

SCS005: R&D for Safety, Codes and Standards: Materials and Components Compatibility

Brian Somerday
Principal Investigator

Chris San Marchi
Team member, SCS manager and presenter

Daniel Dedrick
Hydrogen Program Manager

Sandia National Laboratories

DOE Hydrogen and Fuel Cells Program Annual Merit Review
June 18, 2014

SCS005

This presentation does not contain any proprietary, confidential, or otherwise restricted information

Overview

Timeline

- Project start date: Oct. 2003
- Project end date: Sept. 2014*

* Project continuation and direction determined by DOE annually.

Budget

- FY13 DOE Funding: \$0.8M
- Planned FY14 DOE Funding: \$0.6M
- Total DOE Project Value: \$7.2M

Barriers

- A. Safety Data and Information: Limited Access and Availability
- F. Enabling national and international markets requires consistent RCS
- G. Insufficient technical data to revise standards

Partners

- **SDO/CDO participation:** CSA, ASME, SAE, ISO
- **Industry:** FIBA Technologies, European cylinder manufacturer, Swagelok Company, Hy-Performance Materials Testing, Carpenter Technology
- **Universities:** Boise State
- **International engagement:** I2CNER (Kyushu University, Japan), AIST-Tsukuba (Japan), KRIS (Korea), Mathryce (EU)

Relevance and Objectives

Objective: Enable technology deployment by providing science-based resources for standards and hydrogen component development and participate directly in formulating standards

Barrier from 2013 SCS MYRDD	Project Goal
<p>A. Safety Data and Information: Limited Access and Availability</p>	<p>Develop and maintain material property database and identify material property data gaps</p>
<p>F. Enabling national and international markets requires consistent RCS</p>	<p>Develop more efficient and reliable materials test methods in standards</p> <p>Design and safety qualification standards for components (SAE J2579, ASME Article KD-10) and materials testing standards (CSA CHMC1)</p>
<p>G. Insufficient technical data to revise standards</p>	<p>Execute materials testing to address <i>targeted</i> data gaps in standards and critical technology development</p>

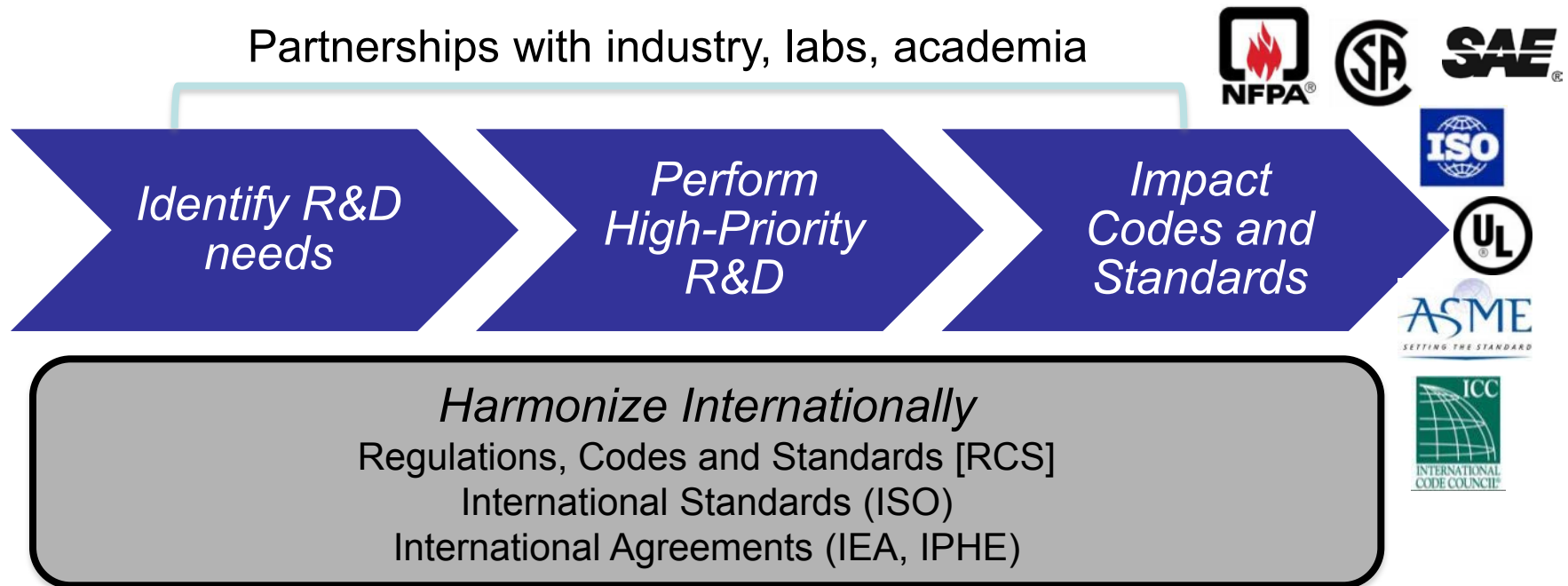
Relevance:

Materials Compatibility and Components project impacts multiple standards

- **CSA CHMC1**
 - Materials testing and data application standard
 - Sandia provides leadership in technical committee and document preparation
 - Working on evaluation of methodology
- **SAE J2579**
 - Hydrogen vehicle fuel system standard
 - Sandia serves as U.S. technical lead on addressing hydrogen embrittlement
- **ASME Article KD-10**
 - Standard on high-pressure hydrogen tanks for transport and storage
 - Sandia provides data on exercising and improving materials test methods
 - Reporting progress on optimizing fatigue crack growth testing to former chair of ASME Project Team on Hydrogen Tanks

