VIII.4 Hydrogen Materials and Components Compatibility

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Project End Date: Project continuation and direction determined annually by DOE

Fiscal Year (FY) 2012 Objectives

- Complete Canadian Standards Association (CSA) Test Method for Evaluating Material Compatibility for Compressed Hydrogen Applications – Phase I - Metals (CHMC1) document
- Issue Sandia report reflecting updated content from Technical Reference website
- Present progress on optimizing fatigue crack growth testing in H_2 gas to American Society of Mechanical Engineers (ASME) Project Team on Hydrogen Tanks
- Develop detailed materials testing program on austenitic stainless steel welds with industrial partner

This project addresses the following technical barriers from the Safety, Codes and Standards section (3.7) of the Fuel Cell Technologies Program Multi-Year Research, Development and Demonstration Plan:

(A) Safety Data and Information: Limited Access and Availability
(F) Enabling National and International Markets Requires Consistent Regulations, Codes and Standards
(G) Insufficient Technical Data to Revise Standards

Contribution to Achievement of DOE Safety, Codes and Standards Milestones

This project will contribute to achievement of the following DOE milestones from the Safety Codes and Standards section of the Fuel Cell Technologies Program Multi-Year Research, Development and Demonstration Plan:

- Milestone 2.5: Develop holistic design strategies. (4Q, 2017)
- Milestone 2.6: Validate inherently safe design for hydrogen fueling infrastructure. (4Q, 2019)
- Milestone 2.16: Publish technical bases for optimized design methodologies of hydrogen containment vessels to account appropriately for hydrogen attack. (Q4, 2014)
- Milestone 2.17: Implement validated mechanism-based models for hydrogen attack in material. (Q4, 2018)
- Milestone 2.18: Demonstrate the use of new high performance materials for hydrogen applications that are cost-competitive with aluminum alloys. (4Q, 2017)
- Milestone 4.1: Identify and evaluate failure modes. (3Q, 2013)

FY 2012 Accomplishments

- Materials compatibility testing and data application standard, CSA CHMC1 Part 1 published May 2012.
- Developed accelerated test methods for measurement of hydrogen assisted fatigue crack growth in Cr-Mo pressure vessel steels. This accelerated test greatly reduces the cost barriers that challenge qualification of new materials in hydrogen service. This directly supports milestone 2.16.
- Developed test methods for evaluating the effectiveness of stainless steel welds in hydrogen. This activity leverages past test methods and identifies methods for evaluating the hydrogen resistance of material under actual use conditions such as welding. This activity directly supports milestones 2.16 and 4.1
- Continued development of international collaborations:
  - Hosted research collaborators from HYDROGENIUS/Japanese National Institute of Advanced Industrial Science and Technology (AIST) at Sandia in February 2012
  - Visited researchers at AIST in June 2012
  - Developing collaborative research goals for FY 2013
  - Developing collaborative research concepts with German industrial partners for FY 2013

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Introduction

Lack of validated safety testing methods in safety standards and insufficient technical data to revise these standards are major barriers to the deployment of hydrogen technologies. The purpose of this project is twofold,

1) provide the technical basis for assessing the safety of hydrogen-based systems with the accumulation of knowledge feeding into the development or modification of relevant codes and standards and, 2) using the development of these test methods to support a broader understanding of material compatibility and thus stimulate technical innovation.

Approach

The focus of the Materials and Components Compatibility project is to optimize materials characterization methodologies, generate critical hydrogen compatibility data for materials to enable technology deployment, and compose the Technical Reference on Hydrogen Compatibility of Materials. Two activities proceed in parallel: generating new data and understanding through materials testing, and identifying and summarizing existing data from technical documents. The high-priority structural materials featured in these activities are low-alloy and carbon steels, austenitic stainless steels, and aluminum alloys. The materials testing activity emphasizes high hydrogen gas pressures (>100 MPa), fatigue crack initiation and propagation test methods, and technology-critical material fabrication (e.g. welds) and service variables (e.g., temperature). The data from materials testing are rigorously reviewed to identify pathways to improve the test methods and to ensure the data are suitable for implementation in structural design.

As part of codes and standards advocacy, Sandia personnel provide leadership in the codes and standards development process through direct participation in organizations such as ASME, CSA, and SAE Inernational. This participation ensures that the standards development organizations have the most current technical information on structural materials compatibility. Sandia personnel provide leadership in the development of both component design standards as well as materials testing standards.

