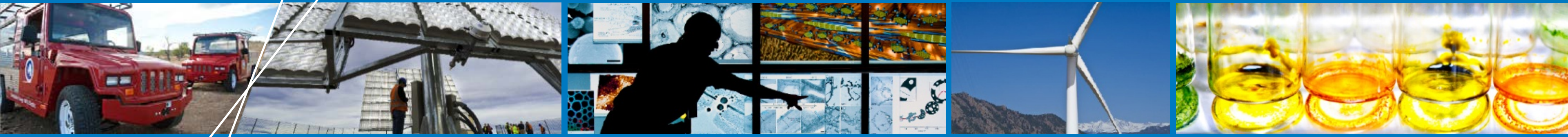
700 bar Hydrogen Dispenser Hose Reliability Improvement



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National Renewable Energy Laboratory

DOE 2016 Annual Merit Review
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Project ID
PD100

Overview

Timeline

Project start date: June 2013

Project end date: October 2016*

*Continued operation after end date on new sample hoses as directed by DOE

Budget

To Date Total Spending: $603k

- **$284k carried over into FY16**
- **Remaining funds covers labor, maintenance**

Total DOE Project Value: $775k

Internal NREL Funding

- **$1.1M – 35/70MPa Fueling Station**
- **$150k – Compression, storage, safety systems and chiller/HXR**

Barriers

- **3.2 – Hydrogen Delivery**

  I. Other Fueling Site/Terminal Operations

  "By 2020, reduce the cost of hydrogen delivery from the point of production to the point of use in consumer vehicles to <$2/gge of hydrogen for the gaseous delivery pathway. (4Q, 2020)"

Partners

- **Spir Star AG**
  - Provided 3 hose assemblies for testing
- **NanoSonic, Inc.**
  - SBIR awardee and potential test articles
- **Sandia National Laboratory**
  - Burst testing on hose assembly
- **Colorado School of Mines**
  - Microscopy and rheology
- **Element One, Inc.**
  - Provided DetecTape leak indicator samples

Relevance - Problem Statement

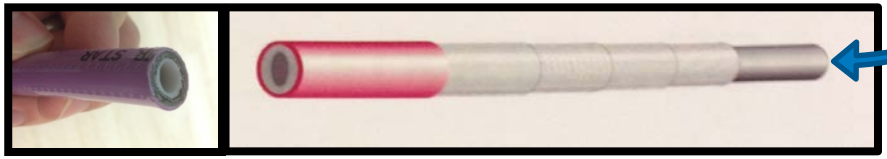
- Limited OEM competition, small FCEV market and extreme operating condition ranges result in high 70 MPa refueling equipment costs
- Low-volume FCEV refueling stations are replacing hoses at higher frequencies than expected. Operators are reporting intervals of a few months
- Other certification standards specify a range of acceptance testing. These test conditions do not reflect the combined stresses of real-world conditions. Accelerated real-world data may be valuable feedback to the OEMs.

Component	 Hose Assembly	 Nozzle		 Breakaway
Primary Supplier	SpirStar (Germany)	WEH (Germany)		WEH (Germany)
Cost	$1,700	$6,800 (non-IR)	$11,000 (IR)	$3,000
Service Interval	~ 6 months	3 years		3 years
Service Cost	$1,700	$1,750 (standard)	$5700 (with IR kit)	$850
10-Year Cost	$34,000	$12,050 (non-IR)	$28,100 (IR)	$5,550
Alternate Suppliers	Yokohama Rubber (Japan)	Walther Präsizion (Germany)		Walther Präsizion (Germany)

Relevance - Project Objective

To characterize and improve upon 700 bar refueling hose reliability under mature market conditions

