

Advanced Barrier Coatings for Harsh Environments

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Project ID: PD132

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Overview

Timeline:

- Project Start Date:
4/1/2015
- Project End Date:
3/31/2017

Budget:

- Phase I: \$149,877
- Phase II: \$999,186

Barriers:

- B. Reliability and cost of gaseous hydrogen compression

Partners:

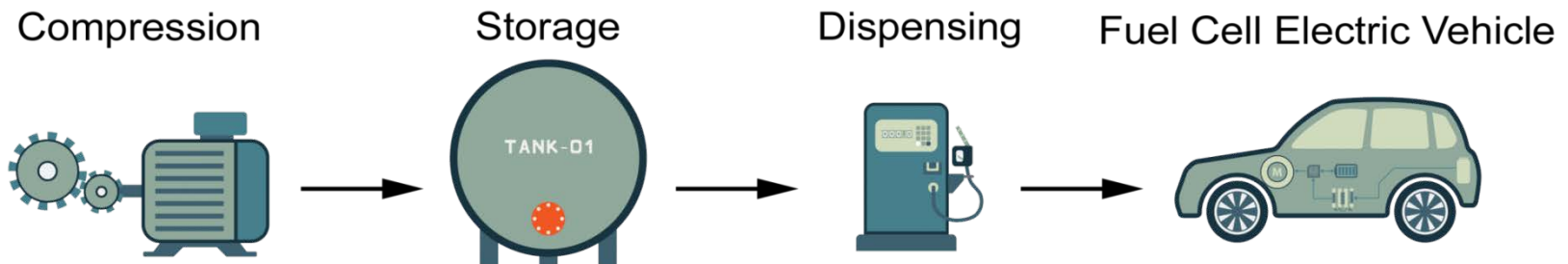
- Greene, Tweed & Co.
- Oak Ridge National Laboratory
- Hydro-Pac Inc.
- Praxair
- PowerTech

Outline

- **Review:** Impact of seal failures on Hydrogen Compression, Storage and Dispensing (CSD)
- **Technology Concept:** Flexible barrier coatings to prevent hydrogen ingress
- **GVD Background & Technology Overview**
- **Program Goals**
- **Progress to Date**

Relevance: Impact of Seal Failures

- Plastic and elastomeric seals are integral to all areas of hydrogen compression, storage, and dispensing (CSD)
- Hydrogen ingress degrades seals
 - Temperature and pressure cycling exacerbate issues
- Wear due to friction in high pressure, high temperature operation degrades seals
 - Frequent seal replacement is required
- Seal failure is a major contributor to process down time
 - Largest cause of unscheduled maintenance
 - >25% of hydrogen leaks
 - Redundant compression often specified



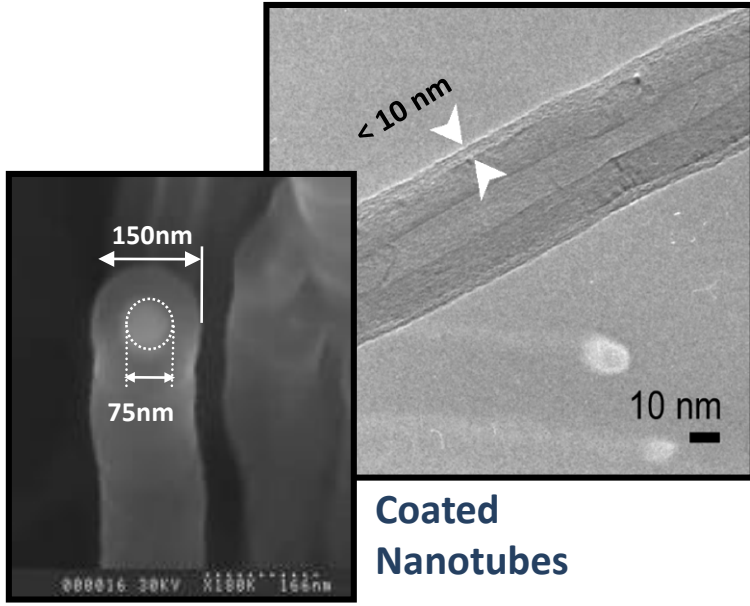
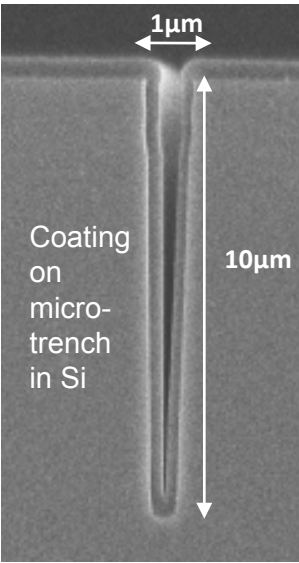
Relevance: Program Goals

- Make tangible improvements in seal life
 - **Lubricious coatings** for **rigid seals** reduce seal wear due to friction
 - **Barrier coatings** for **elastomeric seals** mitigate hydrogen vapor permeation
- Improved seal performance benefits operations and cost
 - Extend seal life from *<1,500 hours* to **>8,000 hours**
 - **5x reduction** in frequency of seal maintenance
 - Help decrease hydrogen CSD cost from *\$3.50/kg* to **\$2.00/kg**

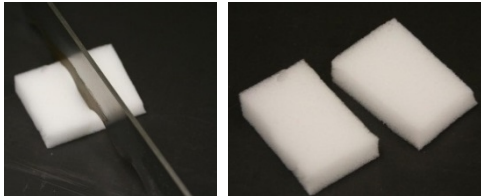
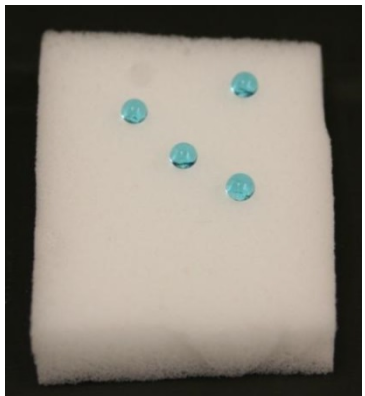
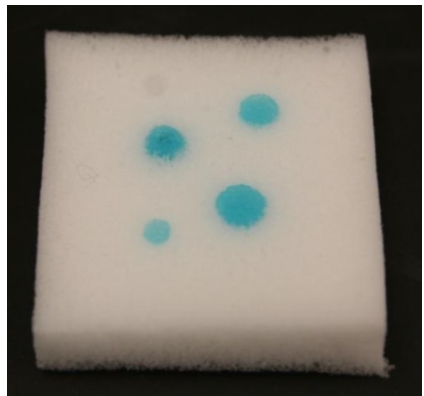
Approach: Polymer Vapor Deposition

A **room-temperature coating** process which produces **thin polymer coatings** on almost any material.

