H2FC Hydrogen and Fuel Cells Program

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

Chris San Marchi, presenter
Brian Somerday, former PI

Sandia National Laboratories

DOE Hydrogen and Fuel Cells Program Annual Merit Review

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Project ID # SCS005

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Overview

Timeline and Budget

- Project start date: Oct 2003
- Project end date: Sept 2016*

* Project continuation and direction determined by DOE annually

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

Budget

- Total Project Budget: $8.6M
  - Total FY16 Budget: $600K
  - Total Partner Share: $50K

Partners

- **SDO/CDO participation:** CSA, ASME, SAE, ISO
- **Industry:** FIBA Technologies, Tenaris-Dalmine, Japan Steel Works (JSW), BMW, Opel, Swagelok
- **International engagement:** AIST-Tsukuba (Japan), I2CNER (Kyushu University, Japan), MPA Stuttgart (Germany), MATHRYCE (EC project), IPHE, KRISS (Korea)

Relevance and Objectives

Objective: Enable technology deployment by providing science-based resources for standards and hydrogen component development and participate directly in formulating standards

Barrier from 2013 SCS MYRDD	Project Goal
A. Safety Data and Information: Limited Access and Availability	Develop and maintain material property database and identify gaps in available material property databases
F. Enabling national and international markets requires consistent RCS	Develop more efficient and reliable materials test methods and work with SDOs (e.g., SAE, CSA, ASME) to validate and incorporate methods in 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

Relevance:

Materials Compatibility and Components project impacts multiple standards

- **ASME Article KD-10**: hydrogen pressure vessels
  - Fracture mechanics approach (fatigue crack growth and fracture)
  - Need: relevant data and improved efficiency of fatigue crack growth testing methods
  - Activity: identifying frequency effects and expanding scope to SA-723 steels
- **SAE J2579**: onboard hydrogen fuel systems
  - Fatigue life approach (includes slow strain rate tensile testing)
  - Need: stakeholders desire test data and international harmonization
  - Activity: developing testing capability for low-temperature fatigue
- **CSA CHMC1**: general test methods in gaseous hydrogen
  - Fracture, fatigue and tensile testing for metallic materials
  - Need: leadership in method development and validation
  - Activity: evaluating methods by exploring parameter space (e.g., temperature, frequency)

Project Approach and Milestones

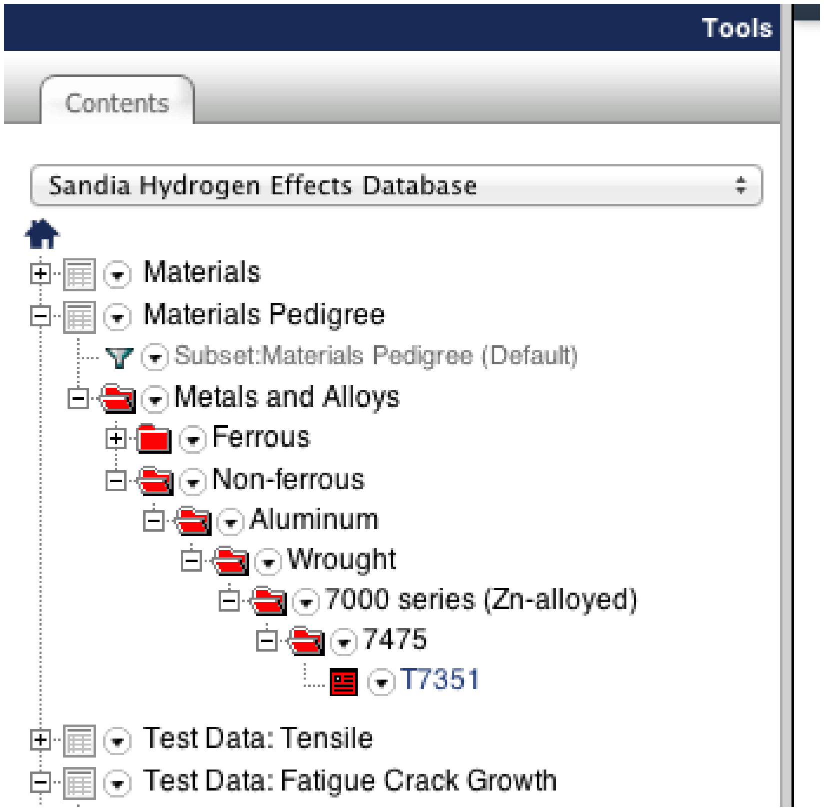
MYRD&D 2013 Barrier	FY16 Milestone	Status
A. Safety Data and Information: Limited Access and Availability	Develop material property database	Trial public access to Sandia Hydrogen Effects Database (using Granta MI)
F. Enabling national and international markets requires consistent RCS	Establish coordinated fatigue life testing activities and data sharing with international stakeholders	• Participating in SAE test definition and coordination activity (FC Safety Task Force) • Promoting IPHE activity for test method validation
G. Insufficient technical data to revise standards	Demonstrate low-temperature fatigue life method for austenitic stainless steels	Final component of low-temperature capability expected in June 2016
	Evaluate relevant Ni-Cr-Mo steels for advanced high-pressure storage	Partnership established with FIBA (US), Tenaris (Europe) and Japan Steel Works