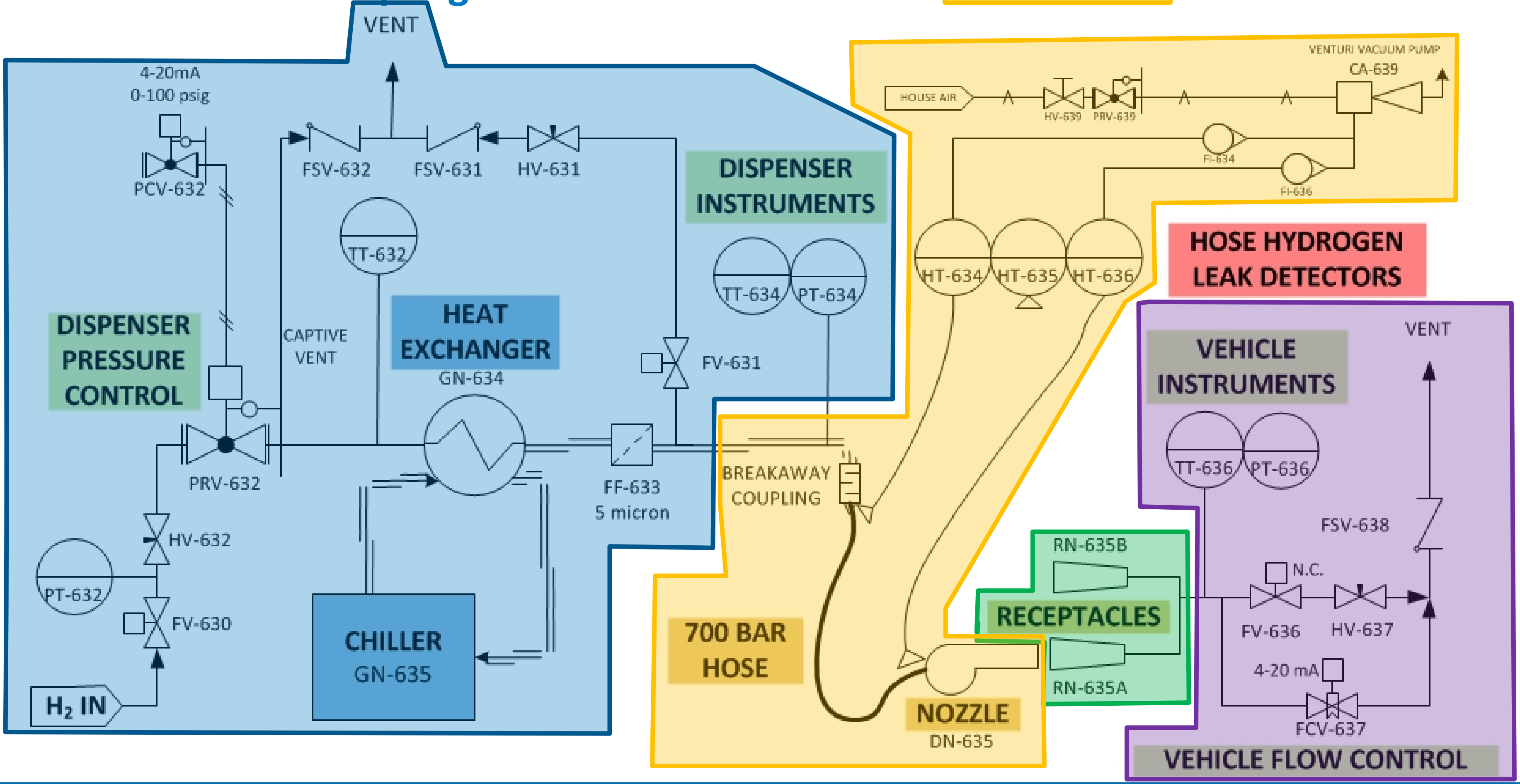
- By working closely with the original equipment manufacturers, the hose reliability project aims to improve the reliability and thus reduce the cost of 700 bar hydrogen refueling hose assemblies by identifying points of failure.
- NREL has designed a high-cycling test apparatus that unifies the four stresses to which the hose is subjected (Pressure, Temperature, Time and Mechanical) to reveal the compounding impacts of high volume FCEV refueling will be revealed.
- The work includes performing physical and chemical analysis on pre- and post-cycled hose material to understand any relative changes in its bulk properties and possible degradation mechanisms.



- This reporting period: High-cycle rates are revealing insights into leak patterns and characteristics of all 700 bar refueling equipment

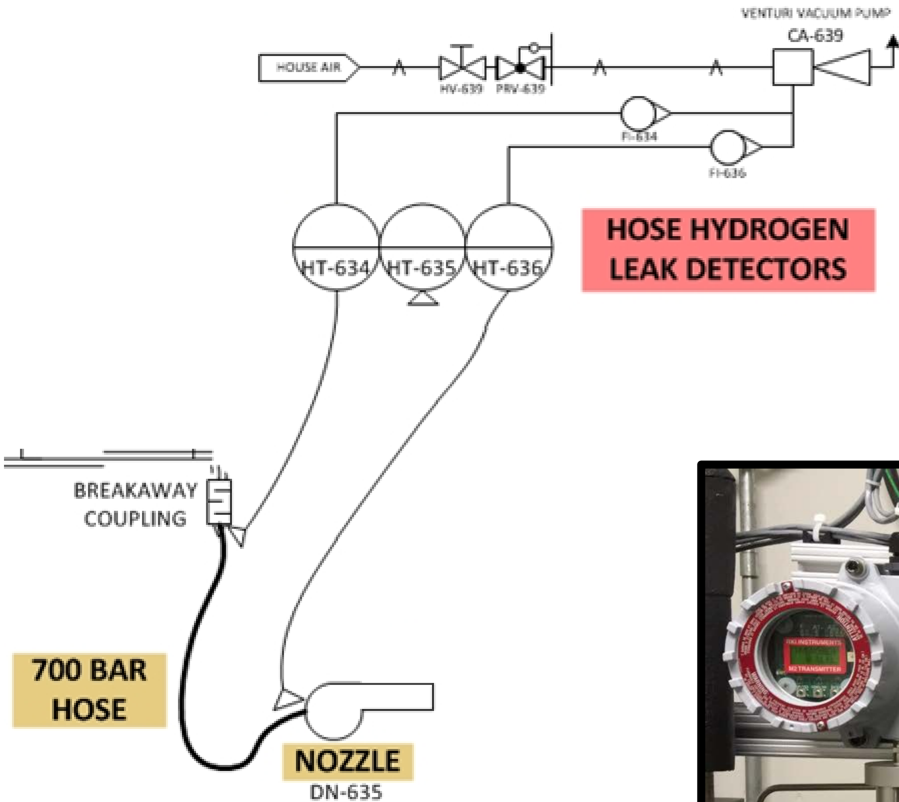
Approach – Hose Test Stand System Design

