Coatings for Compressor Seals

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Project ID: in011

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Program Overview

Timeline:

- Project Start Date: 4/1/2015
- Project End Date: 12/6/2020

Budget:

- Phase I: $149,877
- Phase II: $998,616
- Phase IIA: $999,781

Barriers:

- B. Reliability and cost of gaseous hydrogen compression

Partners:

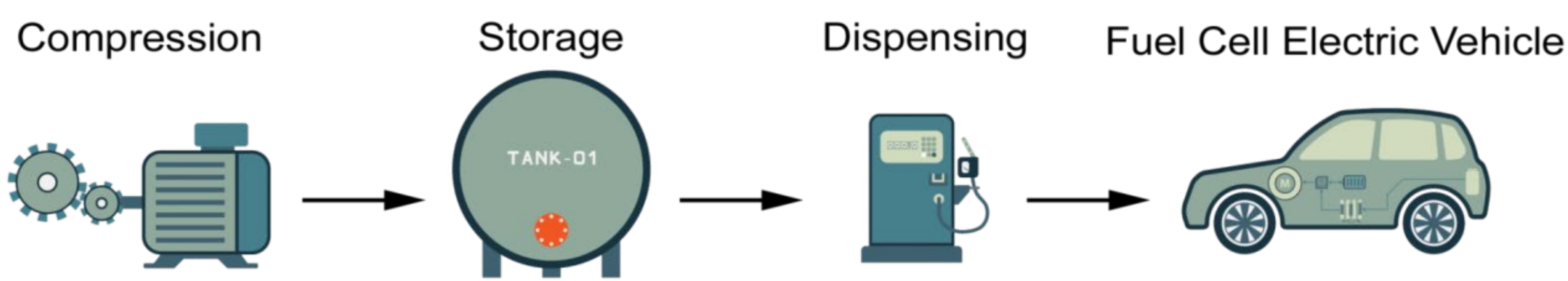
- National Renewable Energy Laboratory
- Takaishi Industries
- Greene, Tweed & Co.
- Oak Ridge National Laboratory
- Hydro-Pac Inc.
- PowerTech

Outline

- **Review**: Impact of seal failures on Hydrogen Compression, Storage and Dispensing (CSD)
- **GVD Technical Approach and Goals:** Flexible barrier coatings to prevent hydrogen ingress & lubricious coatings to reduce wear
- **GVD Objectives and Prior Work**
- **Program Accomplishments in 2018**
- **Proposed Future Work**

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
  - Without improvement, redundant compression necessary



Relevance: Program Goals

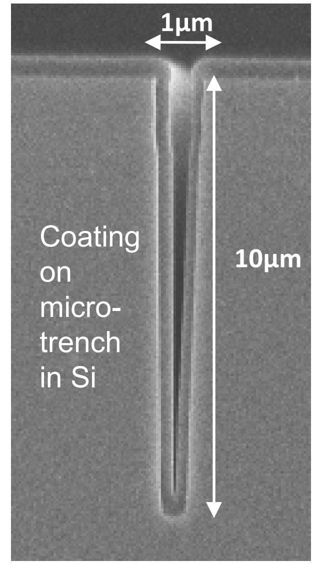
- Objective: Make tangible improvements in seal life with vapor deposited coatings
  - **Lubricious coatings** for **dynamic seals** reduce seal wear due to friction
  - **H_2 Barrier coatings** for **elastomeric seals** mitigate hydrogen vapor permeation
- Improved seal performance benefits operations and cost
  - **3-5x reduction** in frequency of seal maintenance
  - Help decrease hydrogen CSD cost from *$3.50/gge* to **$2.00/gge by 2020**

