SAND2017-5638PE

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

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Sandia National Laboratories, Livermore, CA

2017 DOE Hydrogen and Fuel Cells Annual Merit Review

June 6, 2017

Project ID# SCS005

Sandia National Laboratories is a multi-mission 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.

Overview

Timeline

- Project start date: Oct 2003
- Project end date: Sept 2017*

* Project continuation and direction determined by DOE annually

Budget

- Total Project Budget: $9.16M
  - FY16 DOE Funding: $600K
  - Planned FY17 Funding: $560K

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, Swagelok
- **International engagement:** AIST-Tsukuba (Japan), I2CNER (Kyushu University, Japan), MPA Stuttgart (Germany), MATHRYCE (EC project), KRISS (Korea)

Relevance and Objectives

JJC4

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

Slide 3

JJC4 I think we need to "massage" this objective to align with the R&D goals, while maintaining our focus.

James Jr, Charles, 4/19/2017

Project Approach and Milestones

MYRD&D 2013 Barrier	FY17 Milestone	Status
A. Safety Data and Information: Limited Access and Availability	Provide state-of-the-art materials selection tool for hydrogen compatibility	Terms established for public access to Sandia Hydrogen Effects Database (Granta MI)
F. Enabling national and international markets requires consistent RCS	Demonstrate tensile and fatigue life tests in high-pressure gaseous hydrogen at low temperature	Test plan established with Kyushu University and MPA Stuttgart (in support of th JJC2 JJC3 SAE FC Safety Task Force)
G. Insufficient technical data to revise standards	Demonstrate fracture and fatigue testing methods at low-temperature	Components of low-temperature capability integrated; safety assessment near completion
	Establish scientific basis for fracture mechanics assessment of Ni-Cr-Mo steels with global partners	Partnership & MOU established with FIBA (US), Tenaris (Europe) and Japan Steel Works

Slide 4

JJC2 maybe we can say "in support of the SAE FC Safety Task Force"
James Jr, Charles, 4/19/2017

JJC3 Also, can we strengthen the first and last FY17 milestone.
James Jr, Charles, 4/19/2017

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

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

- *Performance-based methods*: On-board FCEVs
  - Establish materials *performance metrics*
  - Relevant environments
  - SAE J2579, CSA CHMC1
- *Prescriptive methods*: stationary pressure vessels
  - Measure reliable *design data*
  - Relevant environments and mechanics
  - ASME BPVC VIII.3.KD-10 (fracture mechanics)
  - *after ASME BPVC VIII.3.KD-3 ??* (fatigue life, crack initiation)

HL1

SMCW2

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

Slide 5

HL1 Is this supposed to also say KD-10?
Hill, Laura, 4/13/2017

SMCW2 Nope. KD-4 is fracture mechanics using ΔK-da/dN; while KD-3 is fatigue life method using S-N. KD-10 is hydrogen specific and derives from KD-4. The hydrogen specific version of KD-3 is missing.
San Marchi, Christopher W, 5/2/2017

Approach: performance-based methods

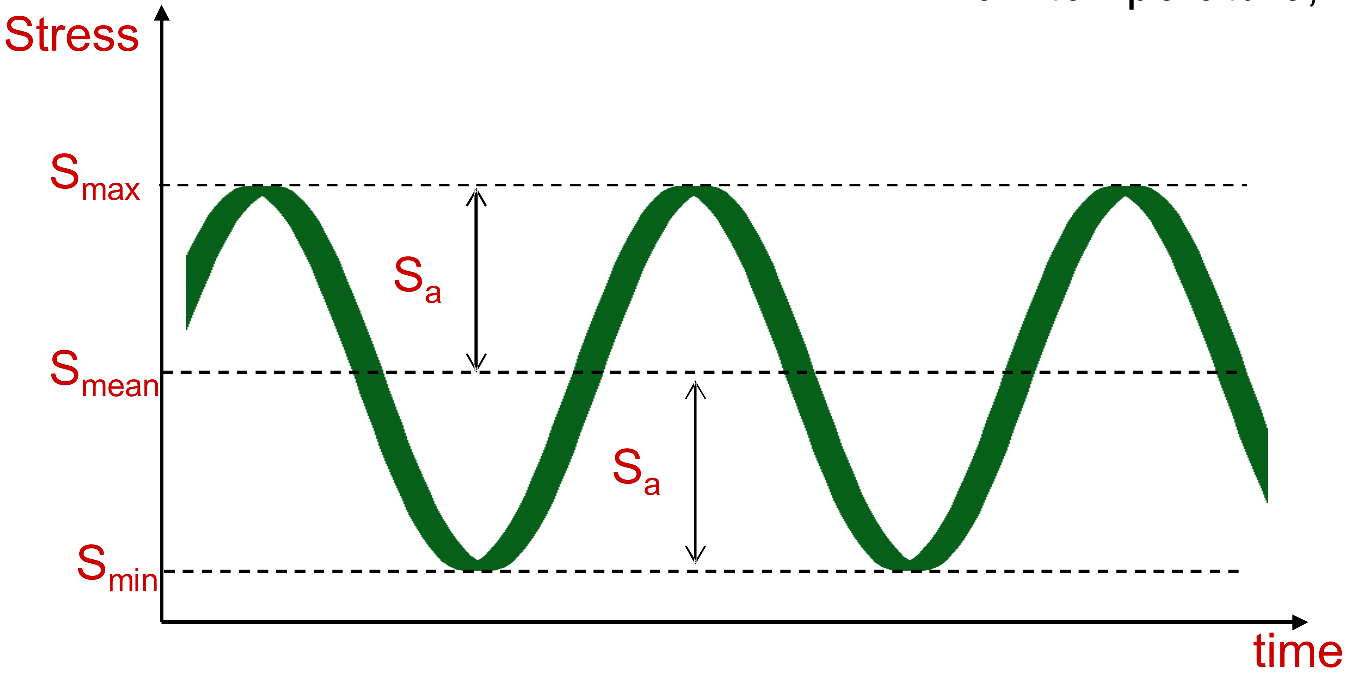
Fatigue life methodology assesses relevant hydrogen-related susceptibilities

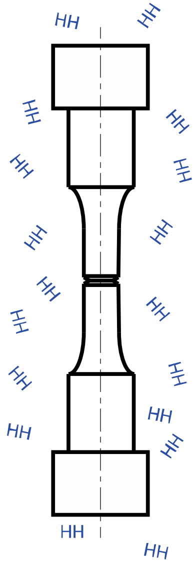
Conventional fatigue life testing

- “smooth” specimens
- Fully reversed loading (R = -1)
- Strain-based for low cycle

Hydrogen fatigue life testing

- “notched” specimens
- Tension-tension loading (R = 0.1)
- Constant stress amplitude (stress-based)
- Low temperature, high pressure



