TITLE: Supported Dense Ceramic Membranes for Oxygen Separation

P.I.: Timothy L. Ward

STUDENTS: Deying Xia (completed M.S.), Hong-Xia Zhang (current)

INSTITUTION: University of New Mexico
Center for Microengineered Materials
Department of Chemical and Nuclear Engineering
Albuquerque, NM 87131
505-277-2067 Fax: 505-277-5433
e-mail: tlward@unm.edu

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ABSTRACT

In the past year, we have focused our investigations on examining alternate paste and slurry formulations to improve the sintering characteristics of supported $SrFeCo_{0.5}O_x$ films. These formulations continued to utilize the spherical submicron powder produced in our laboratory by the aerosol pyrolysis process. We have previously demonstrated that films from these powders can be densified without simultaneous densification of the chemically identical support. The major unresolved obstacle in the work reported earlier in the project was cracking of the $SrFeCo_{0.5}O_x$ films during film drying as well as during sintering. In response to that problem, we have been examining non-aqueous paste and slurry formulations containing binder, dispersant and plasticizer components. We have also begun utilizing spin casting as an alternative to the doctor blade procedure reported earlier. The spin-coating procedure has led to much more uniform coverage over the porous substrate, but required modification to the slurry formulation.

The components and approximate proportions of the paste/slurry formulas were identified from ceramic tape casting formulations found in the literature. The solvent used was mineral spirits and isopropanol in a 2:1 ratio (by weight). To this castor oil was added as a dispersant (0.083 g/g solvent), polyvinyl butyral as a binder (0.125 g/g solvent), and dibutyl phthalate polyethylene glycol (MW 300) and stearic acid as plasticizers (each 0.083/g solvent). The final paste/slurry formulations consisted of this mixture, powder, and additional solvent and/or castor oil in some cases.

The formulation of pastes for doctor blade application were improved by using 25-30 wt% castor oil (in excess of what was present in the solvent mixture), allowing pastes with up to 40 wt% powder to be prepared. Films prepared from these pastes sintered to apparently complete density (by SEM examination), while leaving the support porous. Cracking was much improved, but still existed. We

found that the doctor blade technique did not give good control over thickness, uniformity, or edge-to-edge coverage. Nearly complete film coverage is critical for permeation testing. We therefore began to explore spin coating as an alternative. A modified slurry formulation was developed which utilized extra solvent, with the powder content reduced to 15-18 wt%. With this procedure we find that the film deposit in the initial coating is very thin, and there is evidence of permeation into the porous support. Thicker films were built up by additional depositions, with a sintering treatment done between each layer. A two-layer membrane prepared in this manner revealed nearly complete edge-to-edge coverage of the substrate, with no apparent cracks. However, some remnant pores and defects could still be seen. Room temperature N_2 leak testing revealed a permeance about 4 times lower than the original support. This is the highest quality and most promising membrane we have produced, and the current synthesis methodology is a significant advance toward the goals of the project. We are continuing with characterization and additional deposition on this membrane, and will conduct O_2 permeation if leakage rates are sufficiently reduced.

ARTICLES, PRESENTATIONS AND STUDENT SUPPORT

JOURNAL PUBLICATIONS

- none published at this time

CONFERENCE PRESENTATIONS

- T. Ward,* D. Xia, R. Chitthuri, "The Processing and Mending of Dense Thick Film Ceramic Membranes Using Ultrafine Powders", platform presentation, 1999 Annual Meeting of the American Institute of Chemical Engineers, Oct. 31-Nov. 5, 1999, Dallas, TX.
- T.L. Ward,* D. Xia, and R. Chitthuri, "The Use of Aerosol and Vapor Processing in the Fabrication of Dense Mixed-Conducting Ceramic Films", platform presentation, Vapor Phase Synthesis and Materials III, Engineering Foundation Conference, July 18-23, 1999, Helsinki, Finland.
- D. Xia, H. Zhang, and T. L. Ward*, “The Processing of Dense Thick-Film Sr-Co-Fe-O Membranes Using Aerosol-Derived Powders”, accepted as platform presentation, 2001 National Meeting of the North American Membrane Society, May 16-18, 2001, Lexington, KY.

THESES

- D. Xia, “The Fabrication and Sintering of Sr-Co-Fe-O Membrane Supports and Films”, M.S. Thesis, University of New Mexico, December 2000.

STUDENTS SUPPORTED

- Deying Xia (M.S.)
- Rajarao Chitthuri (M.S.)
- Hong-Xia Zhang (M.S., current)