NREL
NATIONAL RENEWABLE ENERGY LABORATORY

Component Standard Research & Development

2014 DOE Annual Merit Review, Hydrogen Safety Codes and Standards

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National Renewable Energy Laboratory

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Project ID # SCS002

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Overview

TIMELINE

- Start date: 10/1/2013
- End date: 9/30/2014*

*Project continuation and direction determined annually by DOE

BARRIERS

2012 Multiyear RD&D Barriers

- ***A.*** Safety Data and Information : Limited Access and Availability
- ***C.*** Safety is Not Always Treated as a Continuous Process
- ***F.*** Enabling national and international markets requires consistent RCS
- ***G.*** Insufficient technical data to revise standards
- ***H.*** Insufficient synchronization of national codes and standards
- ***J.*** Limited Participation of Business in the Code Development Process
- ***K.*** No consistent codification plan and process for synchronization of R&D and code development

BUDGET

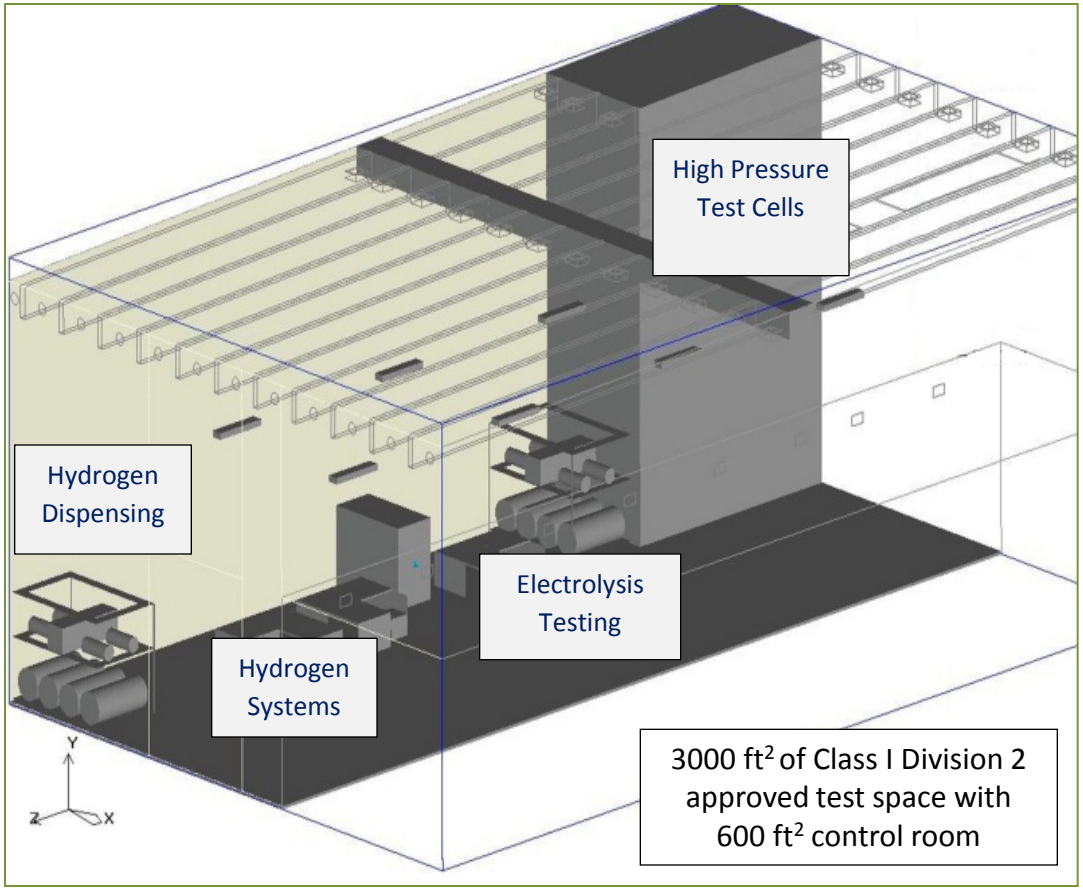
- Funding for FY13: **$150K**
- Planned funding FY14: **$250K**

PARTNERS

- Industry (component manufacturers, automotive OEMs, Station suppliers)
- Laboratories/universities (SNL, CDFA, PNNL, JRC, NHTSA, NIST, CSM, others)
- Codes & standards development organizations (SAE, CSA, ASME, ISO, UL, NFPA, IEC, GTR, ANSI, others)

Component R&D Relevance

Successful deployment of hydrogen infrastructure will require components that are proven to meet existing safety standards. NREL component R&D test efforts are focused on supporting component manufacturers and system installers so that fully tested and certified hardware is available.



