

## III.7 700 bar Hydrogen Dispenser Hose Reliability and Improvement

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Project End Date: Project continuation and direction  
determined annually by DOE

### Overall Objectives

- Working closely with original equipment manufacturers (OEMs) SpirStar and other groups developing advanced high pressure hydrogen hoses, NREL's hose reliability project aims to improve the reliability of 700-bar hydrogen refueling hose assemblies, and ultimately reduce the cost of dispensing hydrogen into fuel cell electric vehicles by identifying and characterizing points of failure.
- Operate a fully automated test system that unifies the four stresses of pressure, temperature, time, and bending. The test apparatus will reveal the compounding impacts of high-volume 700-bar fuel cell electric vehicle refueling that has yet to be experienced in today's low-volume market. Testing includes pre- and post-cycling chemical and physical analysis of the inner hose liner to determine any relative changes in bulk properties and degradation mechanisms due to the stress of repeated fueling events.

### Fiscal Year (FY) 2017 Objectives

- Continue hose cycling towards 25,000 cycles or until failure using the test apparatus that unifies the stresses to which the hose is subjected during high volume fueling events.
- Gather and analyze data on hydrogen leakage rates, timing and sources through the use of a vacuum sampling pump system with combustible gas

detectors and the deployment of chemochromic leak indication tape.

- Use data and observations to help inform preventative maintenance schedules and standards development for hydrogen stations.

### Technical Barriers

This project is conducting applied research, development, and demonstration to reduce the cost of hydrogen delivery systems. 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:

- (I) Other Fueling Site/Terminal Operations
- (J) Hydrogen Leakage and Sensors

### Technical Targets

This project aims to generate data that will help OEMs and hose developers improve reliability and replacement intervals for high-pressure gaseous hydrogen dispenser hoses. This data provided by this project will ultimately reduce the cost of hydrogen delivery from the point of production to the point of use in consumer vehicles by providing robust dispenser operation with less maintenance costs and improved customer satisfaction.

- Target Hose Replacement Interval: 25,000 cycles
- Target Cost of Hydrogen Delivery: <\$2.00/gge by 2020

### FY 2017 Accomplishments

- Completed over 5,000 cycles on Hose Assembly #2 running various cases of SAE J2601 H70-T40 cases with excellent accuracy on pressure controls.
  - Average mass of 200 g consumed per cycle after precooling upgrade completed.
  - Selected cases of H70-T20 and H70-T30 run for comparison of leak rates as a function of hose gas temperature.
- Detected and investigated a leak pattern from crimp fittings on hose at the nozzle end.
  - Vacuum-based active sampling system detected several consistent, but small leaks from the crimp fitting on the nozzle end of the hose starting at Cycle 3033.

- Leaks were observed through all parts of cycles and pressure holds, especially during higher pressures and during venting between cycles.
  - The leak is not isolated to the nozzle end crimp fitting, but was also observed to permeate through the hose layer for up to 25 cm from the crimp fitting, without visible damage or blistering on external surfaces. Chemochromic leak tape provides visual confirmation of this leak activity.
  - Hose continues to pass all standard leak checks, safety interlocks and pressure holds, with a calculated mass loss of up to 500 µg/s.
  - Multiple safety features implemented as part of experiment design allowing hose to be run throughout leaks without risk.
- Results and findings were shared with OEMs, field station operators, and codes and standards groups to help inform inspection requirements and standards development.



## INTRODUCTION

Operation and maintenance costs of dispensing are a large part of the cost of hydrogen stations. NREL has found that about 41% of maintenance hours for hydrogen fueling retail stations are associated with dispensers, with about 10% of those hours attributed to failed parts. This data can be found in NREL's infrastructure composite data products (CDPs) CDP-INFR-21 and CDP-INFR-24 [1]. These CDPs provide an early look at maintenance and reliability issues of the retail 700-bar vehicle refueling stations. Station operators have reported that they are replacing the high-pressure hoses earlier than expected in intervals of a few months. High-pressure hydrogen refueling hoses, compared with their gasoline counterparts, are significantly more expensive, and replacement intervals are much shorter. Hoses are not as high of a capital cost item (\$2,000) compared with the hydrogen refueling nozzle (\$7,000 for nozzles without infrared comms and \$11,000 for nozzles with infrared comms) and breakaways (\$3,000), however, nozzles and breakaway typically can be refurbished or serviced at a lower cost while hoses must be completely replaced. **Currently, the high frequency of hose replacement results in the high-pressure hoses being a significant operations and maintenance cost to the station operator.** Accelerating the cycle rate, monitoring the leakage patterns, and continuing past the point of typical replacement can supply valuable data on post-cycled specimens to share with OEMs and improve reliability through this project.

