III.8 Advanced Barrier Coatings for Harsh Environments

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Overall Objectives

- Optimize polymeric and inorganic layers using initiated chemical vapor deposition (iCVD) and plasma enhanced chemical vapor deposition (PECVD) processes.
- Demonstrate a 10-fold reduction in hydrogen permeation through coated elastomeric substrates.
- Develop a low-friction top coat of polytetrafluoroethylene (PTFE) for friction wear reduction of plastic piston-head seals.
- Demonstrate improved seal life in field testing by a hydrogen compressor end user.
- Develop a coating system to provide conformal seal coatings at reasonable cost.

Fiscal Year (FY) 2016 Objectives

- Optimize polymeric and inorganic layers using iCVD and PECVD processes.
- Select lubricious coating parameters to maximize dynamic seal wear reduction.
- Demonstrate a 10-fold reduction in hydrogen permeation through coated elastomeric substrates.
- Initiate field testing of GVD coatings in hydrogen compression systems.

Technical Barriers

This project addresses the following technical barriers from the Hydrogen Delivery section of the Fuel Cell Technologies Office Multi-Year Research, Development, and Demonstration Plan.

(B) Reliability and Costs of Gaseous Hydrogen Compression

Technical Targets

This project is conducting fundamental studies of coatings to improve seal life for hydrogen compressor systems. Insights gained from these studies will be applied toward the design and manufacture of coatings applied to seals used in hydrogen delivery systems to help meet the following FY 2020 DOE targets.

- Annual Maintenance: 4% of installed capital cost
- Losses: 0.5% of H_2 throughput
- Small Compressor Lifetime: 10 years

FY 2016 Accomplishments

- Designed and optimized a barrier coating deposition system for scalability to enable high throughput and reduced cost of coating application in commercial production.
- Validated optimal barrier coating properties through chemical analysis, adhesion testing and acetone soak testing.
- Initiated head-to-head compressor seal trials using a PTFE coated and uncoated seal, periodically evaluating wear via seal mass and effective compressor operation, to compare seal life improvement in a real-use environment.

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INTRODUCTION

A key cost driver in hydrogen compression systems is the frequent maintenance required to replace seals that fail in operation. Seals fail under a variety of conditions, but primary factors are hydrogen ingress and wear in the high pressure, high temperature dynamic operating environment. Seal failure is a major contributor to process downtime and is the largest cause of unscheduled maintenance, the cause of >25% of hydrogen leaks, and redundant compression is often specified to alleviate the issue.

GVD Corporation specializes in thin-film coatings applied using a low-temperature vapor deposition process. One of these films, a polysiloxane material branded Exilis which is currently used by GVD to provide corrosion protection and electrical insulation to circuit boards, was modified in a publication by GVD's founding laboratory at the Massachusetts Institute of Technology to act as a vapor barrier. GVD identified a clear opportunity to use our expertise with Exilis and commercializing coating technology to pursue a solution to problems plaguing the hydrogen processing industry. This project has resulted in the commercial scale up and manufacture of a production system to deposit this effective barrier coating, previously only evaluated in an academic setting. In partnering with compressor manufacturers and seal designers, GVD will have real-operation data demonstrating improved seal-life which will reduce the annual maintenance costs for hydrogen compressor systems. The project targets an improvement in seal life from the current <1,500 hr to >8,000 hr (a 5X reduction in maintenance frequency) and enables reduced cost for hydrogen operation and delivery.

APPROACH

GVD's approach to improving seal life and reducing maintenance for hydrogen compression systems uses two types of coatings: a vapor barrier and a low friction coating. Initial evaluations validate and optimize these two coatings to provide the most effective solution for improving seal life. The barrier coating is optimized to reduce permeability of hydrogen into the elastomer seal; hydrogen ingress accelerates seal degradation and wear. The low friction coating is optimized to reduce the amount of wear seen in dynamic seal operation, extending the seal life.

Partnership with national laboratories, compressor manufacturers and seal manufacturers allow the development work performed by GVD to be tested in laboratory environments that closely mimic the real world as well as field testing in systems used for production, resulting in meaningful data that demonstrates improved seal life. Hydrogen permeation tests are performed comparing a barrier coated elastomer and an uncoated elastomer made of seal materials typically used in hydrogen compression systems. The best performing barrier coatings are optimized in a scaled coating system developed during this project. Seal wear testing of the low friction coating was initially evaluated using tribometry to identify a coating with optimal lubricious properties. The best performing low friction coating is then tested in a production environment against an uncoated seal, using dynamic seal mass loss as an indicator of reduced wear in operation.

