III.11 700 bar Hydrogen Dispenser Hose Reliability Improvement

Kevin Harrison (Primary Contact), Owen Smith
National Renewable Energy Laboratory (NREL)
15013 Denver West Parkway
Golden, CO 80401-3305
Phone: (303) 384-7091
Email: Kevin.Harrison@nrel.gov

DOE Manager: Erika Gupta
Phone: (202) 586-3152
Email: Erika.Gupta@ee.doe.gov

Subcontractor:
Marc Mann, Spectrum Automation Controls, Inc.,
Arvada, CO

Project Start Date: July 2013
Project End Date: Project continuation and direction determined annually by DOE

Overall Objectives

- Working closely with original equipment manufacturers (OEMs) Spir Star, Yokohama Rubber, and other groups developing advanced high-pressure hydrogen hoses, NREL's hose reliability project aims to characterize and 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 points of failure.
- The project will 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) 2016 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 back-to-back fueling events, following profiles as close to technical specification SAE J2601 fueling protocol as possible with the exception of total mass dispensed per cycle.
- 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.

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 lower maintenance costs and improved customer satisfaction.

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

FY 2016 Accomplishments

- Completed 3,500 cycles on Hose Assembly #1 from Spir Star with 700-bar hydrogen precooled to -40°C (H70-T40) using various SAE J2601 fueling protocol cases with good accuracy on pressure controls.
  - Average mass of 57 grams consumed per cycle after precooling completed.
  - Station upgrades and automation implemented to increase high-pressure hydrogen capacity and recovery and to enable unattended hose testing to increase future cycle rate.
- Detected and investigated a leak pattern from dispenser end of Hose Assembly #1.
  - Vacuum sampling pump system detected several small leaks from the crimp fitting on the dispenser end. The leak pattern was not consistently reproducible between sets.
  - Data gathered shows leak seems to occur mainly on depressurization, only after several cold cycles—no clear pattern identified yet.

– Multiple safety features were implemented as part of experiment design, allowing the hose to be run throughout leaks without risk.

✧ ✧ ✧ ✧ ✧

INTRODUCTION

NREL operates and maintains a unique user facility known as the Energy Systems Integration Facility (ESIF). The ESIF houses a broad array of capabilities and laboratories focused on energy integration research, where fast and flexible swapping of research test articles is a hallmark of the hydrogen infrastructure testing. NREL partners with DOE, as well as hydrogen industry and market stakeholders, in order to provide critical testing and validation all along the product research and development process. NREL's approach to integrated systems testing simplifies the interfaces between hydrogen production, compression, storage, delivery, and end use systems. The hose reliability test stand is housed in the ESIF's High Pressure Test Bay (HPTB), which offers a safe and controlled environment to test components under high pressure to failure while minimizing dangers to personnel or equipment.

Operation and maintenance costs of dispensing are a large part of the cost of hydrogen stations. NREL has found that about 17% of maintenance hours for hydrogen fueling infrastructure are associated with dispensers, with a significant amount attributed to hydrogen leaks or 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 prospective 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. Although high-pressure hoses are not a high capital cost item compared to the nozzle and breakaway, the frequency of replacement could result in the high-pressure hoses becoming a significant lifetime cost over 10 years. By accelerating the cycle rate, monitoring the leakage patterns, and continuing past the point of typical replacement, valuable data on post-cycled specimens can be supplied to OEMs to improve reliability through this project.

APPROACH

This project aims to perform long-duration accelerated life testing using high-pressure, low-temperature hydrogen with commercial or prototype hose assemblies. This work is unique and goes beyond standard OEM and certification standards agency acceptance testing in that it simultaneously stresses the hose assembly with realistic precooled fueling conditions closely following the technical specification SAE J2601-2014 fueling protocol for H70-T40 fills. In addition, the project applies mechanical bending and twisting stress to the hose and nozzle assembly to simulate people refueling vehicles. Finally, the short time in between back-to-back fills, of a yet-to-be-realized high-volume hydrogen refueling market, will simulate a busy station where the dispensing equipment is kept cold most of the time and subjected to frequent decompression and thermal cycles. The main difference between the test plan and a high-volume station is that the mass dispensed per fill will be less than the 3–5 kilograms (kg) of a typical vehicle fill. To prevent overtaxing the production and compression capabilities of the ESIF's hydrogen system, the target mass dispensed per fill is 100 to 200 grams. Back-to-back filling will maintain hose temperatures under the Cold Dispenser cases of SAE J2601. The performance of the hose will be monitored over time using a hydrogen sampling system attached to an outer protective sleeve near each flared crimp fitting to identify leaks as they occur. In FY 2016, chemochromic leak indication tape was also wrapped over the hose end assemblies to further identify exact methods of leakage.

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 specimens in fiscal years 2014 and 2015 include scanning electron microscopy to ultimately identify blistering due to hydrogen permeation and characterization testing, such as Fourier transform infrared spectroscopy, thermogravimetric analysis, differential scanning calorimetry, X-ray spectroscopy, and dynamic mechanical analysis methods, to identify material degradation and compositional changes.