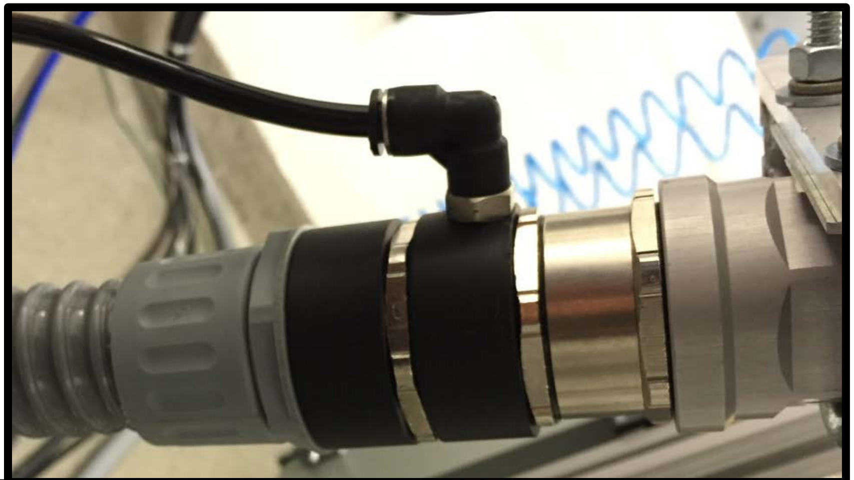
- ‘Dispenser’ subsystem based on commercial dispensers
- Redundant receptacles to give greater path options
- Tankless flow control for low-mass, pre-cooled cycles
- Active leak sampling at hose interconnections



Approach – Hose Leak Detection

- **Vacuum pump sampling system installed on crimp fittings**
- **Constant flow 400 mL/min around hose crimp fittings through hydrogen detectors**





Progress – Current Status

Daily Operations

- 3100+ cycles completed* running various cases of SAE J2601 H70-T40 with good accuracy on pressure controls. *As of 4/18/2016.
- Station upgrades and automation implemented in April to enable unattended operations at rate of up to 1500 cycles/week with higher station capacity for increased target pressures and mass consumption

Leak Status

- Small, infrequent leaks have been observed from the hose crimp end area and at the dispenser valves. The leaks are not consistent.
- Data gathered shows leak seems to occur on depressurization after several cold cycles - no clear pattern identified yet.
- Multiple safety features implemented as part of experiment design allowing hose to be run throughout leaks without risk.

Responses to Reviewers' Comments

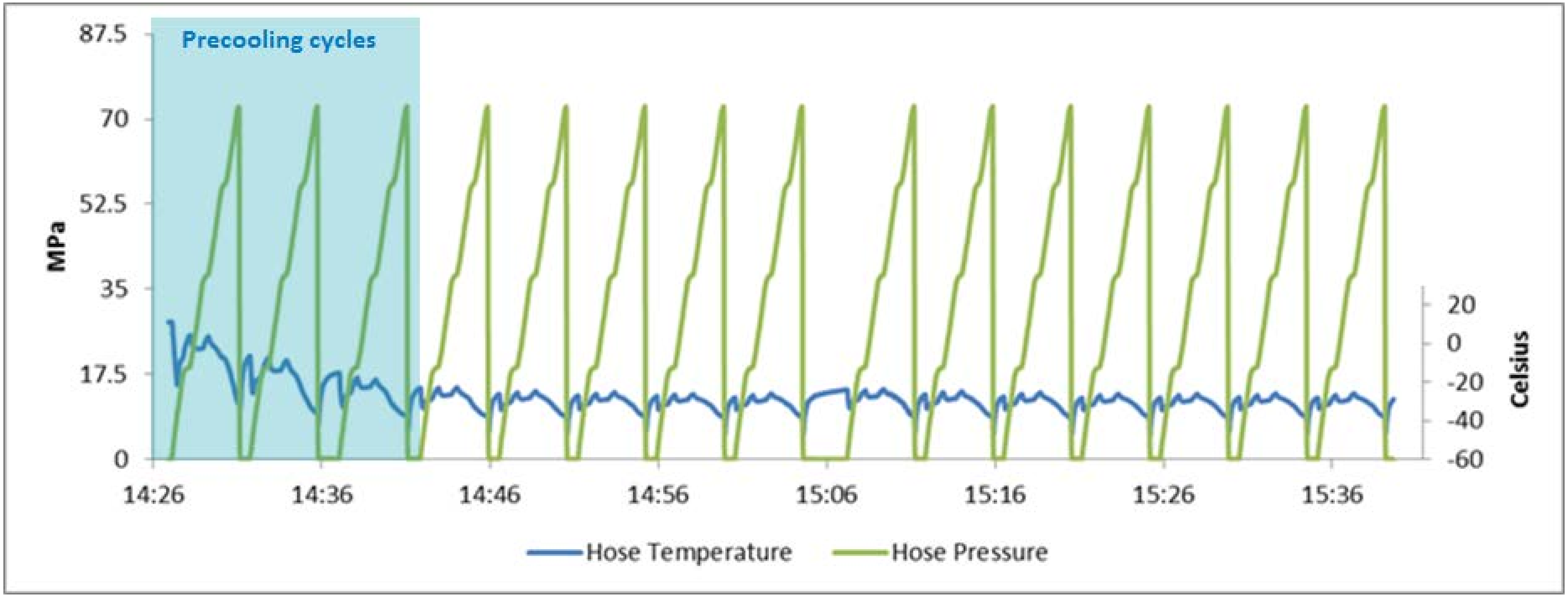
- This project was a non-reviewed poster at the 2015 AMR. Responses to comments from the 2014 AMR are given below:

- *"It may be helpful to include some real-life exposure conditions, such as sunlight, environmental contaminants, etc."*
- *"Nozzles will not last for 25,000-cycle testing; this should be considered as well, because it requires additional torqueing of hose and fittings (and potential damage). It will be interesting to see how fittings/crimps perform under climate extremes (hot and cold) and pressure cycling—the project should look into the fitting/crimps installation process and consider the impact of variations of crimp installation environments on operation in the field."*
  - Stressing the inner hose liner is the focus of this project; not the exterior cover. The hose is protected by corrugated plastic jacket as seen in station installations. This mostly removes the UV resistance variable. Due to the location of the installation in the high pressure test bay, environmental control is not possible and ambient temperature will be room temperature.
  - The nozzle is monitored and replaced as leaks begin to form. During each maintenance event, DetecTape leak indicators are replaced and may show us if a fitting has been stressed during maintenance.

- *"It would be good to see collaboration with station owners in California and distribution centers."*
- *"It may be helpful to include testing of a hose in-service in the field at an existing hydrogen fueling station."*
- *"The testing and comparison of hoses in-service, which may be exposed to real-life conditions, is recommended."*
- *"The project should bring in more data from field installations and operators."*
  - A sample of a failed hose from Burbank Station in California was shipped to us and underwent chemical and physical analysis. The results from this hose sample will be added to the future results from our cycled hoses for further data comparison.

- *"The project should explore collaboration and coordination with Yokohama Rubber in Japan—this company developed a 700-bar hose together with Iwatani Industrial Gases."*
- *"The project should be expanded by adding additional hoses from different manufacturers."*
- *"Domestic refueling hose manufacturers and/or station owners/designers should be brought in to provide input on additional mechanical stress conditions experienced by the hose during normal operations."*
  - Yokohama Rubber and Nanosonic, Inc. have both visited NREL and viewed the Hose Reliability Test Stand and technical service agreements are in place with Nanosonic, Inc. to test samples of their hose assemblies. Yokohama has shown interest in also testing a hose at NREL.
  - Yokohama Rubber has performed their own in-house testing of their 700 bar hose assemblies and shared their information with HySUT. While this testing parallels our current test system, it does not duplicate it and is missing the mechanical stressing introduced by the robot.

Progress – Sample of a Cycle Set



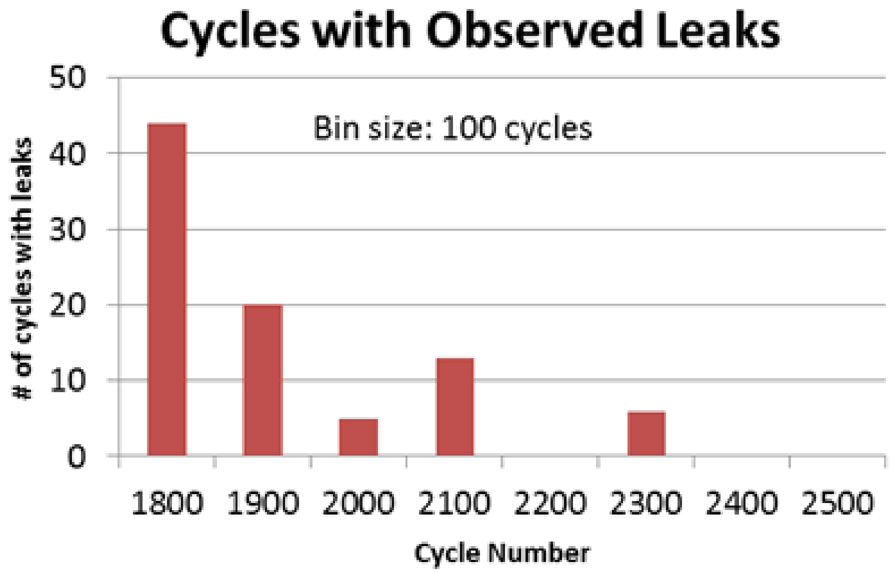
Start of a cycle set with initial temp 10 C (set continues →)

Average mass H2 consumed per cycle	**57 grams**
Average fill time (inc. leak checks)	4 min 2 sec
Average non-fueling time (return to dispenser)	46 sec

- **Successful repetition of tankless filling keeping hose inner liner cold**
- **Regular breaks in fueling provide thermal shock**

