SAND2019- 3280 C

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 2022*

* Project continuation and direction determined by DOE annually

Budget

- Total Project Budget: $10.2M
  - FY18 DOE Funding: $450K
  - Planned FY19 Funding: $550K

Technical 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, Tenaris-Dalmine, Japan Steel Works, BMW, Opel, GM, Swagelok
- **International engagement:** AIST-Tsukuba (Japan), I2CNER (Kyushu University, Japan), MPA Stuttgart (Germany), KRISS (Korea)

Relevance and Objectives

Objective: Enable technology deployment by **performing and applying foundational research toward the development of science-based codes and standards** that enable the deployment of hydrogen technologies

Barrier from 2013 SCS MYRDD	Project Goal
A. Safety Data and Information: Limited Access and Availability	Develop and maintain material property database and informational resources to aid materials innovation for hydrogen technologies
F. Enabling national and international markets requires consistent RCS	Develop science-based materials test methods, working with SDOs and the international community to validate and incorporate methods in globally harmonized testing specifications
G. Insufficient technical data to revise standards	Execute materials testing to address *targeted* data gaps and critical technology deployment • Coordinate activities with international stakeholders

Project Approach and Milestones

MYRD&D 2013 Barrier	FY19 Milestone	Status
A. Safety Data and Information: Limited Access and Availability	Advance state-of-the-art materials database for hydrogen compatibility	Sandia Hydrogen Effects Database (Granta MI) is publically accessible and populated with literature data
F. Enabling national and international markets requires consistent RCS	Negotiate standard language and technical basis with international experts on materials compatibility testing for proposal to GTR IWG	Test method draft negotiated with international experts and presented to GTR IWG; first draft of technical basis document circulated among international experts
G. Insufficient technical data to revise standards	Evaluate test method for fracture resistance of aluminum alloys in high-pressure gaseous hydrogen	Methods discussed with international stakeholders; plan for testing in high-pressure 'wet' gaseous hydrogen established
	Develop test methodology for component-like configurations, such as orbital tube welds	Test geometry concept identified for tube geometry that can interrogate welds consistent with notched fatigue life specimen from SAE J2579

Approach: Establish science-based test methodologies consistent with the requirements of applications

How do we standardize selection methods for materials for H_2 service?

- **High-pressure vehicle fuel system**: *performance-based method*
  - Establish materials *performance metrics*
  - Consider mechanics of the service condition
  - Explore relevant environments and determine dominant conditions
- **Stationary pressure vessel**: *design-based method*
  - Measure reliable *design data*
  - Establish bounding behavior for environment and mechanics
    - balance between testing efficiency and meaningful data
  - Assess data in aggregate to establish global behavior

National Laboratory role: ***Develop and deploy foundational scientific framework to establish and evaluate methods***

Approach: high-pressure vehicle fuel system

Determine relevant performance metrics to establish conservative material behavior for application

What is the limiting material behavior(s) in this application?

- Material definition
  - Microstructure, strength, etc
  - Performance of welds

Critical, but how to define relevant weld geometry?

- Tensile properties
  - Tensile tests in hydrogen do not provide much new information relative to tests in air
    - Yield strength is generally not changed
    - Tensile ductility requirements (elongation, RA)
      - No consensus on criteria
      - Criteria are generally arbitrary
      - Not used quantitatively in design

Do tests in H2 add value?

- Fatigue performance
  - Deep stress cycles associated with refueling

Critical limiting behavior

Accomplishment: high-pressure vehicle fuel system
Critical assessment of limiting fatigue behavior

In vehicle application, pressure cycles due to refueling are typically in the 100s, but theoretically up to ~11,250 refuelings
- 11,200 cycles = refuel once per day for 30 years

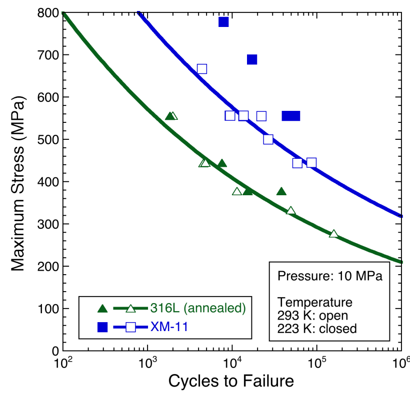
- Fatigue life performance criteria, established to be conservative
  - Two options:
    - 100,000 stress cycles when subjected to stress concentration (notched)
      - Conservative stress state
      - Conservative number of cycles
    - 200,000 stress cycles for tension-compression cycle (smooth)
      - Conservative stress amplitude: 2x typical for stress relieved component
      - Conservative number of cycles