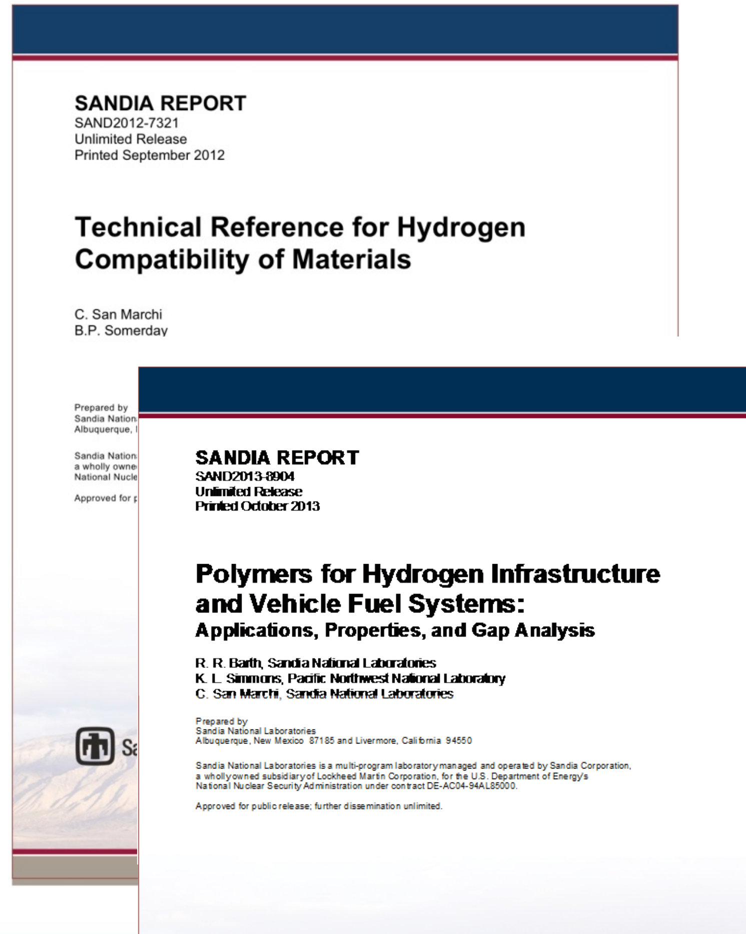
Project Approach and Milestones

MYRD&D 2013 Barrier	FY16 Milestone	Status
A. Safety Data and Information: Limited Access and Availability	**1. Database tools** Develop material property database	Trial public access to Sandia Hydrogen Effects Database (using Granta Mi)
F. Enabling national and international markets requires consistent RCS	**2. Low-temperature testing** Establish coordinated fatigue life testing activities and data sharing with international stakeholders	• Participating in SAE test definition and coordination activity (FC Safety Task Force) • Promoting IPHE activity for test method validation
G. Insufficient technical data to revise standards	Demonstrate low-temperature fatigue life method for austenitic stainless steels	Final component of low-temperature capability expected in June 2016
	3. Advanced storage Evaluate relevant Ni-Cr-Mo steels for advanced high-pressure storage	Partnership established with FIBA (US), Tenaris (Europe) and Japan Steel Works