Specific objectives for FY 2012 include:

- Optimize fatigue crack growth rate measurements for pressure vessel steels in H_2 and report results to ASME
- Evaluate effects of load-cycle frequency on fatigue crack growth rates for 7000 series aluminum alloys in high-pressure H_2
- Measure H_2-affected fracture properties of technologically relevant welds in collaboration with industry partner
- Enable completion of standards through committee leadership and data evaluation
- Develop capability for variable-temperature testing in high-pressure H_2 gas

Results

The accomplishments summarized below are directly related to the objectives and milestones featured in FY 2012: The Materials Compatibility Task accomplished many of the milestones with several more expected for completion by the end of FY 2012.

- The results of optimized fatigue crack growth rate measurements for pressure vessel steels in hydrogen were presented to ASME at their quarterly meeting Nov. 2011. This optimized measurement procedure (illustrated in Figure 1) greatly accelerates the time required to test and validate new pressure vessel steels.
- The results of the evaluation of load cycle frequency effects on fatigue crack growth rates for 7000 series aluminum alloys in high-pressure hydrogen demonstrates that these alloys are resistant to hydrogen embrittlement, enabling qualification of this high-strength aluminum alloy as an alternative to 6061 aluminum.
- Welded tubes were tested after high-pressure hydrogen exposure to produce an initial data set. This work was particularly interesting as the tubing was supplied by an industrial partner and is a representative sample of tubing and welds commonly found in the commercial hydrogen applications. Preliminary testing showed no effect of hydrogen on fracture resistance of the weld (Figure 2). Fatigue testing protocol is under development.
- Several standards have been updated or revised with data generated from this project and through leadership of Sandia personnel in guiding committee experts to appropriately apply the experimental analysis. The new standards are: CSA CHMC1, CSA HPIT1 and the revised standards are: SAE J2579, ASME KD-10.
- Development of the Technical Reference has identified a gap in material properties at temperatures above and

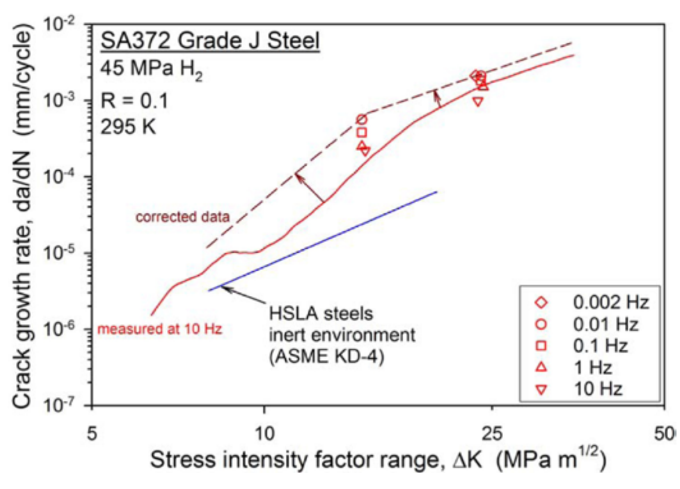

FIGURE 1. Evaluation of various load cycle frequencies for use in developing accelerated test protocol