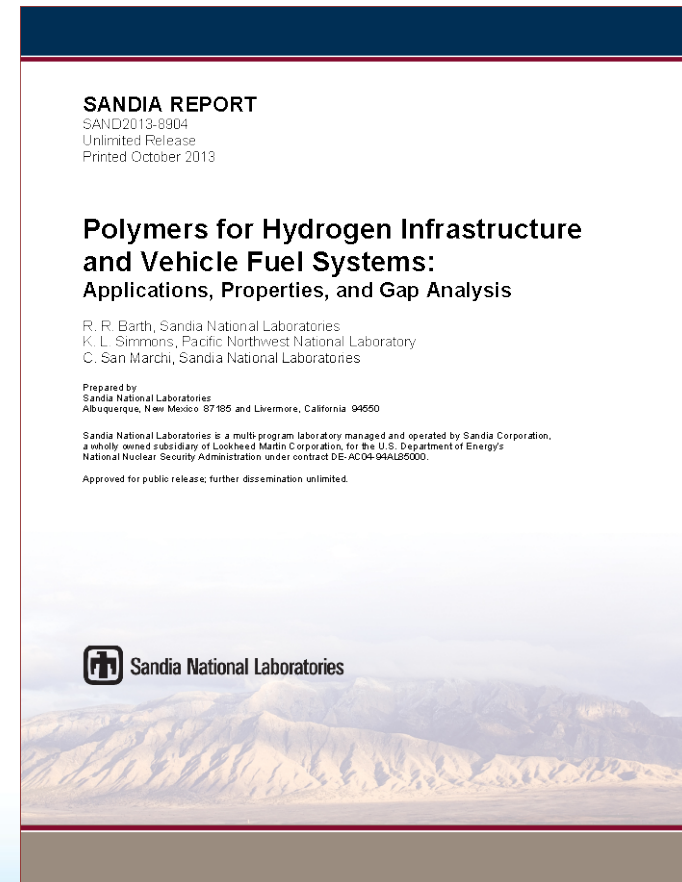
Program Approach

The Safety, Codes and Standards program coordinates critical stakeholders and research to remove technology deployment barriers



Program Accomplishment: Identification of gaps in Hydrogen Compatibility of Polymers

- Objectives:
 - Review polymers in hydrogen infrastructures
 - Survey properties of polymers related to high-pressure hydrogen service
 - Identify gaps to assessing suitability of polymers for hydrogen service
- Motivated by DOE Workshop on *Polymer and Composite Materials Used in Hydrogen Service* (October 2012)
- Collaboration with PNNL
- Collaboration between FCTO program elements: Safety Codes and Standards and Delivery

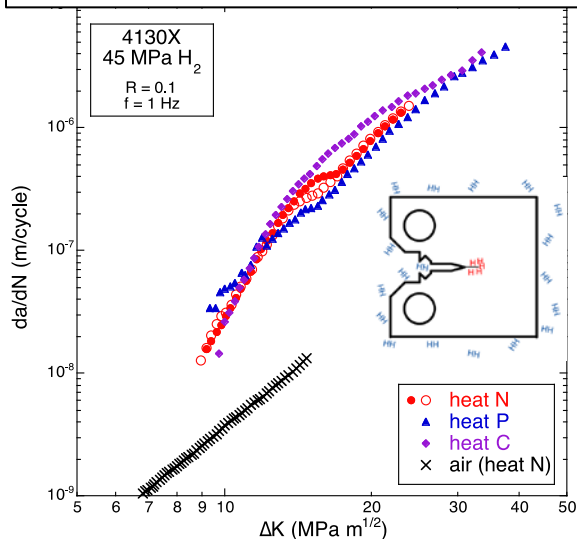


Project Approach and Milestones

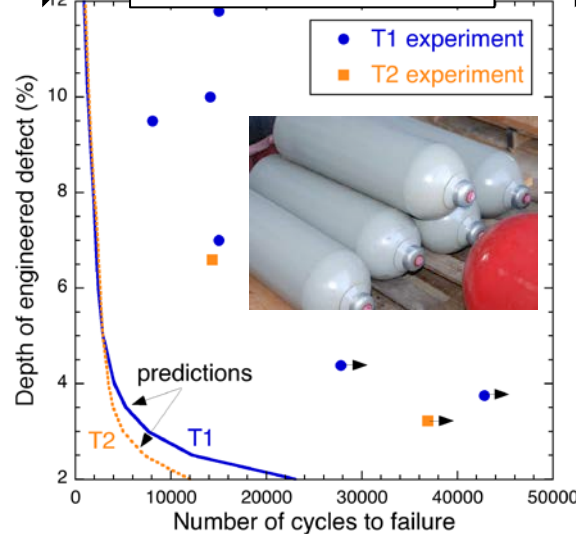
MYRD&D 2012 Barrier	FY14 Milestone	Status
<p>A. Safety Data and Information: Limited Access and Availability</p>	<p>Develop material property database</p>	<p>Working with MDMC and Granta to build schema for hydrogen effects in materials database in Granta MI</p>
<p>F. Enabling national and international markets requires consistent RCS</p>	<p>Preliminary evaluation of stress-based fatigue method outlined in CSA CHMC1 standard</p> <p>Enable completion of standards through committee leadership and data evaluation</p>	<p>Initial data shows promising results for austenitic stainless steels</p> <p>Participating in discussions of materials qualification standard for non-metals</p>
<p>G. Insufficient technical data to revise standards</p>	<p>Evaluate a wider range of welding practice for construction of hydrogen installations</p> <p>Develop capability for variable-temperature testing in high-pressure H₂ gas</p>	<p>Completed room temperature tensile testing of “hand-welded” tubing (same tubing as studied for automated welds)</p> <p>Finalized design plans for remaining hardware purchases</p>

Previous Accomplishment: Integrated materials and component testing program illuminated limitations of methodologies