- “Gentle” application
- low temperature
- dry process
- single-step
- nano- to micro- meter thicknesses
- conformal on nano- and micro- structures



Approach: Conformal, Uniform Coverage



GVD coated foam is cut in half

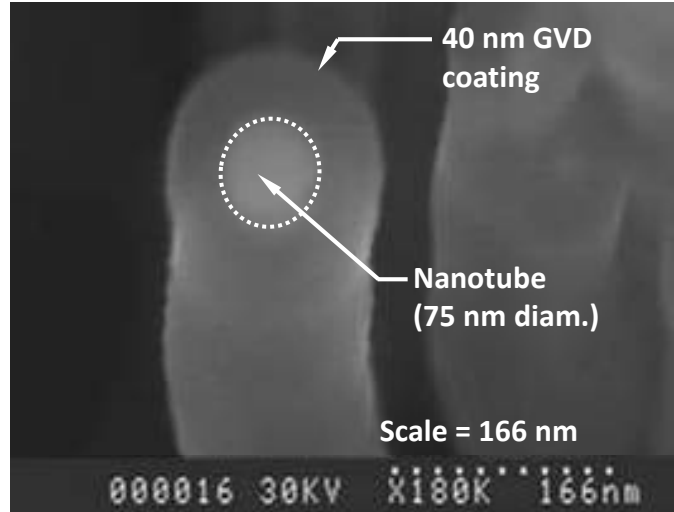
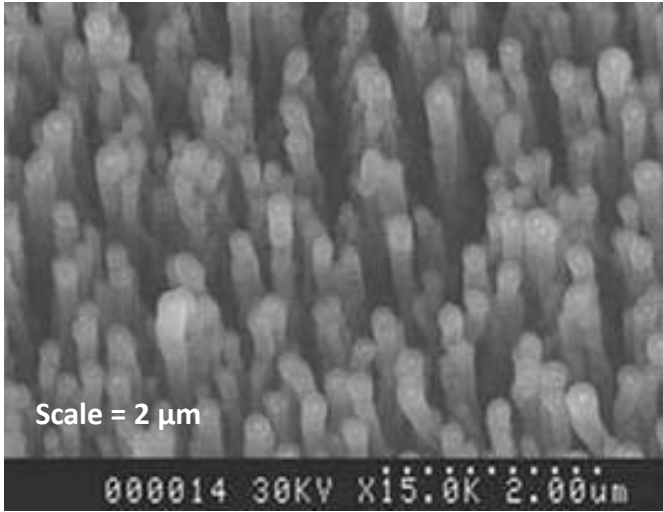
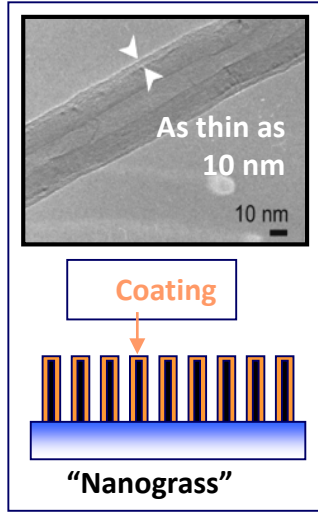


1. Water droplets soak into uncoated foam

2. Water droplets bead-up on GVD Coated Foam

3. Water droplets bead up on the cross-sectioned foam, demonstrating the hydrophobic coating has penetrated deep into the foam.

GVD coatings are conformal down to the nano-scale



Approach: Barrier and low friction coatings

- Vapor Deposition of flexible barrier coatings to prevent hydrogen ingress into elastomeric seals

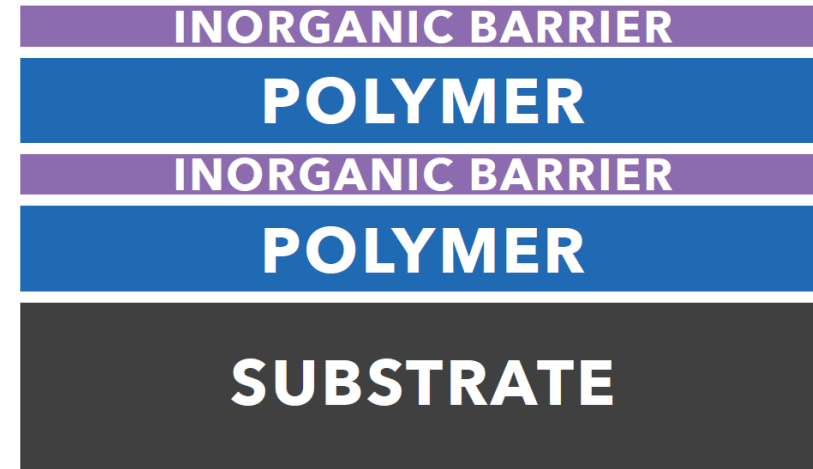
- Thin inorganic layers provide **vapor barrier**
- Polymer layers provide **flexibility**

- Vapor Deposition of lubricious coating to reduce wear on rigid plastic hydrogen seals

- Thin PTFE film provides low coefficient of friction surface for reduced wear

- Vapor deposition advantages:

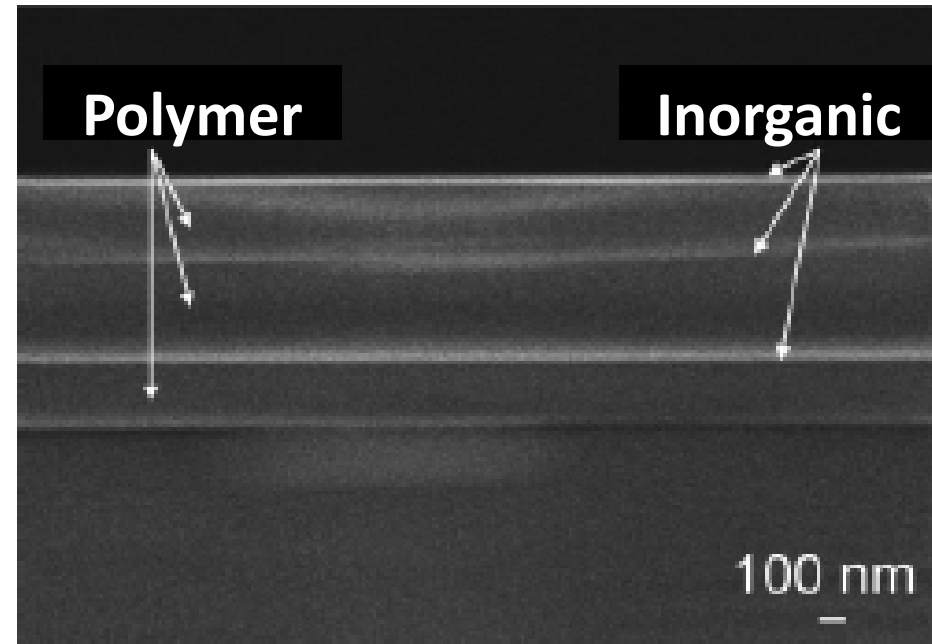
- Conformal coating of **3D seal geometries**
- Barrier layers deposit in the **same chamber** using the **same feed gas**
- **Scalable and manufacturable** compared to competitive solutions



Barrier Coating Stack

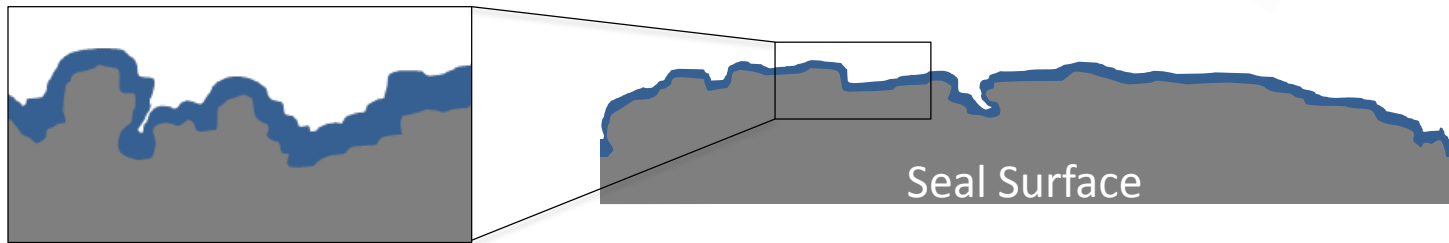
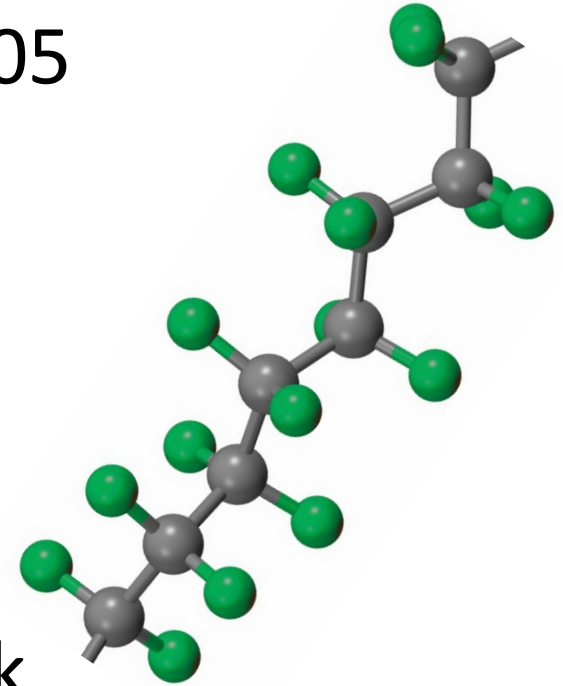
Approach: Barrier Coating

- Initial demonstration of flexible barrier coatings by GVD founder Prof. Karen Gleason at MIT
- Barrier coating flexibility
 - Maintains gas exclusion properties after hundreds of 180° bend cycles
- Barrier properties of coating driven by number of bilayers (dyads)
 - Order of magnitude reduction in water vapor transmission per dyad
 - Permeability reduction of **~1,000 fold with three dyads**



Approach: Low friction coating

- Vapor deposited polytetrafluoroethylene (PTFE)
- Low coefficient of friction, 0.03-0.05
- High chemical resistance
- Deposited at room temperature
- Dry process, solvent-free
- Highly conformal
- 50 nanometers to 10 microns thick