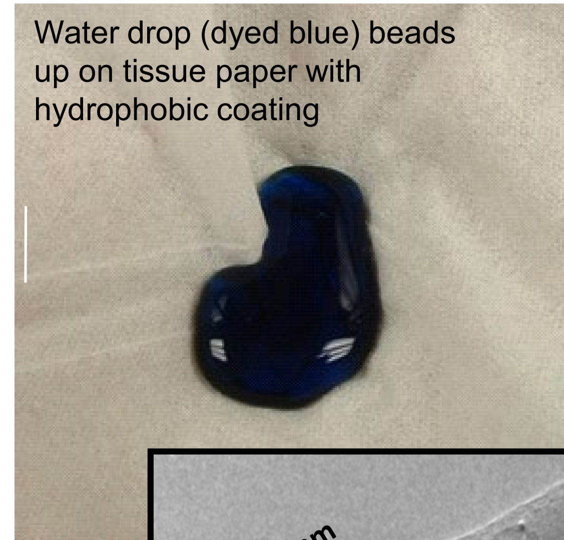
2015 FCTO MYRD&D – Delivery Section

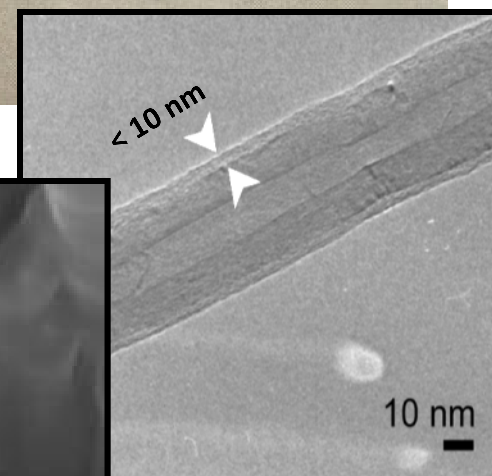
Approach: Chemical Vapor Deposition

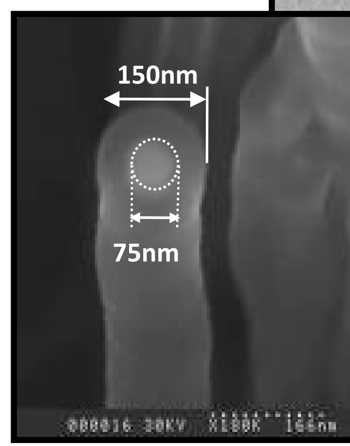
A **room-temperature coating** process which produces **thin coatings** on almost any material.

- “Gentle” application
  - low temperature
  - dry process
  - single-step
- Nano- to micro- meter thicknesses
  - Will not disrupt sealing properties
- Coatings are fully polymerized (no off-gassing, contamination)
- Conformal coating of complex seal geometries
- Scalable and manufacturable compared to competitive solutions





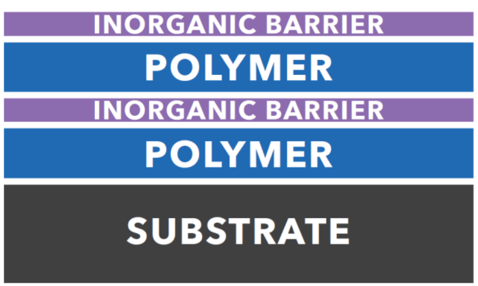




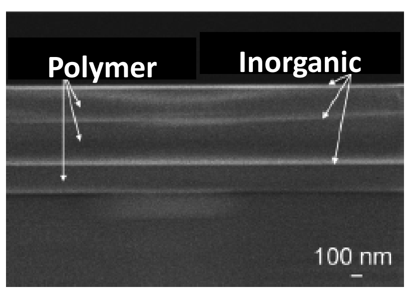
Coated Nanotubes

Approach: H_2 Barrier and Lubricious Coatings

- Flexible H_2 barrier coatings to slow hydrogen ingress into elastomeric seals
  - Thin inorganic layers provide **hydrogen barrier**
  - Polymer layers provide **flexibility**
  - Barrier layers deposit in the **same chamber** using the **same feed gas**
- Initially demonstrated by GVD founder Prof. Karen Gleason at MIT
- Barrier properties of coating driven by number of dyads - 100x reduction in water vapor transmission with three dyads §
- Maintains gas exclusion properties after hundreds of 180° bend cycles§

