

Inorganic Membrane Porous Support Tube Fabrication

Brian Bischoff and Roddie Judkins

Oak Ridge National Laboratory

Presented at

DOE Hydrogen Program Annual Review
Crystal City, VA

May 25, 2005
Project #PDP18

This presentation does not contain any proprietary or confidential information

*Oak Ridge National Laboratory is managed by UT-Battelle, LLC, for the U.S. Department of Energy under Contract No. DE-AC05-00OR22725. Accordingly, the U.S. Government retains a nonexclusive, royalty-free license to publish or reproduce the published form of this contribution, or allow others to do so, for U.S. Government purposes.

Project Objectives

- To develop porous metal supports for hydrogen separation membranes that are compatible with the supported membrane and operational environment

Budget

	Budget (k\$)
FY2004	100
FY2005	0*

*The budget request was \$200K. However, due to changes in EERE priorities the funding zeroed out for FY05.

Technical Targets

➤ DOE Technical Barriers

- A. Fuel Processor Capital Costs
- B. Operation and Maintenance Costs
- AB. Hydrogen Separation and Purification

➤ DOE Technical Targets for 2010

- Purification: 90% at \$0.03/kg Hydrogen
- Palladium Membranes: <\$100/ft² capable of operating at 300-600 °C for 100,000 hrs with at flux of 200 scfh/ft²

Technical Approach

Develop a composite support tube structure especially for palladium membranes

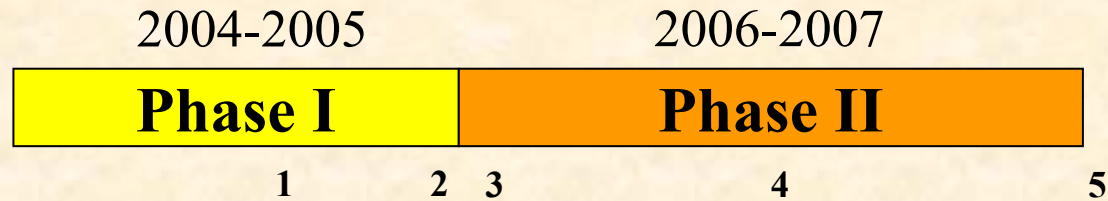
➤ Approach for Porous Support Tube Development

- Establish performance criteria for support tubes for palladium, microporous, ion-transport membranes
- Identify potential support tube materials and down select through a rigorous investigation of potential for fabrication and compatibility with Pd (initially)
- Establish fabrication protocols

Project Timeline

(Project initiated February 2004)

Support Tube Development



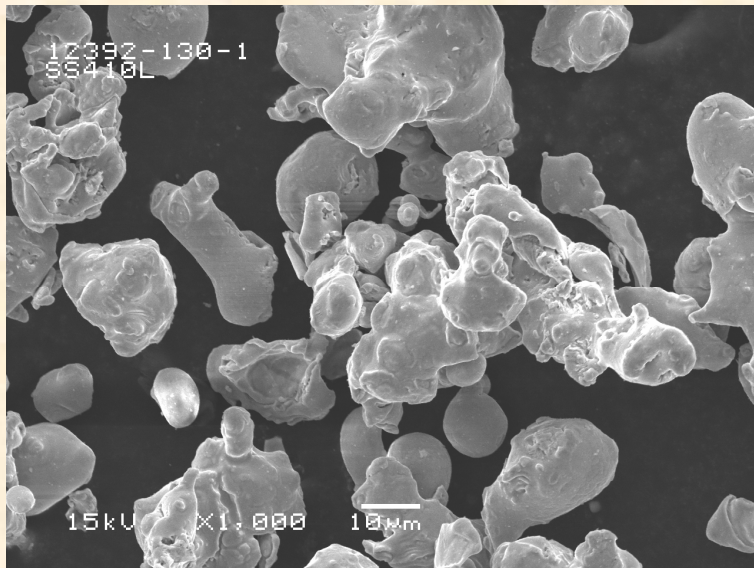
- Phase I: Development and Testing
 - 1 – **Prototype Support Tube**
 - 2 – Complete tests to determine efficacy of tubes to accommodate membrane layer(s)
- Phase II: Optimization, Scale up and Tech Transfer
 - 3 – Composite Support Development (initiate)
 - 4 – Complete tests to determine efficacy of composite tubes to accommodate membrane layer(s)
 - 5 – Technology Transfer

