



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

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

How do we standardize selection methods for materials for H₂ service?

- **Performance-based method:** *high-pressure vehicle fuel system*
 - Establish materials *performance metrics*
 - Consider mechanics of the service condition
 - Explore relevant environments and determine dominant conditions
- **Design-based method:** *stationary pressure vessel*
 - 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***

Example: 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

Example: 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

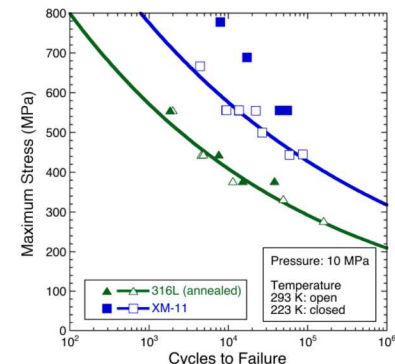
- Fatigue life performance criteria, established to be conservative
 - **Two options:**
 - 100,000 stress cycles when subjected to stress concentration (notched)
 - Conservative stress state
 - Conservative number of cycles
 - 200,000 stress cycles for tension-compression cycle (smooth)
 - Conservative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

Example: 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

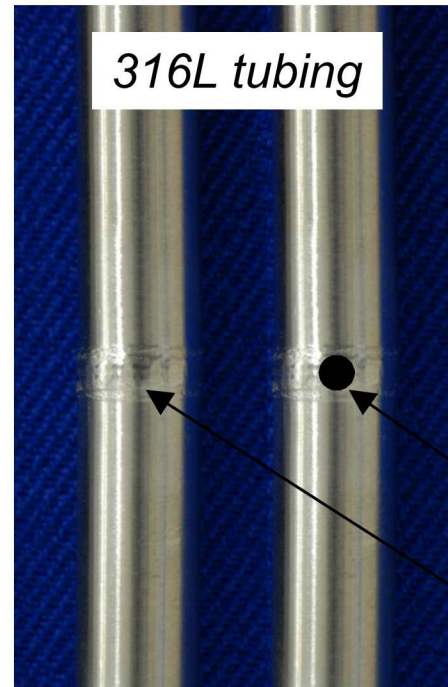
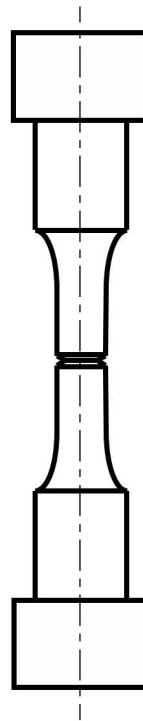
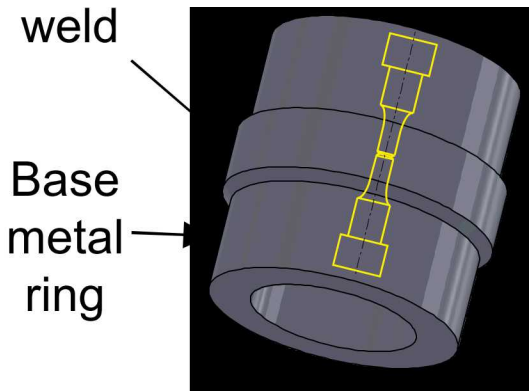