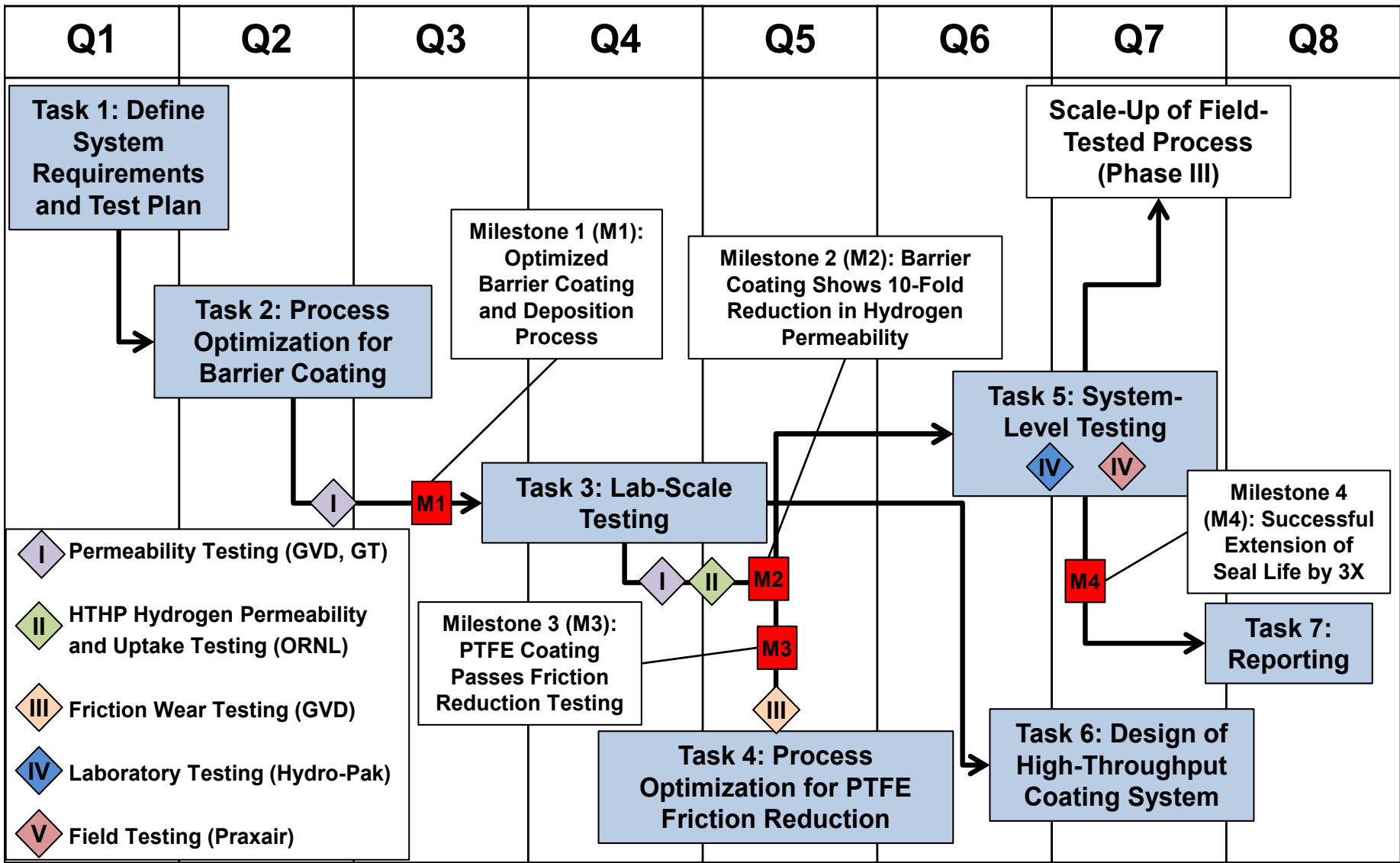
Approach: Phase I Summary

- Barrier coating materials selected to serve as a robust and versatile precursor for both polymer and inorganic barrier layer production
- A organic planarization layer of approximately 2 μm was required for typical seal samples
- A barrier coating thickness of 4 μm was chosen as an optimum coating thickness because it showed a **35% reduction in relative permeability** during preliminary helium testing
 - Helium 7X higher permeation than Hydrogen

Approach: Phase II Objectives

- Optimize organic/inorganic barrier coatings using scaled PECVD processes
- Demonstrate a 10-fold reduction in relative hydrogen permeability of GVD barrier coatings
- Develop a low-friction top coat of polytetrafluoroethylene (PTFE) for friction wear reduction of plastic piston-head seals
- Demonstrate improved seal life (goal of 5X increase) in field testing by a hydrogen compressor end user (PRAXAIR)

Phase II Project Schedule



Simple. Functional. Radical.

Accomplishments: Deposition Optimization

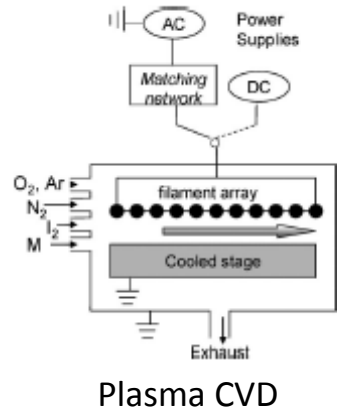
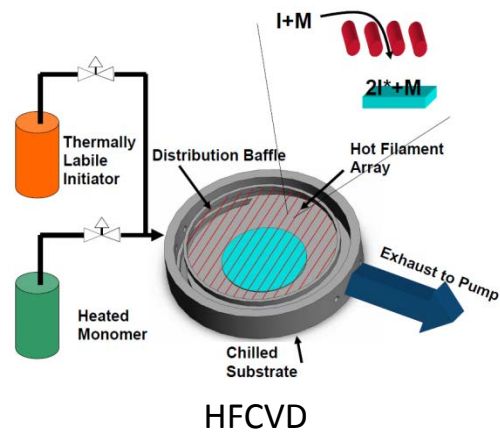
Barrier Coating Process

Transition from hot filament CVD and Plasma CVD to complete Plasma CVD

- Higher deposition rate and easier to scale for high throughput
- Ubiquitous gas volume radical generation, allowing coating on multiple sides of a substrate simultaneously