Technical Progress

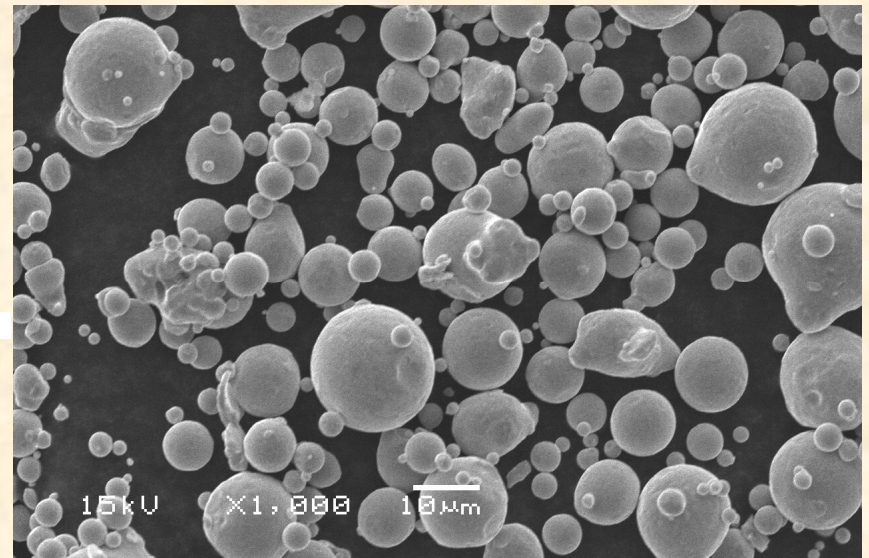
(Porous Support Tube Development)

- Potential support tube materials have been identified and include:
 - 300 and 400 series stainless steels,
 - Iron Aluminide, and
 - Hastelloy X
- Gas (argon or helium) atomized powders have greatest potential for hydrogen membrane supports (powders are spherical and size distribution can be controlled)
- Support tube forming process parameters are being established
- Palladium membranes need barrier layer to prevent intermetallic diffusion of metal atoms from support into palladium membrane layer. Future work will include application of this intermediate layer.

