III.13 700-Bar Hydrogen Dispenser Hose Reliability and Improvement

Kevin Harrison (Primary Contact), Owen Smith, Michael Peters, Krista Isham, Huyen Dinh, and Svitlana Pylypenko
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 Sutherland
Phone: (202) 586-3152
Email: Erika.Sutherland@ee.doe.gov

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

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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, Yokohama Rubber) and 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.
- We will design a fully automated test system that unifies the four stresses of pressure, temperature, time, and bending. The high cycling autonomous 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 changes (if any) due to the stress of repeated fueling events.

Fiscal Year (FY) 2015 Objectives

- Begin operation of a test apparatus that unifies the stresses to which the hose is subjected during high volume back-to-back fueling events
  - The stresses include use of hydrogen gas, high pressure (up to 875 bar), low temperature (≥-40°C), 3–5 minute refueling time and automated mechanical bending and twisting of the hose assembly to simulate the refueling process.
  - SAE International technical specification J2601 pressure and temperature profiles for H70-T40 fills are simulated as closely as possible with the exception of total mass dispensed, which will be minimized via a tankless filling algorithm.
- Identify and perform dynamic mechanical analysis (DMA) tests to characterize pre-cycling and post-cycling structural and thermal viscoelastic properties in response to applied stress

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 improve the robustness of hydrogen dispensers, thereby ensuring a positive customer fueling experience and lowering the cost of station maintenance.

- Target Hose Replacement Interval: 25,000 cycles or two years
- Target Cost of Hydrogen Delivery: <$4/gge by 2015, <$2/gge by 2020

FY 2015 Accomplishments

- Evaluated samples of polyoxymethylene hose liner using two different DMA approaches, time-temperature superpositioning (TTS) testing and torsion rheology testing
  - Torsion testing yielded meaningful characterization data on uncycled hose inner liner material from -60 to 50°C and will be added to the range of post-cycling chemical and physical testing.
- Designed and implemented improved safety systems in the high pressure test bays and hose test apparatus to enable autonomous hose testing in hydrogen
  - Facility upgrades include additional isolation valves, monitoring and feedback of facility

    ventilation status, ultraviolet/infrared detection, video monitoring, and improved visual/audible alarming.
  - Safety measures implemented include a sampling system to rapidly detect hydrogen leaks at each crimp fitting, hose to nozzle, and hose to breakaway.
- Constructed and installed the high pressure, low temperature automated hose reliability test stand at the NREL-funded 700-bar Hydrogen Infrastructure Testing & Research Facility (HITRF) located at the Energy Systems Integration Facility (ESIF)
  - Developed and proved tankless algorithm to simulate SAE J2601 fueling protocol, while conserving mass dispensed

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INTRODUCTION

The hose reliability project aims to lower the capital and maintenance costs of hydrogen dispensers by benchmarking the performance of the current state-of-the-art. Results from this project will demonstrate hose performance in real-world conditions, increasing confidence in their use and giving manufacturers valuable performance data to guide improvements in hose reliability. Previous research at NREL has shown that about 13% of maintenance hours for hydrogen fueling infrastructure are associated with dispensers, with a significant amount attributed to hydrogen leaks or failed parts [1]. Improvement in the reliability of hoses would therefore significantly reduce station down time. Additionally, results on the material properties of today's hoses may facilitate the generation of new hose manufacturers. Only a small handful of companies manufacture hydrogen hoses worldwide today. An increase in the number of manufacturers could significantly advance the technology and lower its cost.

NREL is uniquely positioned to complete this project due to its ESIF. ESIF is a user facility with a broad array of capabilities in energy integration research, from materials testing to supercomputing. Fast and flexible swapping of research test articles is a hallmark of the facility. ESIF is utilized in DOE-funded projects, as well as in partnerships with industry to provide critical testing, validation, and refinement during product research and development. ESIF's high pressure hydrogen test bay (HPTB) integrates hydrogen production, compression, storage, dispensing and end use systems in a safe and controlled environment. The hose reliability test stand is housed in the HPTB due to its ability to test components under high pressure to failure while minimizing dangers to personnel or equipment.