Approach: database tools

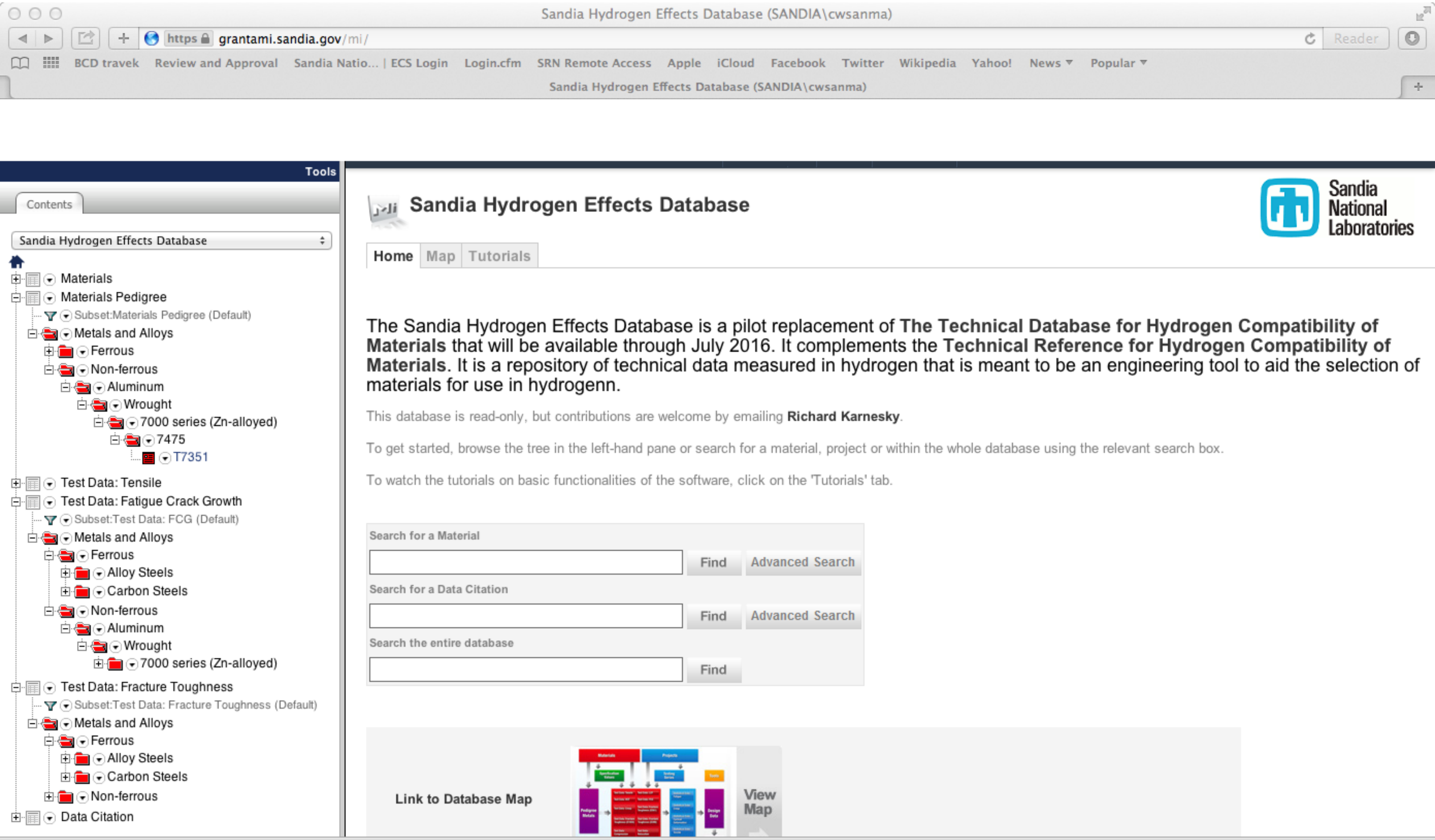
Develop engineering resources to enable materials selection