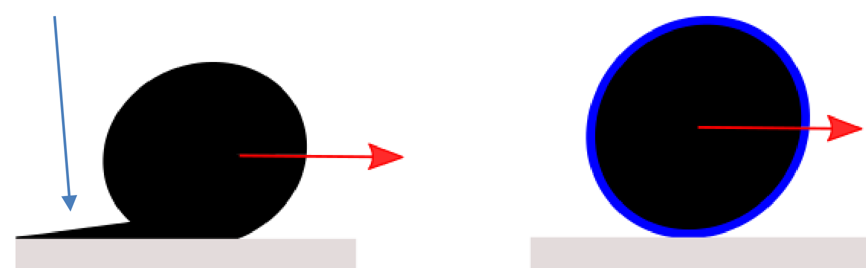


Barrier Coating Stack



- Lubricious coatings to reduce mechanical wear on seals
  - Seals move due to designed motion or swelling, thermal expansion
  - Thin PTFE film provides **low coefficient of friction** surface for reduced wear
  - PTFE's coefficient of friction: 0.03-0.05
- Excellent chemical and thermal stability
- Based on GVD's flagship mold release coating
- Annual revenues from related coatings ~ $5 MM

Material wear



Without coating   *With GVD PTFE coating*

§ Coclite et al., Plasma Process. & Polymers **2010**

Approach: Phase IIA Objectives

- Optimize polymer/inorganic H_2 barrier coatings using scaled PECVD processes
- Demonstrate a 10-fold reduction in relative hydrogen permeability with GVD barrier coatings
- Field validate a low-friction top coat of polytetrafluoroethylene (PTFE) for friction wear reduction of plastic piston-head seals
- Demonstrate improved seal life (goal of 3-5X increase) in field testing by a hydrogen compressor end user (NREL)
- Validate low-friction top coat for wear reduction on elastomeric seals

Approach: Prior Work Summary

H_2 Barrier Coatings

- Helium permeation reduction of 57% observed (compared to 35% previously) on 1 mm silicone.
- Hydrogen permeability did not match the helium permeability – due to particulate inclusion in films caused by modification of deposition chamber.

Lubricious Coatings

- Demonstrated 70% reduction in mass loss rate for hydrogen compressor gaskets with low-friction coating.
- Designed a production tool for commercial scale coating of seals.

2018 Collaborators

NREL
NATIONAL RENEWABLE ENERGY LABORATORY

High-temperature high pressure testing of compressor seals with lubricious coatings

Takaishi Industry Co.,Ltd.
高石工業株式会社

Provision and testing of o-rings

OAK RIDGE
National Laboratory

Hydrogen permeability measurements

Accomplishments 2018: H_2 Barrier Coating

- Eliminated particulate issues by revamping the electrode shape and aspects of the gas flow pattern.
- Improved understanding of coating composition and spatial variations thereof.
- Translated the composition to its likely impact on the permeability.
- Reconfigured chamber to eliminate spatial variations in film composition, achieve lower average permeability over reasonable areas.

Accomplishments 2018: H_2 Barrier Coating

- RF plasma is used to convert precursor gas to coating material
- Extent of conversion of the precursor depends on deposition conditions
- Fully converted films have better structural integrity and ion barrier properties (as measured at GVD)
- **Fully converted films have been shown to have better gas barrier properties (GVD and others)**

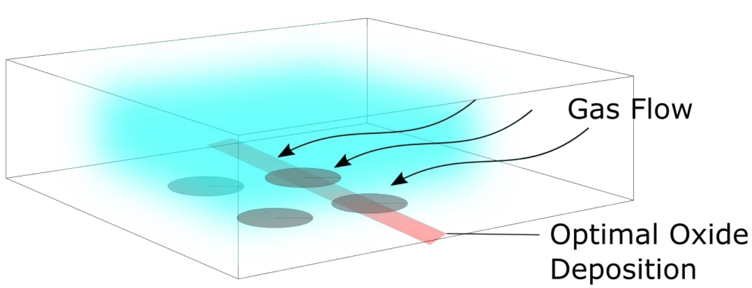
EXTENT OF CONVERSION →

• No coating formed	• Low carbon content • Mixed inorganic/organic • Medium density • Moderate ion exclusion • Poor barrier properties	• Carbon fully removed • Fully inorganic • High Density • Good ion exclusion • Good barrier properties

Deilmann *et al. Surface and Coatings Tech.* **2007**

Accomplishments 2018: H_2 Barrier Coating

- Extent of conversion depends on the deposition conditions *and* the position in the chamber.
- This knowledge led to immediate improvement in barrier properties of films– best permeation reduction seen so far 66% vs. PET (translates to 75% reduction vs. 1 mm silicone).
- Increased effective deposition zone size by 10x *via* a change in the chamber geometry.
- Deposition zone expansion will lead to better performance of coating and higher throughput.