Energy System Integration Laboratory

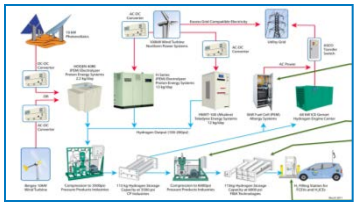
New laboratory facility in NREL's ESIF – Energy Systems Integration Facility

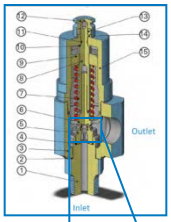
Component R&D Approach

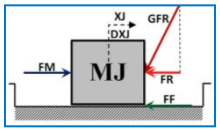
Approach	Barrier*
Work closely with codes and standards technical committees to develop test requirements with sound technical basis	G. Insufficient technical data to revise standards K. No consistent codification plan and process for synchronization of R&D and code development
Integrate DOE/NREL component test projects with Safety Codes and Standards program	C. Safety is Not Always Treated as a Continuous Process
Support hydrogen manufacturers and system suppliers with safety/reliability analysis and testing that can facilitate pre-certification of components and systems	F. Enabling national and international markets requires consistent RCS J. Limited Participation of Business in the Code Development Process
Publish technical reports for general use by stakeholders and NREL outreach activities	A. Safety Data and Information: Limited Access and Availability

* Barriers are based on 2012 DOE MYRD&D SCS Section 3.7.5

Component R&D Approach: Test Hierarchy







	Description	Advantages	Limitations
System Level Field Testing	Data from hydrogen demonstration and other installations including NREL Technology Validation and NREL wind to hydrogen data.	• Large statistical sample size • Actual stresses	• Data fidelity limits • Limited controls on stresses
Component Level Laboratory Testing	Reliability and accelerated life testing at the component level (including production and development hardware)	• Actual hardware • Laboratory control of stresses	• Costly multiple sample run • Proprietary issues • Difficult to measure degradation
Sub-Component Laboratory Testing	Reliability and accelerated life testing at the sub-component level (such as check valves used in hydrogen compressors)	• Actual hardware • Laboratory control of stresses • Less costly than full component testing	• Costly multiple sample run • Proprietary issues • Difficult to measure degradation
Mechanical Element Testing	Fundamental testing of mechanical element root cause failure modes caused by friction, wear, stress, fatigue and other mechanisms.	• Test design flexibility • Statistical sampling • Root cause isolation • Data can be easily shared	• Scaling to component level may be difficult • Special apparatus

ESIF Hydrogen Lab Milestones

- **Laboratory occupancy – *Spring 2013***
- **First laboratory operational – *Summer 2013***
  (Sensor Test Lab was one of the first operational labs in ESIF)
- **House hydrogen electrolysis production startup – *Spring 2014***
- **High pressure test cells operational – *planned Summer 2014***

ESIF - Energy Systems Integration Facility **New NREL laboratory facility, includes sensor lab, high pressure test lab, characterization lab, system integration lab, secure data room and high performance computing.**

AMR 2013 Slide - Future Test Priorities

- **Compressor Reliability**
  - Root cause MTBF (Mean Time Between Failure) analysis and experimental study
- **Materials of Construction in Hydrogen Service**
  - Emeryville pressure relief device extended service pressure testing
- **Flow Meter Accuracy**
  - Partner with manufacturers in developing solutions to dispensed hydrogen metering accuracy
- **Hose Reliability**
  - Failure mode investigation of existing dispenser hose designs and accelerated life testing
- **Receptacle Wear & Nozzle Durability Study**
  - Accelerated life testing , extending CNG results to hydrogen operating conditions
- **Low Temperature Sealing**
  - Mechanical element testing of seal designs at pressure and temperature limits