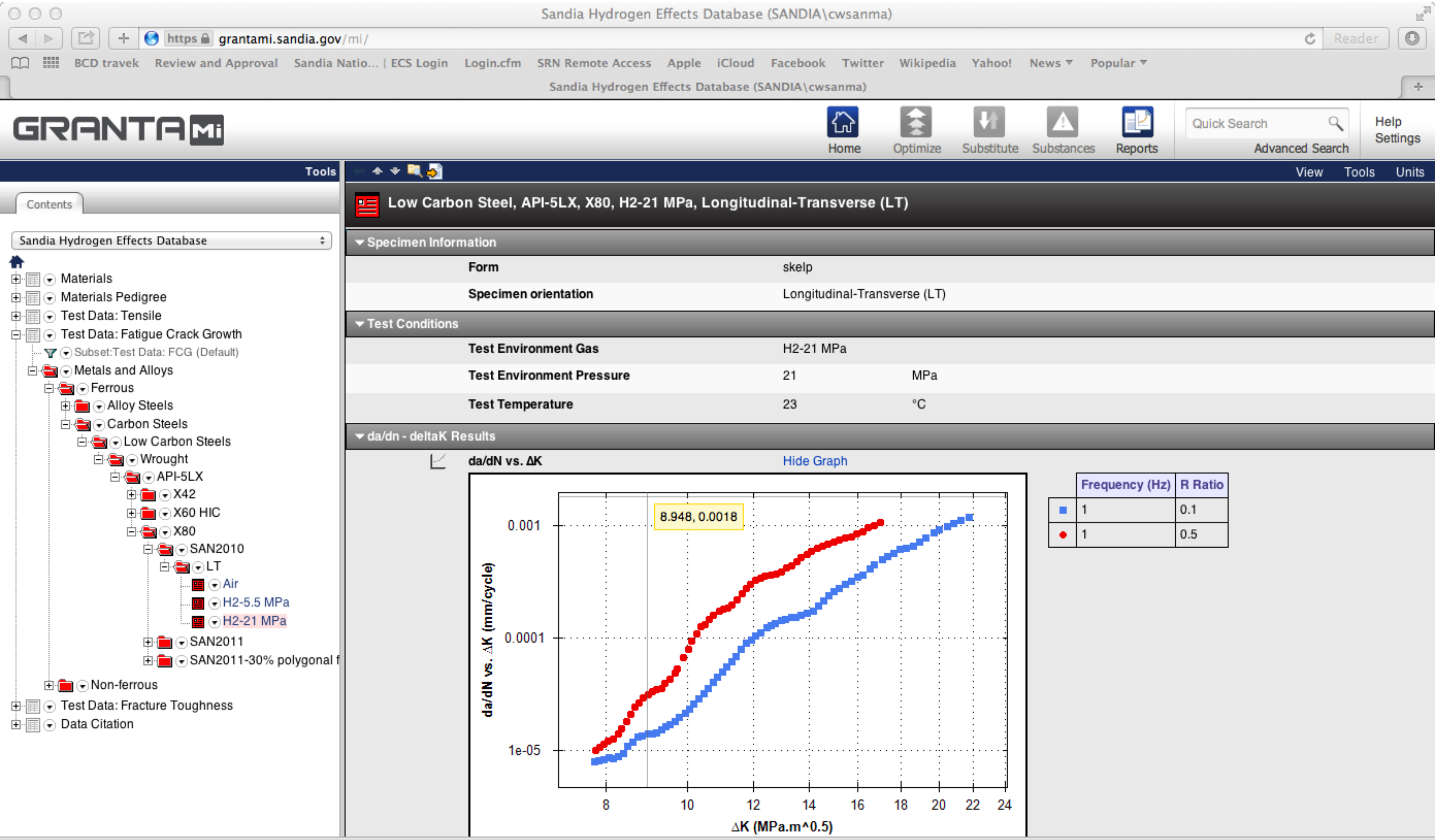
- Use state-of-the-art tools for data distribution
- Enable international comparison/harmonization

Accomplishment: database tools
Technical Database for Hydrogen Compatibility of Materials