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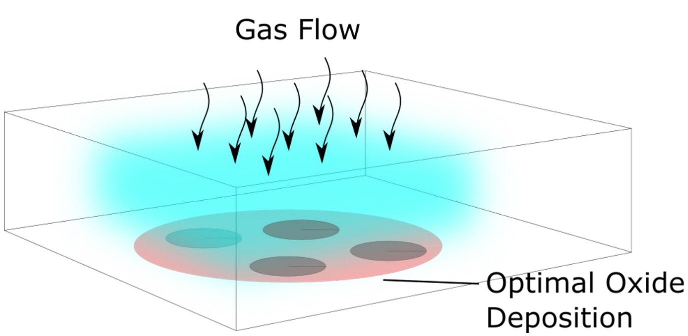
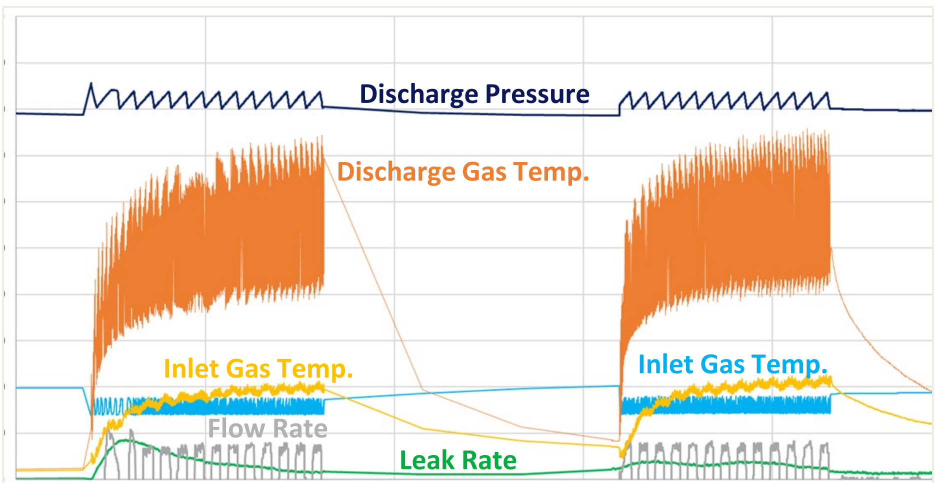


Current

Accomplishments 2018: Lubricious Coating

Aggressive Testing Protocol for H_2 Compressor Gaskets (NREL & GVD)

- Working with NREL to quantify improved lifetime of seals in HydroPac compressors
- Established test conditions for an aggressive thermal cycling with a plan for automated operation to accelerate results.
- ~50 hours of run time logged on control seals to date. Testing delayed due to equipment failure in January 2019.



Test Conditions: 30 min on-time, discharge Temperature: 120 °C, inlet pressure: 15-18 Mpa, max flowrate 7.5 kg/hr,

Endpoint: test until flowrate falls to 90% of initial value

Hydropac compressor similar to that used in NREL testing

NREL NATIONAL RENEWABLE ENERGY LABORATORY

Accomplishments 2018: Lubricous Coating

Elastomeric seals in hydrogen applications (Takaishi and GVD)

- PTFE-coated O-rings provided for testing to Takaishi Industries for hydrogen applications
- Testing in use-conditions showed 40x longer lifetime for coated O-rings and less visible wear than uncoated O-rings
- First commercial sale completed in January 2019

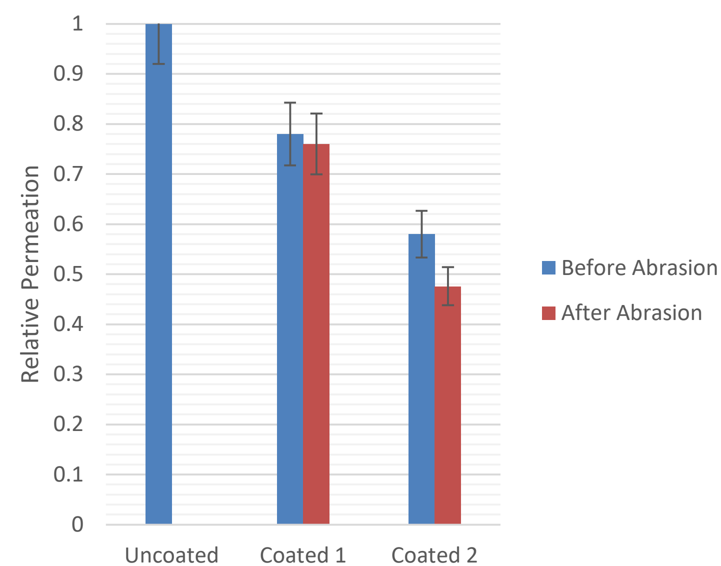
Example of the elastomeric seals coated by GVD