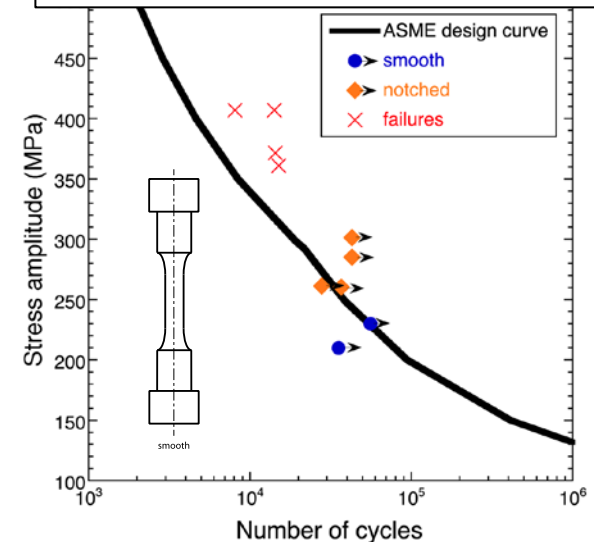
Fatigue crack growth method



Component tests

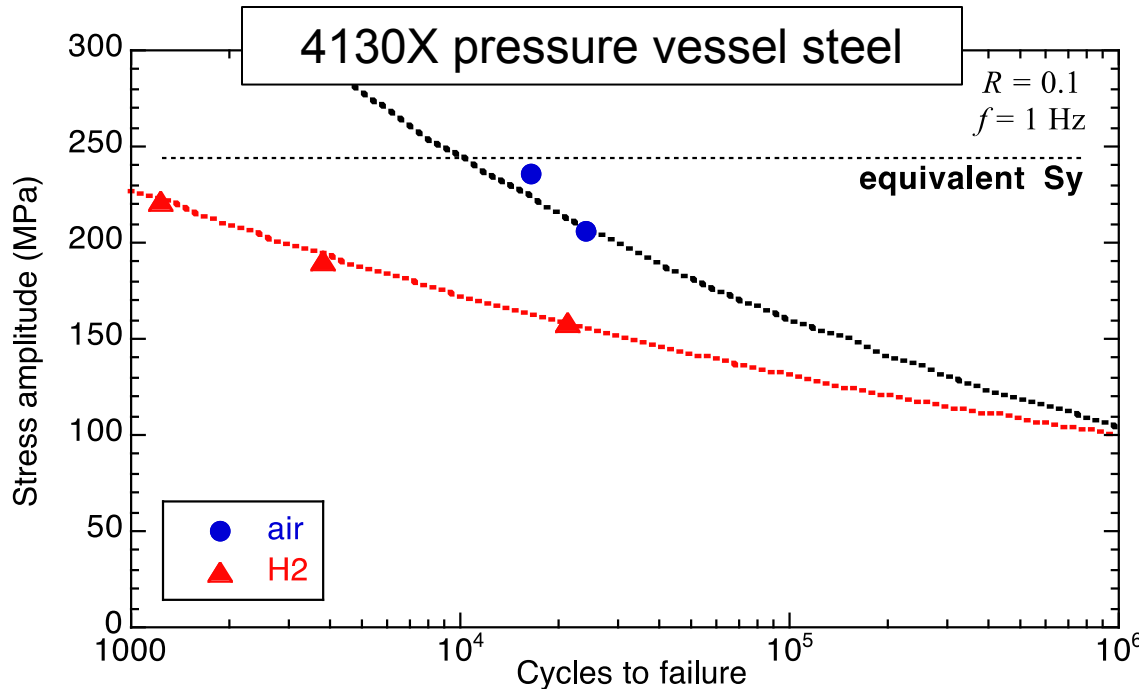


Stress-based fatigue method

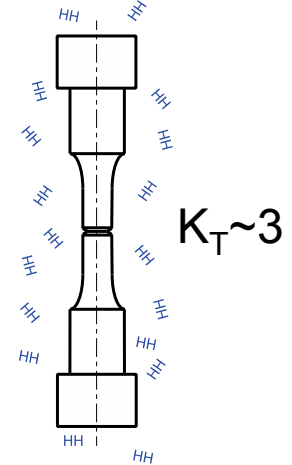


- Predictions using fatigue crack growth testing (ASME BPVC VIII.3 protocol KD-4) is very conservative, if cracks take time to initiate
- Stress-based fatigue method (ASME BPVC VIII.3 protocol KD-3) offers an alternative to fracture mechanics
- Available stress-based fatigue data (S-N curves) in gaseous hydrogen is very limited in the literature

Accomplishment: Adapt stress-based fatigue method to tension-tension configuration in gaseous hydrogen

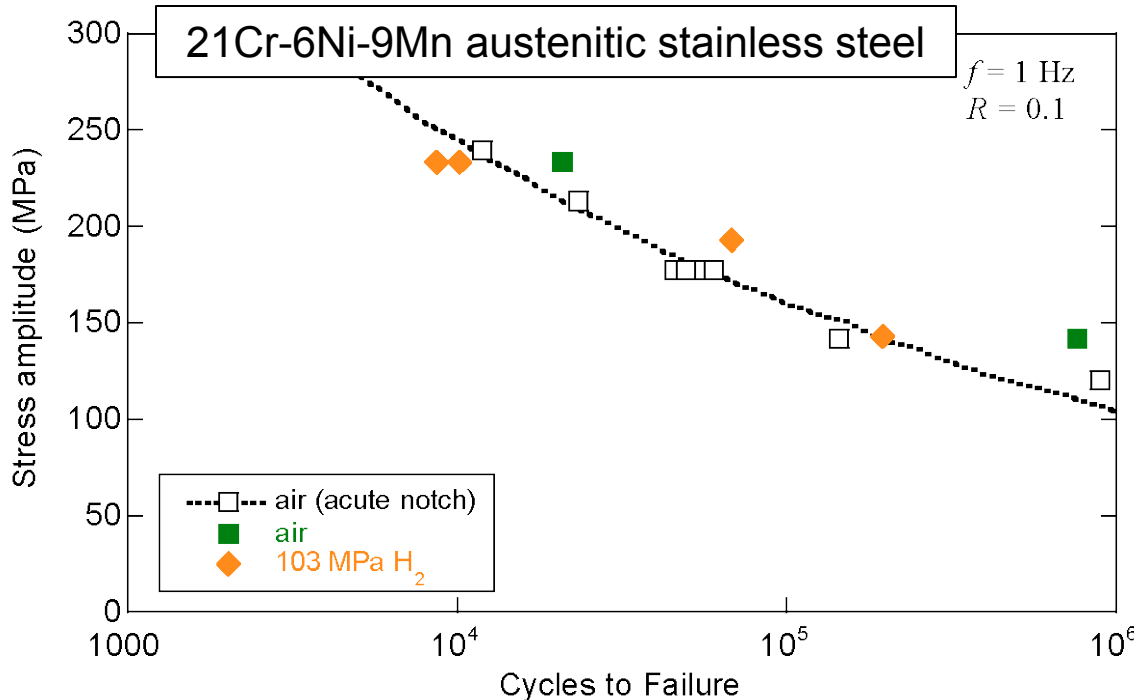
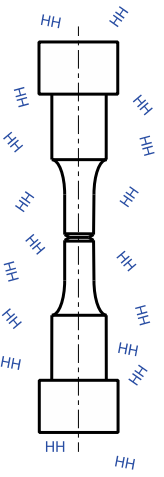