RESULTS

The first phase of this project was to optimize the polymeric and inorganic layers of the barrier coating by changing from an alternating deposition process between iCVD and PECVD to have both layers deposited via plasma-iCVD for increased throughput and processing simplification. GVD developed an alternative plasma-based process for depositing the Exilis polymeric layers. This development was carried out in GVD's iMax system, using a purpose-built radio frequency electrode assembly. Prior plasma deposition was performed using the same hot-filament arrangement used for iCVD of Exilis. Using a hot-filament arrangement is not ideal, since the generated plasma tends to be non-uniform and on both sides of the assembly, resulting in excessive deposition elsewhere in the chamber.

Initial development work focused on choosing the optimal deposition pressure and flow rates for the film precursors as well as radio frequency input power. GVD identified an organic layer deposited by plasma-iCVD with nearly identical chemical structure to the organic layer deposited by iCVD. Optimization of the plasma-iCVD organic layer resulted in:

- Improved coating uniformity, +/-10% over a 10 in by 10 in area.
- Faster growth rates, 25 nm/min, a 4–5X increase over iCVD
- Reduced precursor use, 18 times less compared to iCVD

These improvements will aid in throughput and cost reduction for commercial production. Optimized plasma-iCVD coatings were evaluated for adhesion (Figure 1) and flexibility, with the best performing coating used for vapor permeation testing.

GVD is testing hydrogen permeation with Oak Ridge National Laboratory. Testing methods resulted in damage to the coating on the elastomer material; data showed a permeability of 1.33×10^{-11} mol-m/(m^2-s-Pa) which resulted in only a 5% reduction in hydrogen permeability at 3 µm of coating thickness versus an uncoated substrate. This contrasted starkly with the helium permeability GVD measured on undamaged samples of 5.23×10^{-13} mol-m/(m^2-s-Pa), which would have resulted in a 70–90% reduction at 3 µm of coating thickness. GVD is working with Oak Ridge National Laboratory to optimize

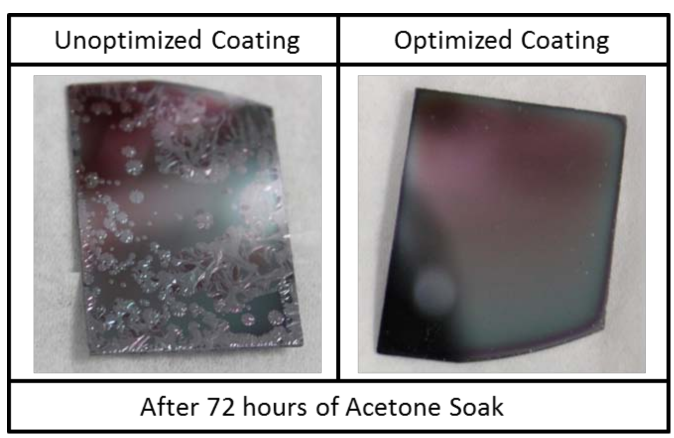

FIGURE 1. Comparison of plasma-iCVD coatings before and after optimization

the testing method to reduce damage to the coating prior to testing and further optimize the barrier coating for rugged use.

GVD has developed a lubricious PTFE coating that is undergoing field testing with a PowerTech, an industrial user of Hydro-Pac's hydrogen compression systems. The PTFE coating was selected after yielding satisfactory results during tribological testing. The lifetime of the seals will be compared to uncoated seals installed simultaneously in a double-ended single stage hydrogen compressor with an inlet pressure of 5,000 psi and hydrogen compression pressure of 13,500 psi. Mass measurements are periodically taken to demonstrate wear between 50 hr and 200 hr of use, at 500 hr, then after failure. Approximately 100 hr of use has been achieved thus far; testing is in too early a stage to expect any meaningful difference between coated and uncoated seals.

GVD has completed the construction of a low-volume scaled system for plasma-iCVD. This system is adapted from an existing system which had been used as a high-vacuum oven. Figure 2 shows the system after removing the high-vacuum components (diffusion pump, ion gauge, etc.). The chamber is a cylinder, and has a deposition zone of approximately 35 in by 18 in. This new deposition chamber has been dubbed Omega.