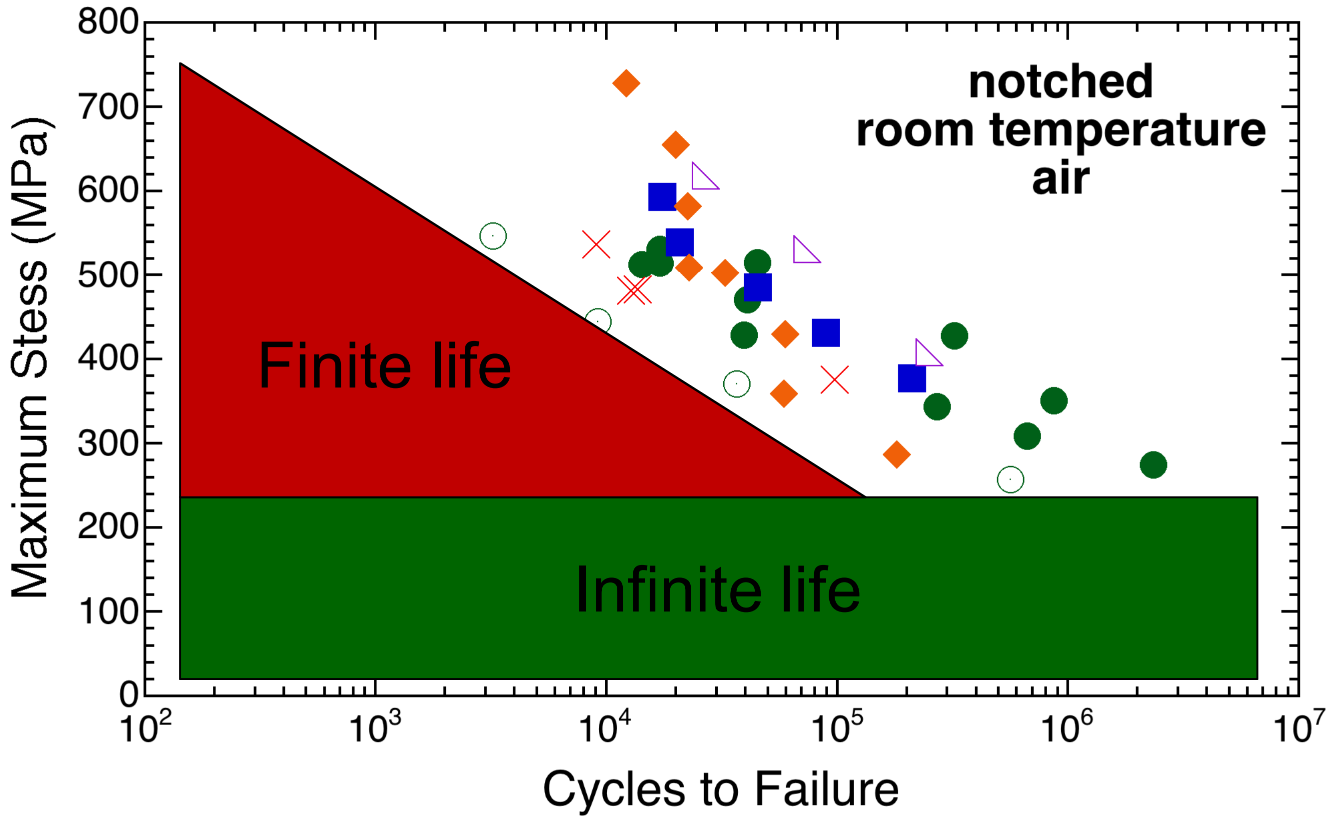


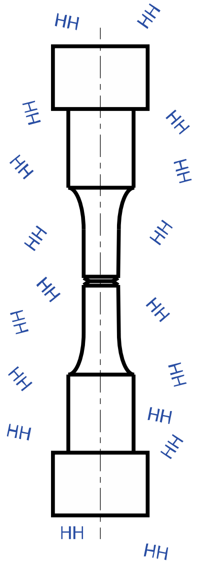
$R = S_{min} / S_{max}$

$S_{max} = 2S_a/(1-R)$

Approach: performance-based methods

How can fatigue life data be used in a *performance-based* methodology?

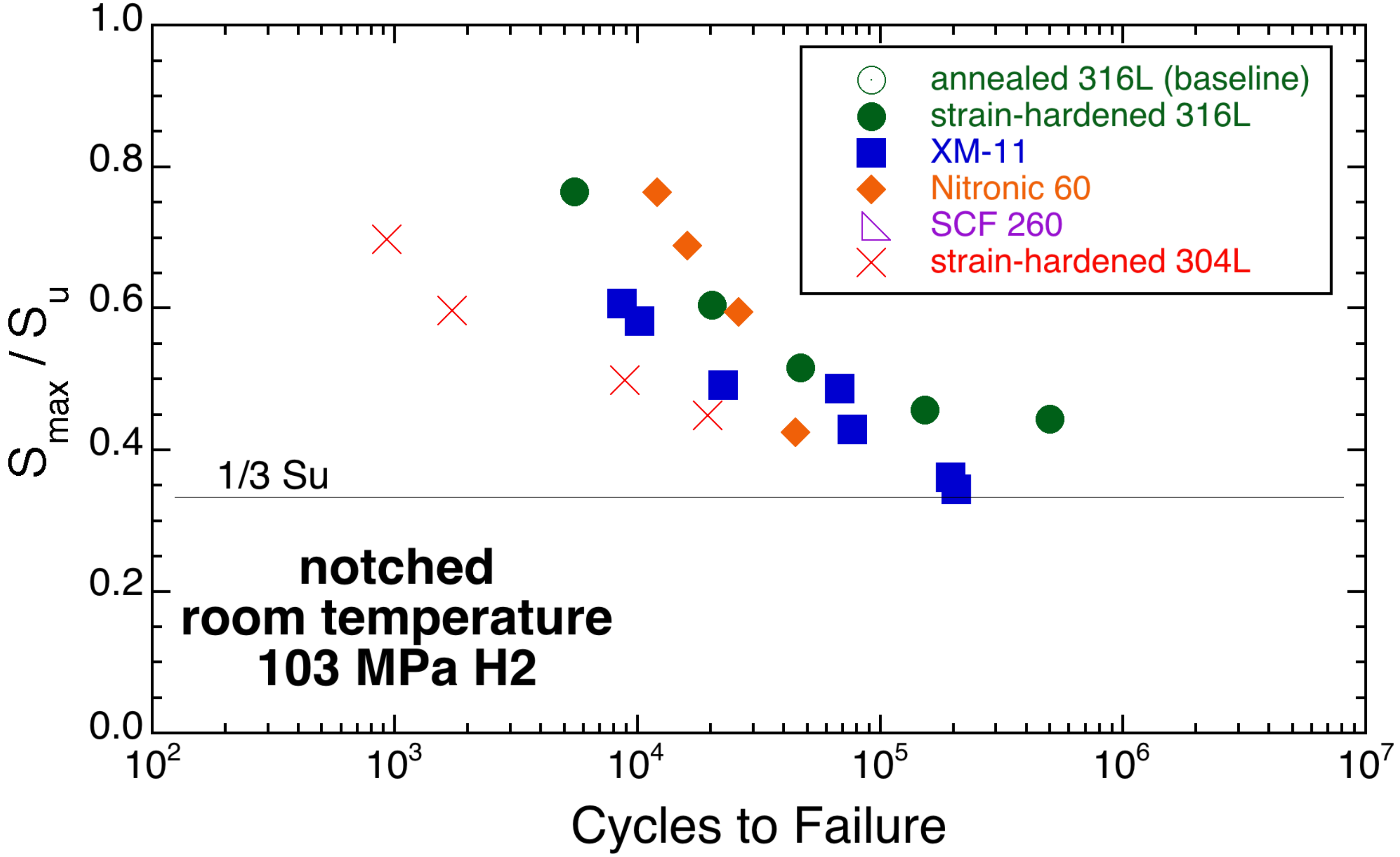




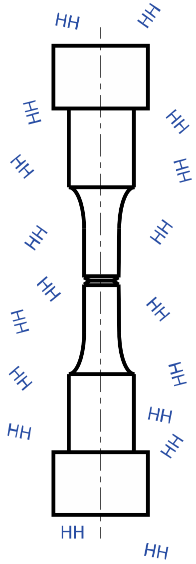
- Test data is being generated in storage program (ST113)
- Capability deployment for low-temperature testing in high-pressure gas and application to SCS is purview of this program

Accomplishment: performance-based methods

Normalization with tensile strength enables comparison of a wide range of materials