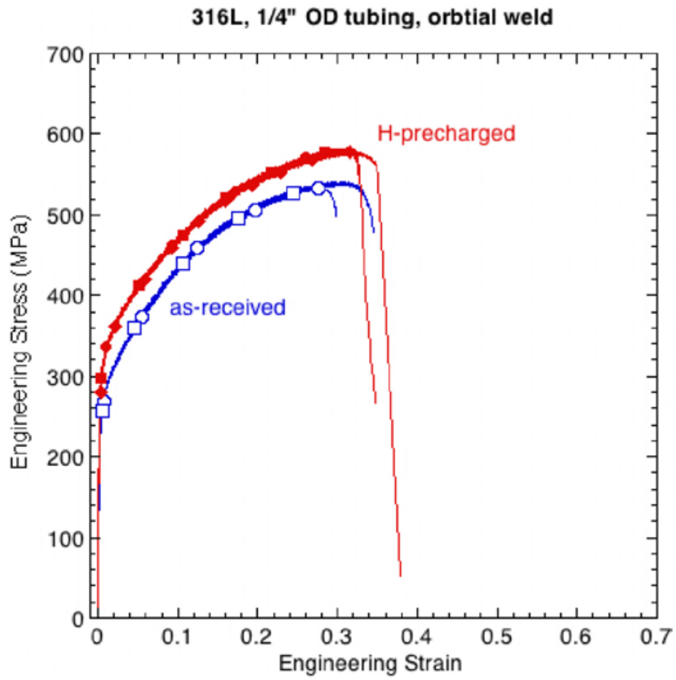

FIGURE 2. Preliminary results of welded tube tensile testing

below ambient. Construction of a new automated gas manifold that will support a future capability for variable temperature testing is nearly complete. This future capability will increase testing throughput at ambient temperatures and provide a new testing capability at sub-ambient and elevated temperatures.

Conclusions and Future Directions

FY 2012 brought about the conclusion of several multi-year efforts in the publication and revision of several standards. These publications represent hundreds of testing hours and hundreds of hours dedicated to standards development meetings and communication regarding the appropriate application of testing results to the standards development

FY 2013

- Measure fatigue crack initiation resistance of H_2-exposed stainless steel tube welds.
- Develop validated methodology to account for fatigue crack initiation life in steel H_2 pressure vessels for consideration in ASME Article KD-10.
- Develop research and development project with industry partner to improve resistance of high-strength pressure vessel steel to H_2-assisted fatigue crack growth.
- Procure pressure vessel to complete variable-temperature testing in H_2 gas system.
- Leverage results on fatigue crack growth of steels in H_2 to advance international coordination with AIST and International Institute on Carbon-Neutral Energy Research on materials testing and basic science.
- Develop a Hydrogen Materials Collaboration Database as a resource for the broader hydrogen community. It will include discussion forums, archives of open-source content and reports from conferences or meetings, and new results on hydrogen compatibility testing that have not yet gone through peer review for incorporation into the Technical Reference. The Hydrogen Materials Collaboration Database will enable global harmonization of test methods, facilitate research coordination, and lead to accelerated deployment of hydrogen systems.

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FY 2012 Publications/Presentations

1. R. Gangloff and B. Somerday, Eds., *Gaseous Hydrogen Embrittlement of Materials in Energy Technologies*, Woodhead Publishing, Cambridge, UK, 2012.

2. C. San Marchi, "Hydrogen Embrittlement of Austenitic Stainless Steels and Their Welds", in *Gaseous Hydrogen Embrittlement of Materials in Energy Technologies*, R. Gangloff and B. Somerday, Eds., Woodhead Publishing, Cambridge, UK, 2012, pp. 592-623.

3. K. Nibur and B. Somerday, "Fracture and Fatigue Test Methods in Hydrogen Gas ", in *Gaseous Hydrogen Embrittlement of Materials in Energy Technologies*, R. Gangloff and B. Somerday, Eds., Woodhead Publishing, Cambridge, UK, 2012, pp. 195-236.

4. K. Nibur, B. Somerday, C. San Marchi, J. Foulk, M. Dadfarnia, P. Sofronis, "The Relationship Between Crack-Tip Strain and Subcritical Cracking Thresholds for Steels in High-Pressure Hydrogen Gas", *Metallurgical and Materials Transactions A*, 2012, accepted for publication.

5. C. San Marchi, A. Harris, M. Yip, B. Somerday, K. Nibur, "Pressure Cycling of Steel Pressure Vessels with Gaseous Hydrogen", Proceedings of the ASME 2012 Pressure Vessels and Piping Division Conference, July 15–19, 2012, Toronto, Canada, PVP2012-78709.

6. K. Nibur, B. Somerday, C. San Marchi, J. Foulk, M. Dadfarnia, P. Sofronis, "The Relationship Between Crack-Tip Strain and Subcritical Cracking Thresholds for Steels in High-Pressure Hydrogen Gas", Joint HYDROGENIUS and I^2CNER International Workshop on Hydrogen-Materials Interactions, Fukuoka, Japan, February 2012.

7. K. Nibur and B. Somerday, "Effect of Crack Tip Strain on the Subcritical Cracking Thresholds for Steels in High-Pressure Gaseous Hydrogen", MS&T Conference 2011, Columbus OH, October 2011.