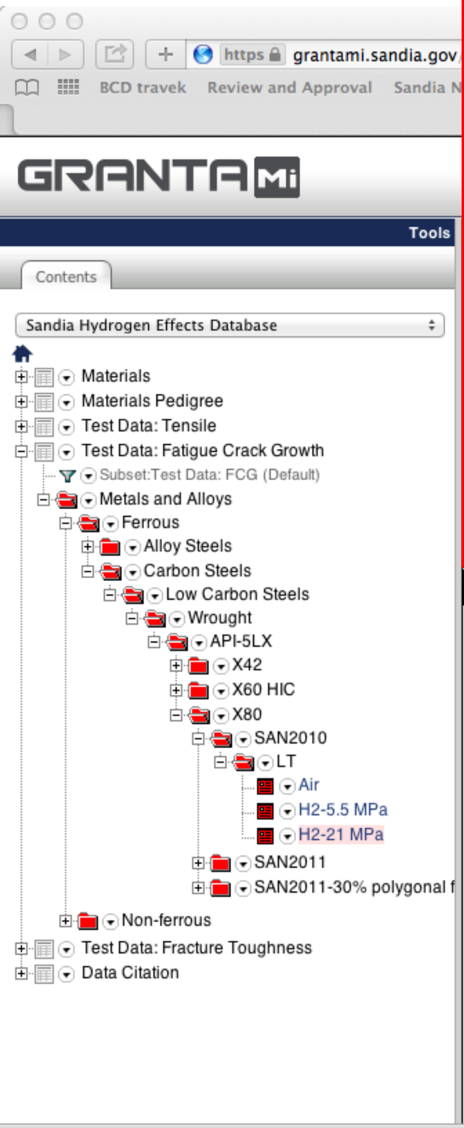
Accomplishment: database tools

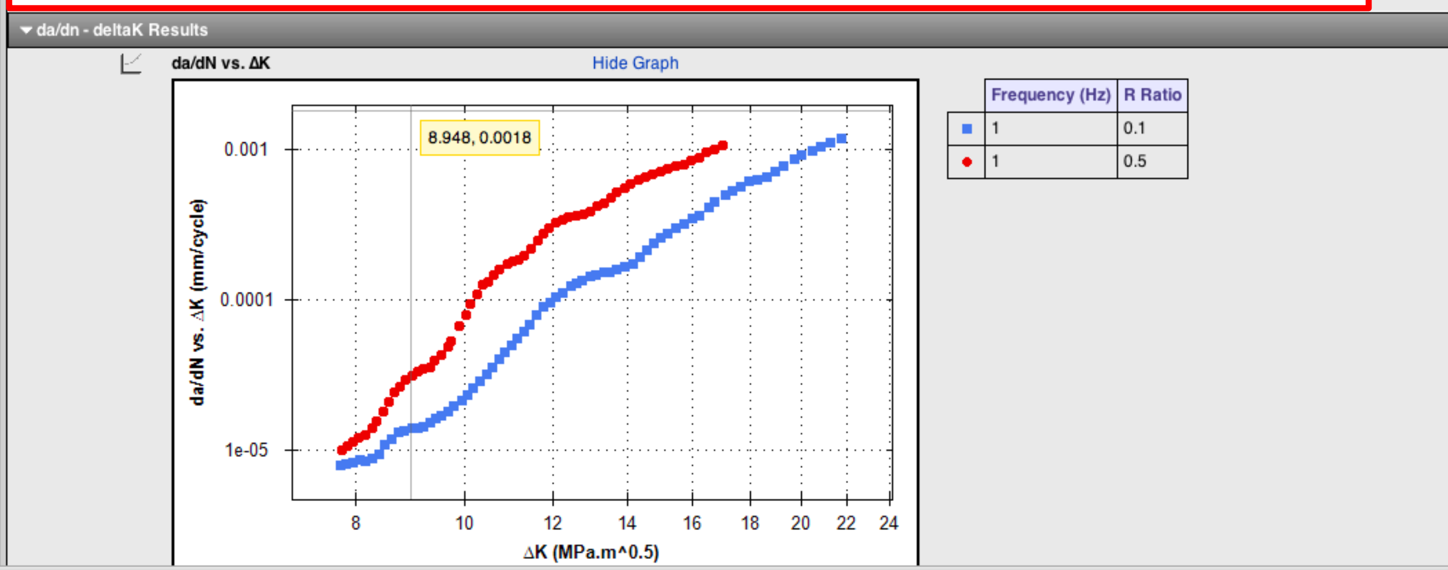
Technical Database for Hydrogen Compatibility of Materials



Accomplishment: database tools
Technical Database for Hydrogen Compatibility of Materials

- Granta is leader in data management tools
- Hydrogen effects database will be made available to Granta-users
- *Trial program*: web-based, public interface to all interested users
  - https://granta-mi.sandia.gov