FIGURE 2. Original system

The system's gas distribution was designed using fluid flow simulation in SolidWorks. A simple inlet tube without any diffusion would result in vortices and regions of stagnation, particularly near the door of the chamber (Figure 3). Several different types of gas distributors were designed in SolidWorks and then tested in simulation. A gas distributor was finally settled on as a solution that is easier to manufacture while still resulting in good laminar flow in the chamber (Figure 4).

CONCLUSIONS AND FUTURE DIRECTIONS

GVD has demonstrated the scalability of the technology by:

- Transitioning from a two-system deposition process to a single-system deposition process, including validation of coatings.
- Designing, building and validating a production coating system.

GVD has demonstrated the efficacy of the coating to improve seal life by:

- Optimizing low friction coatings for use in high temperature high pressure hydrogen compressor use environments.

Future work includes:

- Characterize hydrogen permeability of the barrier coating with a revised test method at Oak Ridge National Laboratory.
- Continue to generate wear and seal-life data with the low-friction coating.
- Design a plasma tumble-coating process for the production system.

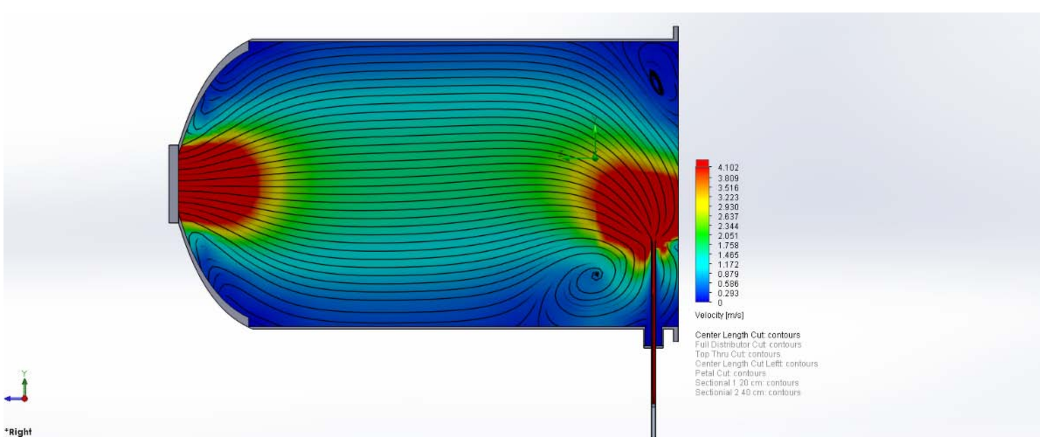

FIGURE 3. Gas flow simulation in Omega system, no gas distribution

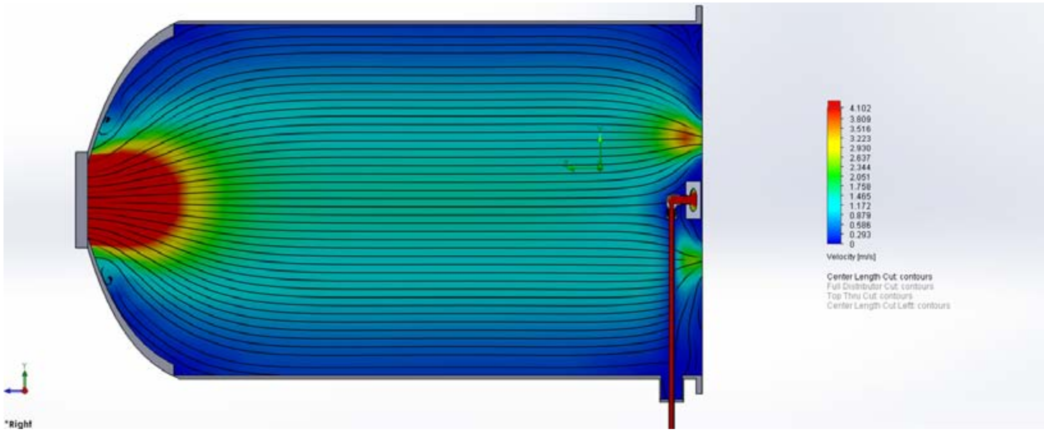

FIGURE 4. Gas flow simulation in Omega system with selected gas distributor

FY 2016 PUBLICATIONS/PRESENTATIONS

1. W. O’Shaughnessy, “Advanced Barrier Coatings for Harsh Environments,” 2016 Hydrogen and Fuel Cells Program Annual Merit Review. Presentation.