Seals used for testing

Takaishi Industry Co.,Ltd.
高石工業株式会社

Accomplishments: Responses to Reviewers

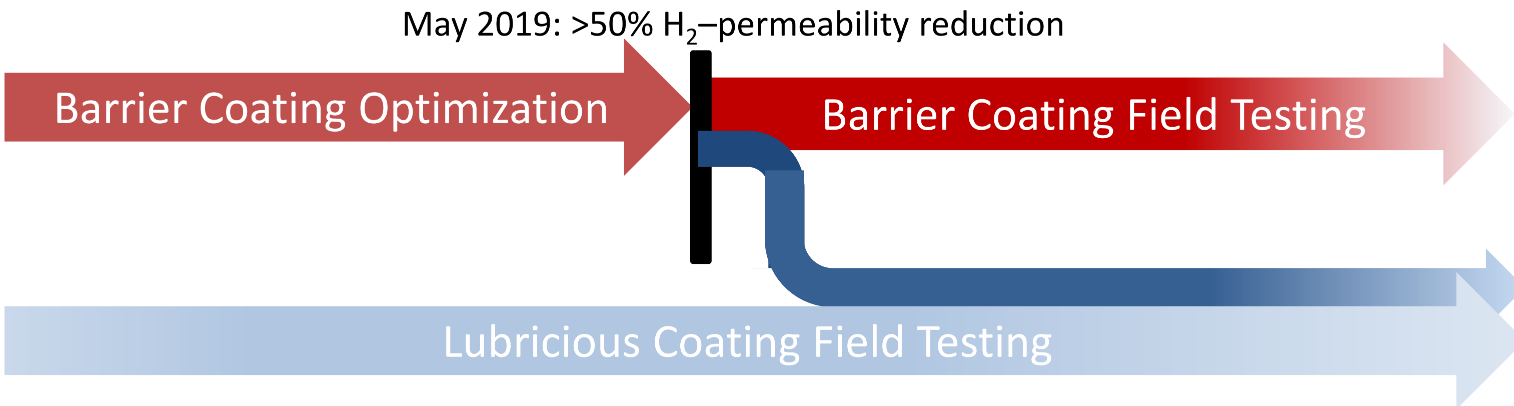
- Application-induced Defects of the Coatings
  - Coatings maintained barrier properties after abrasion.
- Thermal Stability
  - Polymer in barrier coatings stable between -40 °C and 200 °C.
  - Barrier layer undergoes no change in thickness or composition (densification) up to 300 °C.
  - No decomposition of PTFE until 400 °C. HTHP conditions include discharge temps of 200 °C.
- Coating Uniformity
  - Macroscopic uniformity explored in the work done this year.
  - Major improvements expected from improving the uniformity of composition and deposition rate.



Patrick, Nature 1958
Wall, Michaelson, National Bureau of Standards 1956

Proposed Future Work

GVD will prioritize commercialization-readiness with a stage gated work plan.



- Barrier Coatings (goal: 90% H_2 permeation reduction)
  - If barrier properties at low pressure helium are reflected in high pressure H_2 permeability measurements, GVD to pursue scale-up and field testing
  - GVD has previously designed a large scale coating tool for this application
- Lubricious Coatings (goal: 3-5x lifetime improvement)
  - Continued testing planned at NREL in high temperature/high pressure compression
  - Pursuing expansion of testing with Takaishi
  - Currently building commercialization partnerships e.g. in communication with an interested hydrogen fuel cell forklift system manufacturer

Any proposed future work is subject to change based on funding levels.