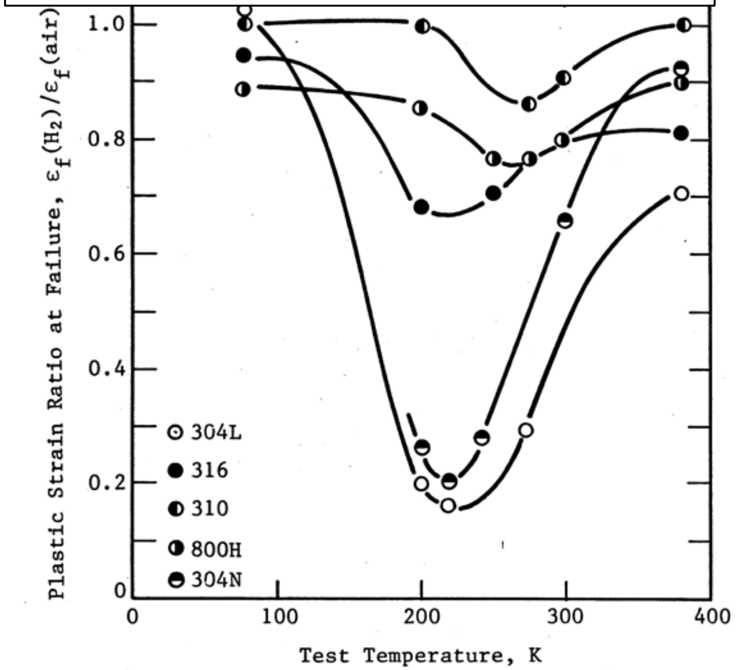




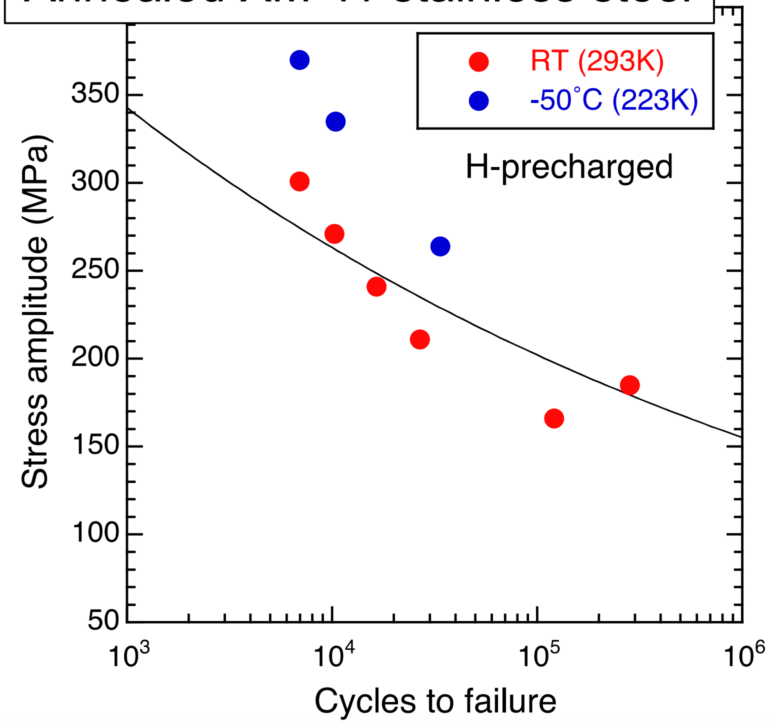
Approach: low-temperature testing

Develop testing capability and methods for understanding hydrogen effects at low temperature

Tensile ductility:
Various stainless steel



Fatigue life:
Annealed XM-11 stainless steel



Fatigue response not necessarily limited by performance at low temperature (unlike tensile ductility)

Collaborations/Future Work: low-temperature testing

Growing demands for low-temperature testing in high-pressure hydrogen

- BMW and Opel active in coordinating activity to provide data to industry
  - Coordination through SAE

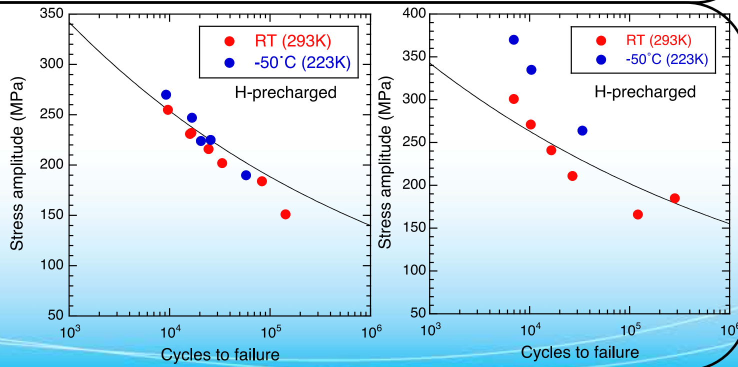
parameter	3. option: comparison with the new high pressure test equipment and proof of comparatibility of test results
test method	1st SSRT & 2nd Fatigue Life Test
Indicator for HEE	RRA
test numbers per medium	3-5 (Depending on Scattering)
material (*)	1.4435 (12,5-12,7% Ni) (***) TBC, PREFERENCE LOW NICKEL
heat treatment	solution annealed
material condition	uncharged
specimen manufactor	MPA or SANDIA
specimen diameter	4 mm
specimen surface condition	precision-turned
notch factor	smooth
temperature	-65°C (to be verified)
H2 pressure	700 bar
H2 quality	6N
strain rate	5,5e-5 /s (0,1 mm/min)

(*) using materials with significant HEE
(**) AVIF Var. 2 (internal description of MPA)
(***) HEE expected with this testing conditions and first new results with 700 bar H2 generated

Phase	Temperature (K)	Hydrogen Pressure (MPa)	Maximum fatigue stress (MPa)	Approximate maximum load (kN)	Number of specimens
1	293	10 MPa H2	Monotonic loading to failure		3
			500	6.3	4
			400	5.0	4
2	220	100 MPa H2	Monotonic loading to failure		3
			500	6.3	4
			400	5.0	4