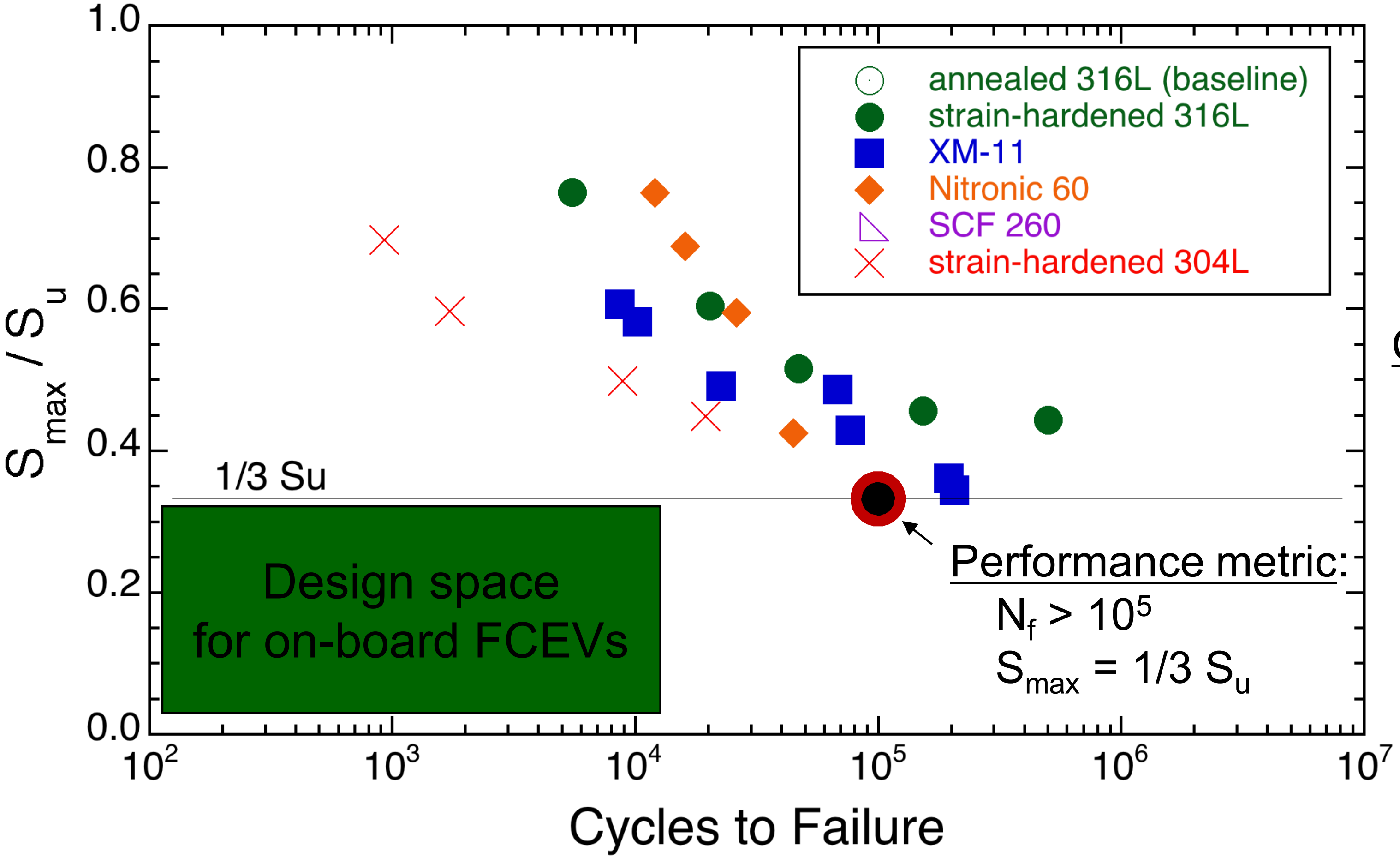


S_u = tensile strength



Accomplishment: performance-based methods

Simple performance metric established based on relevant design space and common design criterion



Performance metric:

$N_f > 10^5$

$S_{max} = 1/3\ S_u$

S_u = tensile strength

S_y = yield strength

Common design criterion:

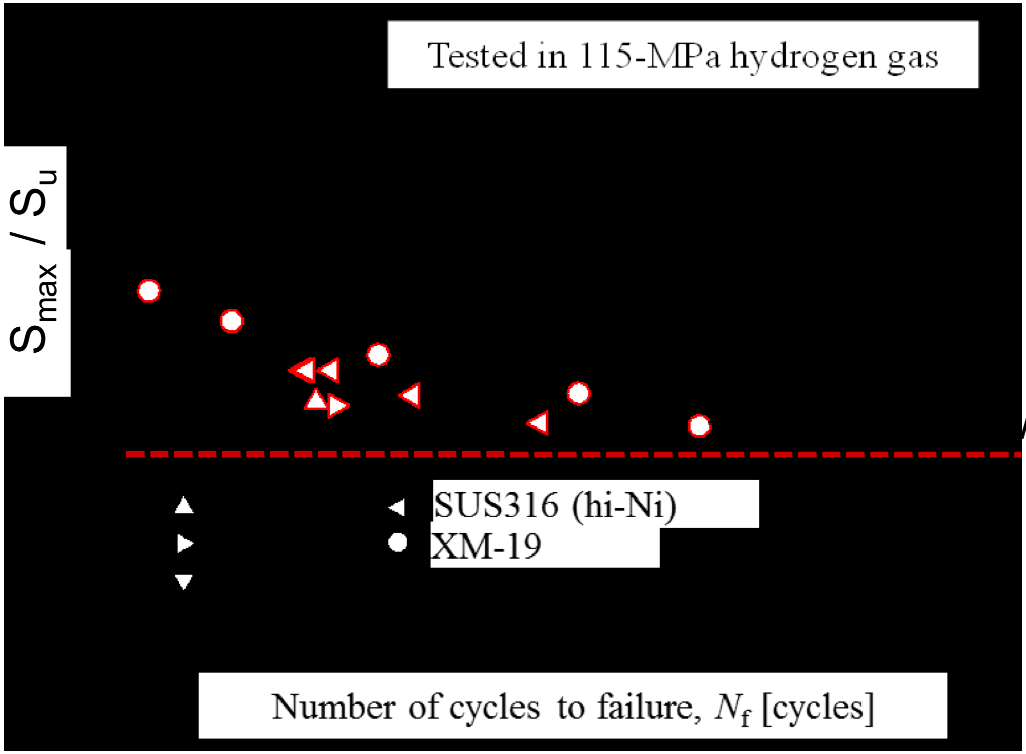
$S \leq 1/3\ S_u$ or

$S \leq 2/3\ S_y$

S = allowable stress (or design stress)