Proposed Future Work: Technology Transfer Activities

- Commercial Interest and Inquiries:
  - Hydrogen Compression & Storage
  - Hydrogen Refueling Stations
  - Hydrogen Purification
- Potential Commercial Partnerships
  - Seal manufacturers
  - Hydrogen compressor manufacturers
  - Valve manufacturers
  - H_2 Fuel Cell Systems Providers
- Intellectual Property
  - IP established based on MIT proof of concept
  - Additional GVD patents to be submitted as required

Any proposed future work is subject to change based on funding levels.

Summary

Objective: Reduce costs to Hydrogen Fuel Cell Electric Vehicles and hydrogen processing systems associated with Hydrogen Compressor seal failure. Improve seal life 3-5X.

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 static seals, preventing premature failure. Low friction coatings that reduce wear of dynamic seals, extending seal life.

Accomplishments: GVD's coatings have demonstrated significant reduction in helium permeation through polymer material and significant reduction in mass loss for dynamic seals.

Future Work: Drive toward commercialization by demonstrating barrier performance in relevant testing environments (HTHP Hydrogen) and demonstrating reduced wear for low friction coatings in a full operational environment.

Thank you

Questions?

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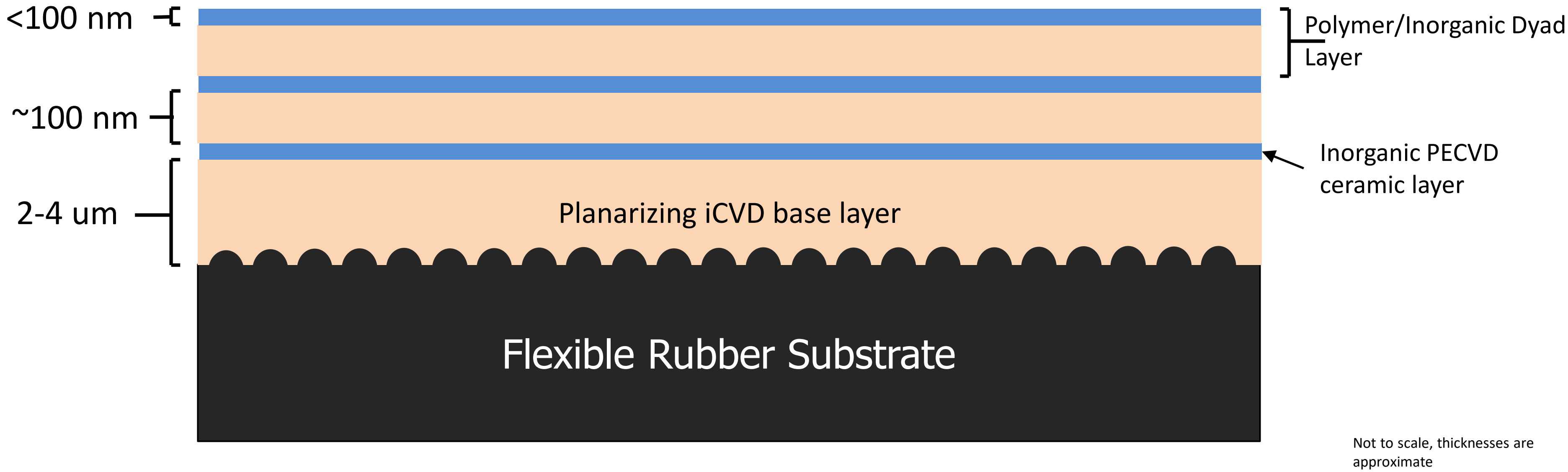
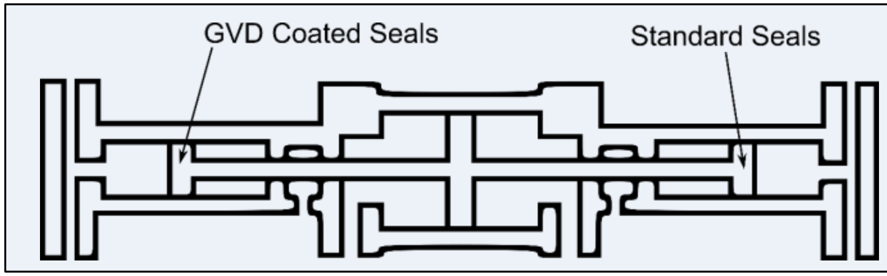
Technical Backup Slides