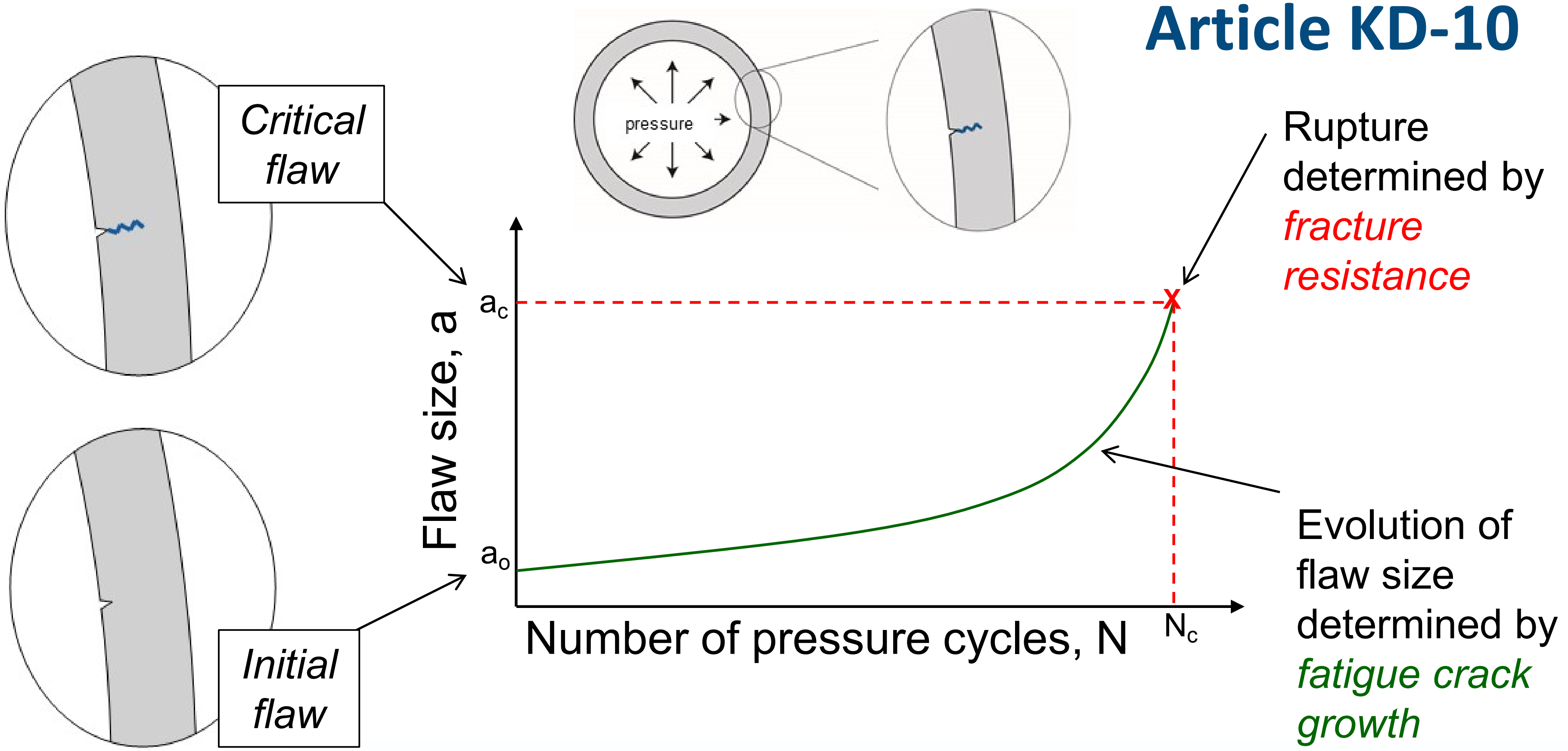
- IPHE round robin testing to establish fatigue testing methods
  - Draft test plan has been developed

- H2 Storage project on materials for BOP
  - Fatigue test matrix established with industry partners