## APPROACH

This project aims to perform long-duration accelerated life testing on commercial or prototype hose assemblies using high-pressure, low-temperature hydrogen to achieve realistic precooled fueling conditions closely following the SAE J2601-2014 fueling protocol. This work is unique and goes beyond standard OEM and certification standards agency acceptance testing in that it simultaneously stresses the hose assembly by applying mechanical bending and twisting stress to the hose and nozzle assembly to simulate people refueling vehicles. The short cycle time achieved with this system models the demand of a busy gas station where the dispensing equipment is kept cold most of the time and subjected to frequent decompression and occasional thermal cycles.

The main difference between the test plan and a high volume station is that the mass dispensed per fill is less than the 3–5 kg of a typical vehicle fill. To prevent overtaxing the production and compression capabilities of NREL's hydrogen system, the target mass dispensed per fill is 100–200 g. Back-to-back filling maintains hose temperatures under the Cold Dispenser cases of SAE J2601. Due to the low viscosity and mass of hydrogen, the additional shear stress on the hose wall from mass flow is negligible compared to the thermal stress and radial stress from pressurization cycles.

A hose reliability test stand, shown in Figure 1, was developed to support full 700-bar fueling simulation capabilities. The test stand uses a six-axis robot using pre-programmed motion paths to capture normal and realistic stresses resulting from friendly human interaction with the hose assembly while maintaining a compact footprint to safely operate in the 9.3 m<sup>2</sup> High Pressure Test Bay, which offers a safe and controlled environment to test components under high pressure to failure while minimizing risk to personnel or equipment. The test stand closely mirrors an actual dispenser in its design and pressure ramping capabilities. A tankless control algorithm was successfully developed using the interaction of an air-loaded pressure regulator on the dispenser side of the test apparatus and flow control valves on the vehicle side.

The leakage rate of the hose is monitored using a vacuum-pump sampling system attached to an outer protective sleeve near each flared crimp fitting to identify hydrogen leaks during the fueling cycles. The sampling flow rate is set to 400 mL/min and was calibrated to measure delayed response times. Chemochromic leak indication tape is also wrapped over the hose end assemblies to further identify exact methods of leakage, and potential inspection methods are verified at regular intervals. Data collected from the hose reliability test stand include pressure, temperature and real-time leakage rates from crimp fitting areas. These data can be used to explore dependency of leak intensity on current pressure and temperature. Permeation of hydrogen



**FIGURE 1.** Hose reliability test stand

through the polymer inner layers is a potential source of non-destructive leakage. Permeation activity typically follows the Arrhenius rate equation and is reduced with lower temperatures. However, temperatures that drop close to the glass transition temperature increase the brittleness of the polymer and the likelihood of internal damage to the polymer, allowing for easier permeation. Plotting the natural logarithm of the permeability rate and the inverse temperature allows correlation to the Arrhenius relationship and insights into the activation energy, or the ease of permeation of hydrogen through the polymer. Similar relationships have been tabulated for comparable polymers but a study has not been carried out for hydrogen and polyoxymethylene [2].

The project also includes analysis of the physical and chemical property changes of the inner hose liner due to long duration hydrogen cycling. Chemical tests previously identified and performed on pre-cycled inner hose specimens include scanning electron microscopy to ultimately identify blistering due to hydrogen permeation and characterization testing to identify material degradation and compositional changes such as Fourier transform infrared spectroscopy, thermogravimetric analysis, differential scanning calorimeters, X-ray spectroscopy and dynamical mechanical analysis methods. These tests will be repeated on post-cycled inner liner samples to compare any changes or degradations in the polymer properties.

## RESULTS

The hydrogen detection system began sensing leaks on the nozzle-side crimp fitting at Cycle 3,033. While some leaks were also detected on the dispenser side crimp fitting earlier at Cycle 1,856, those leaks have been inconsistent. The

amount of hydrogen lost per cycle is relatively small, with a mass loss rate of up to 500  $\mu\text{g/s}$ , originating primarily from the nozzle-side crimp fittings. Since the leak rate is smaller than many instrumentation tolerances, pressure checks pass and these leak rates almost certainly go undetected in the field. There is only one current code and standard that has defined numerical criteria for leakage thresholds, currently set as 10 ccN/h, or 3,235  $\mu\text{g/s}$  [3]. On the NREL hose test apparatus, over 1,500 cycles have been completed on the same hose assembly that exhibit signs of leakage without failure. The leak patterns can be seen in Figure 2, trending upwards with pressure and spiking during depressurization and motion back to the dispenser. This could possibly indicate leakage from the plastic to metal seat during venting, blistering of the inner layer from depressurization, or cracking of the inner liner during motion.