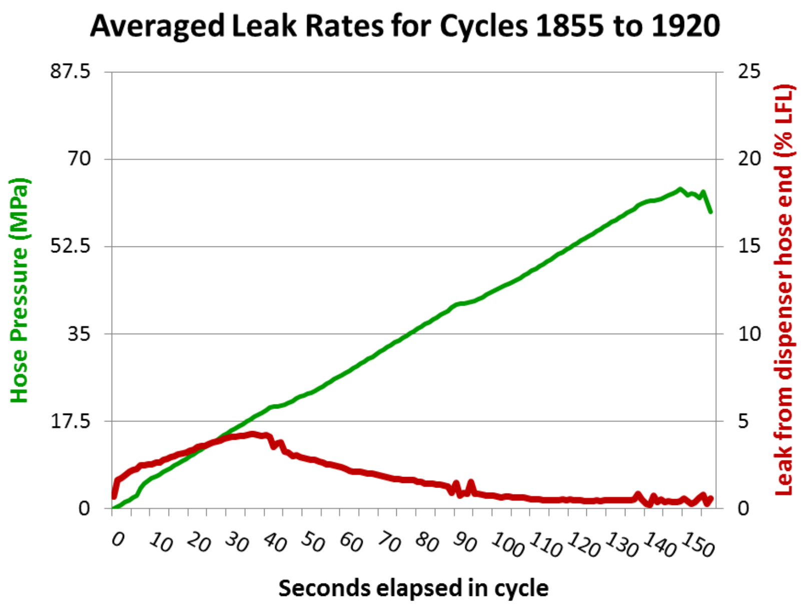
Progress – Hose Fitting Leak Detection

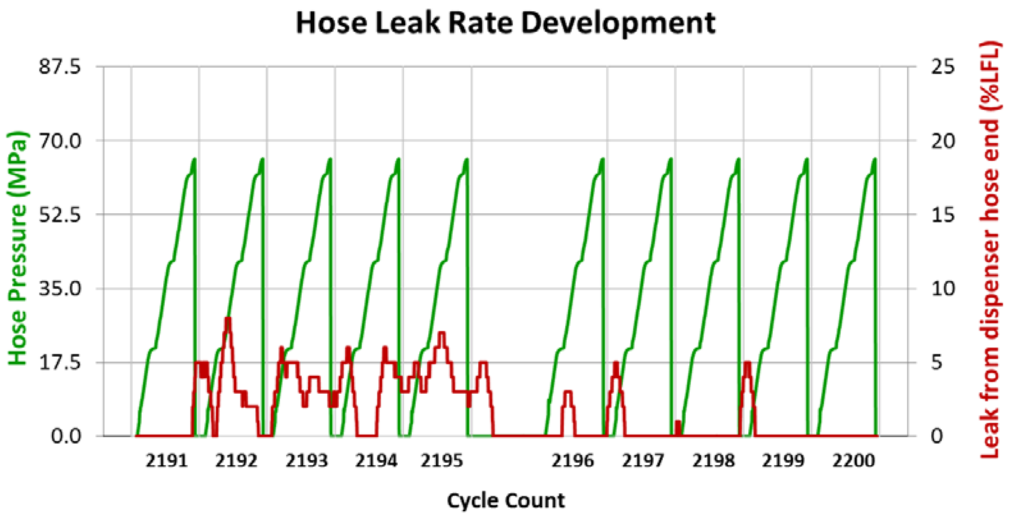
- Hose leak active detector system mounted on dispenser hose end started registering leaks at Cycle 1856
- Nozzle hose end registered leaks at Cycle 3033
- Leak tended to develop after several chilled cycles and persist through multiple cycles with little day-to-day consistency
- Small amount H_2 released
  - ~1 milligram H_2 through detector per cycle (400ml/min)
  - Hose is not considered failed



Progress – Detected Hose Leak

- Leak appears to mainly occur upon hose depressurization, motion after hose gets very cold
- Often leaks over consecutive cycles
- Can stop leaking after cycle paused
- No clear pattern identified yet

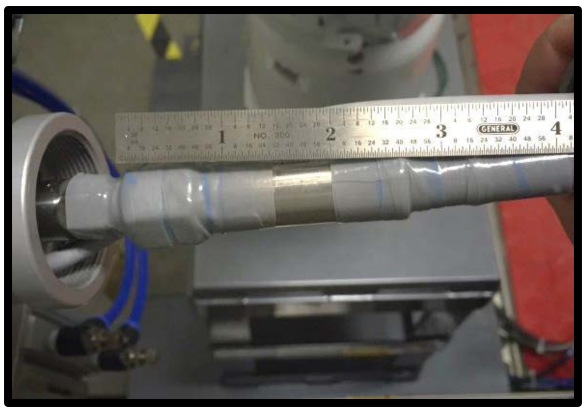




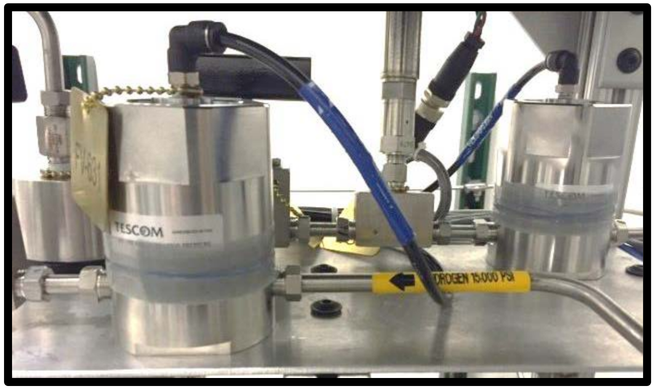
- Nozzle hose end leak detected at Cycle 3033-3050
- Smaller than dispenser hose end leak but follows similar pattern

Challenges – Leak Site Identification

- Handheld detectors confirmed leak from vicinity of hose end at dispenser but not exact location
- Hydrogen detection tape installed over hose crimps, metal fittings and secondary leak sources (valves)
- Comparison photographs after several leaking cycles may show exactly where leak occurs



DETECTAPE Hydrogen Leak Detection H_2



Chemochromic tape that darkens when exposed to hydrogen leaks.
- Provided by Element One, Inc.

Note: DetecTape has not yet been approved for use by the manufacturer under these conditions and this project is not intended to draw any conclusions on the performance of the tape.