Gas Atomized Powder is More Spherical

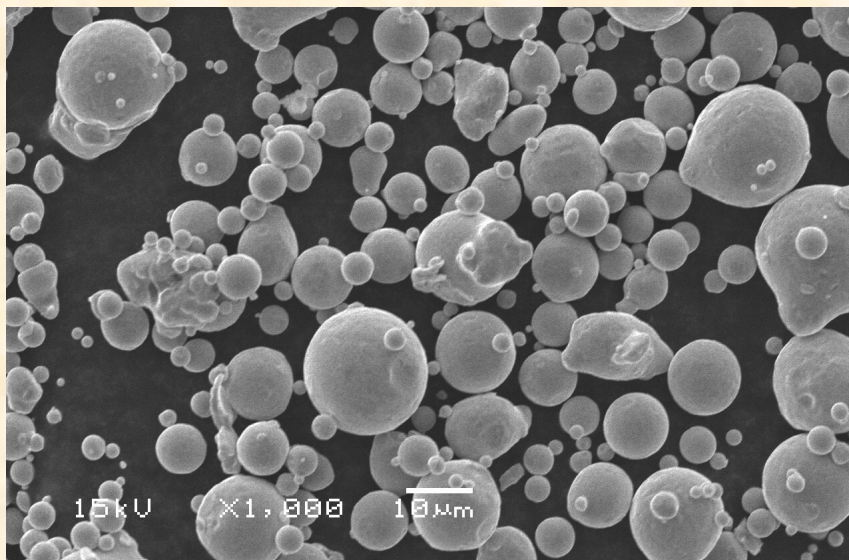


Water Atomized 410 Stainless Steel Powder

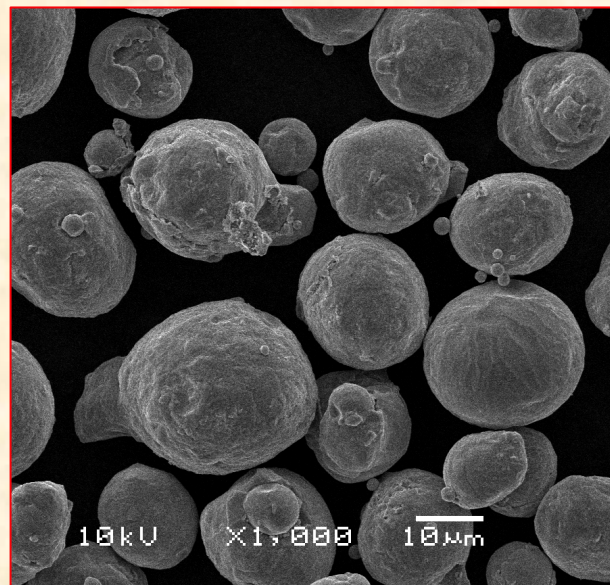


Gas Atomized 410 Stainless Steel Powder

Uniform Particle Size is Key to High Quality Supports



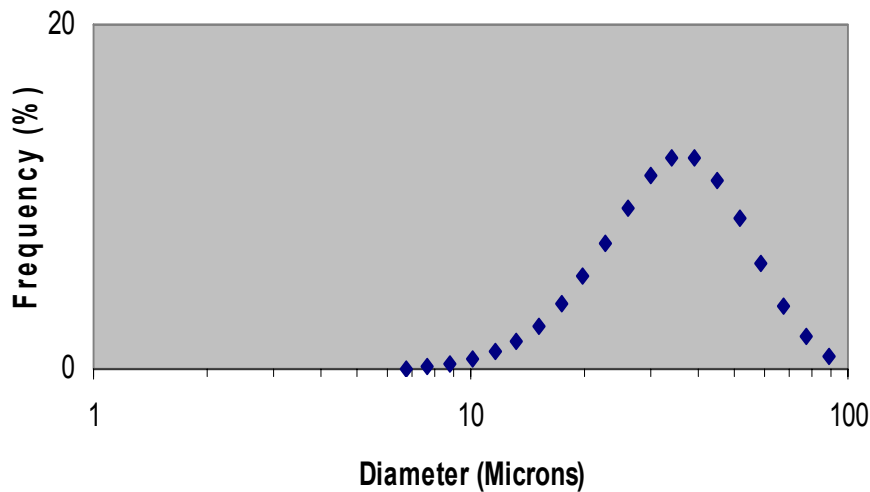
Standard Gas Atomization Produces
Broad Size Range of Powder



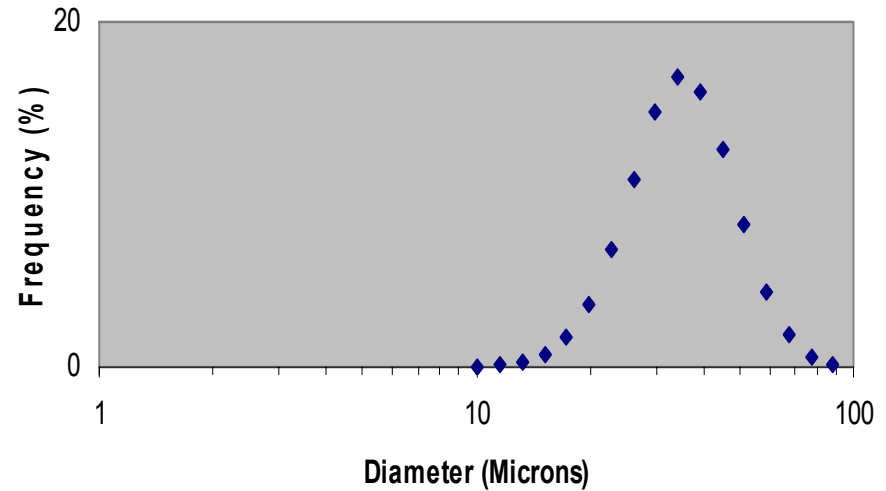
Gas Atomization at Ames
Laboratory Produces Uniform
Particle Size

Ames Laboratory Can Fabricate Spherical Particles With a Very Narrow Size Distribution

Particle Size Distribution of Water Atomized Powder



Particle Size Distribution of Gas Atomized Powder from Ames



Interactions and Collaborations

- **Ames Laboratory:** providing novel materials for support tubes
- **Worcester Polytechnic Institute:** discussions were planned to have WPI deposit Pd membranes on ORNL support tubes
- **NETL:** initial discussions on collaborative effort
- Discussions on implementation of technology are ongoing with
 - **ConocoPhillips, ChevronTexaco, Pall Corp., and Praxair**

Future Work

- Porous Support Tube Development
 - Continue to identify and characterize materials for support tube fabrication
 - Establish fabrication parameters and fabricate support tubes FY2005
 - Characterize support tubes for strength, permeance, and high temperature stability
 - Expand activity to include composite structure support tubes

Project Safety

The most significant hazard is the use of pure hydrogen in our membrane test systems

Our approach to ensuring safe operation includes:

- Project has undergone “Integrated Safety Management Pre-Planning and Work Control” (Research Hazard Analysis and Control)
- Each work process is authorized on the basis of a Research Safety Summary (RSS) reviewed by ESH subject matter experts and approved by PI’s and cognizant managers
- The RSS is reviewed/revised yearly, or sooner if a change in the work results in a need for modification.
- Experienced Subject Matter Experts are required for all Work Control for Hydrogen R&D including periodic safety reviews of installed systems
- Results of Work Control Process requires:
 - Monitoring hydrogen concentration at ceiling above test system. Alarm sounds at 50% LEL.
 - Personnel be present at all times when using hydrogen.
 - Evacuation of gas lines of air or purging with inert gas prior to introduction of hydrogen
 - Exhaust of gas lines containing hydrogen using eductors instead of electrically driven vacuum pumps.