RESULTS

Automated Hose Reliability Test Stand

A hose reliability test stand 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 realistic stresses resulting from human interaction with the hose assembly while maintaining a compact footprint to safely operate in the 100-ft^2 blast-rated HPTB. 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 pressure ramp is controlled using a proportional, integral and derivative (PID) method set to SAE J2601 average pressure ramp rates (APRRs) and target pressures, similar to commercially available dispensers. The temperature is controlled using an air operated valve for faster precooling flow to reach the target gas temperature (-33°C to -40°C) within 30 seconds per SAE J2601 protocols. After this target temperature is attained, the air operated valve closes, but flow continues through a bypass motorized needle valve at a rate just enough to maintain the temperature inside the hose. Good automated control of both pressure and temperature was obtained using a minimum of dispensed mass using hydrogen at full pressure scales, shown in Figure 1. This example set, after precooling, had an average mass dispensed of 57 grams, an average hose temperature of -30.5°C, and a cycle time of 4 minutes 2 seconds with 46 seconds of non-fueling time. The current design takes about two to four cycles of increased flow for components to reach full operating temperature, then uses less than 100 grams per cycle and is able to maintain temperature. The software control program allows for a variety of cases to be manually input from SAE J2601 tables.

Hose Fitting Detected Leak Characteristics

The hose reliability test stand features a leak detector system consisting of open-area combustible gas detectors and two active vacuum pump sampling systems (Figure 2). The Venturi vacuum pump aspirates air from two sampling chambers installed around both crimp fittings at the hose ends. The aspirated air passes through a cell attached to a combustible gas detector and a flowmeter set at 400 mL/min. The sampling chambers are not airtight; thus, a constant suction flow is created instead of a stable vacuum. The system has been measured to have a delayed response of 1.5 seconds on the dispenser-side hose end and a delayed response of 9.5 seconds on the nozzle-side hose end due to the pickup tubing and flow rate. The hydrogen levels detected are accurate as measured by calibration gasses at 25% and 50% lower flammable limit (LFL).

The detector system on the dispenser-side hose end first started to register leaks at Cycle 1856, while the nozzle-side hose end did not start to register the first leaks until Cycle 3033. The amount was relatively small, with about 1 milligram of hydrogen that was captured by the detectors per leak incident. This hose might not be considered failed in a field deployment, although it is possible users may hear the leak. The leaks were inconsistent from day to day but exhibited some patterns. Specifically, the leaks would only occur after several chilled cycles had already been completed, and the highest frequencies of leaks happened during the depressurization and motion back to the dispenser.

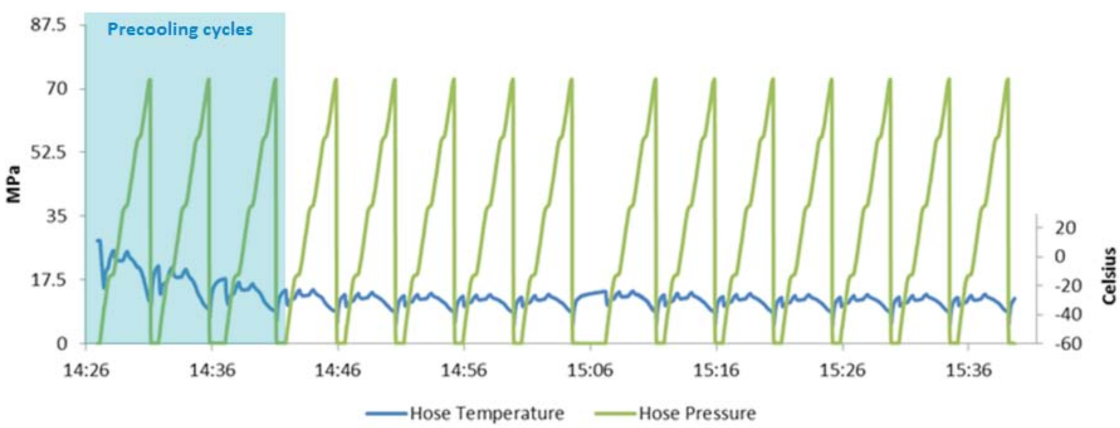

FIGURE 1. Example set of a cycle showing precooling phase and back-to-back fueling

FIGURE 2. Leak detection system with fixed-area detector and two vacuum pump active sampling detectors