Accomplishment: high-pressure vehicle fuel system

Simple performance requirements established for SAE J2579 based on relevant design space (proposed to GTR IWG)

Test configuration		Evaluation parameter	Requirements of tests performed in H2
Fatigue life tests (must satisfy 1 of 2 options)	Option 1 (3 tests): Smooth, R= -1	Cycles to failure	Each > 200,000 cycles
	Option 2 (3 tests): Notched, R = 0.1	Cycles to failure	Each > 100,000 cycles

- Test requirements have substantially evolved to simple performance-based metrics to demonstrate suitability for application
  - Discussion to remove Slow Strain Rate Tension (SSRT) test
  - Fatigue life test conducted at room temperature only (i.e., low-temperature, high-pressure tests removed)
    - Data show that the fatigue life of austenitic stainless steels is greater at low temperature than at room temperature

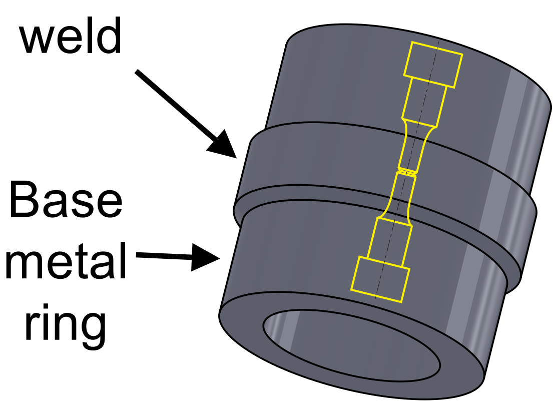


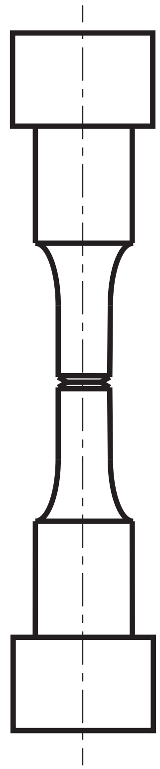
Accomplishment: high-pressure vehicle fuel system

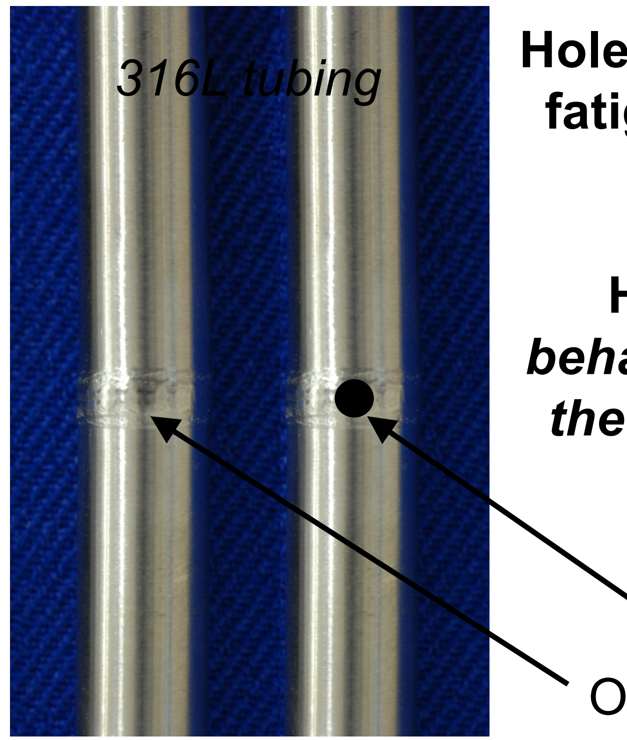
Developing geometries and test methods for welded structures in components (in progress)