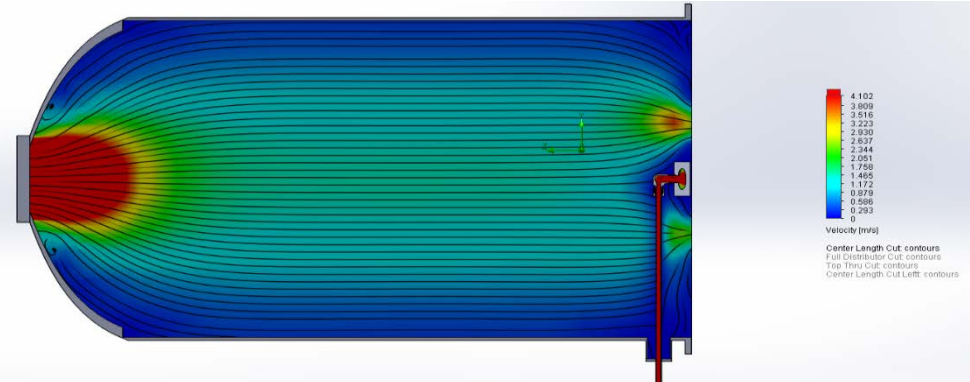
Coating properties optimization and validation through chemical analysis, adhesion testing, and acetone soak testing

Modified standard GVD deposition hardware to allow single chamber hot filament CVD and plasma CVD



Unoptimized Coating	Optimized Coating
After 72 hours of Acetone Soak	

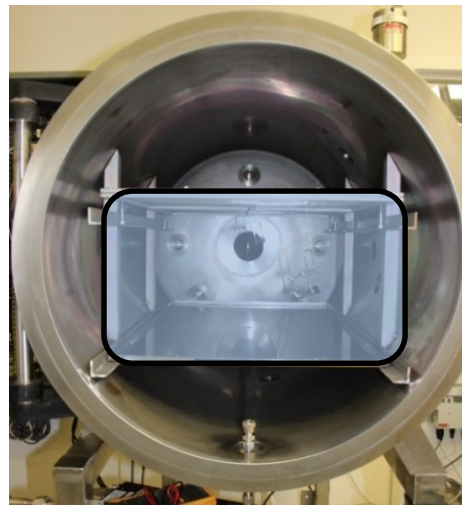
Accomplishments: Large Coating System



Gas flow simulation was used to optimize vapor flow in the system



Large Area Coating System



Coating Region (blue shading)

Cylindrical design allows the system to be upgraded to “tumble-coat” large quantities of seals.

Collaborators

<p>Greene, Tweed & Co.</p>	<p>Manufacturers of advanced seals and gaskets</p>	<p>Providing seal samples for experimentation & performing helium permeability tests</p>
<p>Oak Ridge National Lab</p>	<p>Leaders in evaluation of hydrogen permeability in polymers</p>	<p>Evaluating GVD barrier coating performance in hydrogen permeability tests</p>
<p>HydroPac Inc.</p>	<p>Manufacturers of advanced hydrogen compression equipment</p>	<p>Testing the life of coated dynamic piston bore seals</p>
<p>Praxair</p>	<p>Industrial user of hydrogen fuel cell vehicles</p>	<p>Field testing of coated seals</p>
<p>PowerTech</p>	<p>Turnkey designers and manufacturers of hydrogen fueling stations</p>	<p>Testing the life of coated dynamic piston bore seals</p>



Remaining Barriers and Challenges

•• For technology validation:

- Complete field and representative testing of barrier and low friction coatings
- Optimize process parameters for a high-throughput plasma coating system based on test data

•• For meeting industry technical targets and commercialization:

- Demonstrate 10 fold reduction in hydrogen permeability
- Demonstrate 3x extension of seal life in representative testing or field operation
- Complete design for high throughput plasma coating system

Future Work: Testing Underway

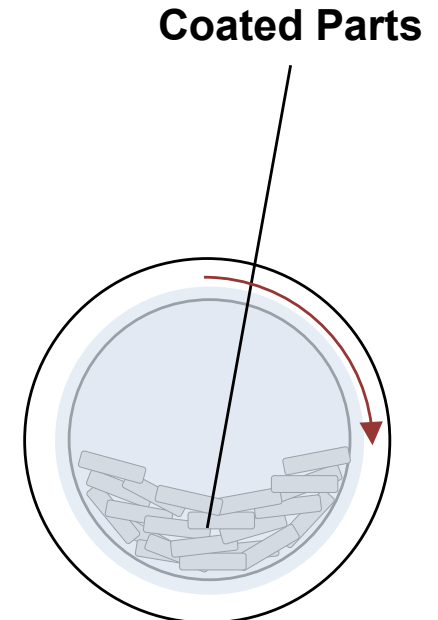
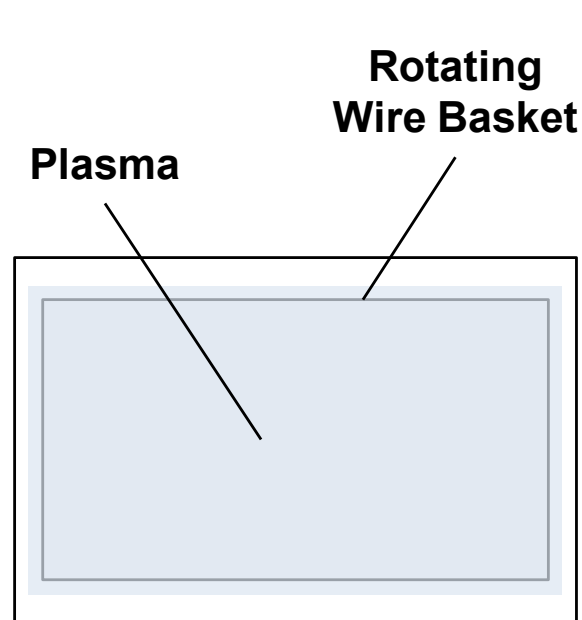
Hydrogen Permeability

- Oak Ridge National Labs
- Performed on ONRL's High Pressure Temperature Cycling (HPTC) apparatus
 - 700-875 bar on the upstream side
 - -50°C to 200°C temperature range

Wear Reduction

- PowerTech Labs
- Low friction PTFE coated seal tested in a double-ended single stage hydrogen compressor side-by-side with an uncoated dynamic piston bore seal
 - Hydrogen compression pressure 13,500psi
 - 5k psi inlet
 - Evaluations of coated and uncoated seal weight between 50-200 hours of operation, at 500 hours, then after failure

Future Work: Tumble Coating Design



- GVD will explore and develop a Plasma CVD tumble-coating process for components
 - Deposition of both coating layers within single system
 - High volume processing

Technology Transfer Activities

• Commercial Interest and Inquiries:

- Hydrogen Compression & Storage
- Hydrogen Refueling Stations
- Downhole Oilfield

• Potential Commercial Partnerships

- Seal manufacturers
- Hydrogen compressor manufacturers
 - Testers as earlier adopters

• Intellectual Property

- IP established based on MIT proof of concept
- Additional GVD patents pending

Summary

Objective: Reduce costs to Hydrogen Fuel Cell Electric Vehicles and hydrogen processing systems associated with Hydrogen Compressor seal failure. Improve seal life from <1,500 hours to >8,000 hours.