Accomplishment: performance-based methods

Performance metric is also consistent with smooth-specimen fatigue data



Data from Kyushu University

- Smooth specimens
- R = -1

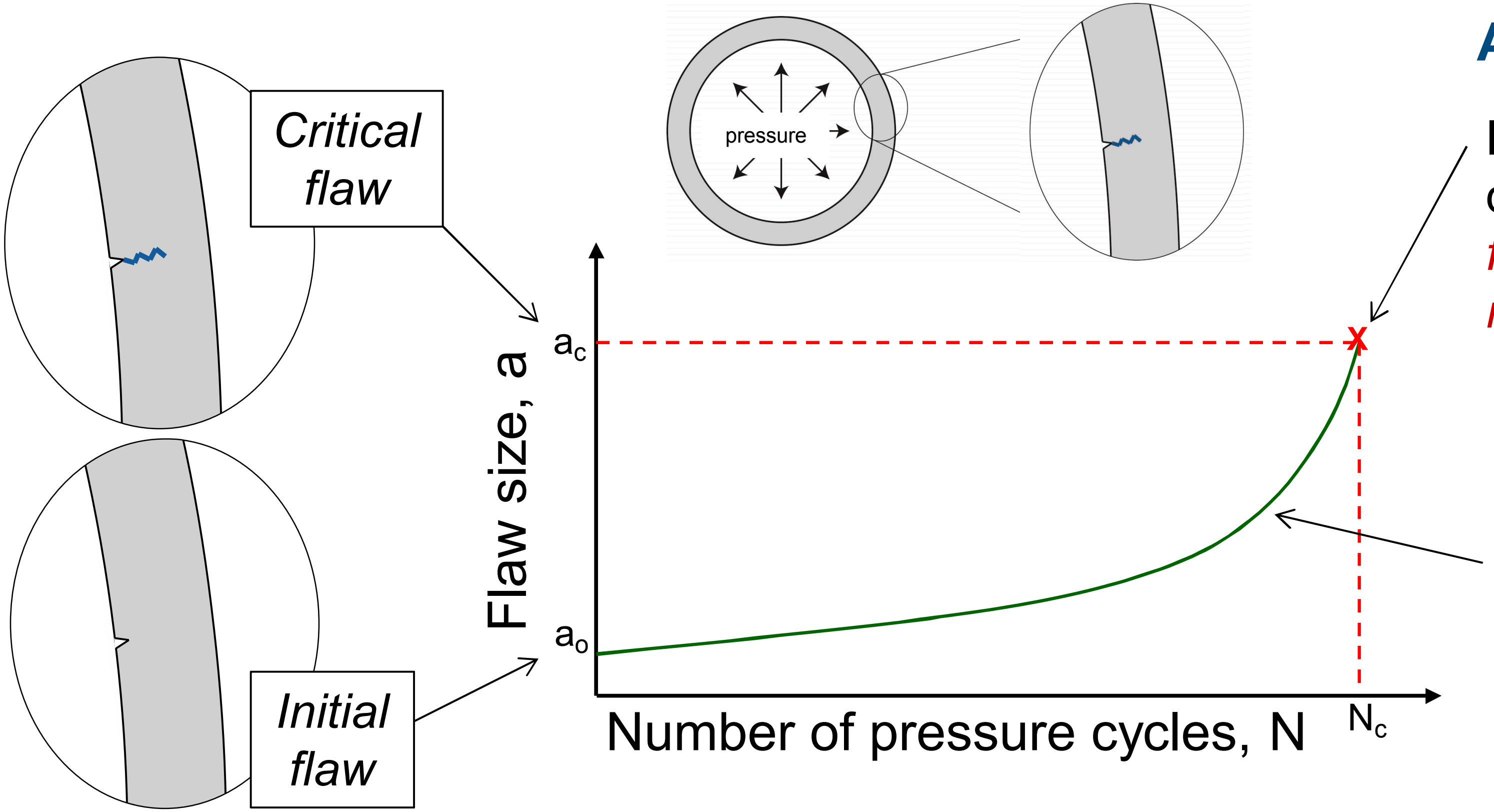
Simple performance metric for fatigue life accommodates different testing methodologies

- **SAE FC Safety Task Force is assessing performance metric**
- **Performance metric is proposed as the basis for materials selection in the Phase II UN GTR No. 13**

Approach: prescriptive-based methods

Use scientific principles to improve existing standardized methods for fracture mechanics-based design

ASME BPVC VIII.3
Article KD-10



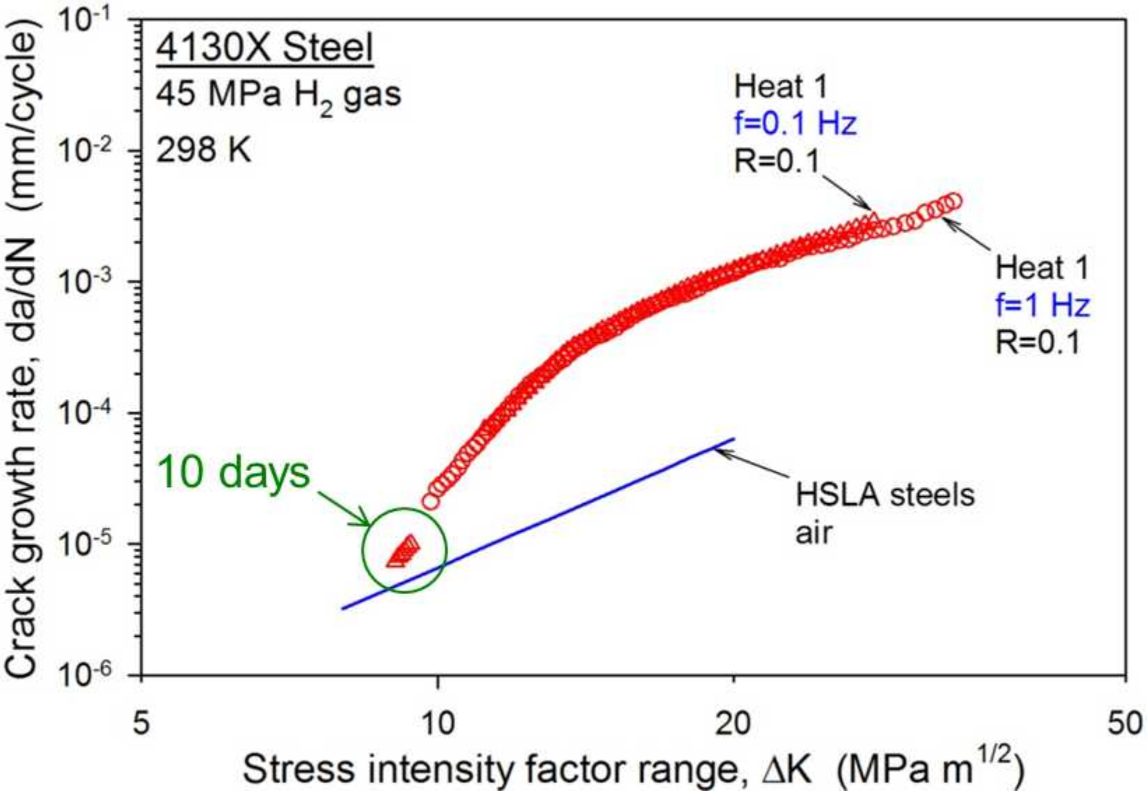
Rupture determined by *fracture resistance*

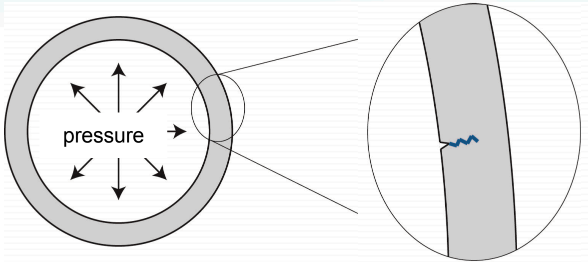
Evolution of flaw size determined by *fatigue crack growth*

Industry needs and uses fracture mechanics to optimize pressure vessel designs for stationary storage

Approach: prescriptive-based methods

Life prediction depends on fatigue crack growth measurements





$$\Delta K = \Delta P \times f(a, t, R)$$

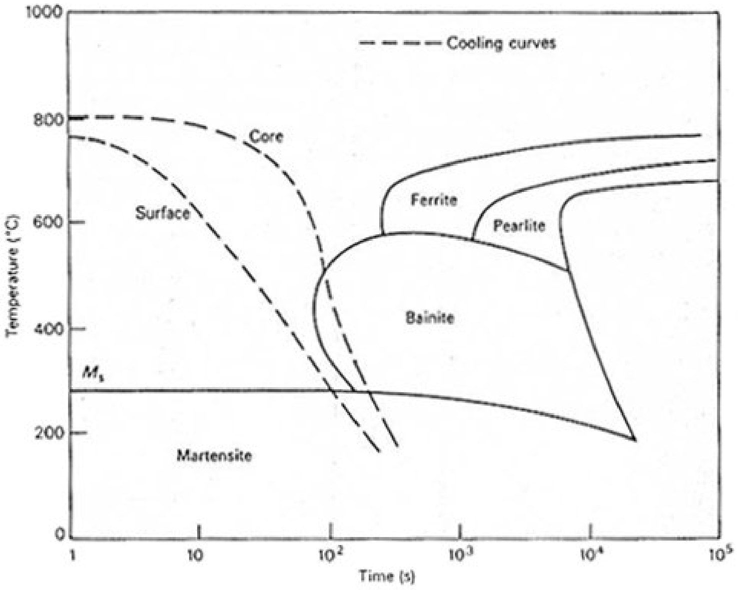
$$da/dN = C \Delta K^m$$

$$a = a_i + \left(da/dN\right)^{a=a_i} \Delta N$$

Efficient methods for generating fatigue crack growth data are needed to enable conservative predictions