Tension-tension fatigue of standard notched tensile specimen (after ASTM G142)



- Initial results for pressure vessel steel follow anticipated trends
- Additional data is needed to demonstrate reproducibility and consistency, as well as to coordinate with efforts in the international community
- Test results serve as a means to evaluate one of the testing options in the new CSA CHMC1 standard

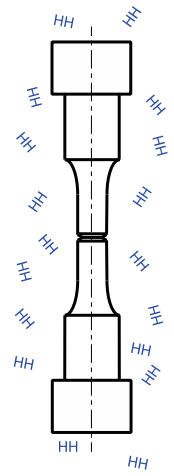
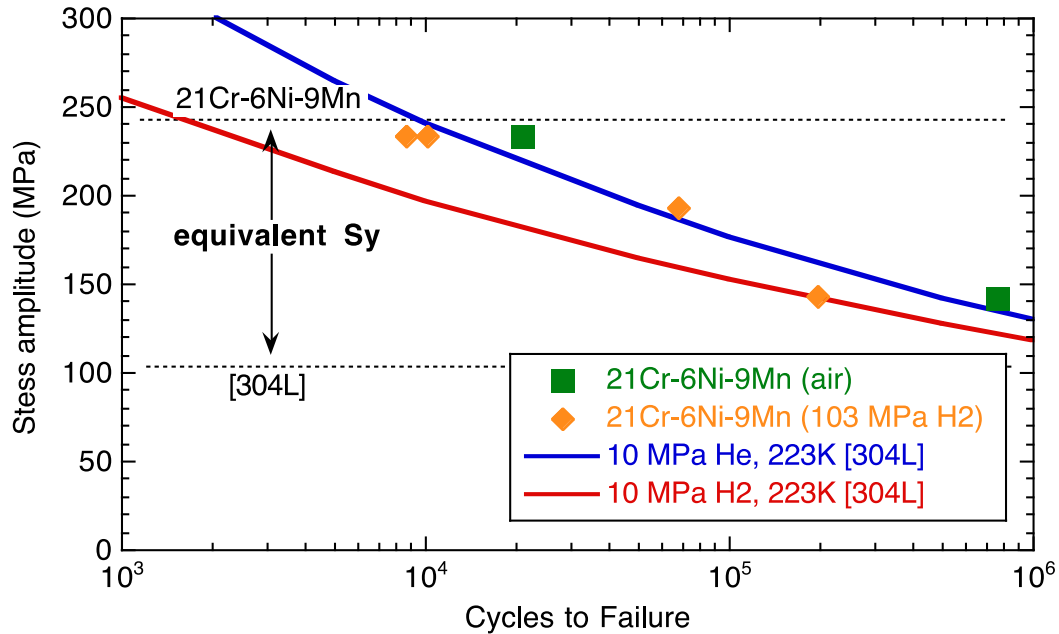
Accomplishment: Extend stress-based method in CSA CHMC1 to austenitic stainless steels



- Strength of annealed 21Cr-6Ni-9Mn is >2x strength of annealed type 316L
- Cost of 21Cr-6Ni-9Mn bar material is ~80% of type 316L bar

- Hydrogen reduces total fatigue life
- High fatigue stress can be achieved with cycles to failure greater than 10,000 cycles
- Broader evaluation of methodology requires testing under combination of low temperature and high pressure

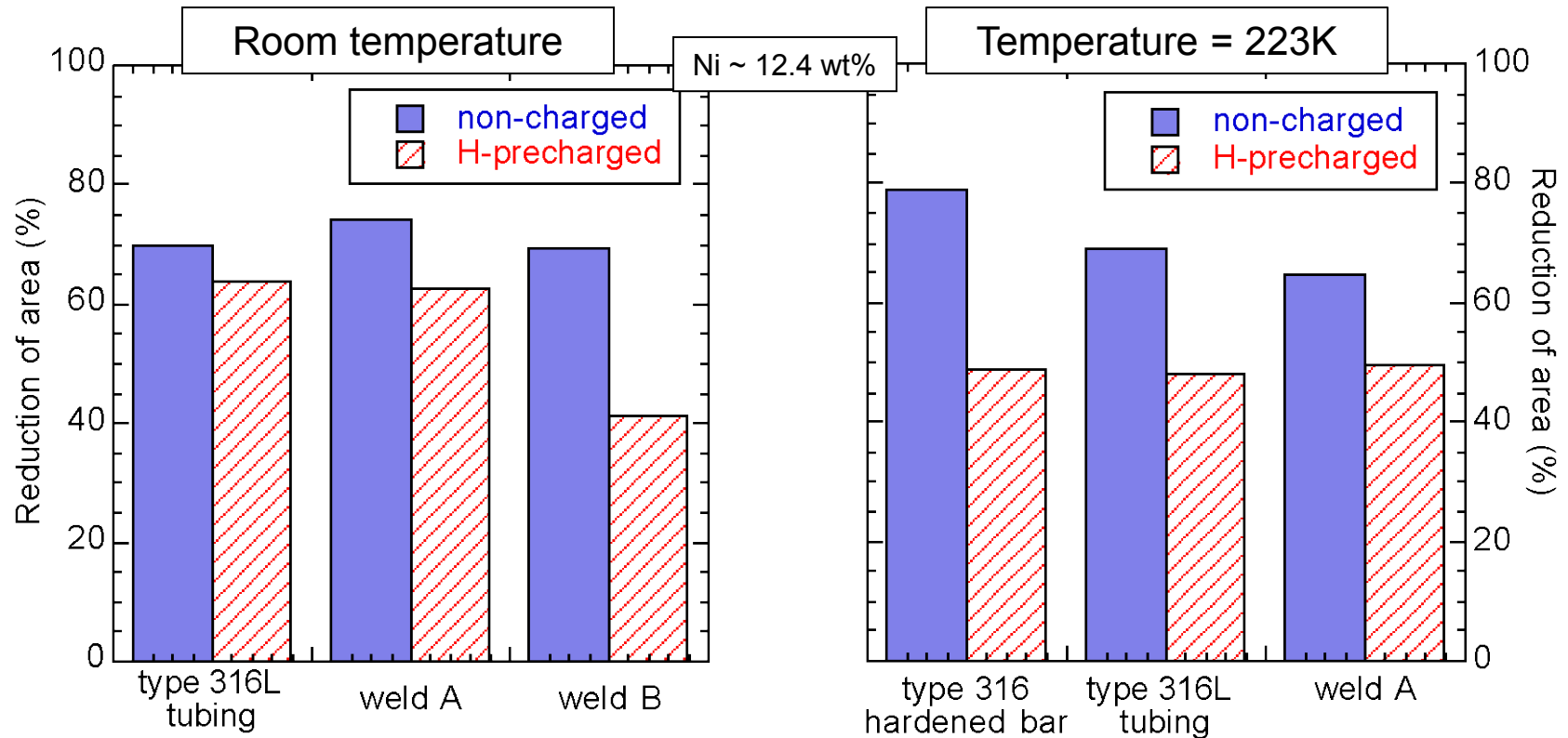
Accomplishment: Demonstrate use of stress-based fatigue data for design of hydrogen components (e.g., automotive)