Example: high-pressure vehicle fuel system

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**

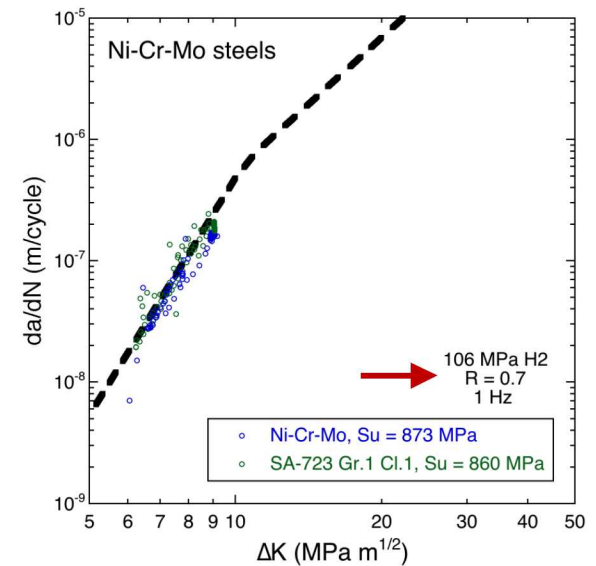
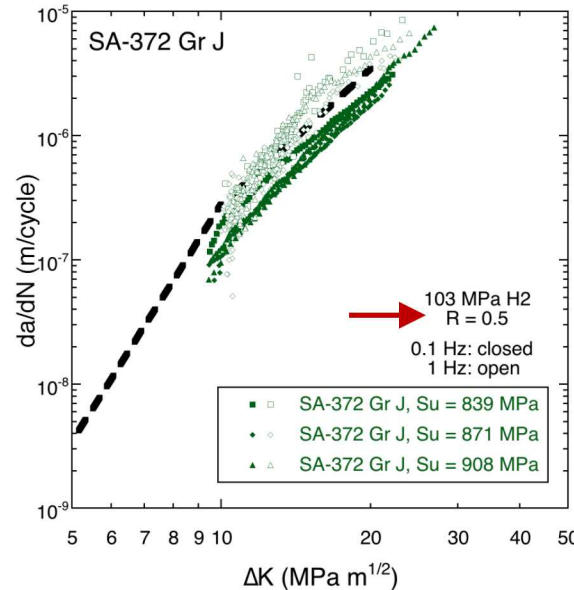
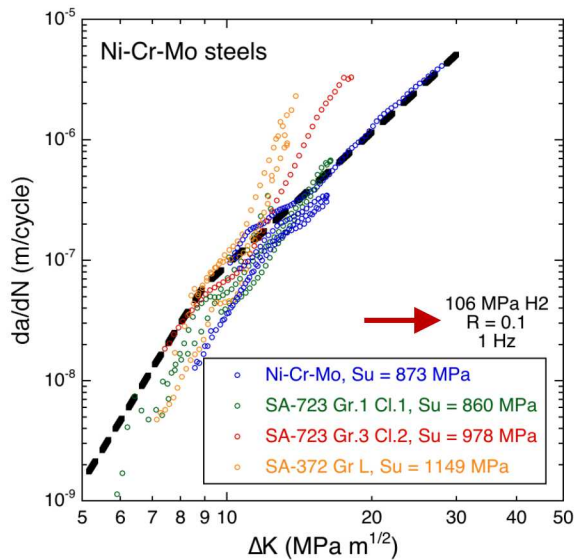
through hole
Orbital tube weld

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

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

Example: stationary pressure vessels ASME Code Case 2938 approved

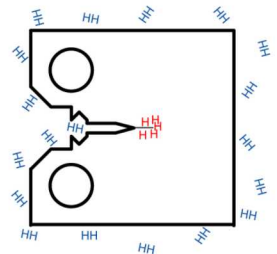
“Technical basis for proposed master curve for fatigue crack growth of ferritic steels in high-pressure gaseous hydrogen in ASME section VIII-3 code” (PVP2019-93907), Proceedings of the 2019 ASME Pressure Vessels & Piping Conference, 14-19 July 2019, San Antonio TX. (manuscript in review)



- 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



Example: 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 $TS \leq 915$ 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 $R < 0$, $K_{min} = 0$

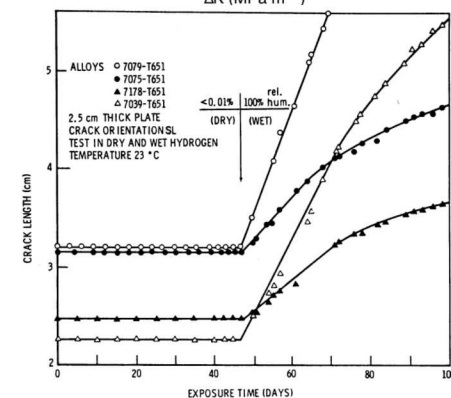
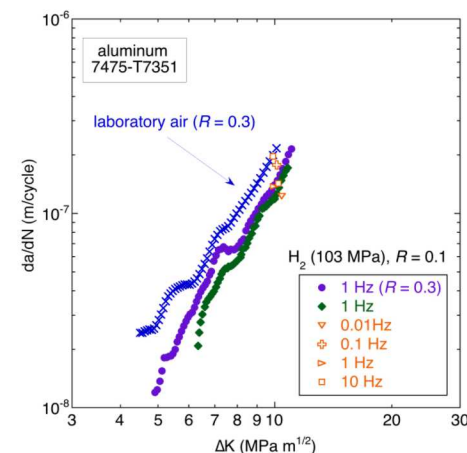
Hardware and methods are being developed for high-pressure testing at low K_{max} and negative K_{min}

Example: 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

Example: 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

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



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)