NREL Designed Gravimetric/Volumetric Hydrogen Dispensing Apparatus

Component testing is being coordinated through multiple subprograms to leverage NREL's core capabilities

FY2014 Approach

- **Compressor Reliability**
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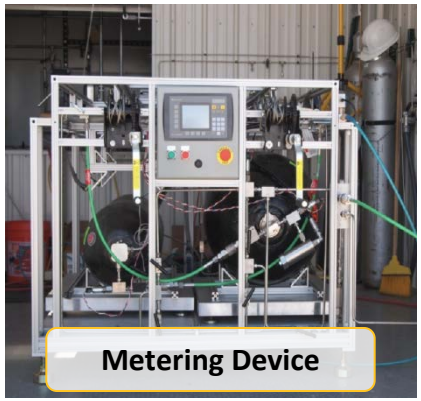
- **Compressor Reliability funded through FCTO Technology Validation**
- **Pressure Relief Valve failure mode investigation funded through FCTO Safety Codes and Standards**
- **Flow Metering project completed through leveraged funding from the State of California**
- **Hose Reliability project funded through FCTO Delivery Program**
- **Wear and Durability data will be outcome of hose reliability testing using robotic connect-disconnect**
- **Low Temperature sealing data is outcome of other component testing and ESIF systems operations**

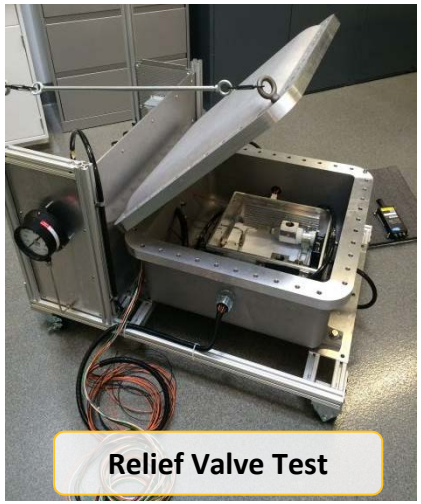
Component testing is being coordinated through multiple subprograms to leverage NREL's core capabilities

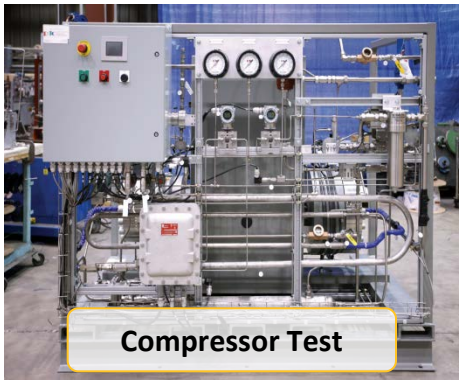
Component R&D Crosscutting Accomplishments

- Codes and standards test protocols used to develop component test plans
- Technical Committee contacts utilized for hose manufacturer participation
- SCS funds provided metering apparatus test support in California
- Test results will be used as basis for next generation of codes and standards requirements