Technical Back-Up: Barrier Coating Construction



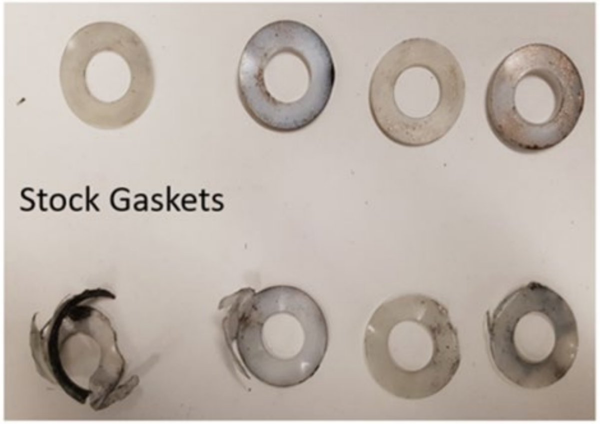
- Multiple dyads force the H_2 molecules to forge a tortuous path through nanodefects in the coating surface
- Number of dyads and coating thicknesses were optimized for maximum flexibility, planarization and minimum Hydrogen ingress

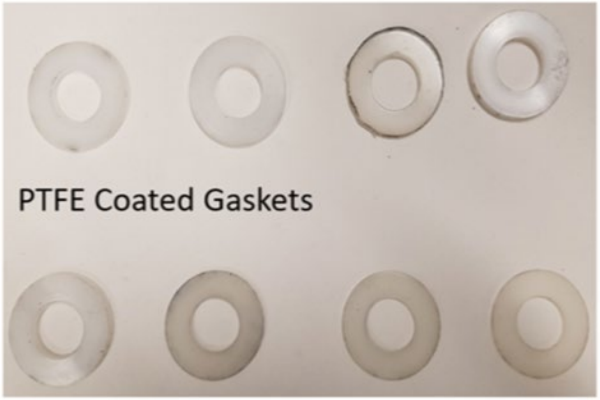
Tech. Backup: Reduction in Compressor Seal Wear



Schematic of gasket seal locations in Hydro-Pac compressors. Tested by Powertech

- Side-by-side testing of coated versus uncoated hydrogen piston gaskets
  - >70% reduction in mass loss observed
- No failure of coated gaskets during testing
  - Samples pulled at uncoated failure per protocol
  - Future testing will allow independent lifetime assessment





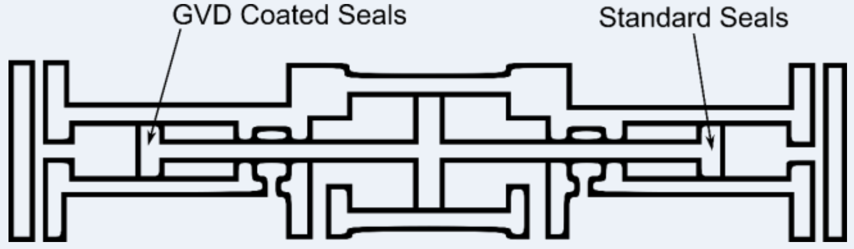
Technical Backup: Helium Permeability Testing

Sample	Average Permeation Reduction	Standard Deviation	SiOx/Exilis (nm/nm)	Bilayer Thickness (nm)
Neat Silicone	0.0%	-	-	-
Barrier Coating	48.8%	7.6%	100/100	2000
Barrier Coating	50.7%	3.3%	70/100	2000
Barrier Coating	54.2%	8.0%	50/100	2000
Barrier Coating	**57.3%**	9.7%	35/100	2000

- Samples prepared and sent to ORNL for H_2 testing
  - No improvement in permeability seen
  - Plan to perform helium testing at ORNL in Phase IIA for validation before further H_2 work

Technical Backup: PowerTechCompressor Seal Testing

- Abbreviated Testing Procedure
  - Weigh gasket before cycling
  - Record compressor run time until failure
  - Measure weight after failure



Schematic of gasket seal locations in Hydro-Pac compressors. Tested by Powertech

Example of a Hydro-Pac seal received by GVD prior to coating.