APPROACH

This project aims to perform accelerated life testing using high pressure, low temperature hydrogen with ramp rates and fueling times that comply with SAE J2601 protocols. (Hydrogen stations being installed today for use by passenger vehicles are designed for compliance with SAE J2601.) This work is unique and goes beyond standard OEM testing in that it simultaneously stresses the hose assembly with realistic precooled fueling conditions closely following the SAE International technical specification SAE J2601 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 and subjected to frequent decompression cycles. The only difference between the test plan and a high volume station is the mass dispensed per fill will be less than the 3–5 kg of a typical vehicle fill. To prevent overtaxing the production and compression capabilities of the HIRTF, the target mass dispensed per fill is 100–200 g. 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.

The project also includes analysis of the physical and chemical property changes in the inner hose liner due to long duration cycling. Chemical tests previously identified and performed on pre-cycled specimens in FY 2014 include scanning electron microscopy to detect blistering due to hydrogen permeation and testing to characterize material degradation and compositional changes. Characterization testing included Fourier transform infrared spectroscopy, thermogravimetric analysis, differential scanning calorimeters, and X-ray spectroscopy. In FY 2015, DMA methods were investigated based on feedback received from industry experts and other stakeholders. As a result, TTS and torsion tests were performed on pre-cycled polyoxymethylene (POM) inner liner.

RESULTS

Automated Hose Reliability Test Stand

A new hose reliability test stand was developed to simulate SAE J2601 700-bar fills in a footprint small enough to fit in NREL's HPTB. The test stand closely mirrors an actual dispenser in its design and pressure ramping capabilities. A small six-axis robot was also installed and programmed to simulate human movement of the nozzle from 'dispenser' to one of two 'vehicle' receptacle positions. A hydrogen leak sampling system, using vacuum pumps

FIGURE 1. Completed hose test stand installed in HPTB

drawing air from chambers around each hose crimp fitting through a combustible gas detector, was installed to monitor for likely leak points. The completed hose test stand assembly is shown in Figure 1.

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 (APRR) and target pressures, similar to commercially available dispensers. Temperature control (-33°C to -40°C within 30 s of fueling, per SAE J2601 protocols) is achieved using an air operated valve that controls hydrogen flow. 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 fills was demonstrated with nitrogen gas dispensed at 7.0 MPa (Figure 2). The tankless PID algorithm was successful in remaining within the SAE J2601 APRR and temperature tolerance bands for the duration of the fill except for a slight overshoot at the end. The total mass of nitrogen dispensed during this fill was approximately 150 g. Nitrogen gas was used to develop the process to avoid unnecessarily wasting hydrogen. The control algorithm is being retuned for 70 MPa hydrogen gas and additional controls will be put in place to prevent overshoots.

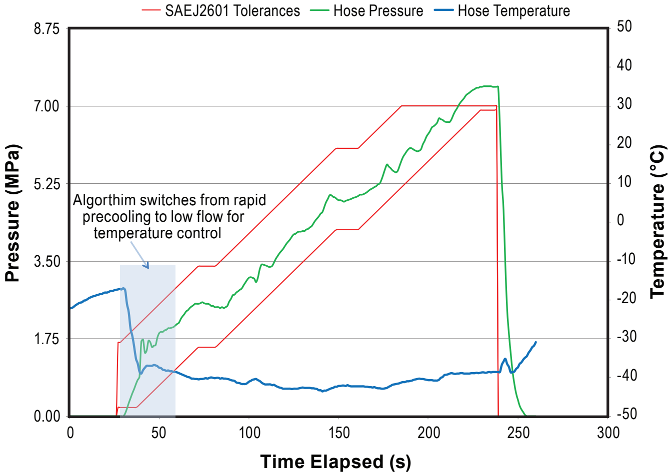

FIGURE 2. Preliminary pressure profile control with leak check holds, precooling, and temperature stabilization

Enhanced facility safety improvements were required by NREL's authority having jurisdiction causing some delays to full system operation. During this time, the team has worked to increase monitoring of potential leak points and enhance safety systems on the test apparatus. Safety systems now include hydrogen detectors, video camera feedback to the control room, automatic shut-off of the robotic arm in case of collision, and room ventilation monitoring.