Approach: prescriptive-based methods

International partnership provides common basis for development of advanced storage options



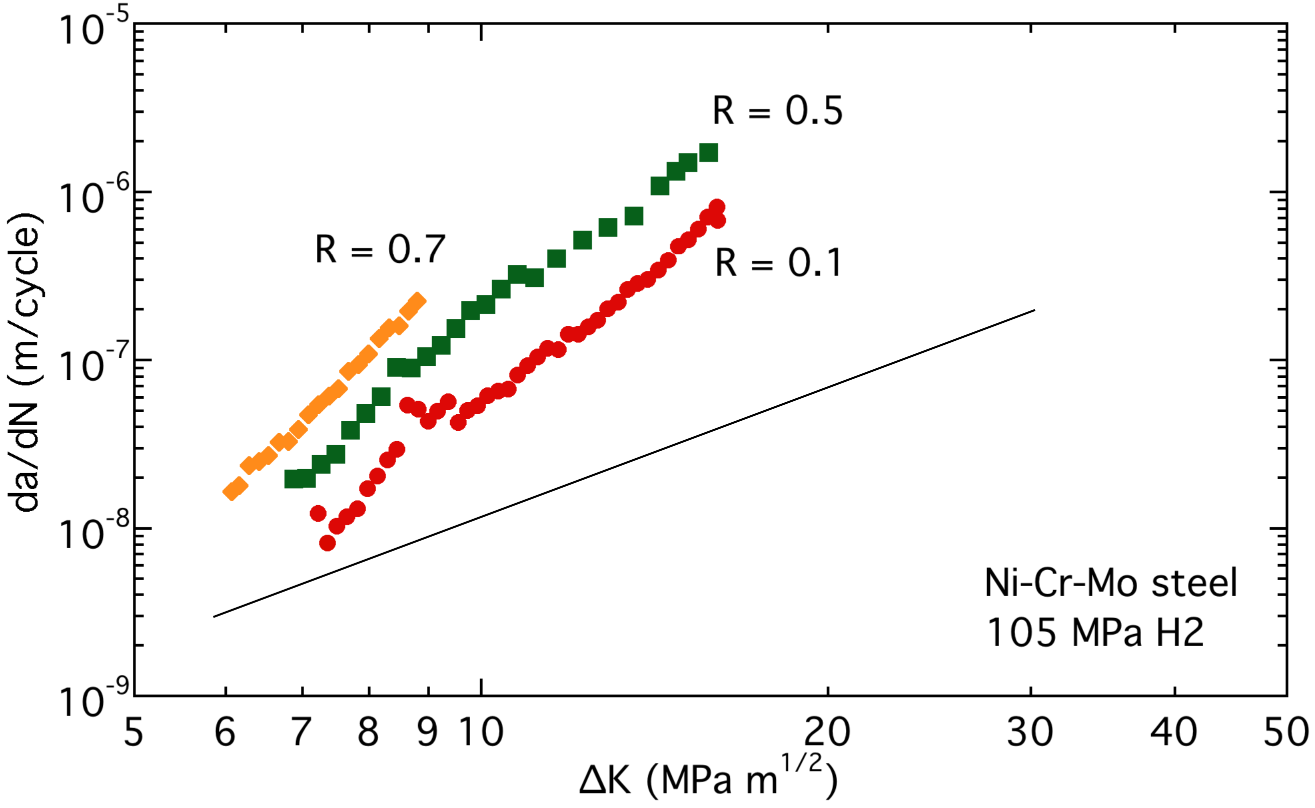
- Pressure vessel steels are quenched to achieve uniformity of desired properties through the wall thickness
- “Hardenability” of Cr-Mo steels limited to <38mm wall thickness

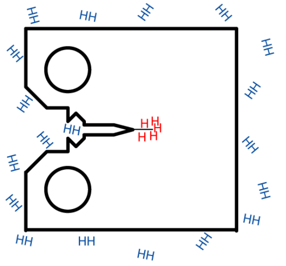
- Ni-Cr-Mo pressure vessel steels provide superior hardenability
  - Reduces variability in thick-walled steel vessels
  - Enables design with greater inner diameter (greater volume)
- International MOU established for evaluating Ni-Cr-Mo pressure vessels
  - Fiba Technologies (US)
  - Tenaris-Dalmine (Europe)
  - Japan Steel Works (Asia)

Scientific objective: ***Establish scientific basis for fracture mechanics assessment***
Engineering objective: ***Increase storage volume of stationary pressure vessels***

Accomplishment: prescriptive-based methods

Results for Ni-Cr-Mo steels in high-pressure gaseous hydrogen are consistent with other steels





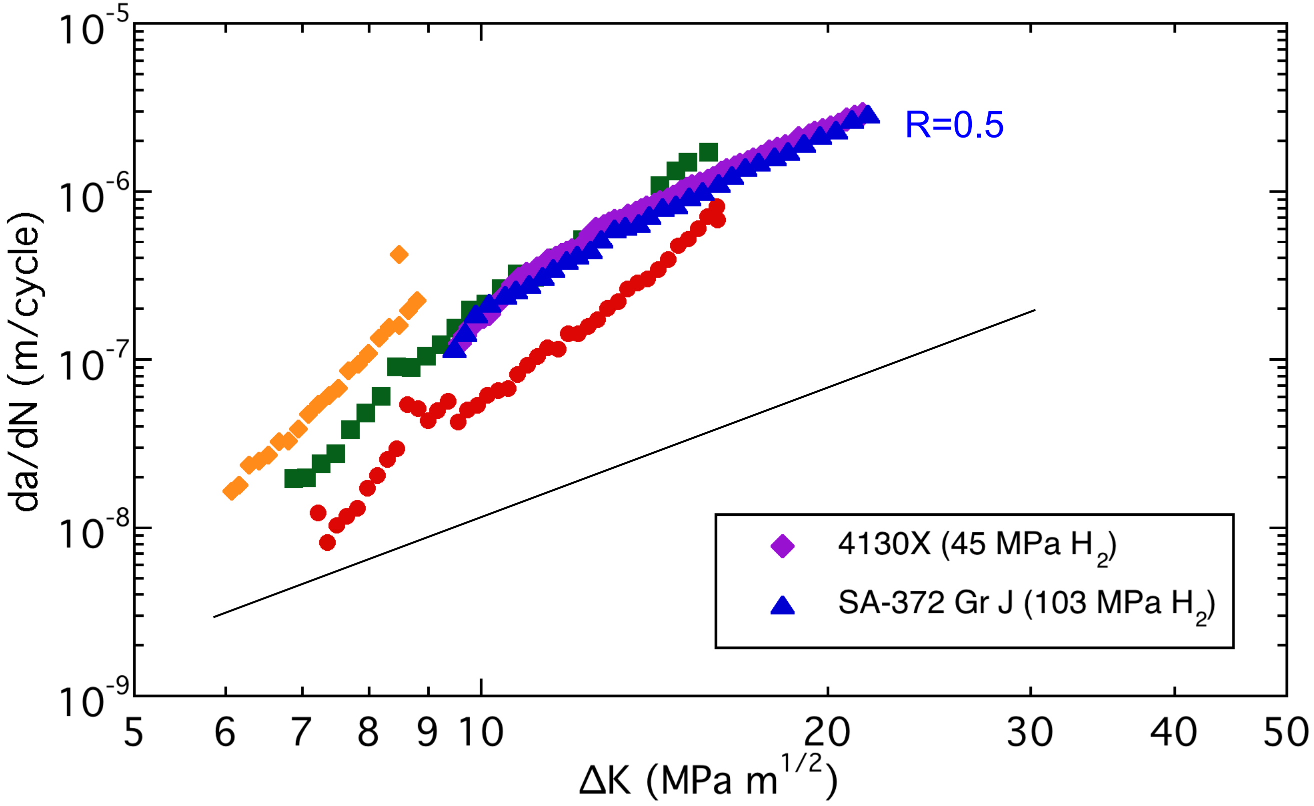
Cascade storage employs PVs operating in narrow pressure ranges:

- ΔP∝ΔK is small
- R is large ($=P_{min}/P_{max}$)

The effect of load ratio (R) follows expected trends, emphasizing hydrogen-accelerated FCG at low ΔK

Accomplishment: prescriptive-based methods

Ni-Cr-Mo pressure vessel steels compare favorably to Cr-Mo pressure vessel steels



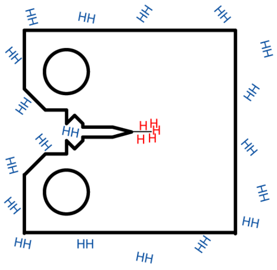
Common PV steels

Cr-Mo

- SA-372 Gr.J (ASME)
- 4130X (DOT)

Ni-Cr-Mo

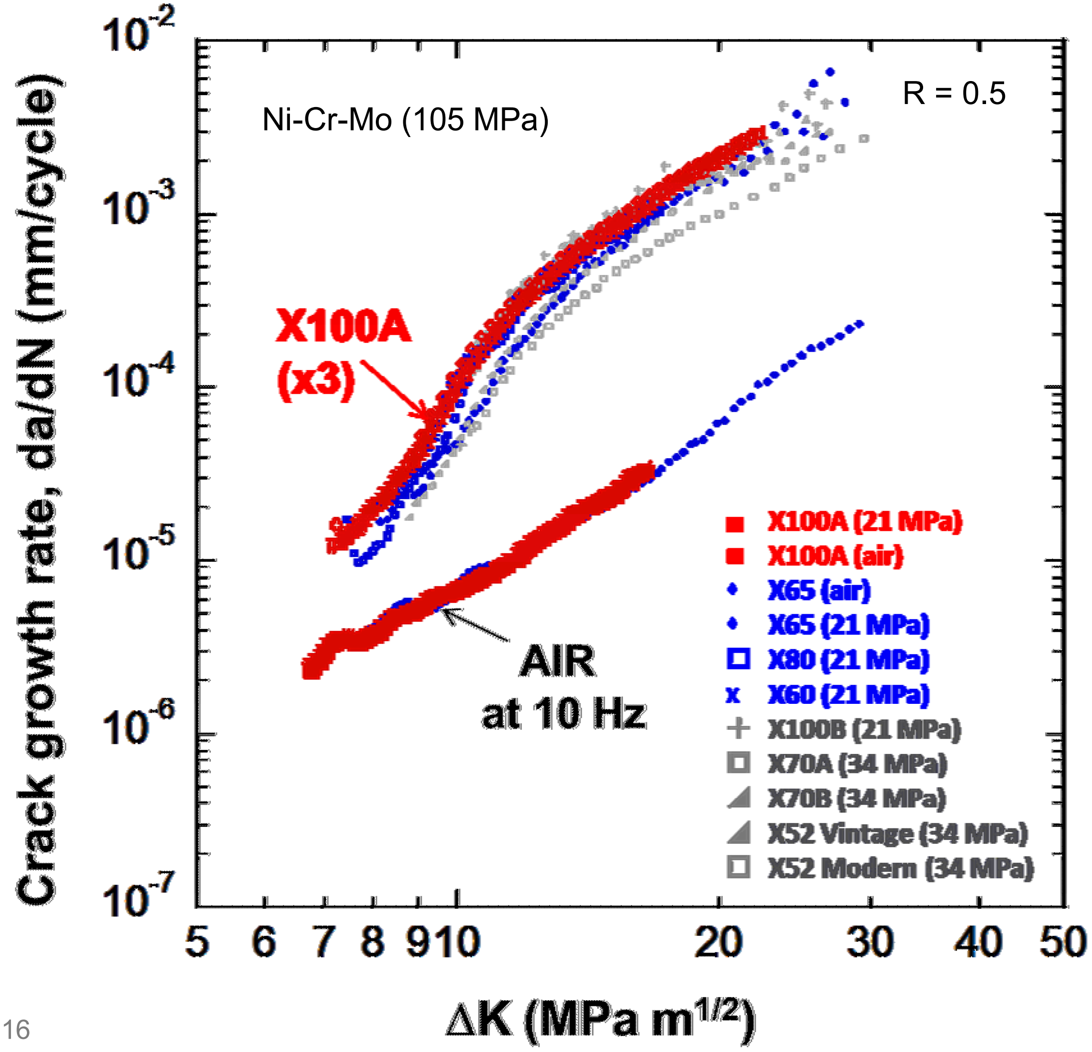
- SA-372 Gr.L (ASME)
- SA-723 (ASME)



Fatigue crack growth appears to be relatively insensitive to pressure, in comparison to other parameters

Accomplishment: prescriptive-based methods

Low strength steels tend to show very similar fatigue crack growth rates in gaseous hydrogen



- A wide variety of pipeline steels display nominally the same fatigue response as HSLA steels
- The effect of pressure on fatigue is generally within the scatter
- Lack of data at low ΔK
  - *the majority of the life of small cracks occurs at low ΔK*

Response to Previous Year Reviewers' Comments

- *FY16 Reviewer Comment:* "Improve industry collaboration and conducting industry-led testing."
  - The partnership to evaluate Ni-Cr-Mo pressure vessel steels includes industry participation from the US, Europe and Asia. Partners have provided steels for evaluation and are eager to make use of the data.
  - The program provides leadership in SAE's FC Safety Task Force for establishing methods and performance metrics for on-board FCEV refueling systems with input from all interested industry participants, including active participation from major (international) automotive manufacturers. DOT also participates and has requested additional outreach to other nations expected to participate in Phase II of the UN GTR no. 13.
- *FY16 Reviewer Comments:* there is a general theme of "better definition of future work and priorities", such as: "A statement of specific areas in which future work can support standards development would be helpful..."; "Progress and remaining key actions should be explained because it is difficult to distinguish the relevance and new information…"
  - We have tried to be more mindful of developing and communicating a long-term strategy for the work and packaging the strategy succinctly by maintaining consistent themes and vocabulary throughout the slide set

HL2

Slide 17

HL2 While I get what you're saying here, is there a better way to say it? I read the comments as being more that the distinction between current work and future priorities wasn't clear last year.

Hill, Laura, 4/13/2017

Collaborations

- Standards Development Organizations (SDOs)
  - Sandia technical staff participate on committees engaged in materials testing and selection for hydrogen service when the SDOs are active
  - Low-temperature fatigue studies are being actively being shared with the FC Safety Task Force at SAE
- 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 informs development of advanced high-pressure storage
- 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)
    - shared learning on capability deployment and testing methods

Remaining Challenges and Barriers

- Determine simple metrics for materials selection that are independent of design philosophy
  - Generalized metrics remain elusive for environmental-assisted fracture and fatigue
- Demonstrate low-temperature, high-pressure capability for standardized materials characterization
  - One-of-a-kind system design incorporates several unique innovations, thus timeline for full commissioning is uncertain (international interactions and shared learning are aiding deployment)
- Establish internationally harmonized fatigue life test methods
  - Europe and Asia embrace different test methods/parameter space
  - Significant progress on conservative criteria (“infinite life”); less conservative performance-based metrics will be a challenge (“finite life”)
- Formulate partnerships for effectively defining and performing high-impact R&D activities
  - Lack of sustained engagement from broader community