Relevance: Seal failure is a major contributor (25%) to hydrogen compressor maintenance, adding significant downtime and cost to operation.

Approach: Improve seal life through two types of coatings. Barrier coatings that mitigates hydrogen ingress into the seal, preventing premature failure. Low friction coatings that reduce wear of rigid seals, extending seal life significantly.

Accomplishments: GVD's barrier coating has demonstrated significant reduction in helium permeation through elastomeric seal materials. Optimized coating process for high-throughput manufacturing and constructed a prototype system.

Future Work: Demonstrate barrier performance in relevant testing environments (HPHT Hydrogen). Demonstrate reduce wear for low friction coatings in an operational environment. Design tumble-coating system for high-throughput manufacturing.

Thank you

Questions?

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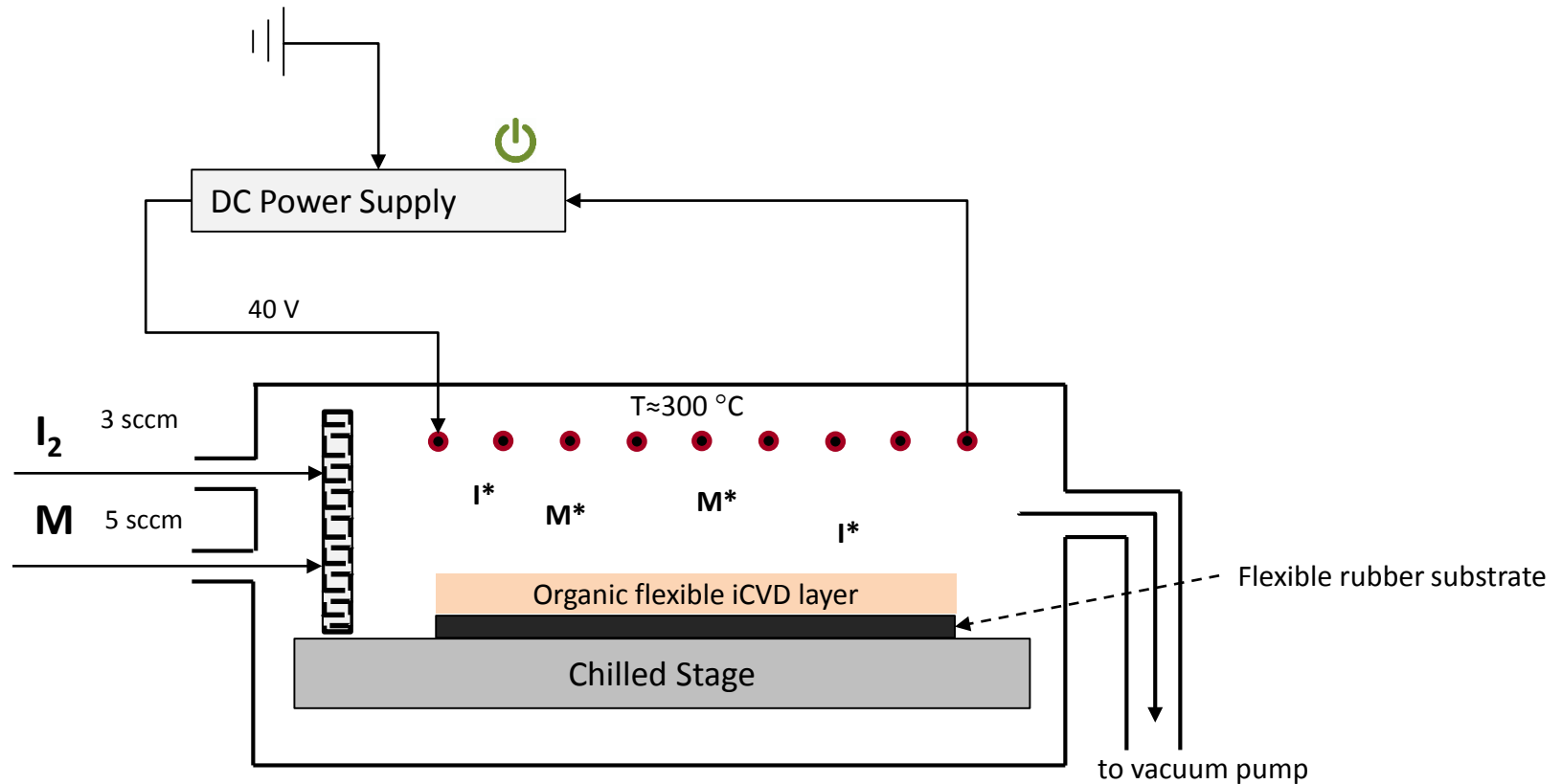
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Technical Back-Up