Baseline (Pre-Cycled) Inner Hose Material DMA

Uncycled hose samples, supplied by SpirStar, were used to conduct DMA testing using two different methods. The TTS method involved cutting narrow strips of the cylindrical POM hose liner and placing it in a solids analyzer equipped with a three-point bending clamp. The sample was soaked in a nitrogen-purged forced convection oven from 30 to 100°C at a soak rate of 3 minutes for each 5°C step with frequency sweeps from 0.001 to 10 Hz. The TTS test results were affected by sample geometry and the semicrystalline nature of the POM nature, showing no usable time–temperature relationships. Analysis may be possible at low temperatures but useful only to predict short timescales or high frequencies. TTS DMA will therefore not be repeated for future pre-cycling or post-cycling characterization of the POM inner liner.

The data, shown in Figure 3, characterizes the transition in material structural rigidity and viscous response under applied stress and over the full temperature range possible in operation. Two separate cycled samples showed good repeatability and clear relationships between the storage modulus and loss modulus. These relationships indicate that the hose material retains structural integrity from -60°C to 50°C. The torsion DMA tests will be repeated on the hose material after cycling to detect any shifts in the onset of glass transition or softening due to hydrogen permeation, mechanical fatigue, or other effects of the cycling.

The data, shown in Figure 3, characterizes the transition in material structural rigidity and viscous response under applied stress by measuring the ratio of the storage modulus and loss modulus over a full temperature range. Two separate cycled samples showed good repeatability and clear relationships between the two moduli, defining a stable region from -60°C to 50°C where the structural integrity is maintained. Post-cycled specimens undergoing torsion DMA tests will be able to show any shifts in the onset of either glass transition or softening caused by hydrogen permeation, mechanical fatigue, or other effects of the hose reliability test stand.

CONCLUSIONS AND FUTURE DIRECTIONS

- **Conclusion:** Completed design, development and fabrication of 700-bar hydrogen hose reliability test stand using a low volume (i.e., tankless) SAE J2601 filling algorithm to achieve realistic H70-T40 cooled fills while mechanically stressing and monitoring likely leak points of the hose assembly.
  - **Future:** The system will move quickly from attended to unattended mode of operation to accomplish high cycle counts of the first hose assembly until 25,000 cycles, excessive leak or hose failure.