- Curves are fit to data from the literature for relatively high-Ni (11.4%) austenitic stainless steel [304L-like] at temperature of 223 K
- Broader evaluation of methodology requires testing at low temperature and high pressure

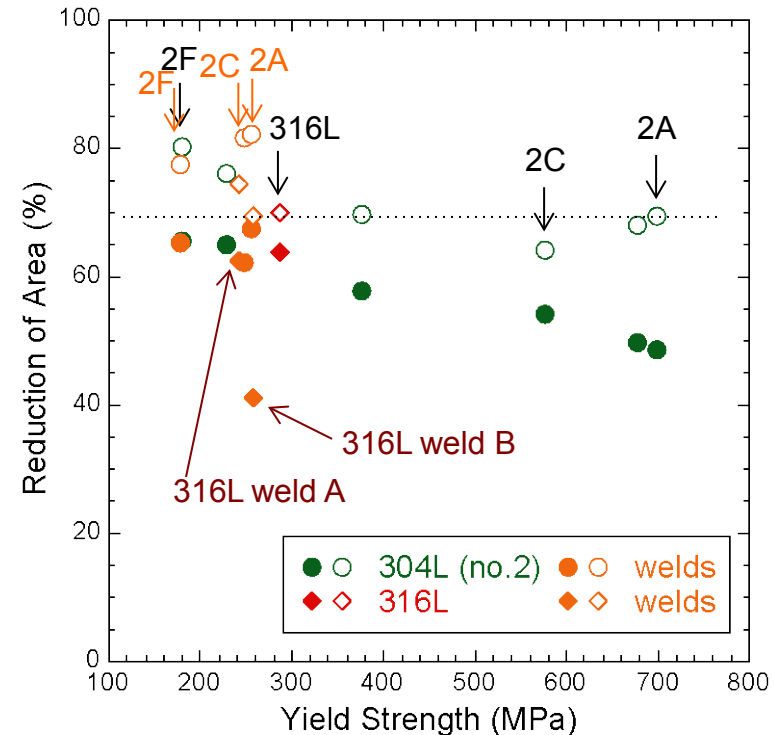
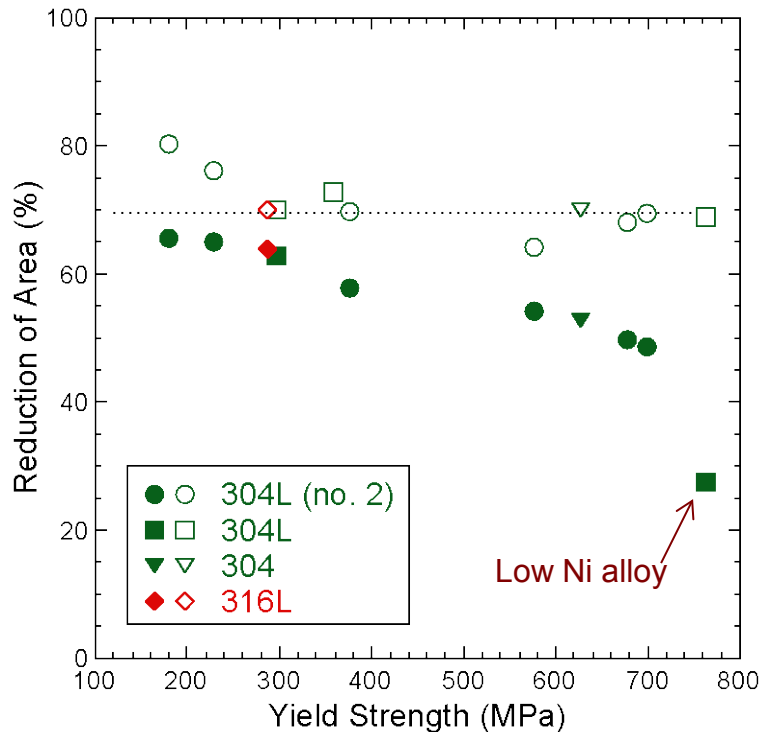
- Results are consistent with data from the literature on lower strength austenitic stainless steels
- Higher yield strength alloys allow access to higher applied fatigue stress
 - Higher allowable stress = less material (lower cost, lower weight)
- Combined higher allowable stress and lower cost materials = potential savings of >50% compared to 316L baseline

Previous Accomplishment: Automated welds behave similarly to base materials



- Hydrogen-assisted fracture is similar for base material, tubing and automated welds
- Low-temperature response (T=223K) is similar for welds and tubing (and base material)