Metering Device

Hose/Dispenser Test

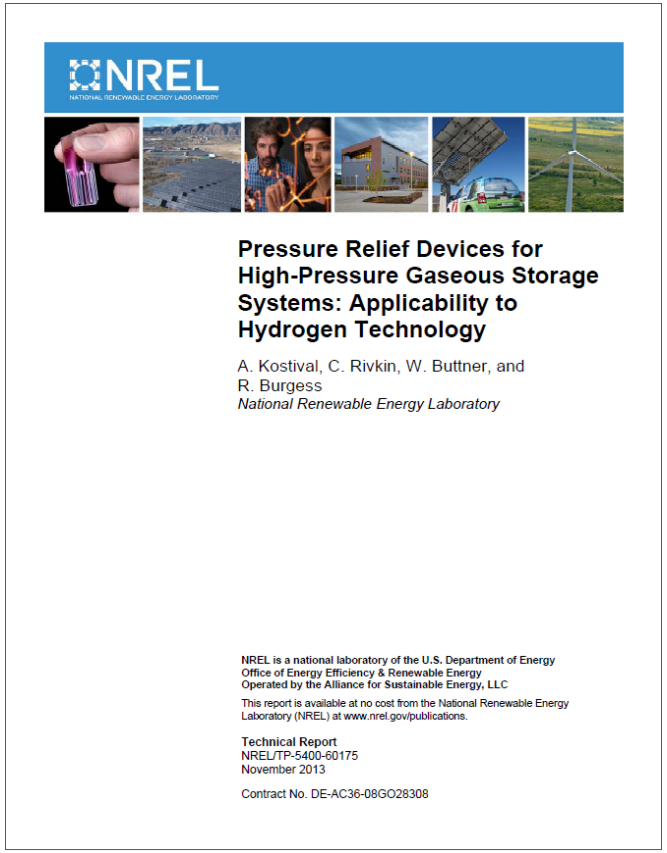

Relief Valve Test


Compressor Test

SCS subprogram is providing interface between standards development organizations and DOE supported component test programs

Pressure Relief Valve Accomplishments at NREL

- **NREL Technical Report**
  - "Pressure Relief Devices for High-Pressure Gaseous Storage Systems: Applicability to Hydrogen Technology"
    - Peer reviewed report on relief valve applications in hydrogen environments
- **PRV Accelerated Life Testing**
  - Qualitative reliability testing
  - Failure mode investigation



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Pressure Relief Devices for High-Pressure Gaseous Storage Systems: Applicability to Hydrogen Technology

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Technical Report
NREL/TP-5400-60175
November 2013

Contract No. DE-AC36-08GO28308

http://www.nrel.gov/docs/fy14osti/60175.pdf

Relief Valve Failure Mode Investigation

- Replicate known failure under controlled laboratory conditions
- Use valves that are “designed to fail” (use of high strength material under tensile loading)
- Phase I Investigation: Laboratory control of stressors include pressure loading and temperature cycling
- Phase II Investigation (if needed to produce failure): Additional stressors include pressure cycling, humidity, vibration and induced flaws

Project Timeline

- **JULY 2013 – FEBRUARY 2013**
  - Phase I Experiment Design
    - Safety Review
    - Experiment Setup
    - Preliminary Equipment Testing (No pressure)
    - Initial PRD Inspections
- **MARCH 2014 – AUGUST 2014**
  - Phase I Pressure Relief Device Testing
    - Conditions Tested:
      - Constant Elevated Pressure (7000 psi) in 100% Hydrogen Environment
      - Temperature Cycling – Simulated Annual Temperature Cycles
    - Regular PRD inspections to assess any crack growth and development
  - Phase II Experiment Design
- **AUGUST 2014 AND BEYOND**
  - Depending on results of Phase I testing, begin implementing Phase II testing to potentially include: Pressure Cycling, Humidity Cycling, Minor Vibration Exposure, Chemical Exposure and Flaw Testing

Experiment Operation Plan

Temperature:

- Control temperature to simulate extremes over an annual cycle to mimic accelerated service conditions
  - ≤ 0 °C and ≥ 50 °C
- Ramp/soak to temperature set point
  - Hold for ≈1 hour
  - Change set point, repeat
- 10-12 cycles per day
  - Acceleration ratio of 10:1 or higher

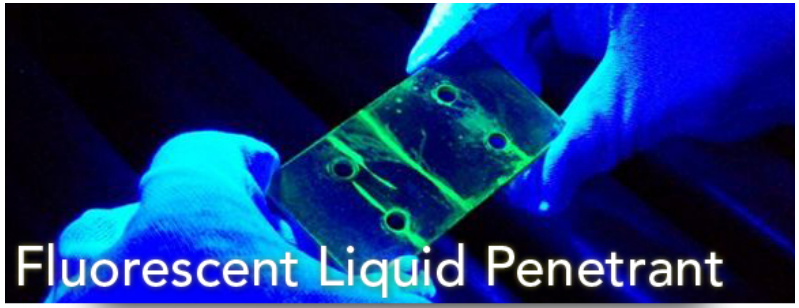
Pressure:

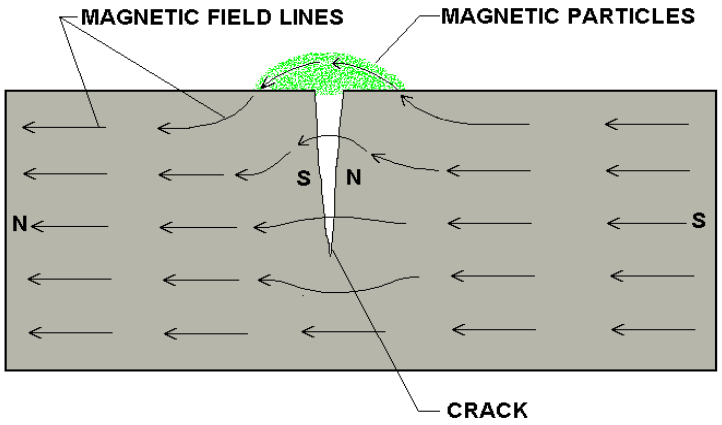
- Initial fill to 7,000 psi
- Expect some pressure change from temperature cycles
  - (6,800-7,200 psi?)
- Expect gradual pressure loss during testing
  - Automatic hydrogen filling with air operated valves
    - Ex. Fill when pressure drops below 6,000 psi for >5mins

Part Inspection/Evaluation

Collaboration with Colorado School of Mines Metallurgy Lab

- Regular inspection of samples for crack growth and propagation using:
  - Dye Penetrant Inspection
    - Use penetrating UV reactive liquid to inspect part for cracks
  - Ultrasonic (Pulse-Echo) Inspection
    - Use high frequency sound waves to map internal structure of component
  - Magnetic Particle Inspections
    - Similar to dye penetrant, but magnetic field is applied to part and fluorescent iron filings are used to indicate surface/subsurface cracks





Potential Experiment Results

Scenario A:

Crack propagation leading to failure similar to Emeryville

- Shows hydrogen embrittlement was primary factor in failure, indicates need to search out and remove these devices from service before additional failures occur
- Indicates manufacturing damage only accelerated primary failure mechanism

Scenario B:

No crack growth or device failure

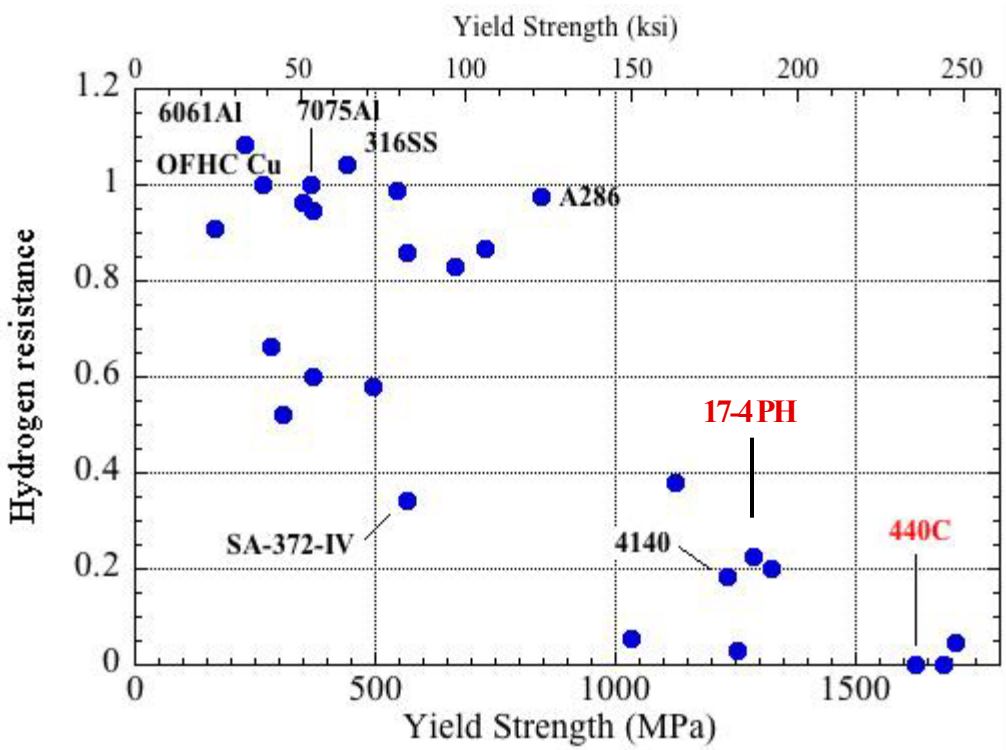
- Indicates manufacturing damage was driving factor in Emeryville failure; hydrogen embrittlement alone wasn't enough to cause failure
- Could result in asking the question: What would cause a failure?
  - Implement Pressure & Humidity Cycling, Vibration Exposure