Challenges – Downtimes and Limitations

Leakage from other sources and unplanned downtime

- **Breakaway leaked within 100 cycles at cold temps**
  - Older model that was sent back to manufacturer for refurbishment with improved seals to match current field deployments
- **Valve body seal failures**
  - Valves having difficulty with sustained cold temperatures – frequent maintenance required. Configured leak detector to minimize background leaks from valves
- **Other equipment failures – high pressure compressor (gas booster)**
  - Gas booster housed in same space as hose tester. Leaks from gas booster have resulted in early triggering of the safety failsafe systems, and additional downtime.

Safety Systems Upgrades

- High Pressure Test Bay – Hose test apparatus outside original design intent of the space. Required additional review and upgrades to safety systems

Station Capacity

- In the past year, limited to high pressure gas booster (~1 kg/hr) and two high pressure tanks (32 kg) sharing time with multiple FCEV fills
  - Limited the cases possible based on target pressures and flow rates

Progress – Station Upgrades

- **Major Systems Integrations and Commissioning**
  - Additional high pressure storage tanks – 32 kg @ 875 bar (Total 60 kg)
  - High pressure compressor – up to 20 kg/hr to 875 bar

  - 16 hp chiller and heat exchanger – will provide -40°C at hose with higher flow
  - Together, the station upgrades allow for increased cycle rate, higher target pressure, higher flow rates and less downtime due to shared projects

Future Work – Milestones and Testing

Qtr	Due Date	Milestones, Deliverables, or Go/No-Go Decision	Status
Q1	12/31/2015	Complete 1,000 cycles on Hose Assembly #1 or until failure (NREL Task 4)	Completed
Q4	Estimated FY16	**Perform accelerated hydrogen cycling to 700 bar (nominal) on a hose sample to failure or 25,000 cycles, whichever occurs first.**	On Target
Q4	Estimated FY16	Perform post-cycling physical and chemical testing including DMA, SEM, XPS, FTIR and other tests previously identified	On Target

- Search for a pattern of hose leak development while cycling
  - Vary downtimes between individual cycles and cycles sets, add pre-fueling pressure pulses
- Cycling rate greatly increased with unattended operation, made possible by multiple safety features to allow for continued operation even with minor leaks
- Hose sample will be considered “failed” upon inability to hold pressures long enough to complete cycles or when safety features consistently stop cycling.
  - Likely will occur much later in life than when hose would “fail” a commercial dispenser NFPA leak check - provides clearer perspective into material changes
- Test additional hoses, including from other manufacturers – FY17- TBD

Collaborations

- **SpirStar AG**
  - Provided dispenser hose test material and assemblies. Provided feedback on project results.
- **NanoSonic, Inc.**
  - Candidate for future hose tests— SBIR Phase II *“Cryogenically Flexible Low Permeability Thoraeus Rubber™ Hydrogen Dispenser Hose”* (Presented at 2016 AMR as **PD101**)
- **ISO Technical Committee 197**
  - Requested results for the standardization of ISO-19880-5 WG22
  - Gaseous hydrogen fueling station hoses
- **Colorado School of Mines**
  - Performed SEM imaging and torsion rheology benchmark testing
- **Sandia National Laboratories**
  - Performed hose hydrostatic burst testing
- **Element One, Inc.**
  - Provided chemochromic hydrogen leak indicating DetecTape samples
- **NREL Chemists and Hydrogen Sensor Group**
  - Performed TGA/DSC/FTIR chemical analysis, H_2 detector calibrations

Summary

Relevance & Impact

- Characterize reliability and improve 700 bar refueling hoses under mature market conditions. Project is expected to reveal the compounding impacts (P, T, t and mechanical) of high volume 700 bar FCEV refueling that has yet to be experienced in today's low-volume market.

Progress & Accomplishments

- Over 3,100 cycles with low-mass flow are meeting pressure, time and temperature limits set by SAE J2601 fueling protocols
- Hose fitting leak monitoring from hose crimp-ends seem to indicate infrequent leak development upon depressurization after several cycles.

Future Work

- Production and station upgrades will increase capacity for higher pressures and flows per cycle.
- Develop and implement test plan with variations to attempt to further characterize leak development and pinpoint leak sources with H_2 leak indicator tape.
- Unattended cycling developed for greatly increased cycling rate.

Technical Back-Up Slides

Approach – Pressure and Temperature

- **Pressure profile closely follows SAE J2601 (2014)**
  - H70-T40 Type fills, APRR ~20 MPa/min w/ Flow control
  - T_{fuel} -33°C to -40°C within 30s (measured prior to hose), T_{amb}= 20°C
  - Cold Dispenser (Communications) tables primarily used, variety of cases
- **3 – 5 minute fills using ~ 100 g H_2 / cycle**
  - Hydrogen not recycled
  - Target fill amounts 100g per fill to sufficiently cool gas
- **Sets of rapid back to back cycles with warm-up periods**
  - Delay times can be added between cycles to study leaks
- **6-axis robotic arm to provide repetitive mechanical stress of routine consumer refueling**
  - Automated control of entire fueling process
- **H_2 leak monitoring of crimp fittings on each end**
  - Focus on ability to stay sealed while cold under high pressure and depressurization cycles