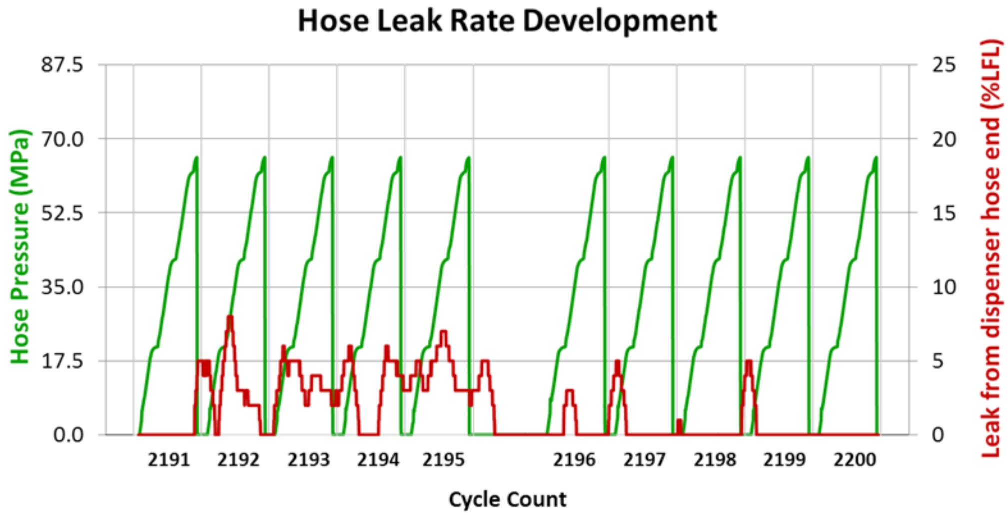

FIGURE 3. Example pattern of hose leak rate development as measured by sampling detector

This could possibly indicate leakage from the plastic-to-metal seal during venting or cracking of the inner liner during motion. The leaks tended to occur over several cycles and often did not reoccur after extended non-fueling periods. An example can be seen in Figure 3.

Leaks were confirmed from the vicinity of the hose end with a handheld detector, but the exact leak site was unable to be identified. After the leaks were detected and recorded, researchers partnered with Element One, Inc., a manufacturer of chemochromic leak indication tape, to deploy their tape to test it on extremely cold surfaces. The tape has been wrapped around the crimp fitting as shown in Figure 4, and around other potential background leak sources such as valves. The permeable chemochromic tape will darken when hydrogen passes through it, and photographs may help identify if the leak is from the metal fitting, the crimp fitting, or if it is leaking from the inner liner through the permeable outer jacket. This will also help identify specific areas to study with post-cycle testing.

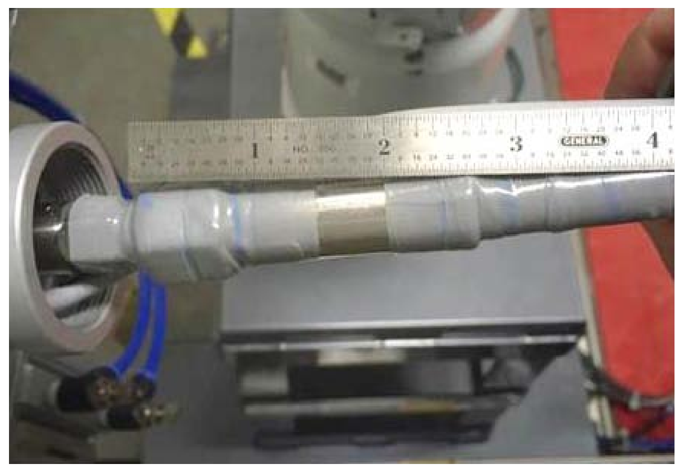

FIGURE 4. Deployment of chemochromic leak indication tape on nozzle-side hose end

CONCLUSIONS AND FUTURE DIRECTIONS

- **Conclusion:** Operated 700-bar hydrogen hose reliability test stand using a low-volume (i.e., tankless) SAE J2601 filling algorithm to achieve over 3,300 realistic H70-T40 cooled fills while mechanically stressing the hose and monitoring likely leak points of the hose assembly.
  - **Future:** Upgrading hydrogen station automation, compression, and storage capacities will allow for increased cycling rate, a wider range of cases with higher target pressures, and unattended operation of the hose test apparatus to be accomplished in FY 2016.
- **Conclusion:** Hydrogen leaks first detected at Cycle 1856 from hose near the breakaway end suggest possible patterns related to decompression or motion of hose after hose has been cooled for several cycles. Hydrogen leaks were first detected at the nozzle end of the hose at Cycle 3033. The amount of hydrogen leaking from these events is in the range of 1 mg as captured by the sampling system.
  - **Future:** Develop test plan with variations to characterize leak development and use chemochromic leak indication tape to pinpoint leak sources and identify specific hose sections for further post-cycling studies.
  - **Future:** Collaborate with industry partners like NanoSonic, SpirStar, and Yokohama Rubber. In the case of NanoSonic, there may be a potential

opportunity to test prototype hoses from their Small Business Innovation Research Phase II project, “Cryogenically Flexible, Low Permeability Thoraeus Rubber™ Hydrogen Dispenser Hose.”

FY 2016 PUBLICATIONS/PRESENTATIONS

1. International Hydrogen Energy Development Forum 2016 International Symposium of Hydrogen Polymers Team, HYDROGENIUS & I2CNER Joint Research Symposium, Kyushu University, Fukuoka, Japan – February 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 2015.” Hydrogen Fueling Infrastructure Analysis. May 2016. http://www.nrel.gov/docs/fy16osti/66580.pdf.