Determine necessary and sufficient conditions required to produce known field failure

Case Study: Brittle material design

- **Brittle materials are used for properties such as strength, conductivity, optics, hardness etc.**
- **Understanding hydrogen effects on materials**
  - Sandia National Lab: Technical Reference for Hydrogen Compatibility of Materials, source: http://www.sandia.gov/matlsTechRef/
- **Design factors for brittle materials**
  - Compressive loading will suppress crack growth
    - No tensile loading or residual tensile load
  - Non pressure bearing structure
  - Manufacturing quality control
  - Understanding failure modes and effects

Hydrogen Effect on Ductility (reduction in area)

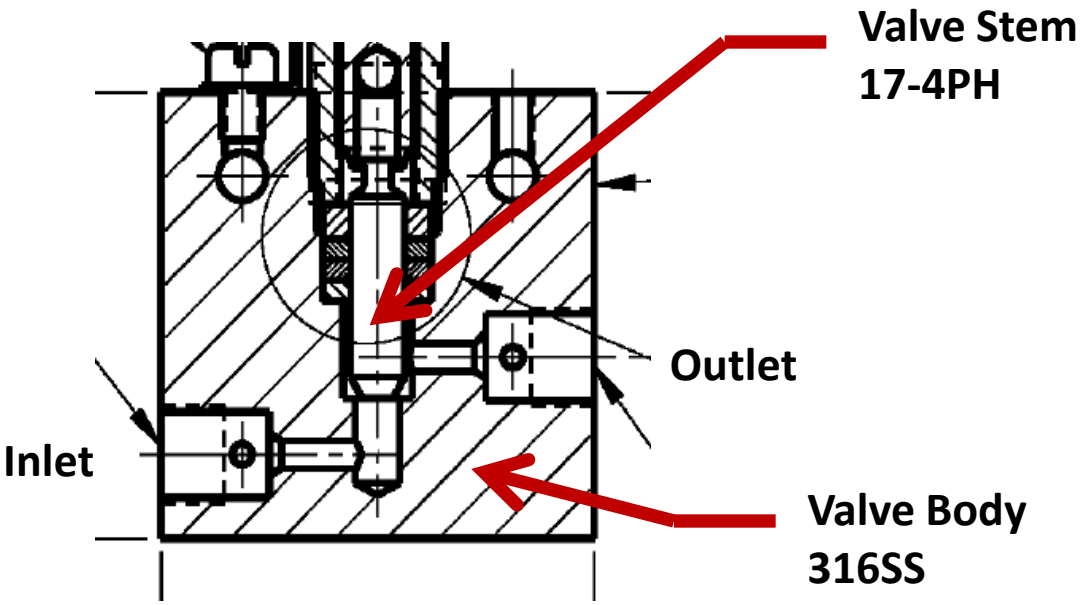


Hydrogen susceptibility as a function of yield strength for a range of structural alloys. The susceptibility is the ratio of reduction of area in a tensile test measured in gaseous hydrogen at pressure of 69 MPa relative to measurement in helium at the same pressure.