Typical cycles run with T20, T30, and T40 temperature profiles and longer-term (10 min) pressure holds at 82 MPa were analyzed for permeation effects. The typical cycles were observed to have leak rates that generally increased with pressure and decreased with temperature, with a notable increase in leak activity at temperatures around  $-40^\circ\text{C}$ , close to the glass transition temperature previously measured as  $-50^\circ\text{C}$ . Many pressure holds on the hose assembly, typically those immediately following a cold cycle, exhibit a strong exponential relationship between leak rate and temperature. Other, warmer holds performed during idle periods still leaked, but showed weaker correlations with temperature as shown in Figure 3. Activation energies of 21.9–43.8 kJ/mol were reported for the sample pressure holds shown.

Inspection methods were explored to help inform preventive maintenance requirements and codes and standards development, with a particular interest in finding out if this type of leak may be indicative of early failure of

### Sample of Typical T40 Cycle Set

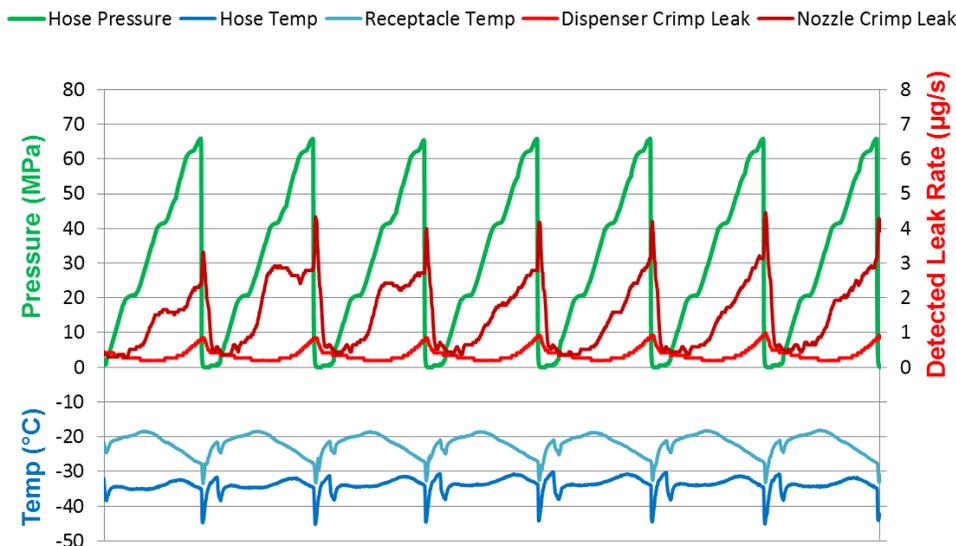


FIGURE 2. Sample of hose cycle pressure profile set, showing leak trends

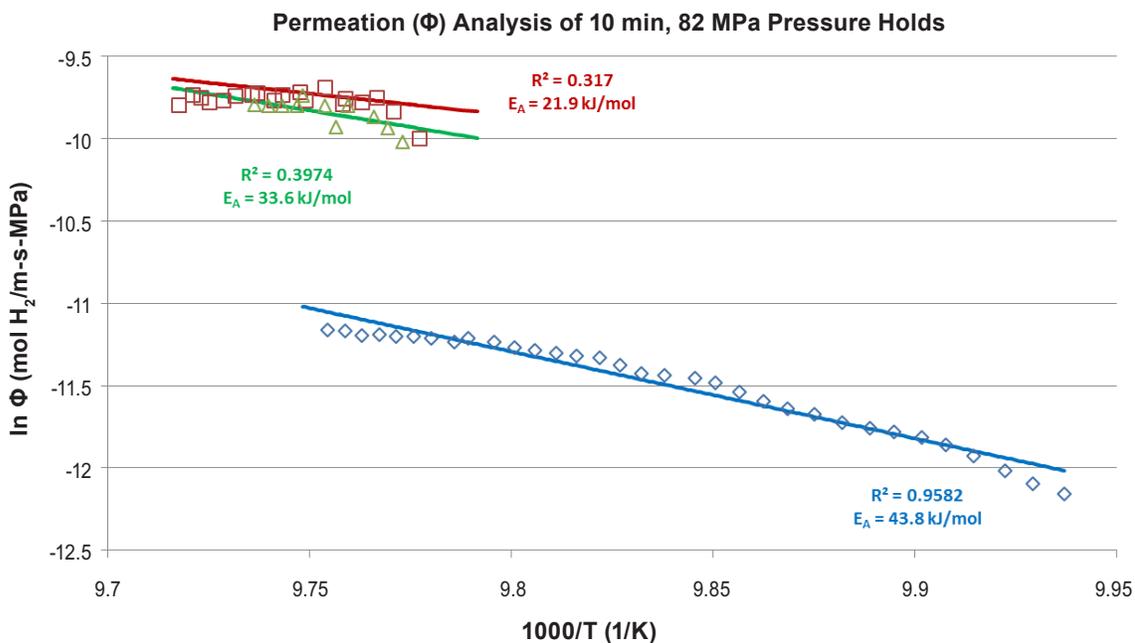


FIGURE 3. Analysis of permeation activity dependency on hose temperature

hose assemblies. This level of leak being reported is too small to be audible by operators and there is no visible damage on the outer layer. However, commonly available handheld combustible gas detectors can reliably detect leaks of this size. Hose sample #2—the one currently under test—shows stronger leak intensity near the crimp fitting, decreasing up to 25 cm from the crimp fitting. Hose Assembly #1 was burst

tested at the beginning of the project to confirm the hoses from this batch were meeting the manufacturer’s criteria.

A reliable inspection method may be available by using chemochromic leak indication tape that darkens upon contact with hydrogen. A sample was donated by ElementOne and deployed on both ends of Hose Sample #2, with control tapes deployed on NREL’s hydrogen dispenser as well. After

1,000 cycles on the test apparatus, the tape clearly showed heavy leaking from the nozzle side crimp fitting through the pinpricks designed to relieve pressure from permeation of hydrogen, shown in Figure 4. The dispenser side crimp fitting, which had inconsistently leaked smaller amounts, showed fainter but still legible leak marks. In comparison, NREL's dispenser hose, which had filled about 150 times during the same time frame with similar mechanical stressing, had zero leak indications, confirmed with a handheld detector. Chemochromic tape may be very useful to station operators as an inspection method to identify weakened hoses before failure.

