SAND2015-6404R

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

Brian Somerday (Primary Contact), Chris LaFleur, Chris San Marchi
Sandia National Laboratories
P.O. Box 969, Livermore, CA 94551-0969
(925) 294-3141
bpsomer@sandia.gov

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-AC04-94AL85000.

DOE Manager

Will James
Phone: (202) 287-6223
Email: Charles.James@ee.doe.gov

Project Start Date: October 1, 2003
Project End Date: Project continuation and direction determined annually by DOE

Overall Objectives

- Optimize the reliability and efficiency of test methods for structural materials and components in hydrogen gas
- Generate critical hydrogen compatibility data for structural materials to enable technology deployment
- Create and maintain information resources such as the "Technical Reference for Hydrogen Compatibility of Materials"
- Demonstrate leadership in the international harmonization of standards for qualifying materials and components for high-pressure hydrogen service

Fiscal Year (FY) 2015 Objectives

- Extend fatigue crack growth testing on Cr-Mo pressure vessels steels in high-pressure hydrogen gas to include data at lower ΔK levels
- In collaboration with vendor, complete engineering drawings and initiate manufacturing of pressure vessel for variable-temperature testing in high-pressure hydrogen system
- Foster growth of international collaboration and leadership on materials science of hydrogen embrittlement, in particular within the International Institute for Carbon-Neutral Energy Research (I2CNER)
- In partnership with AIST, complete fracture threshold measurements on Japanese Cr-Mo pressure vessel steel as a function of hydrogen gas pressure and loading rate

Technical Barriers

This project addresses the following technical barriers from the Safety, Codes and Standards section of the 2012 Fuel Cell Technologies Office Multi-Year Research, Development and Demonstration Plan (section 3.8):

(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

Contribution to Achievement of DOE Technology Validation/Safety, Codes & Standards/Education/Manufacturing R&D/Systems Analysis Milestones

This project will contribute to achievement of the following DOE milestones from the Safety Codes and Standards section of the 2012 Fuel Cell Technologies Office Multi-Year Research, Development and Demonstration Plan:

- Milestone 2.9: Publish technical basis for optimized design methodologies of hydrogen containment vessels to account appropriately for hydrogen attack (4Q, 2013)
- Milestone 2.16: Demonstrate the use of new high-performance materials for hydrogen applications that are cost-competitive with aluminum alloys. (4Q, 2017)
- Milestone 2.3: Implement validated mechanism-based models for hydrogen attack in materials (4Q 2016)
- Milestone 3.3: Reduce the time required to qualify materials, components, and systems by 50% relative to 2011 with optimized test method development. (1Q 2017)
- Milestone 3.4: Develop hydrogen material qualification guidelines including composite materials (Q4, 2017)
- Milestone 4.8: Completion of the GTR Phase 2. (1Q, 2017)
- Milestone 5.2: Update materials compatibility technical reference (4Q, 2011-2020)
- Milestone 5.4 Develop and publish database for properties of structural materials in hydrogen gas. (2Q, 2013)

FY 2015 Accomplishments

- Completed measurements of fatigue crack growth rate vs. frequency at two lower-ΔK levels for SA-372 Grade J and 34CrMo4 pressure vessel steels in 45 MPa hydrogen gas
- In collaboration with vendor, completed engineering drawings for pressure vessel in variable-temperature testing in hydrogen gas system
- In partnership with AIST, completed fracture threshold measurements on Japanese Cr-Mo steel SCM 435 as a function of hydrogen gas pressure and loading rate

Introduction

A principal challenge to the widespread adoption of hydrogen infrastructure is the lack of quantifiable data on its safety envelope and concerns about additional risk from hydrogen. To convince regulatory officials, local fire marshals, fuel suppliers, and the public at large that hydrogen refueling is safe for consumer use, the risk to personnel and bystanders must be quantified and minimized to an acceptable level. Such a task requires strong confidence in the safety performance of high pressure hydrogen systems. Developing meaningful materials characterization and qualification methodologies in addition to enhancing understanding of performance of materials is critical to eliminating barriers to the development of safe, low-cost, high-performance high-pressure hydrogen systems for the consumer environment.