Proposed Future Work

Remainder of FY17

- ***Low-temperature, high-pressure testing capability for fuel systems and refueling infrastructure***
  - Continue shakedown of "first of its kind" testing system: working with vendors to address component failures
  - Complete low-temperature system demonstration testing for SAE in partnership with Kyushu and MPA Stuttgart
- ***Advanced stationary storage options***
  - Establish scientific basis for fracture mechanics assessment of Ni-Cr-Mo steels for larger pressure vessels with global partners

FY18

- ***Advancing design tools and harmonized methods***
  - Enhance coordination activity with international partners (e.g., GTR no. 13)
  - Develop hardware designs for reverse loading and strain-based methods to extend test method development to negative load ratios
  - *Necessary for conventional design methodologies and to characterize autofrettage*
- ***Scientific basis for finite life design strategies***
  - Extend test methods to characterize crack initiation and short crack behavior
  - *Necessary for design at high stresses and for finite life*

Summary

- Definitive *database tools* for materials selection
  - Agreement for international access to state-of-the-art data management tools to complement the *Technical Reference for Hydrogen Compatibility*
- *Performance-based methods*: materials for on-board FCEVs
  - Simple fatigue performance metric proposed within SAE Safety Task Force
    - Performance test could be the basis for materials selection in GTR Phase II
  - General performance metric for ”finite” life will be a challenge
- *Prescriptive-based methods*: stationary pressure vessels
  - Ni-Cr-Mo steels appear to display similar fatigue crack growth behavior in hydrogen as other pressure vessel and pipeline steels
    - Thick-walled stationary storage vessels of Ni-Cr-Mo steel are feasible
  - More data at high load ratio and low ΔK is needed, as these conditions represent a combination of most critical and most relevant
- 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)

Technical Back-Up Slides

Joint test matrix of SAE H2 Compatibility Expert Team

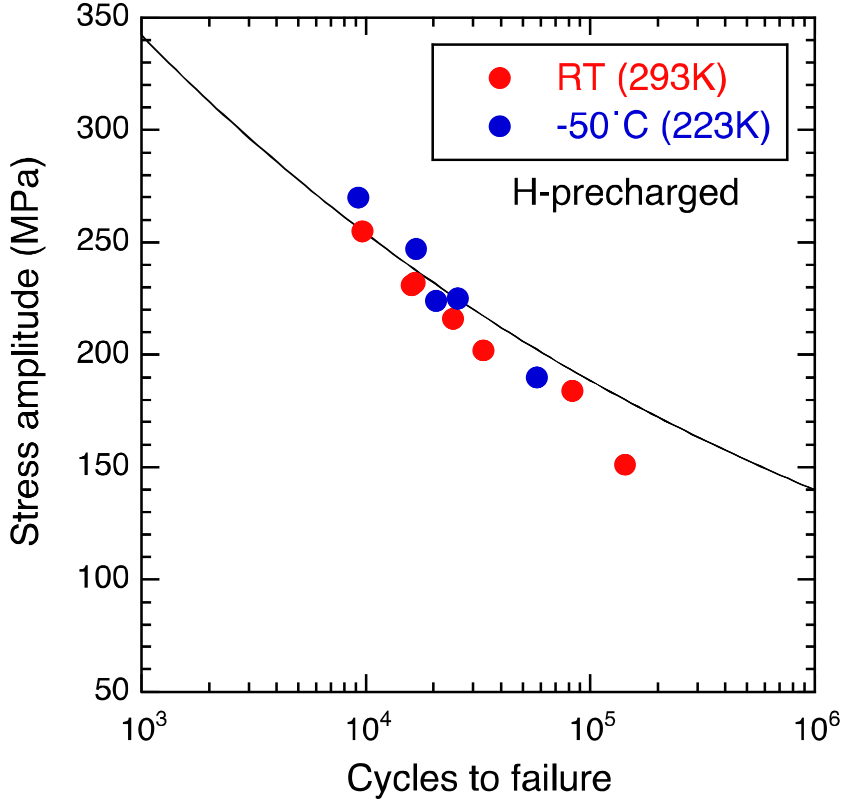
Objective: develop shared experience and knowledge of test methods

Test	Test conditions	Environment	Number of tests each institute†
SSRT	<5 x10^{-5} s^{-1}	Control -40˚C	3
		90 MPa H2 -40˚C	3
Notched fatigue	Sa = 200 MPa R = 0.1 1 Hz	Control -40˚C	3
		90 MPa H2 -40˚C	3
Smooth fatigue	Sa = **300** MPa R = -1 1 Hz	Control -40˚C	3
		90 MPa H2 -40˚C	3

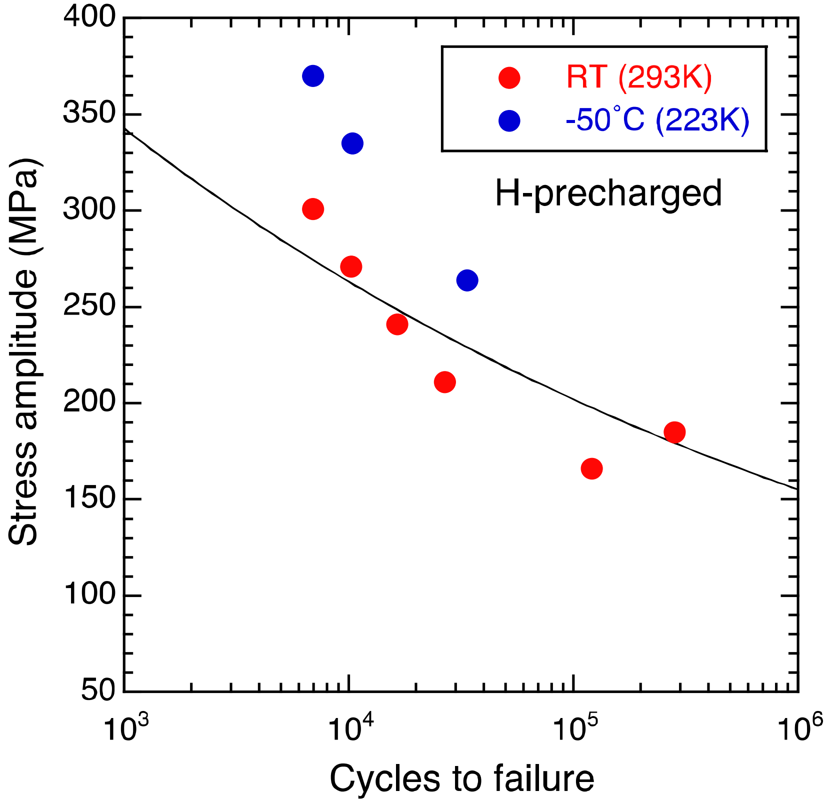
†Participants: Sandia National Laboratories, Kyushu University, MPA Stuttgart

Fatigue life measurements indicate tensile trends cannot be extrapolated to design

Strain-hardened type 316L (12wt% Ni)



Annealed XM-11 (6.2 wt% Ni)



RRA (RT) ~ 80% RRA (223K) ~ 50%	Tensile ductility	RRA (RT) ~ 60% RRA (223K) ~ 30%