Approach – Mechanical Bending/Twisting

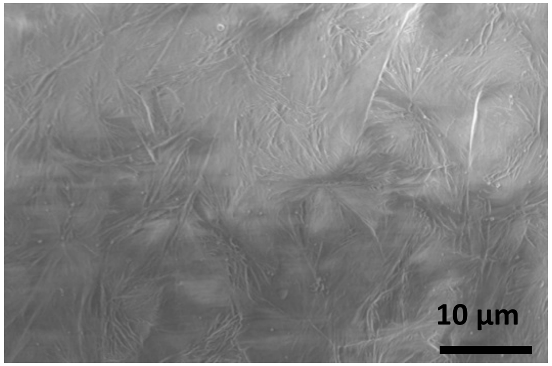
- **Epson six-axis robot installed and programmed with realistic human motions from 'dispenser' to receptacles – total distance ~3 feet**
- **Double receptacle system to provide options for greater randomization of paths from dispenser**

- **High repeatability, over torque tripping capability, anchored frame and calibration help reduce stresses on nozzle and receptacle**
  - Focus on crimped hose end connections and their ability to remain sealed under bending/twisting stress while cold.
  - Nozzle and other components are replaced as needed if they wear out.

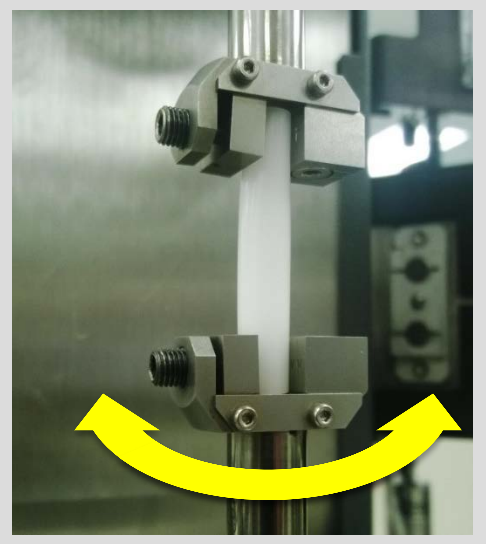
Approach – Materials Testing

Chemical and mechanical testing previously identified and performed on pre-cycled specimens

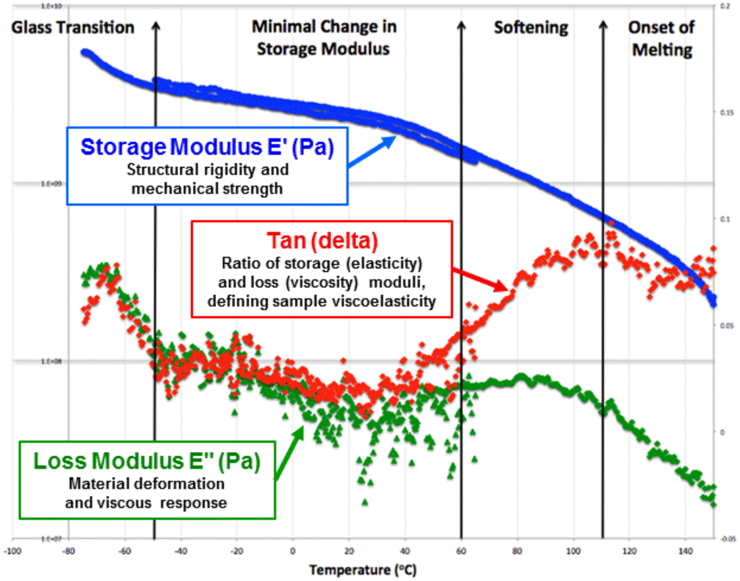
- Physical – hydraulic bursting (SNL), rheology, scanning electron microscopy
- Chemical – FTIR, DSC, XDS, XPS, TGA