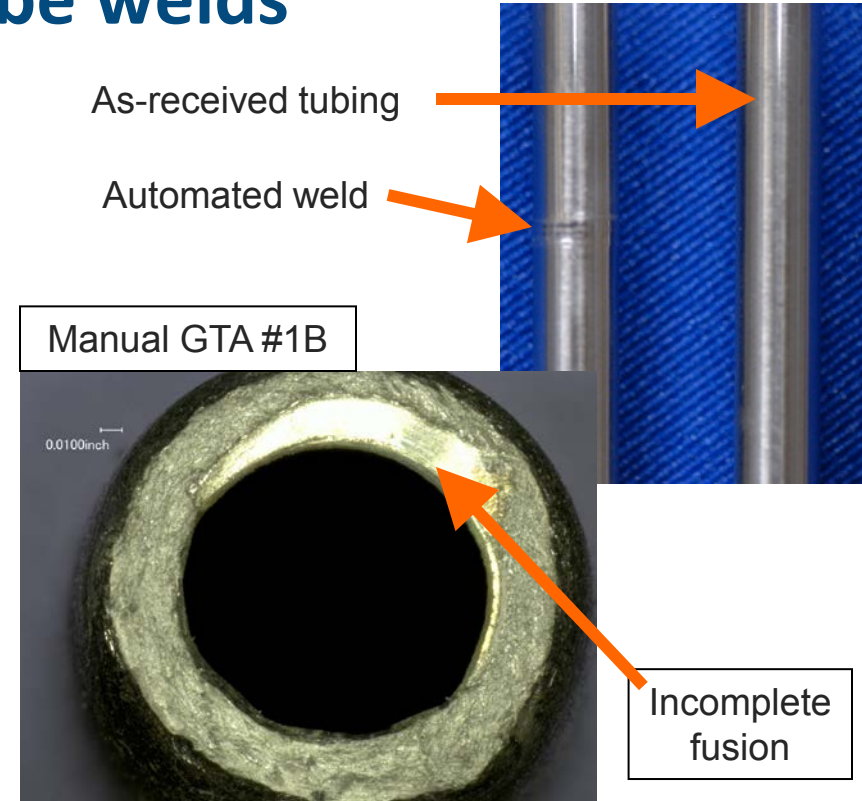
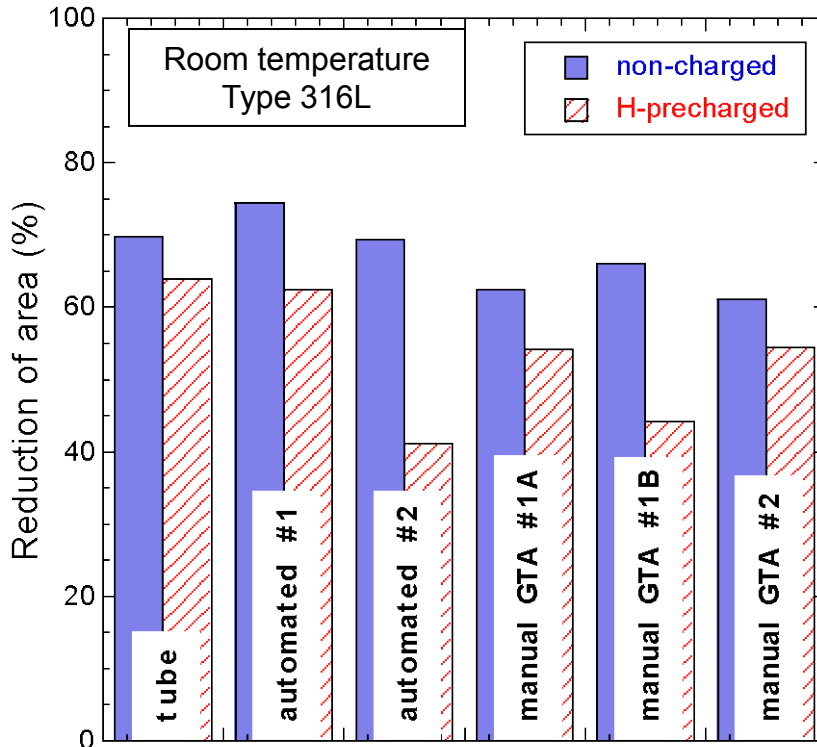
Accomplishment: Leverage other programs to show broad applicability of results for automated tube welds



- Results for a variety of tubing (including high-strength tubing) are consistent with premium 316L tubing and studies on raw materials
- Welds of type 304L behave similarly independent of initial strength of the tubing

from: "Hydrogen compatibility of austenitic stainless steel tubing and orbital tube welds" accepted to IJHE

Accomplishment: Evaluate welding variability by comparing manual tube welds to automated tube welds



- Tube butt welds have been produced by experts and non-experts using automated and manual welding procedures (single-pass GTA welds)
- Welds remain ductile when saturated with hydrogen
- In general, manual welding is more susceptible to incomplete fusion which affects ductility

Accomplishment: Update information resources

- Added comprehensive literature review on polymers to technical resources
 - SAND2013-8904
- Developing informational hydrogen resource pages to augment Technical Reference website
 - includes resources for other hydrogen programs at Sandia as well
- Working with the Materials Data Management Consortium (MDMC) and Granta to build schema for hydrogen effects in materials database in Granta MI

SANDIA REPORT

SAND2012-7321
Unlimited Release
Printed September 2012

**Technical Reference for Hydrogen
Compatibility of Materials**

C. San Marchi
B.P. Somerday

SANDIA REPORT

SAND2013-8904
Unlimited Release
Printed October 2013

**Polymers for Hydrogen Infrastructure
and Vehicle Fuel Systems:
Applications, Properties, and Gap Analysis**

R. R. Barth, Sandia National Laboratories
K. L. Simmons, Pacific Northwest National Laboratory
C. San Marchi, Sandia National Laboratories

Prepared by
Sandia National Laboratories
Albuquerque, New Mexico 87185 and Livermore, California 94550

Sandia National Laboratories is a multi-program laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Corporation, for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-AC04-04OR21400.

Approved for public release; further dissemination unlimited.