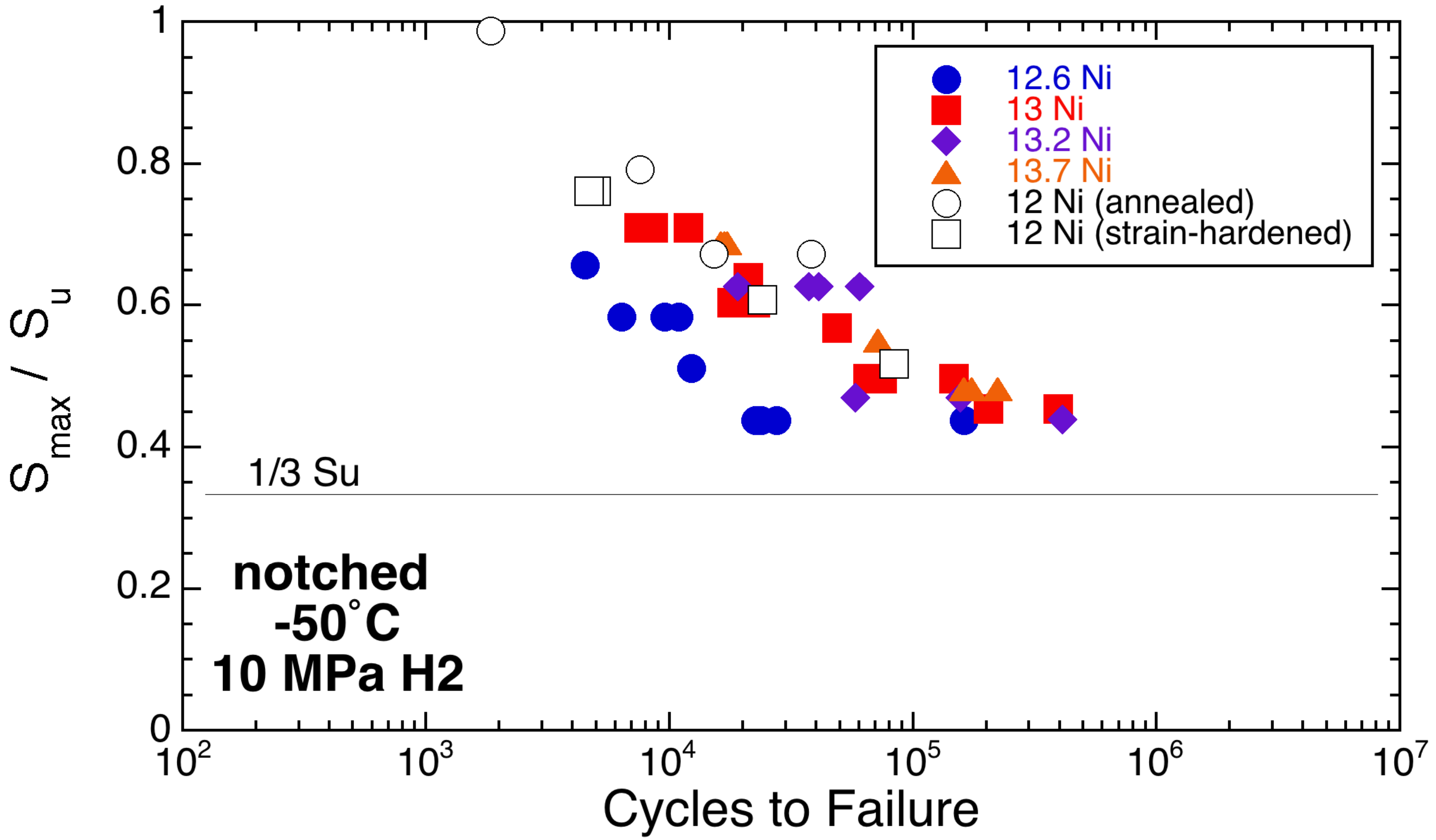
Range of austenitic stainless steels are being evaluated by fatigue life methodology in the storage program

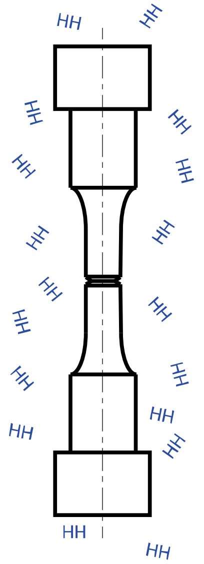
material	S_y (MPa)	S_u (MPa)	Cr	Ni	Mn	N
316L	280	562	17.5	12	1.2	0.04
CW 316L	573	731	17.5	12	1.2	0.04
304L	497	721	18.3	8.2	1.8	0.56
XM-11	539	881	20.4	6.2	9.6	0.26
Nitronic 60	880	1018	16.6	8.3	8.0	0.16
SCF-260	1083	1175	19.1	3.3	17.4	0.64

Wide range of strength (i.e., weight)

Wide range of Ni content (i.e., cost)

Data from the literature is consistent with performance criterion based on $1/3S_u$





Colored symbols from Michler et al. *Intern J Fatigue* **96** (2017) 67-77.

Reviewer-Only Slides

Critical Assumptions and Issues

1. One principal focus of the materials testing is assessing the effects of load-cycle frequency on fatigue crack growth rates of steels in high-pressure hydrogen gas. Sandia has developed one of the only specialized laboratory capabilities for conducting these fatigue crack growth measurements in the U.S. However, Sandia currently has only one such laboratory capability, and thus testing can only be conducted serially. In addition, the fatigue crack growth testing must cover a wide range of load-cycle frequencies, including frequencies less than 1 Hz. Consequently, test durations in hydrogen gas can be rather extended, e.g., days to weeks. The extended test times coupled with only one current testing apparatus may lead to limitations on the amount of testing that can be accomplished. For this reason, it is important to coordinate activities with other research institutions, such as HYDROGENIUS/AIST (Tsukuba, Japan) and I^2CNER (Kyushu University, Japan).

2. We are dependent on stakeholders to supply technologically relevant materials for testing. It is imperative that we generate data for materials that represent those used in service. To date, we have been able to receive ample materials through our interactions with industry partners, e.g., FIBA Technologies, Swagelok, as well as both European and Japanese pressure vessel manufacturers. We must maintain and expand relationships with industry partners and SDOs not only so that we have a supply of materials but also access to their input into materials testing parameters.

Publications and Presentations

- C. San Marchi, E.S. Hecht, I.W. Ekoto, K.M. Groth, C. LaFleur, B.P. Somerday, R. Mukundan, T. Rockward, J. Keller, C.W. James: "Overview of the DOE hydrogen safety, codes and standards program, part 3: Advances in research and development to enhance the scientific basis for hydrogen regulations, codes and standards". *Intern J Hydrogen Energy* (proof online).
- B.P. Somerday, J.A. Campbell, K.L. Lee, J.A. Ronevich, C. San Marchi: "Enhancing safety of hydrogen containment components through materials testing under in-service conditions". *Intern J Hydrogen Energy* (proof online).
- B. An, Z. Hua, T. Iijima, C. Gu, J. Zheng, C. San Marchi: "Scanning Kelvin probe force microscopy study of hydrogen distribution and evolution in duplex stainless steel" (PVP2017-66121), Proceedings of the 2017 ASME Pressure Vessels & Piping Conference, 16-20 July 2017, Waikoloa, HI.
- T. Michler, C. San Marchi, K. Berreth, J. Naumann, R.K. Mishra, R.C. Kubic, "Microstructure, deformation mechanisms and influence of hydrogen on tensile properties of the Co based super alloy DIN 2.4711/UNS30003". Mater Sci Eng A662 (2016) 36-45.
- 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, Livermore CA, April 2016
- L. Zhang, B. An, T. Iijima, C. San Marchi, "Effect of gaseous hydrogen charging on nanohardness of austenitic stainless steels", Proceedings of the ASME2016 Pressure Vessels & Piping Conference, PVP2016-63390, Vancouver BC, Canada, July 2016.
- B.P. Somerday, J.A. Campbell, K.L. Lee, J.A. Ronevich, and C. San Marchi (presentation). "Enhancing Safety of Hydrogen Containment Components Through Materials Testing Under In-Service Conditions". In Proceedings of the International Conference on Hydrogen Safety (ICHS 2015), Yokohama, Japan, October 19-21, 2015
- C. San Marchi, E. S. Hecht, I. W. Ekoto, K. M. Groth, C. LaFleur, B. P. Somerday, R. Mukundan and T. Rockward. "Advances in research and development to enhance the scientific basis for hydrogen regulations, codes, and standards." In Proceedings of the International Conference on Hydrogen Safety (ICHS 2015), Yokohama, Japan, October 19-21, 2015
- 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
- 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.
- 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.