Approach

The Materials and Components Compatibility project leverages decades of experience in high-pressure hydrogen systems, well-developed industry partnerships, and an enabling capability in hydrogen-materials interactions anchored by the Hydrogen Effects on Materials Laboratory to focus on three critical activities: 1) optimize materials characterization methodologies, 2) generate critical hydrogen compatibility data for materials to enable technology deployment, and 3) provide international leadership by assembling and maintaining a technical reference that is populated with vetted data and includes a technical assessment of the data and its application.

Results

Optimizing fatigue crack growth measurements in hydrogen gas
An activity featured in FY13 was fatigue crack growth testing on two commercial Cr-Mo pressure vessel steels as a function of stress-intensity factor range (ΔK), hydrogen gas pressure, and load-cycle frequency. The objective of this testing was to explore an optimized method, in which measurements could be performed over a broad ΔK range while limiting test duration. The

proposed approach was to measure the fatigue crack growth rate (da/dN) vs. ΔK relationship at high load-cycle frequency (10 Hz) and then apply a correction based on supplemental da/dN vs. frequency data. During FY15, these data were comprehensively analyzed and documented in a manuscript submitted to the ASME 2015 Pressure Vessels & Piping Division Conference. As a result of this analysis, it was determined that the fatigue crack growth data were insufficient for assessing the viability of the proposed modified approach. Specifically, since the baseline da/dN vs. ΔK relationships measured at 10 Hz must be corrected based on da/dN vs. frequency data, the latter data must be measured at appropriate constant-ΔK intervals. The gap in the data set revealed during analysis was the absence of da/dN vs. frequency measurements at lower constant-ΔK levels.

To address this gap, additional da/dN vs. frequency measurements were initiated in FY15, focusing on two lower ΔK levels for both pressure vessel steels at 45 MPa hydrogen gas pressure. Figure 1displays the original data (baseline da/dN vs. ΔK relationships at 10 Hz and da/dN vs. frequency data at two higher constant-ΔK levels) and the latest da/dN vs. frequency measurements at two lower ΔK levels in 45 MPa hydrogen gas. These latest data emphasize that the dependence of fatigue crack growth rate on frequency persists to ΔK levels lower than expected. The implication is that corrections to the baseline da/dN vs. ΔK relationships to reflect upper-bound behavior must include da/dN vs. frequency data in the lower ΔK range. Based on the more comprehensive data sets in Figure 1, a more definitive correction can be applied to the baseline da/dN vs. ΔK relationships to represent upper-bound behavior (dark-red dashed lines in Figure 1).

System for variable-temperature testing in hydrogen gas
Sandia maintains an enabling capability in hydrogen embrittlement of structural materials, in which the Hydrogen Effects on Materials Laboratory is the central asset. This laboratory features several specialized systems for measuring the mechanical properties of materials in high-pressure hydrogen gas. One essential system for providing materials data to support hydrogen and fuel cell technology deployment allows dynamic mechanical loading of materials (i.e., fatigue) in high-pressure hydrogen gas at room temperature. However, it is well known that certain materials such as austenitic stainless steels are most susceptible to hydrogen embrittlement at temperatures near 233 K. Thus, it is imperative to develop a system for dynamic mechanical loading of materials in hydrogen gas with a mechanism for varying temperature.

The remaining effort for completing the new materials test system is primarily focused on a pressure vessel with variable-temperature mechanism. In FY14, an internal cooling mechanism was identified and validated for the concept pressure vessel. Based on this internal cooling mechanism, the Sandia team has been working collaboratively with a pressure vessel design and manufacturing company to create a set of engineering drawings. These drawings have been completed, and the pressure vessel vendor is determining a cost estimate for the manufacturing.

International collaboration with I2CNER
On December 20, 2014, Dr. Brian Somerday led a coordination meeting at Sandia National Laboratories in Livermore, CA for the I2CNER Hydrogen Materials Compatibility division. The purpose of this meeting was twofold: 1) promote interaction and coordination among researchers in the Hydrogen Materials Compatibility division, and 2) refine research roadmap for the division and ensure research activities align with roadmap. The foundation of the agenda was 12 short overview presentations of research projects in the division. The presenters included professors, post-docs, and graduate students having affiliations with I2CNER.