 Sandia National Laboratories

Response to Previous Year Reviewers' Comments

- ***FY13 Reviewer Comment: “The project team must show the capability to test at -50°C to have credibility for embrittlement testing.”***
 - We have made significant progress toward developing a system at Sandia for dynamic testing at low temperature in gaseous hydrogen (see technical backup slides).
 - At this point, we are prepared to procure the final components in this system.
- ***FY13 Reviewer Comment: “The relevance of testing weldments (1) under pre-charged conditions rather than directly in a hydrogen environment and (2) in short-term tensile tests only should be explained and justified.”***
 - The question being addressed is whether welds are more susceptible to hydrogen embrittlement than the base metal.
 - Previous work has demonstrated that testing stainless steels in hydrogen gas vs. testing hydrogen-precharged specimens yields similar trends; therefore comparative studies address this question.
- ***FY13 Reviewer Comment: “It would be preferable for SNL to collaborate with NIST and other researchers to avoid duplication ...”***
 - SNL does collaborate with NIST on materials compatibility, primarily through the Hydrogen Delivery sub-program.
 - To our knowledge, US-funded materials testing activities are complementary, not duplicative
 - SNL has effectively leveraged testing capabilities and expedited data generation through its international partnerships, such as with AIST

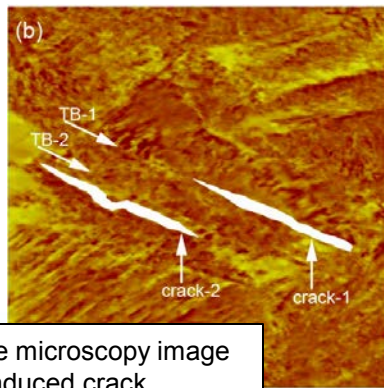
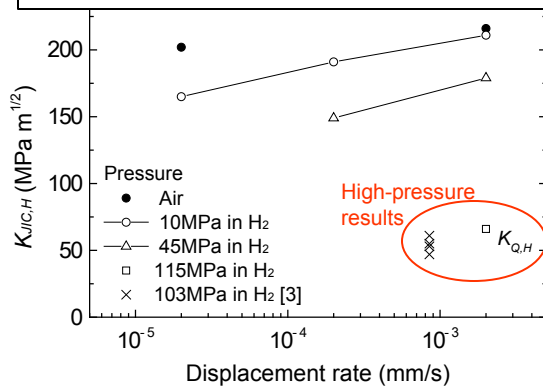
Collaborations

- Standards Development Organizations (SDOs)
 - Sandia technical staff lead and serve on committees (CSA, SAE, ASME)
- Industry partners
 - Partners provide technology-relevant materials and input into materials testing conditions (FIBA Technologies, Swagelok, cylinder manufacturers, Carpenter Technology)
- Universities
 - Student intern conducted prototyping and analysis for variable-temperature testing in high-pressure gaseous hydrogen system (Boise State)
- International research institutions
 - International Institute for Carbon-Neutral Energy Research (I²CNER)
 - Dr. Brian Somerday (Sandia) serves as Lead PI for Hydrogen Compatible Materials Division of I²CNER with influence on and access to *basic research* to complement *applied research* in Safety, Codes and Standards (e.g., predictive models for H₂-assisted fatigue)

Collaborations:

AIST-SNL collaboration actively pursuing two tasks to harmonize test methods and standards

Comparison of AIST and Sandia results for ASME SA372 Gr. J pressure vessel steel



Magnetic force microscopy image of hydrogen-induced crack initiation in stainless steels

- Task 1: Measurement of Ferritic Steel Fracture Properties in H₂ Gas
 - Despite different test procedures, “rising displacement” thresholds measured at AIST similar to previous results from Sandia
 - Results contribute to technical basis for developing “rising displacement” fracture threshold testing standard
- Task 2: Identifying Mechanisms for Hydrogen-Assisted Fracture in Stainless Steels
 - AIST characterized formation of microcracks in H₂-exposed 304 stainless steel using advanced surface science methods
 - Fundamental understanding of deformation and fracture behavior can lead to materials innovations, i.e., lower-cost, H₂-compatible stainless steels
- Joint publications (ASME PVP-2014 conference)
- AIST-SNL Joint Workshop (Livermore, Jan 2014)

Proposed Future Work

Remainder of FY14

- Complete first round of stress-based fatigue measurements in gaseous hydrogen for stainless steel and pressure vessel steels
- Interface with international programs (e.g., EU-supported Mathryce project) on development of stress-based fatigue methodologies for pressure vessels
- Formalize schema for material property database in Granta MI

FY15

- Identify needs of community for more comprehensive use for Technical Reference; for example, add stress-based fatigue data to TR
- Evaluate low-cost austenitic stainless steels using stress-based fatigue testing methodology with emphasis on relating performance bounds in hydrogen to material characteristics (e.g., high strength, low nickel, high nitrogen)
- Critically evaluate test methods in CSA CHMC1, including rate effects (AIST collaboration) and “safety factor method” option
- Develop R&D program with industry partner(s) to evaluate and improve resistance of high-strength structural metals to H₂-assisted fracture
- Procure remaining variable-temperature testing subsystems (with cost sharing), integrate subsystems, and demonstrate functionality