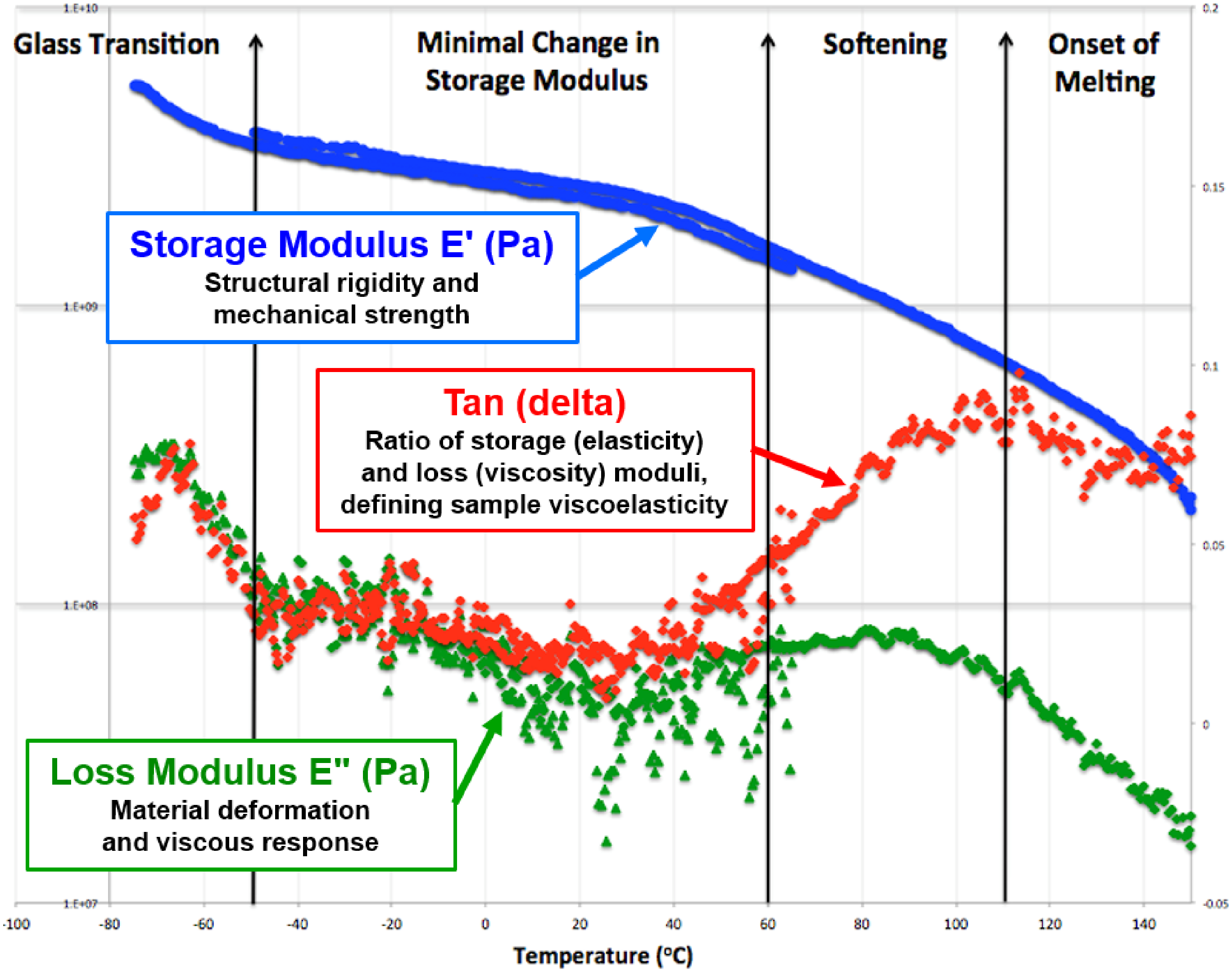

FIGURE 3. Torsion DMA results showing relationship between storage and loss moduli of two polyoxymethylene hose liner samples

  - **Future:** Methods to expedite warm-up rest periods between the 70 back-to-back fill sets will be developed. These periods are meant to introduce realistic thermal stressing that would be expected if the hose assembly were unused overnight.

- **Conclusion:** DMA, aimed at characterizing transitions in material structural rigidity between pre- and post-cycled hose material, was completed. Torsion DMA testing was selected for future testing because it showed stable and repeatable results in the region between -60°C and 50°C for the polyoxymethylene hose inner liner material before glass transition or softening occurred. TTS DMA test method will not be utilized in the future due to the inability to prepare a sample of the cylindrical hose liner in a way conducive for reliable data results.
  - **Future:** Perform post-cycling of hose assembly #1 using torsion DMA alongside suite of previously identified chemical analysis methods to reveal any changes in bulk material properties and elemental material composition.

- **Future:** Continue to work with industry partners like NanoSonic, SpirStar, and Yokohama Rubber. In FY 2016, NREL plans to test prototypes of hoses developed by NanoSonic through its Small Business Innovation Research Phase II project, “Cryogenically Flexible, Low Permeability Thoraeus Rubber™ Hydrogen Dispenser Hose.”

FY 2015 PUBLICATIONS/PRESENTATIONS

1. 1-Page Fact Sheet - http://www.nrel.gov/docs/fy14osti/61091.pdf

2. YouTube Video - http://www.youtube.com/watch?v=Rbc7f01oP8kA

REFERENCES

1. Sprik, Sam, Jennifer Kurtz, Chris Ainscough, and Mike Peters. “Next Generation Hydrogen Station Composite Data Products - Data through Quarter 4 of 2014.” Hydrogen Fueling Infrastructure Analysis. May 2015. http://www.nrel.gov/docs/fy15osti/64317.pdf.