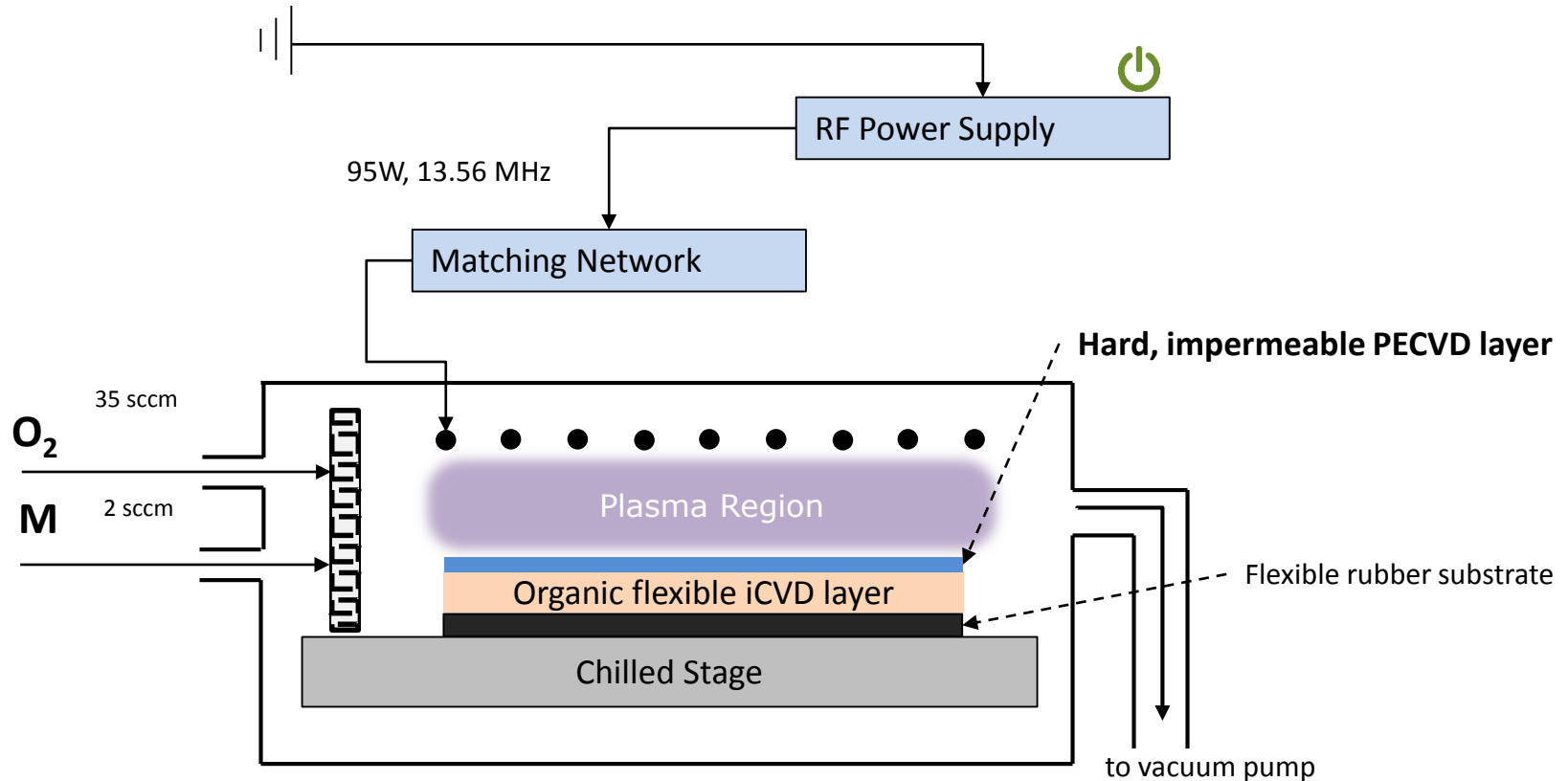
Single Chamber Barrier Coating Process: iCVD



- Gentle hot filament iCVD conditions allow for the growth of a soft and flexible polymer base layer

Technical Back-Up

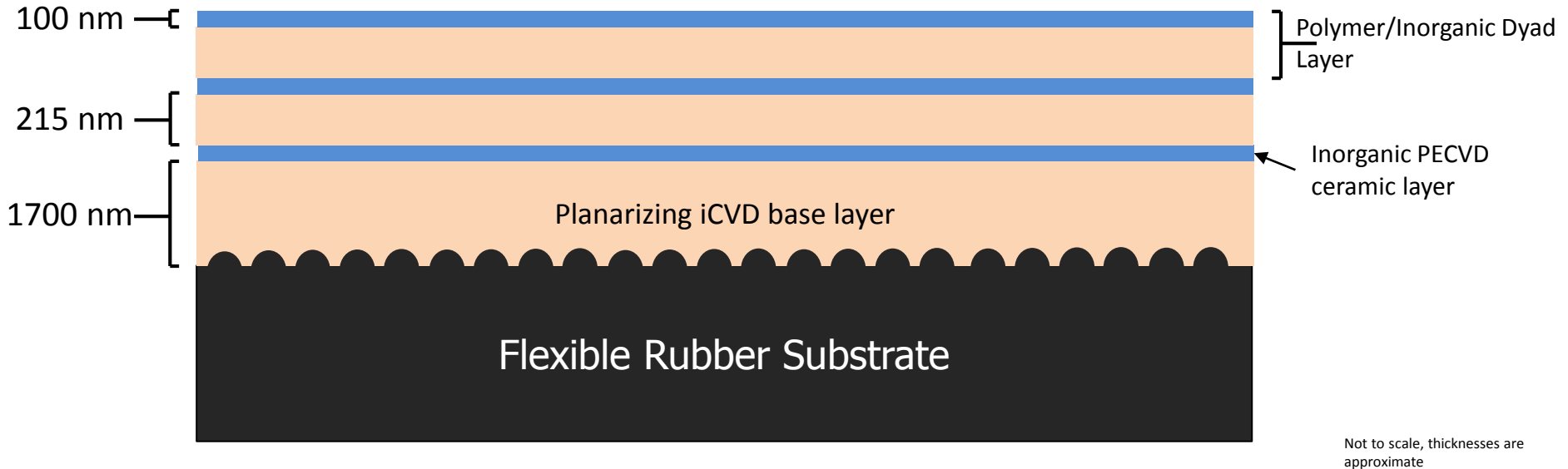
Single Chamber Barrier Coating Process: PECVD