Notched fatigue specimen
Kt ~ 3

Established for bar and plate materials







Hole-drilled tubular fatigue specimen
Kt ~ 3

Hypothesis:
behaves nominally the same as bulk specimen

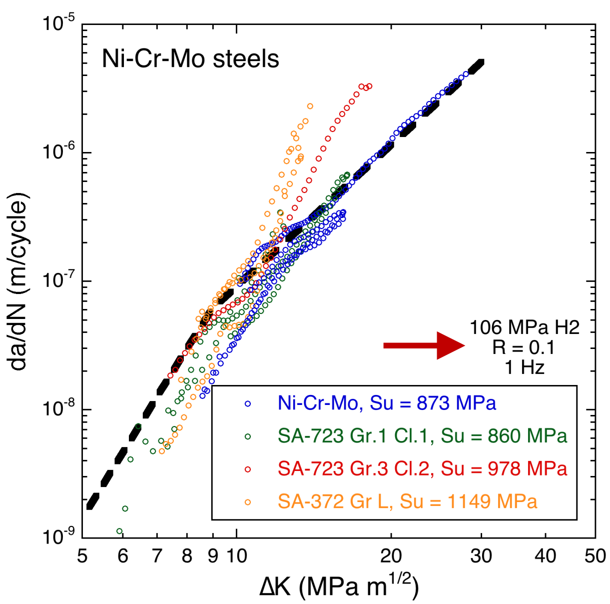
Easily applied to large welds: GTA welds and (potentially) EB welds

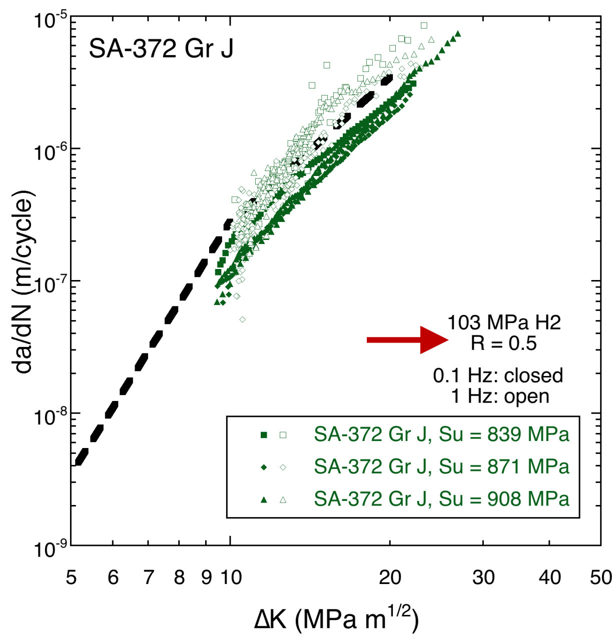
If true, ideal for evaluation of very common weld configuration: orbital tube weld

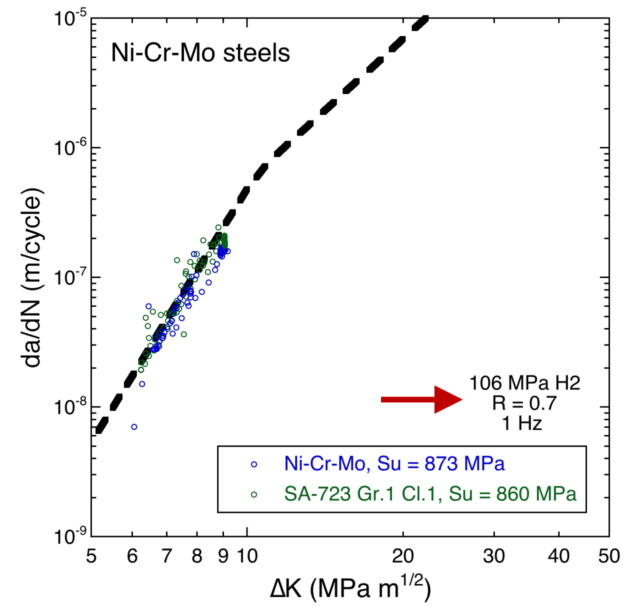
Accomplishment: stationary pressure vessels

ASME Code Case 2938 approved

"Technical basis for proposed master curve for fatigue crack growth of ferritic steels in high-pressure gaseous hydrogen in ASME section VIII-3 code" (PVP2019-93907), Proceedings of the 2019 ASME Pressure Vessels & Piping Conference, 14-19 July 2019, San Antonio TX. (manuscript in review)

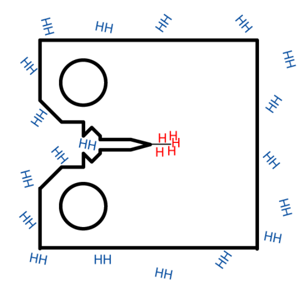






- **Provides design curve** $$\frac{da}{dN} = C\left[\frac{1 + C_H R}{1 - R}\right]\Delta K^m$$

  based on data and analysis from this program



Accomplishment: stationary pressure vessels

Design curves based on best available data, however a few questions remain (in progress)

- **High-strength steels show low fracture resistance in H2**
  - Fracture resistance becomes uncomfortably low, when tensile strength is >950 MPa
  - CC limits TS ≤ 915 MPa

> - *Steels with TS between 915-950 MPa are being re-evaluated*
> - *High-strength steels considered in H-Mat*