Dr. Brian Somerday served as co-organizer of the Joint HYDROGENIUS and I2CNER International Workshop on Hydrogen-Materials Interactions at the International Hydrogen Energy Development Forum in Fukuoka, Japan on February 4, 2015. The workshop featured invited presentations on research related to hydrogen effects on mechanical properties of structural metals. The speakers invited to this international workshop represented the U.S., Germany, France, Norway, and Japan.

Leveraging partnership at AIST

AIST (Tsubuka, Japan) and Sandia continued to perform collaborative R&D in FY15 to evaluate test methods for measuring fracture thresholds of pressure vessel steels in high-pressure hydrogen gas. The primary testing activity was measuring the fracture thresholds for a Japanese steel (SCM 435) as a function of loading rate and hydrogen gas pressure (Figure 2). These results in conjunction with measurements performed on a U.S. steel (SA-372 Grade J) in FY14 constitute the most comprehensive fracture threshold data set for Cr-Mo pressure vessel steels in hydrogen gas. Given the value of these data, Dr. Takashi Iijima from AIST presented the results at the ASTM subcommittee meeting on Environmentally Assisted Cracking (E08.06.02) in November 2014. The data are contributing to the technical basis for formulating an ASTM standard on rising-displacement fracture thresholds measurement in hydrogen gas through ASTM subcommittee E08.06.02. Although these fracture threshold measurements are recognized as having relevance for life prediction of pressure vessels in hydrogen gas service, there is currently no ASTM standard guiding such measurements in hydrogen gas. The fracture threshold measurements featured in the AIST-Sandia collaboration have been documented in jointly authored publications submitted to the 2014 and 2015 ASME Pressure Vessels & Piping Division Conference.

Conclusions and Future Directions

- Establishing an efficient method for measuring the fatigue crack growth relationship for Cr-Mo steels in hydrogen gas over a broad ΔK range can lead to more favorable calculated fatigue lives for hydrogen pressure vessels.
- Progress in completing the variable-temperature testing in hydrogen gas system bolsters the enabling capability in materials compatibility and assures critical testing can be performed on technologically pivotal materials such as stainless steels.
- International partnerships with I2CNER and AIST provide access to basic science related to materials behavior in hydrogen as well as data that enable the development of international standards for materials testing and component qualification.
- (future) In collaboration with the EC-supported project MATHRYCE, perform fatigue crack initiation and growth testing on a Cr-Mo pressure vessel steel in 100 MPa hydrogen gas to complement measurements by MATHRYCE partners at lower pressure
- (future) Commission variable-temperature testing in hydrogen gas system: integrate subsystems and demonstrate functionality.
- (future) Evaluate suitability of high-hardenability steels for stationary high-pressure hydrogen vessels in partnership with industry stakeholder consortium
- (future) In collaboration with international partners, initiate activity (e.g., round robin testing) to define test methods and augment database for stainless steels in high-pressure hydrogen gas

FY 2015 Publications/Presentations

1. C. San Marchi , "Development of Hydrogen Safety Standards in the United States", presentation at Workshop on International Trends in Hydrogen Safety Standards, Seoul, Korea, Nov. 2014.
2. C. San Marchi , "Trends in Hydrogen Research in the United States", presentation at Korean Society of Mechanical Engineers, GwangJu, Korea, Nov. 2014
3. B. Somerday, "Measurements of Subcritical Cracking Thresholds and Fatigue Crack Growth Rates for Steels in H_2 Gas", presentation at Tenaris-Dalmine, Dalmine, Italy, Oct. 2014.