Summary

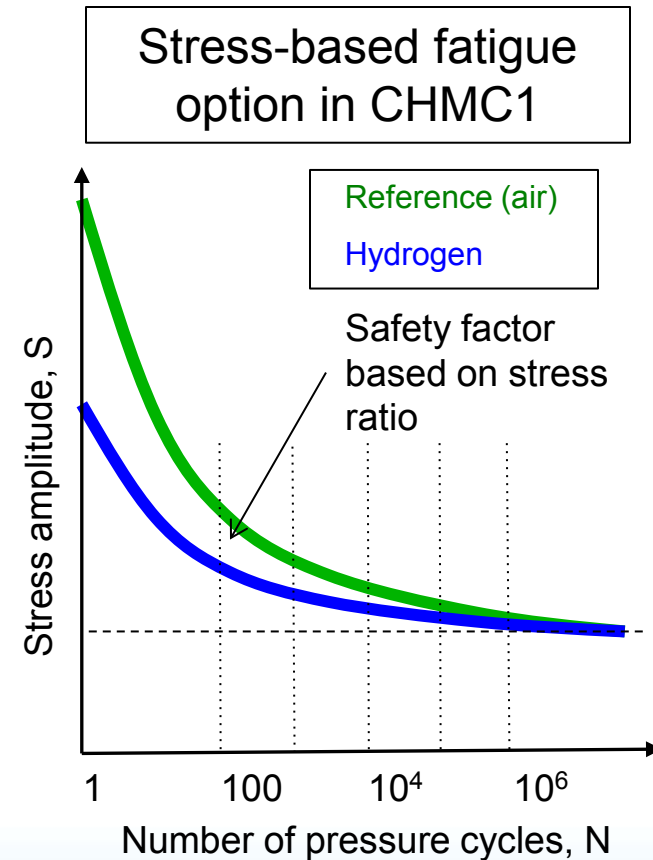
- Materials testing motivated by standards development and technology needs
 - Evaluating stress-based fatigue testing methodology
 - Measuring tensile ductility of H₂-exposed tube welds
- Working with experts toward structured database on hydrogen effects in metals
- Demonstrating leadership in materials testing
 - Developing new variable-temperature system
 - Leading international dialogue with OEMs on testing standard
- Concrete progress in developing standards that address hydrogen compatibility of components
 - CSA CHMC1 revision has been published
 - Initial discussions with CSA and CHMC committee chair on standard for non-metallics
- Maintaining active international collaborations
 - AIST (Tsukuba, Japan)
 - I²CNER (Kyushu University, Japan)
 - HYDROGENIUS (Kyushu University, Japan)

Technical Back-Up Slides

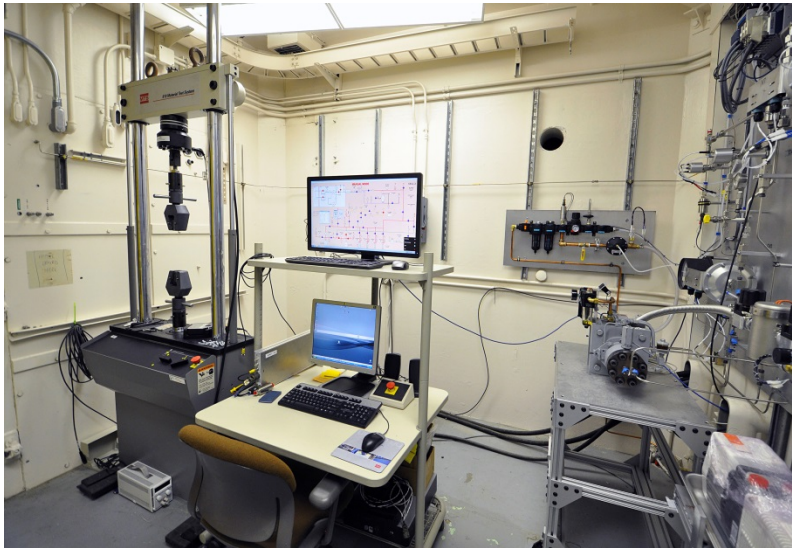


Accomplishment: Hosted informational meeting with Japanese OEMs on CSA CHMC1 methodology (November 2013)

- Goal: establish common understanding of materials qualification using CSA CHMC1 with emphasis on global harmonization
- Attendees: ~12 people: representation from CSA Group, I2CNER, JARI, OEMs, as well as from the CSA CHMC1 committee
- Output:
 - Communication on CHMC1 methodology
 - Clarification of the basis (and limits) of applicability of CHMC1 testing method
 - General rules for qualification of material classes
 - Clarification of rules in Japan



Accomplishment: Variable-temperature testing in high-pressure hydrogen: 2 of 3 subsystems installed



- Procured test frame, test controller, hydraulic pump, testing software

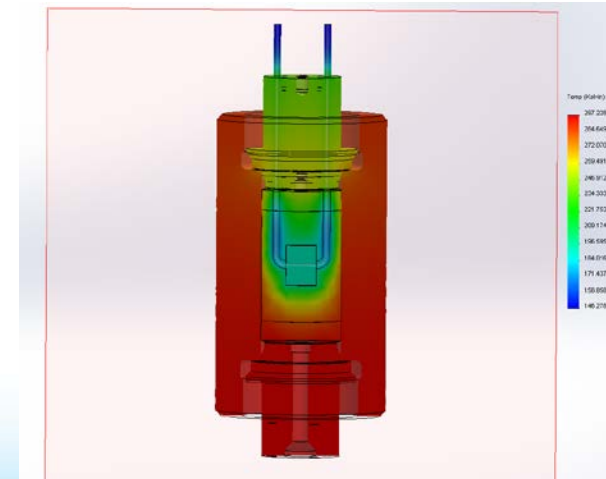


- Gas handling manifold designed and installed
- Software for manifold automation developed and exercised

Remaining subsystem: pressure vessel with variable-temperature function

Accomplishment: Design specifications for variable-temperature pressure vessel finalized

- March 2013 workshop Advancing Materials Testing in Hydrogen Gas provided idea for internal cooling concept
- Internal cooling components specified from prototype testing and analysis
 - Optimized tube ID for effective cooling
 - Optimized tube wall thickness to sustain stress from external pressure
- Determined temperature distribution in concept pressure vessel with simulation
- Boise State University student intern conducted prototyping and analysis



Concept pressure vessel thermal gradient simulation at -50° C

SNL and I²CNER leverage applied and basic research for common goal



Friction and Wear



Sugimura



Sawae



Tanaka



Somerday (Lead PI)



Kirchheim

Materials Processing



Takaki



Tsuchiyama



Nakada



Macadre

Optimize cost, performance, and safety of H₂ components

- Predictive models based on physics of gas-surface interactions, H migration, and material degradation
- Advanced methods for characterizing hydrogen-induced degradation in materials

- Next-generation H₂ compatible materials having lower cost and higher strength levels

Fatigue and Fracture



Sofronis



Robertson



Kubota



Matsunaga



Yamabe



Ritchie



Xu



Nagao



Aravas