- **Fatigue behavior is pressure sensitive**
  - Empirical pressure term fits data for pipeline steels at low pressure

> *Testing is being considered to evaluate broader applicability of design curves*

- **Fatigue behavior near threshold and with negative load ratio are not well documented**
  - CC assumes that a fatigue threshold does not exist in H2
  - CC allows assumption that for $R < 0$, $K_{min} = 0$

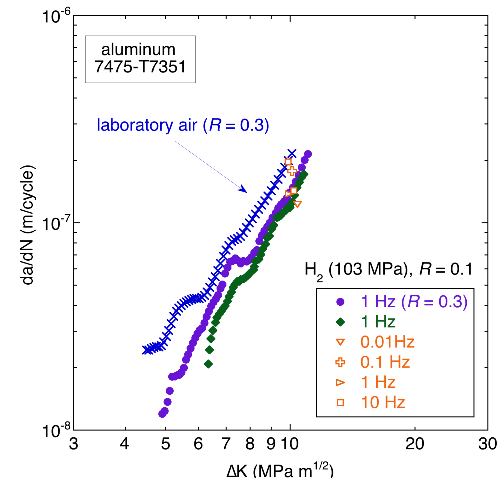
> *Hardware and methods are being developed for high-pressure testing at low K_{max} and negative K_{min}*

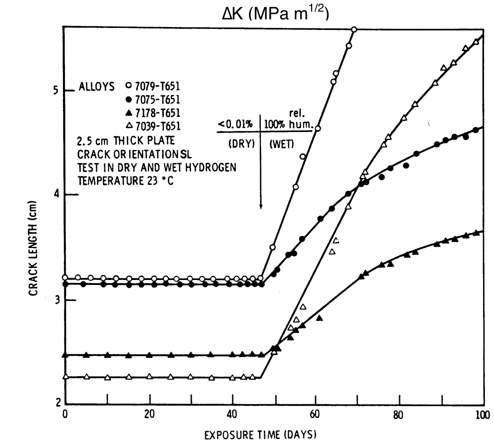
Approach: test methods for aluminum alloys
Critical assessment of existing test methods, relevance of environments and physical phenomena in aluminum alloys

- Previous work has shown no effect of dry hydrogen on fracture and fatigue of aluminum alloys
- Stress corrosion cracking (SCC) in high-strength aluminum is well known
  - Cracking is apparent in 'wet' hydrogen

- High-Pressure Institute of Japan has proposed SCC test method to evaluate aluminum in 'wet' air as a surrogate for 'wet' high-pressure gaseous hydrogen (HPIS E 103:2018)
  - Method has not been validated experimentally against testing in a relevant environment

From: San Marchi et al, ASME PVP-2011 conf.

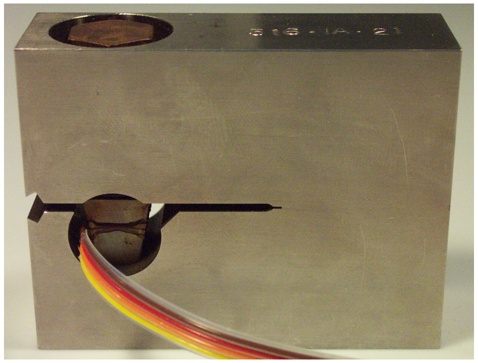




From: Speidel, Hydrogen Embrittlement and Stress Corrosion Cracking, 1984

Accomplishment: test methods for aluminum alloys
Informal partnership with international stakeholders to establish behavior of aluminum in hydrogen (in progress)

- **Sharing data/plans with Fuel Cell Safety Task Force (SAE)**
  - Includes OEMs, component manufacturers, and other international stakeholders
  - JARI coordinating testing of aluminum in low-pressure 'wet' H2
  - MPA Stuttgart coordinating tests using 'wet' air
  - Sandia performing SCC tests in high-pressure hydrogen with 100 ppm water
- **Establish benchmark for SCC in the presence of 'wet' hydrogen**
  - If *cracking is observed*, evaluate lower (more relevant) water content
  - If *cracking is not observed*, evaluate kinetic barriers, for example through fatigue testing