Data from these studies have been shared with the hose OEM, field station operators, and with standards groups such as ISO 19880-5 WG22 to help inform development.



**FIGURE 4.** Chemochromic leak indication tape on nozzle-side hose end

## CONCLUSIONS AND UPCOMING ACTIVITIES

- Conclusion: Hydrogen leaks were first detected at the nozzle end of the Hose Sample #2 at Cycle 3,033, consistently leaking throughout several hundred cycles at a rate of about 500  $\mu\text{g/s}$ . This leak rate does not fail standard leak checks and pressure holds, and passes current code criteria. However, it may indicate increased permeation and risk of eventual failure due to weakened inner material.
- Conclusion: Inspection methods were verified, with handheld gas detectors and chemochromic leak tape being shown as highly reliable and easy to check. Station operators may be able to use these methods to help identify hoses at risk of failure.
- Upcoming: With remaining funding, continue testing Sample #2 beyond 5,000 cycles until failure or significant degradation, and perform post-cycle scanning electron microscopic imaging, physical dynamical mechanical analysis and chemical composition testing previously identified.
- Upcoming: With future external funding, hoses from additional manufacturers could be tested and verified under similar test conditions and materials testing.

## FY 2017 PUBLICATIONS/PRESENTATIONS

1. Joe Pratt, Chris Ainscough, Danny Terlip, Terry Johnson, Mike Peters, and Ethan Hecht, "H2FIRST Tasks and Impact," presented to the California Fuel Cell Partnership, December 2016.
2. Kevin Harrison and Owen Smith, "700 Bar Hydrogen Hose Reliability Project," presented to the Hydrogen Delivery Tech Team, November 2016.

## REFERENCES

1. Sprik, Sam, Jennifer Kurtz, Chris Ainscough, Genevieve Saur, Michael Peters and Matthew Jeffers. "Next Generation Hydrogen Station Composite Data Products - Data through Quarter 4 of 2016." Hydrogen Fueling Infrastructure Analysis. May 2017. <http://www.nrel.gov/docs/fy17osti/66555.pdf>.
2. San Marchi, C., B.P. Somerday. "Technical Reference for Hydrogen Compatibility of Materials," Sandia National Laboratory Technical Report SAND2012-7321. September 2012.
3. CSA Group. "Hoses for compressed hydrogen fuel stations, dispensers and vehicle fuel systems," ANSI/CSA HGV 4.2-2013. 2013.