

# Standardization of Methods for the Characterization of Inorganic Membranes

# **Project Objectives**

- To indicate the <u>practical capabilities</u> of physical and physico-chemical properties for, specifically, the characterization of inorganic membranes
- To determine an <u>efficient methodology</u> for the determination of inorganic membrane properties of practical importance
- To draw attention to unresolved or unaddressed issues that deserve experimental attention

# Background

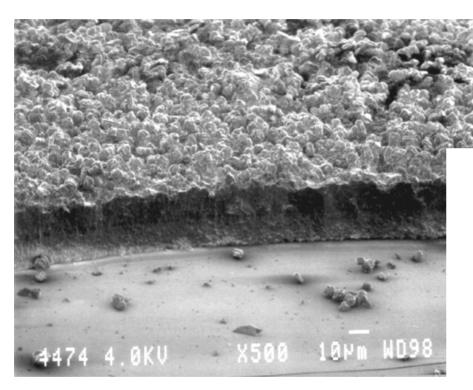
In the past decade, there has been a rapid growth in the number of publications dealing with synthesis and characterization of inorganic membranes. Furthermore, the increased industrial interest has been demonstrated by the recent formation of two multiconsortiums to develop inorganic membranes for partial oxidation of methane to synthesis gas. Such a large increase in the interest in the development of a new generation of inorganic membranes is, at least, due in part to their excellent chemical, thermal, and mechanical stability.

Therefore, the **development of standard characterization procedures** for the evaluation and comparison of the important characteristics of inorganic membranes is essential to facilitate information exchange among researchers and industrial practitioners and to enhance the membrane development.

### Procedure

- Organization of two workshops to address the issues and techniques
- Formation of a Task Group consisting of researchers from both academia and industry
- Implementation of surveys to obtain additional information on characterization techniques and suggestions for new techniques
- Selection of a preliminary list of suggested standardized characterization methods
- Distribution of the preliminary list to researchers for comments and suggestions
- Formulation of the final recommendations to IUPAC for review and approval

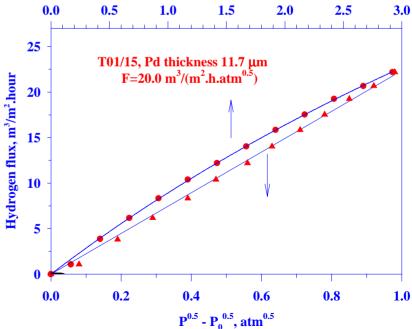
#### making sense of inorganic membrane characterization



SEM micrograph of the cross section of a silicalite membrane supported on  $\gamma$ -alumina disk

Hydrogen flux for a Pd membrane as a function of the difference of the square root of pressure (Sievert's law) at 350°C





# **Project Contents**

- Characterization of physical properties of inorganic membranes (e.g., porosity, pore size distribution, membrane thickness) With an emphasis on well-established general techniques (e.g., mercury porosimetry, adsorption and desorption Isotherms, calorimetric measurements, SEM, TEM, NMR, and AFM)
- Transport properties characterization by dynamic techniques (e.g., bubble point, liquid/gas permeance measurements, and permporometry)

# Contents (continued)

- Development of standards for protocols, data analysis, and reporting formats
- Development of standards for experimental conditions for characterizations (e.g., temperature, pressure, standard gases, and mixture gas measurements)

# First Workshop

- Chairmen:
   Professors Louis
   Cot and Yi Hua Ma
- Place:
   Montpellier, France
   In conjunction with the
   3rd International
   Conference on Inorganic
   Membranes
- Date: June 25, 2000
- Number of Participants: ~ 100
- Number of Invited Presentations: 6

• Microstructure, Structure, and Defect Structure Characterization in Mixed Conductor Membrane Materials

#### R. Bredsen, Norway

• Present Status of the "CHARMME" Network: Harmonization of Characterization Procedure for Porous Membranes

#### C. Guizard, France

 An Overview of Techniques for Membrane Characterization

#### T. Tsotsis, USA

 Characterization of Inorganic Membranes by Radiation Scattering and Spectroscopic Techniques

#### J. Ramsay, France

- Status of Zeolite Membrane Characterization R. Noble, USA
- Characterization of Pd and Pd/Alloy Membranes

#### J. D. Way, USA

## **Current and Future Activities**

- Organization of the Second Workshop
- Continuation of the formation of the Task Group with emphasis on recruiting more industrial members
- Formation of subgroups within the Task Group and assignment subareas to each subgroup
- Implementation of surveys and selection of a preliminary list
- Distribution of the preliminary list for comments and suggestions from academic and industrial researchers
- Formulation of the final recommendations to IUPAC for review and approval

#### **Task Group Chair:**

Ed Yi Hua Ma

TG Members: L. Cot, J.A. Dalmon, I. Dekany, D. Fain, J. Falconer, J.H. Fendler, H. Fleming, G.R. Gavalas, W.S. Winston, E. Kikuchi, W.J. Koros, Y.S. Lin, V. Linkov, S. Nakao, R.D. Noble, J. Pellegrino, J.D.R. Ramsay, J. Santamaria, T.T. Tsotsis, J. D. Way, N. Xu

- Part of the IUPAC Strategic
   Initiative on Materials
- Proposed Duration: 2 years (started in June 2000)
- TG to be completed by addition of members from both industry and academia