WOL specimen for SCC testing

Response to Previous Year Reviewers' Comments

- *FY18 Reviewer Comment: "The project scope is good, although aluminum should be considered in the near term."*
  - Efforts on aluminum are being ramped up both here and with the H-Mat program. While the activity for SCS is focused on test methods for standards/codes, the H-Mat effort focuses on the physics of hydrogen-aluminum interactions from a mechanistic perspective. The latter informs and strengthens the technical basis for the former.
- *FY18 Reviewer Comments: "The project lacks mechanics modeling to account for and assess the influence of the microstructure of austenitics on fatigue life and crack growth."*
  - This is a legitimate criticism of the portfolio and a motivation for the H-Mat effort. The development of codes and standards requires a significant investment to participate with the committees, to evaluate engineering data and to formulate a firm technical basis for engineering decision-making. This leaves very little resources for investigating the physics hydrogen-microstructure interactions, which the codes and standards committees do not value and for which it is difficult to show impact in the near term. The H-Mat consortium, however, is addressing more fundamental questions such as the relationships between microstructure and materials response in hydrogen. And we hope industry and academy will partner with the H-Mat teams.
- *FY18 Reviewer Comments: "The weakness of the project is the uncertain impact of the results."*
  - SAE J2579 appendix B is a direct outcome of this project. The only proposal for materials compatibility testing at the GTR no. 13 Phase II IWG is a direct outcome of this project.
  - Code Case 2938 (ASME BPVC.VIII.3) is a direct outcome of results from this project.

Collaborations

- Standards Development Organizations (SDOs)
  - Test method for SAE J2579 and proposed method for GTR no. 13 Phase II is based on extensive international discussion with organization stakeholders and automotive OEMs
  - Code case adds design guidance to Article KD-10 (ASME BPVC)
- Industry partners
  - Partners communicate materials testing gaps/needs and provide technology-relevant materials (FIBA Technologies, Tenaris-Dalmine, JSW, BMW, Opel, Swagelok)
  - International MOU for evaluation of Ni-Cr-Mo PV steels motivated Code Case for ASME BPVC and future testing plans (threshold and $R < 0$)
- International research institutions
  - Fatigue testing at low temperature is focus of R&D collaboration within the context of SAE and international participants with complementary programs in Japan (Kyushu Univ) and Germany (MPA Stuttgart)
    - Joint publication for ASME PVP conference (July 2018)
    - Expanding participation to Korea and China

Remaining Challenges and Barriers

- Long-time scales (kinetics) associated with hydrogen-materials interactions challenges our ability to interrogate the materials response
  - Acceleration of fatigue testing is challenging and generally requires equal parts creativity and patience
  - Surface effects are difficult to characterize and even more difficult to quantify – thus establishing bounding behavior can be challenging
- Stationary pressure vessels remain a design challenge
  - Conventional steels are necessarily limited to relatively low strength
  - Design strategies are conservative with limited allowance for life extension
- Next generation materials/microstructures cannot be identified without fundamental understanding of the physical processes
  - Advanced scientific computing, coupled with controlled experimentation are needed to develop mechanistic understanding of hydrogen effects and inform materials design hypotheses

H-Mat SM Hydrogen Materials Compatibility Consortium

Proposed Future Work

Remainder of FY19

- ***Welded austenitic stainless steels relevant to vehicle application and infrastructure***
  - Exercise methodology for fatigue testing of orbital tube welds and compare to base materials
  - Share weld data with internal community in support of SAE & GTR
- ***Test methods for aluminum alloys***
  - Evaluate proposed method (HPIS) in high-pressure environments
  - Coordinate testing on moisture effects in high-pressure hydrogen with MPA Stuttgart and JARI (Japan) to develop/validate test method

FY20 (project continuation and direction determined by DOE annually)

- ***Test methods for low ΔK and negative load ratio***
  - Develop hardware designs for reverse loading and strain-based methods to extend test method development to negative load ratios
- ***Comprehensive revision of Technical Reference***
  - Recent advances in test methods, standards, and relevant data will be added to existing ”handbook” informational resources to reflect state of knowledge

Summary

- **Motivation of SCS materials work:**
  - *Establish science-based test methodologies consistent with the requirements of relevant applications as well as tools for engineering*
- ***High-pressure vehicle fuel system***
  - International coordination on simple metric for materials testing: SAE J2579 and UN GTR no. 13
  - Developing test configuration (and data) for ***welds*** and unique characteristics of **aluminum**
- ***Stationary pressure vessels***
  - ASME Code Case 2938: consolidation of data into **simple design curve**
  - Need to address **high-strength materials**, pressure sensitivity, fatigue threshold and **negative load ratio**
- Extensive ***international partnerships***
  - *Research institutions*: AIST (Japan) , Kyushu University (Japan), KRISS (Korea), MPA Stuttgart (Germany)
  - *Industry*: Japan Steel Works, Tenaris-Dalmine (Italy), FIBA Technologies (US)