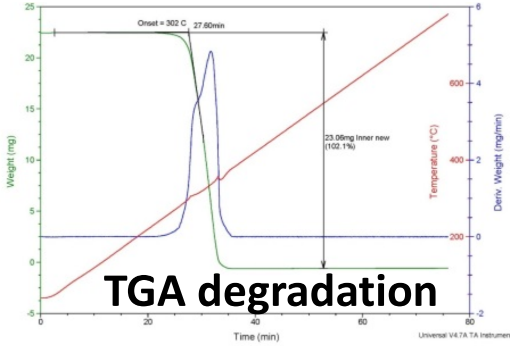


SEM imaging



Torsion rheology

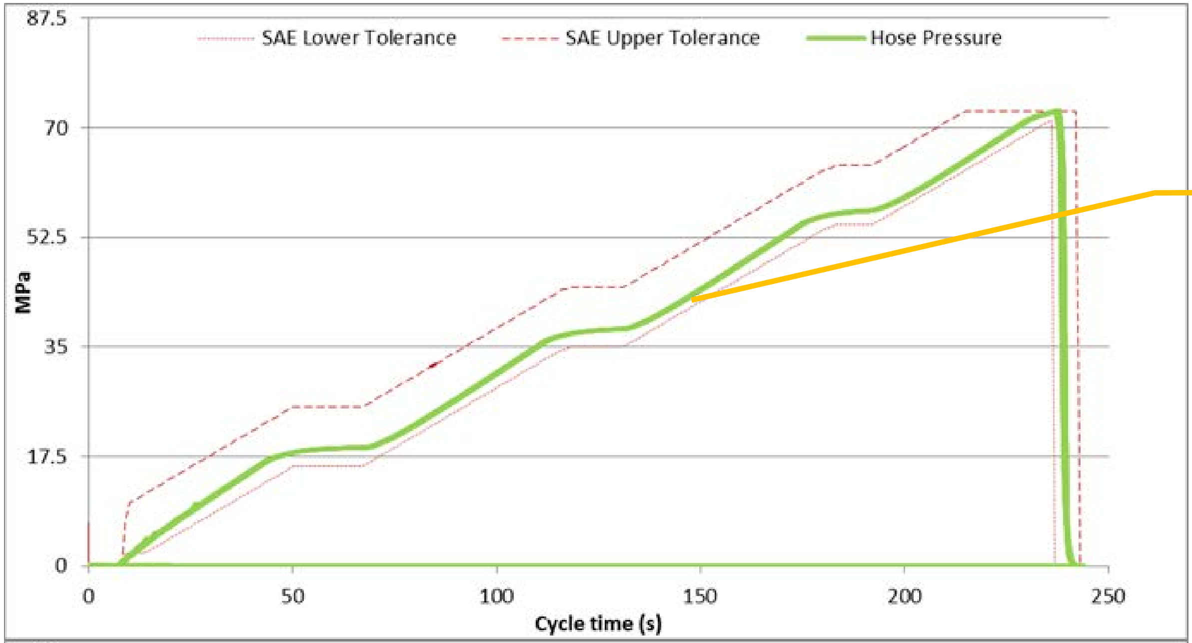


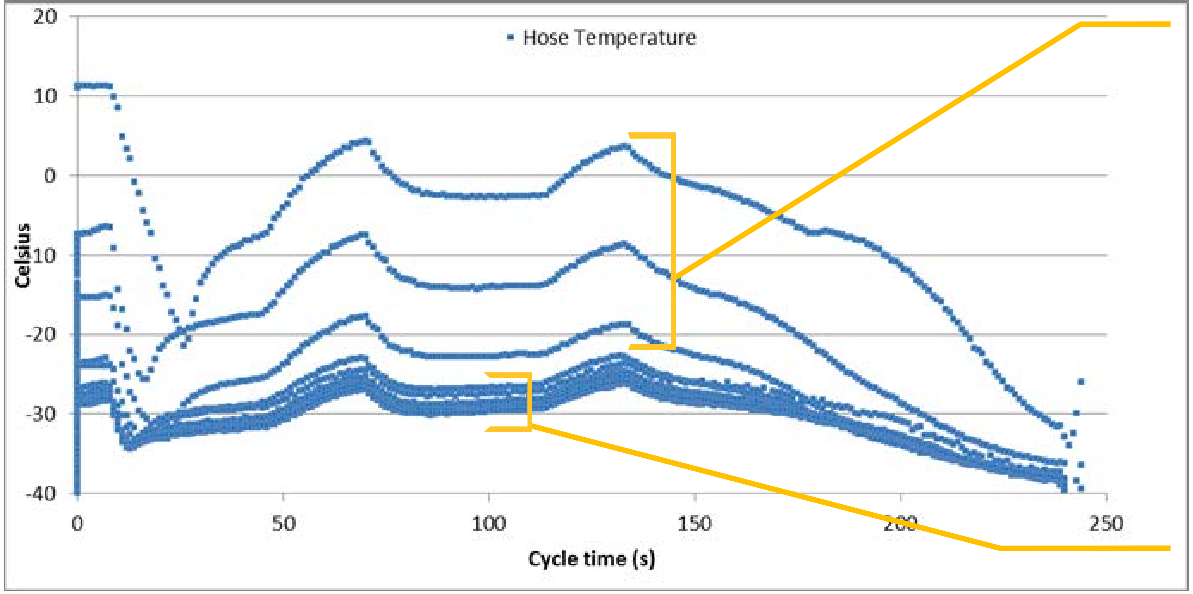


TGA degradation

- **All tests to be repeated on post-cycled materials**

Progress – Sample of a Cycle Set





Set of 15 cycles has high repeatability and remains within specified cycle tolerance at all times

First 3 cycles:
Average hose temp -17.5 C

Next 12 cycles (and beyond)
Average hose temp -30.5 C

Progress – J2601 Fueling Cases

SAE J2601 cases are changed to improve variation in pressure cycles and to work within existing station pressures

SAE J2601 Case	Cycles Completed* *as of 4/8/2016
H70-T20 2-4kg non-communication fill Standard, 0.5 MPa start pressure, 0 C ambient temp Target pressure 59.2 MPa, APRR 15.3 MPa/min	200
H70-T40 2-4kg non-communication fill Standard, 0.5 MPa start pressure, 10 C ambient temp Target pressure 66.1 MPa, APRR 27.0 MPa/min	690
H70-T40 4-7kg non communication fill Cold Dispenser 0 C, 2 MPa start pressure, 25 C ambient temp Target pressure 72.7 MPa, APRR 22.9 MPa/min	375
H70-T40 4-7kg non communication fill Cold Dispenser -10 C, 2 MPa start pressure, 20 C ambient temp Target pressure 71.6 MPa, APRR 27.7 MPa/min	900
H70-T40 2-4kg non communication fill Cold Dispenser 0 C, 2 MPa start pressure, 10 C ambient temp Target pressure 73.8 MPa, APRR 28.5 MPa/min	300+ (current case)
H70-T40 7-10kg communication fill Cold Dispenser -10 C, 0.5 MPa start pressure, 25 C ambient temp Target pressure 81.4 MPa, APRR 19.9 MPa/min	150