Stock Gaskets from Hydropak

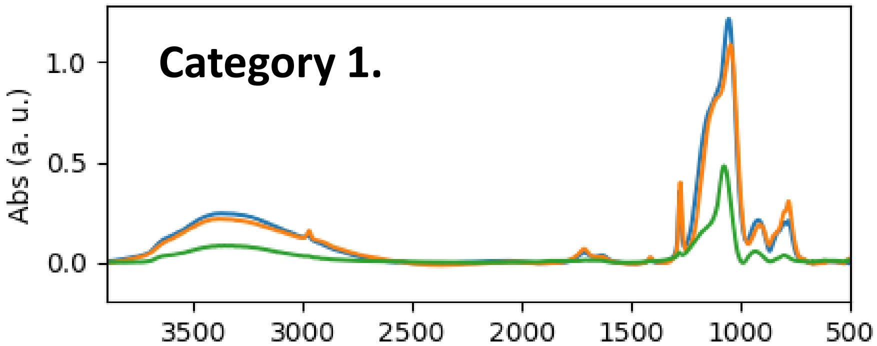
Total Initial Mass	11.76g
Total Final mass	10.95g
Ttl. Delta:	0.81g
Average Delta:	0.10g
Run Time	20.63h
Depletion rate	4.933mg/h

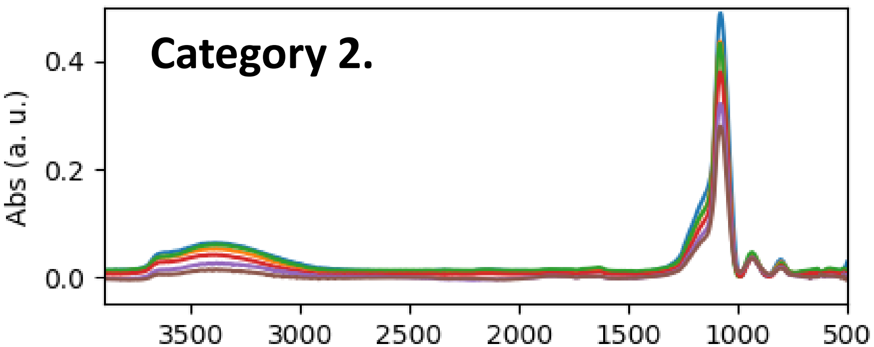
GVD Coated Hydropak Gaskets

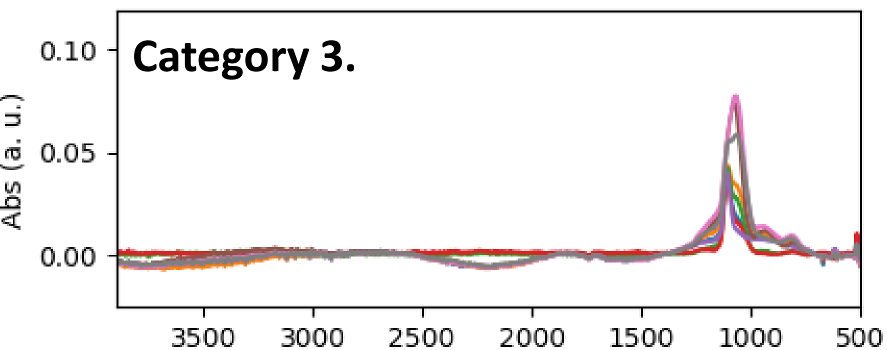
Total Initial Mass	11.79g
Total Final mass	11.61g
Ttl. Delta:	0.18g
Average Delta:	0.02g
Run Time	20.63h
Depletion rate	1.095mg/h

$$Depletion\ Rate\ \left({}^{mg}/_{hr}\right) = \frac{Initial\ Mass\ (mg) - Final\ Mass\ (mg)}{Runtime\ (hr)}$$

Tech Backup: FTIR – Structural categorization







FTIR of SiOx films allows us to distinguish three categories of films.

- ❑ Category 1 – Highly organic
  - ◆ Still has features of C-H and O-H bonds.
  - ◆ Strong peak near 1010 cm-1 – (indicates SiO bonding of precursor is intact).
- ❑ Category 2 – middle range
  - ◆ OH bonds still present, C-H minimal.
  - ◆ SiO bonding peak shifts to 1080 cm-1.
- ❑ Category 3 – highly glassified
  - ◆ Complete disappearance of OH and CH.
  - ◆ SiO region indicates major restructuring.
  - ◆ Category 3 corresponds to PASSING BOTH pinhole tests.