Reference: Data from Rocketdyne, Walter, R. & Chandler, W. (1968, October), *Effect of High Pressure Hydrogen on Metals,* ASM Conference, Detroit, MI

Case Study: ESIF pneumatically operated valve

- Collaboration with SANDIA for material selection
- Dissimilar materials are required to provide reliable sealing surface with a low risk of galling
- Work with manufacturer to understand operational history of valve in h_2 service and verify compressive load
- ESIF lab used to provide real world operating conditions
- Failure mode is safe release of hydrogen up vent stack



High strength 17-4PH stem seating against dissimilar 316SS

Material selection in hydrogen is dependent on many design factors

Component Outreach Accomplishments

- **Codes and standards technical committee support**
  - NREL is hosting joint NFPA 2/52 meetings, July 2014
  - Continued monitoring of C&S activities to identify gaps and needs
  - Contract support of DOT GTR validation
- **NREL/Sandia H2 FIRST implemented to provide structure for stakeholder input and prioritization of critical tasks**
- **NREL Component Webinar planned for May 2014**
  - Provide educational information to key stakeholders
  - Solicit input during Q&A session

NREL Component Collaborations

- Codes and Standards Development Organizations - SAE, CSA, ASME, ISO, UL, NFPA, IEC, GTR, ANSI, others
- Laboratories/universities - SNL, CDFA, PNNL, JRC, NHTSA, NIST, CSM, others
- CRADA (Cooperative Research and Development Agreement) in place with compressor manufacturer
- H2FIRST collaboration in place with Sandia National Labs includes component safety/reliability
- Collaboration across DOE subprograms including Technology Validation program for systems level safety/reliability integration

Accomplishments and Progress: Responses to Previous Year Reviewers' Comments

- **"To qualify a meter, the testing device needs to be at least 10 times more accurate. For example, this means to qualify a meter at 1.5%, the qualifying device needs to be 0.15% accurate. Someone in the project needs to do this…"**
  - Response: NREL has leveraged metering efforts with CDFA, and it should be clarified that the 10 times statement applies to MMQ (minimum measured quantity). A qualifying standard should be "less than one-third of the applicable device tolerance" per NIST Handbook 44, Appendix A, Section 3.2 (and as adopted by the California Code of Regulation)
- **"It would be good to see tighter partnering with other laboratories in this area-specifically the materials science team at Sandia"**
  - Response: NREL has worked closely with Sandia National Laboratory in putting together the H2FIRST collaboration. This partnership with Sandia will strengthen our mutual DOE projects and provide a framework for feedback and cooperation
- **"In situ PRD testing is needed and has been requested for several years"**
  - Response: Project started in FY14 to evaluate a known failure mode and to provide qualitative reliability data

Remaining Challenges and Barriers

- **Harmonization of national and international RCS**
  - Harmonization with international standards is a challenge requiring participation with organizations such as NOW, GTR, ISO and others. Use of sound test results as a basis for RCS will help accelerate acceptance
  - R&D results can be utilized to develop improved RCS, but coordination with revision cycles can result in significant time lag to implementation
- **Limited industry engagement**
  - Component suppliers will increase involvement and investment in parallel with hydrogen market growth. Availability of components at the early stages of market growth is a potential barrier requiring DOE support
  - Industry proprietary knowledge concerns may limit participation. By modeling our collaborations after the successes of the sensor lab industry participation, we will help assure that industry partners are confident that critical data is properly protected
- **Safety tends to be reactive**
  - Through collaboration with stakeholders and end users, and through data collection Technology Validation activities, a proactive approach to component R&D will provide benefits to circumventing future safety incidents
- **Material selection knowledge gaps**
  - In situ testing at the component level has added challenges of three dimensional stress state variations with corresponding effects on material properties
  - Component reliability testing with a statistically significant sample size typically requires a dozen or more parts, which creates a challenge based on available resources

Proposed Future Work

- **FY14 activities, completion date September 2014**
  - Complete Phase I PRV testing, determine Phase II plans
  - Formal agreement in place with hose manufacturer for participation in R&D hose reliability study
  - Formal agreement in place with additional compressor manufacturer with plans for expanded onsite 70 MPa compression for ESIF dispensing
- **FY15 future work priorities**
  - Pressure relief valve phase II testing – extreme environment tests
  - Dispenser flow meter testing in conjunction with CDFA
  - Ultrasonic flow meter testing on 70 MPa dispensing system, in collaboration with NIST flow measurement division
  - Collaboration with NRTL's to help enable NRTL/Manufacturer component certification efforts
  - Receptacle wear and nozzle durability accelerated life testing
  - Low temperature sealing testing and publication of best practices

Summary

Relevance: Safe deployment of hydrogen fuel cell technologies is dependent on components that are proven to perform safely and reliably as measured against new safety and performance standards

Approach: NREL will work with manufacturers, installers and NREL's Technology Validation Program to prioritize gaps, then work toward closing those gaps by conducting hydrogen component R&D and performance validation.