4. B. Somerday, "Enhancing Reliability of Hydrogen Assisted Cracking Properties Measured in Hydrogen Gas", presentation at Hydrogen Embrittlement-Multi-scale Modelling and Measurement: What is the Impact?, National Physical Laboratory, Teddington, UK, Oct. 2014.
5. B. Somerday and C. San Marchi, "R&D for Safety, Codes and Standards: Materials and Components Compatibility", presentation at Joint Delivery-Codes & Standards Tech Team Meeting, Sacramento, CA, Jan. 2015
6. L. Zhang, B. An, T. Iijima, C. San Marchi, and B. Somerday, "Hydrogen Transport and Hydrogen-Assisted Cracking in SUS304 Stainless Steel During Deformation at Low Temperatures", Proceedings of the ASME2015 Pressure Vessels & Piping Conference, PVP2015-45211, Boston, MA, July 2015.
7. T. Iijima, H. Itoga, B. An, C. San Marchi, and B.P. Somerday, "Fracture Properties of a Cr-Mo Ferritic Steel in High-Pressure Gaseous Hydrogen", Proceedings of the ASME2015 Pressure Vessels & Piping Conference, PVP2015-45328, Boston, MA, July 2015.
8. B. Somerday, P. Bortot, and J. Felbaum, "Optimizing Measurement of Fatigue Crack Growth Relationships for Cr-Mo Pressure Vessel Steels in Hydrogen Gas", Proceedings of the ASME2015 Pressure Vessels & Piping Conference, PVP2015-45424, Boston, MA, July 2015.

Acronyms

AIST – Advanced Industrial Science and Technology
ASME – American Society of Mechanical Engineers
I2CNER – International Institute for Carbon-Neutral Energy Research

Figure Captions

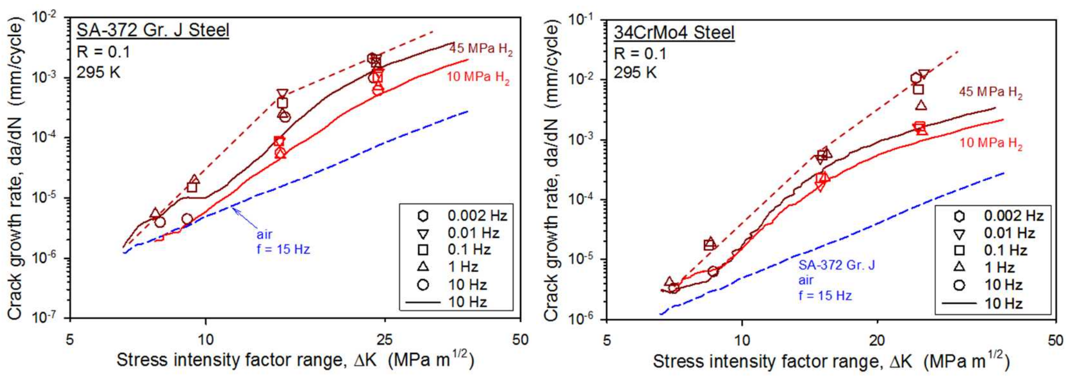

Figure 1. Composite data showing fatigue crack growth rate (da/dN) vs. stress-intensity factor range (ΔK) and da/dN vs. frequency for two Cr-Mo pressure vessel steels in 10 and 45 MPa hydrogen gas. The dark-red dashed lines indicate corrections applied to the baseline da/dN vs. ΔK relationships based on the da/dN vs. frequency data to reflect upper-bound behavior.

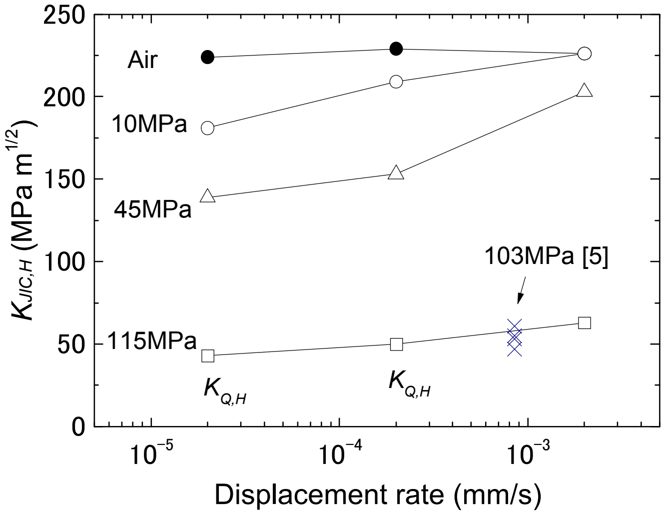

Figure 2. Rising-displacement fracture thresholds measured for SCM 435 pressure vessel steel in air and high-pressure hydrogen gas as a function of loading rate. The cross symbols are previous measurements performed at Sandia on a similar Cr-Mo pressure vessel steel (SA-372 Grade J) in 103 MPa hydrogen gas.