Plasma initiation scheme creates a thin, rigid ceramic barrier layer

Technical Back-Up

Barrier Coating Construction

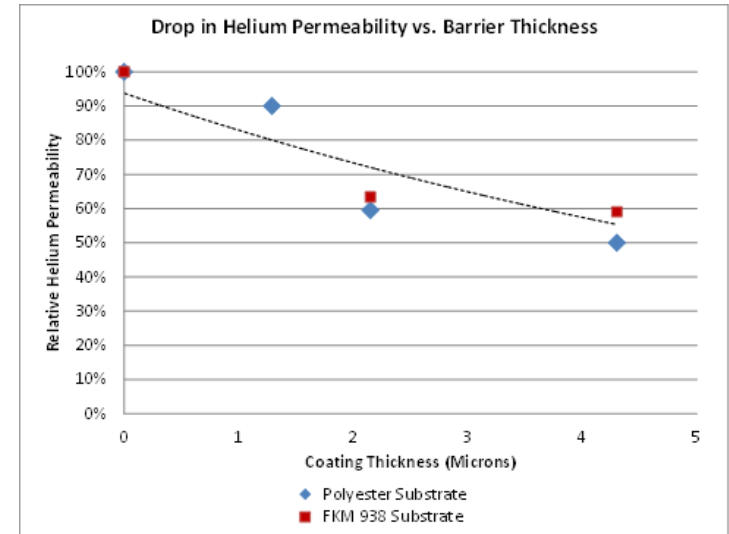


- Multiple dyads force the H_2 molecules to forge a tortuous path through nanodefects in the coating surface
- Number of dyads and coating thicknesses were optimized for maximum flexibility, planarization and minimum Hydrogen ingress

Technical Back-Up

Helium Permeability Reduction

- Steady state helium permeability testing performed at multiple coating thicknesses
- Reduction of 35% seen in helium permeability through polyester and fluorinated elastomer
 - Helium 1.6-7 X more permeable than hydrogen
- Coating provides significant improvement vs. most materials used in hydrogen compressor seal construction



Material	Helium Permeability (Barrier)
FKM 983	6.9
Viton	12.8
Polyethylene	1.1-7.3
PTFE	570
4um GVD Barrier Coating (tested on FKM 938)	5.0 +/- 0.6

Technical Back-Up

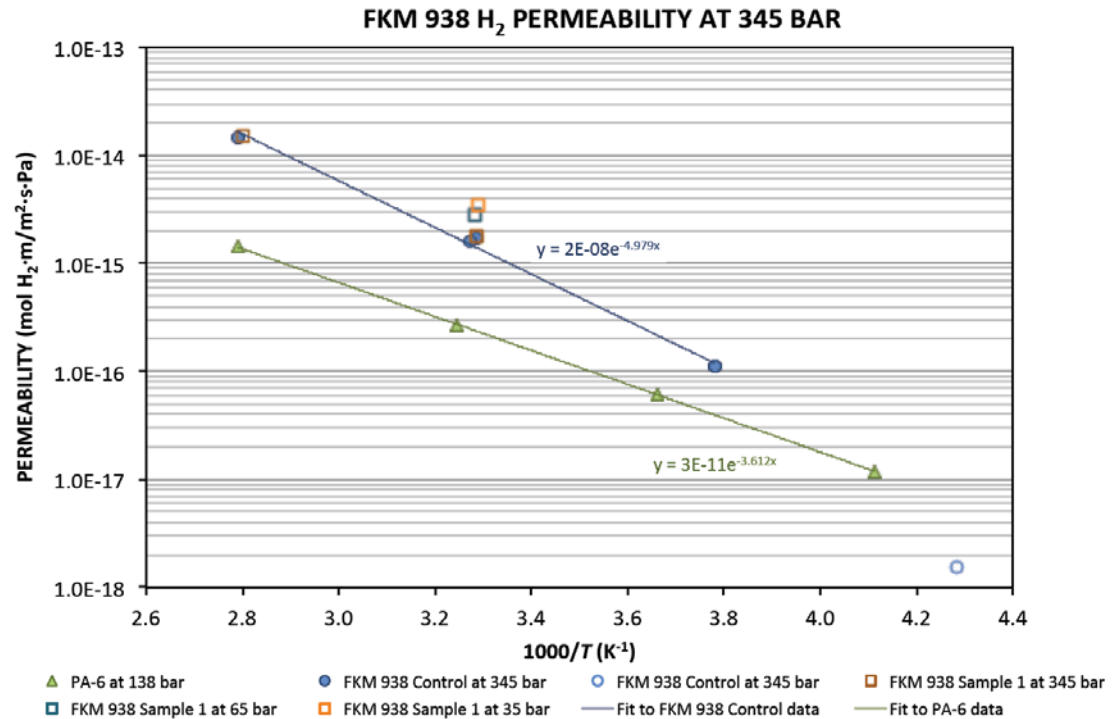
ORNL Barrier Coating Permeability Testing

Initial Testing

- 4um GVD coating on Greene Tweed FKM 2mm thick fluoroelastomer
- Initial testing shows no statistical difference in permeability
 - Test data dominated by FKM lack of permeability

Future Planned Testing

- GVD will coat commercial Viton for testing
 - Current elastomer used as energetic seal
 - Permeability multiple orders of magnitude higher than FKM
 - Lower temp testing required
 - Future seal material development for high temp (collaborate with GTC)



Critical Assumptions and Issues

Assumption: Technical success for barrier properties and low friction will provide desired increase in hydrogen compressor reliability

- Mechanical damage to the barrier coating can cause premature failures
 - Develop detailed installation procedures
- Demonstration in operation is required to determine whether the technology will be useable commercially
 - Testing before commercial launch with potential early adopters

Critical Assumptions and Issues

Assumption: Single chamber deposition of both coating layers will provide cost-effective commercial product

- Required permeability reduction is achievable at reasonable coating thickness
 - Validate reduction in hydrogen permeation with HPHT testing at ORNL
 - Optimize deposition process to allow for commercially viable coating cycle times
- Convince customers of value proposition
 - Field demonstration of increased seal life to justify coating cost
 - Cost/benefit trade off against novel seal materials