Accomplishments & Progress: NREL's is leveraging component R&D accomplishments, having provided a sound technical basis for new hydrogen codes and standards and is now operating under a new MYPP to conduct root cause analysis and R&D testing to improve safety and reliability of hydrogen system components.

Collaborations: Collaboration with codes and standards technical committees, component manufacturers, industrial partners and hydrogen fuel cell applications experts has been a key part of NREL's success in advancing component program objectives

Proposed Future Work: NREL will continue to work with codes and standards technical committees to identify R&D gaps and to utilize the ESIF laboratory to conduct basic engineering R&D aimed at closing technology gaps.

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Technical Back-Up Slides

ESIF Facility Utilization

Testing Area:

- ESIF C341/342
  - Dedicated high pressure testing zones
- One of the first facility "users", project will help ESIF Operations team to evaluate user facility procedures

PRV Testing Apparatus Design

- **Project Specifications:**
  - Temperature Control
    - (≤ 0 °C and ≥ 50 °C)
  - Allow simultaneous testing of two PRDs
  - Portable/Configurable
  - Safe

- **Subsystems:**
  - External Housing
    - Explosion Proof Enclosure
  - Heat Transfer System
    - Thermoelectric modules for cooling, resistance heater
  - Automation/Data Collection
    - Fully automated operation
  - High Pressure Components
    - 7,000 psi operation
  - Mounting Chassis
    - 8020 Frame/Housing

ESIF Component Test Utilization

Energy Systems Integration Facility

Electricity Laboratories

1. Power Systems Integration
2. Smart Power
3. Energy Storage
4. Electrical Characterization
5. Energy Systems Integration

Thermal Laboratories

6. Thermal Systems
7. Thermal Storage Materials
8. Optical Characterization and Thermal Systems

Fuel Laboratories

9. Energy Systems Fabrication
10. Manufacturing
11. Materials Characterization
12. Electrochemical
13. Energy Systems Sensor
14. Fuel Cell Development
15. High-Pressure Testing

Data, Analysis, and Visualization

16. ESIF Control Room
17. Visualization Room
18. Secure Data Center
19. High Performance Computing

- Component Testing Laboratory Capability within ESIF

Component R&D Summary

- Work with codes and standards technical committees on revision efforts as these technical documents are vetted through early market system operation
- Identify root cause safety/reliability issues by utilizing statistical data provided through NREL Technology Validation
- Conduct component safety analysis and testing
- Develop user facility capabilities in new NREL ESIF building

ESIF - Energy Systems Integration Facility
New NREL laboratory facility, includes sensor lab, high pressure test lab, characterization lab, system integration lab, secure data room and high performance computing.

ESIF Component Test Utilization

Work Scope

	Energy Systems Integration Laboratory	Material Characterization Laboratory	Energy Systems Sensor Laboratory	High Pressure Testing Laboratory	Secure Data Center Technology Validation	High Performance Computing
4.1 Compressor Reliability	x	x		x		x
4.2.Materila of Construction in Hydrogen	x	x	x	x		
4.3.Hose Reliability	x	x	x	x	x	
4.4.Flow Meter Accuracy	x			x		
4.5.Low Temperature Sealing	x	x	x	x	x	
4.6.Technology Validation Study					x	x
4.7.Receptacle Wear and Nozzle Durability	x	x	x	x		
4.8.Temperature Activated Pressure Relief Device	x	x	x	x		
4.9. Certification & Listing of Components	x		x	x		
4.10.Localized Fire Scenarios	x	x				x
4.11.COPV Production and Reliability	x	x		x		x
4.12.Hydrogen Safety Sensor Performance	x	x	x	x		x