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

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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), KRISS (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

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

Identify R&D needs

Perform High-Priority R&D

Impact Codes and Standards

Partnerships with industry, labs, academia

Harmonize Internationally
Regulations, Codes and Standards [RCS]
International Standards (ISO)
International Agreements (IEA, IPHE)
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 *Polymers 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

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

- 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

- 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

**21Cr-6Ni-9Mn austenitic stainless steel**

![Stress-Strain Curve](chart)

- Stress amplitude (MPa) vs. Cycles to Failure
- **f = 1 Hz**
- **R = 0.1**

**Conditions:**
- Air (acute notch)
- Air
- 103 MPa $H_2$

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

- 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

- 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
Automated welds behave similarly to base materials in terms of hydrogen-assisted fracture, and their 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

<table>
<thead>
<tr>
<th>Room temperature</th>
<th>Type 316L</th>
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<tr>
<td></td>
<td>non-charged</td>
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<tr>
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<th>Reduction of area (%)</th>
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<tbody>
<tr>
<td>tube</td>
<td>60</td>
</tr>
<tr>
<td>automated #1</td>
<td>50</td>
</tr>
<tr>
<td>automated #2</td>
<td>40</td>
</tr>
<tr>
<td>manual GTA #1A</td>
<td>30</td>
</tr>
<tr>
<td>manual GTA #1B</td>
<td>50</td>
</tr>
<tr>
<td>manual GTA #2</td>
<td>60</td>
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</tbody>
</table>

- 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
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

• 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

![Stress-based fatigue option in CHMC1](image)

<table>
<thead>
<tr>
<th>Reference (air)</th>
<th>Hydrogen</th>
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<tr>
<td>Safety factor based on stress ratio</td>
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![Graph](image)
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 -50º C
Collaborations:

SNL and I²CNER leverage applied and basic research for common goal

Friction and Wear

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

Materials Processing

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

Fatigue and Fracture

- Optimize cost, performance, and safety of H₂ components

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Takaki  Tsuchiyma  Nakada  Macadre

Sofronis  Robertson  Kubota  Matsunaga  Yamabe  Ritchie  Xu  Nagao  Aravas