temperature Gas Separation.

U.S. DOE Grant No: DE-FG22-93MT93008

2. **Investigators:**

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3. **Project Objectives and Scope:**

To develop a new class of permselective inorganic membranes, we have identified electroless plating as potential a route to deposit a thin metal film on porus substrate. Electroless plating is a controlled autocatalytic deposition of continuous film on the surface of a substrate by the interactions of a metal salt and a chemical reducing agent. This method can give thin films of metals, alloys and composites on both conducting and nonconducting surfaces. The objective of this project is to develop thin film palladium membranes for separation of hydrogen in high temperature applications. We plan to use electroless plating to deposit thin palladium films on microporus ceramic and silver substrates. We plan to characterize the membrane in terms of permeability and selectivity for gas separation. To accomplish the research objective, the project requires three tasks:

i. Development of a Process for Composite Membrane Formation

The work will involve the selection of methods for thin metal/metal-alloy film deposition on inorganic and ceramic microporus substrates. To assure reproducible film, optimization of the electroless deposition will be required. This step will involve determination of the optimum configuration of the plating bath, which consists of studying the effect of parameters like pH, temperatures, concentrations, type of reducing agent and sensitizing solution on the rate of deposition of the film.

ii. Characterization of Fabricated Composite Membrane

The work for this task will start with the fabrication of composite membrane. The membrane will be characterized by measurements and evaluation of physical properties of composite (film composition by EDAX, film thickness by SEM). The measurements will

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also include permeability and steady state diffusion, and the effect of thermal cycling.

iii. Development of Theoretical Model For Hydrogen Gas Separation

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

4. Technical Highlights and Milestones:

This report covers the quarter ending November 30, 1994. During this quarter, we attempted to measure the diffusivity and permeability of hydrogen gas through the palladium composite membrane. Microporous ceramic disc was used as a substrate for electroless plating of palladium film. We developed a gas chromatographic method to measure the concentration of gases in the gas streams. In the diffusion cell, graphite based packing material is used as seal to stop leakage of hydrogen gas. While running the diffusion measurements at elevated temperature and pressure, leakage of hydrogen was observed. This is a serious problem and it needs to be resolved. Currently, we are working on this problem.

During this quarter, we designed a diffusion cell to test thin-film palladium membrane in tubular structure. The diffusion cell is being fabricated and assembled by a local machine shop. During next quarter, we plan to fabricate thin-film palladium membrane in tubular structure using ceramic tubes as substrate. Future work will focus on developing methods for evaluating these membranes for permeability, selectivity and structural integrity.

At the November 1994 AIChE Annual Meeting at San Francisco, we presented some preliminary results of this work in a poster paper.