Approach: advanced storage

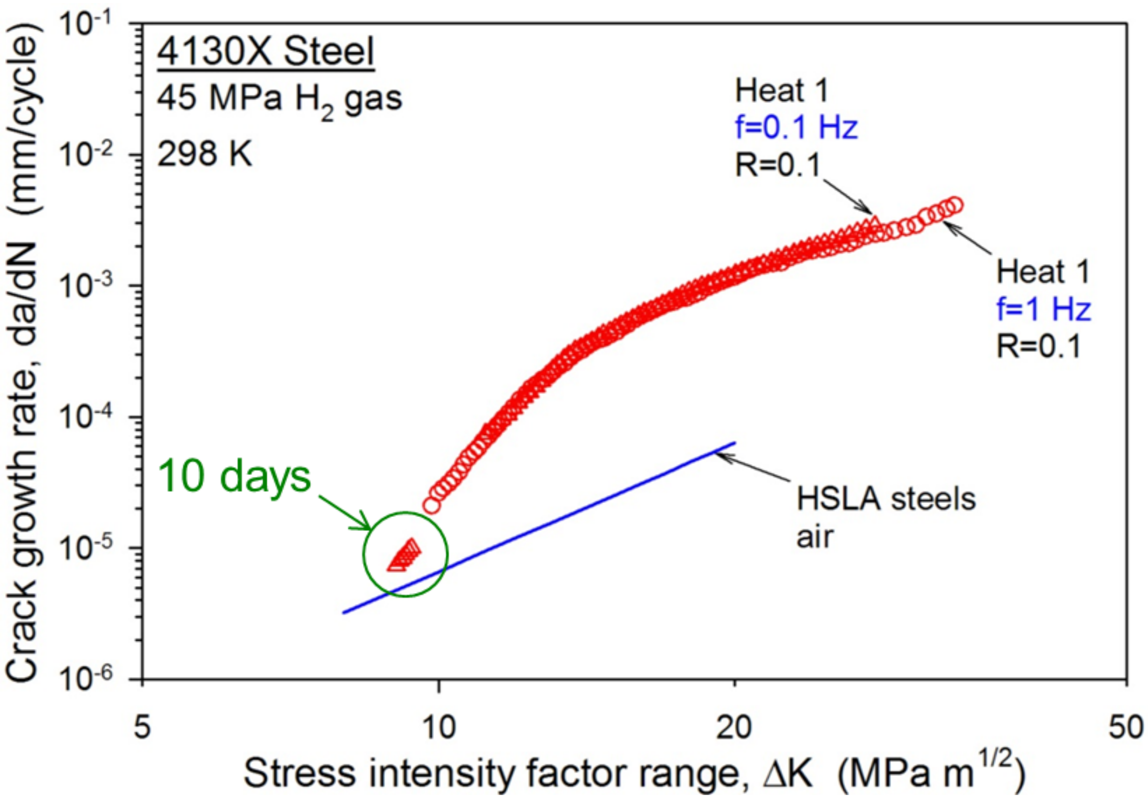
Use and improve existing standardized methods for fracture mechanics-based design: ASME BPVC VIII.3 Article KD-10

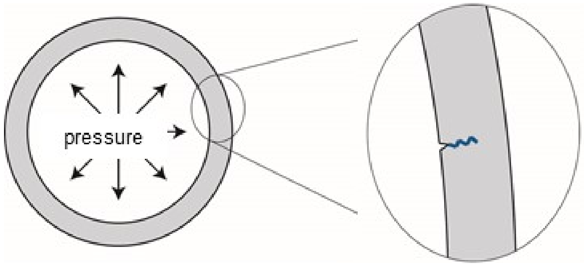


Industry needs and uses fracture mechanics to optimize pressure vessel designs for stationary storage

Approach: advanced storage

Life prediction depends on fatigue crack growth measurements





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

$$\frac{da}{dN} = C\,\Delta K^{m}$$

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

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

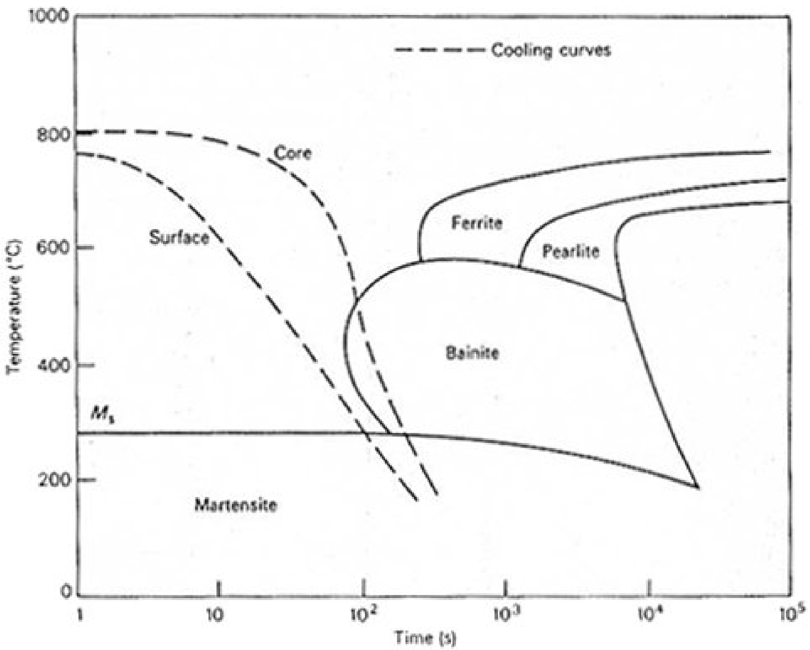
Approach: advanced storage

Partnership with industrial stakeholders enables evaluation of relevant parameter space

Category	Steel	S_y (MPa)	H_2 pressure (MPa)	Load ratio range	Designation
Cr-Mo pressure vessel steels	SA372 Gr. J	760	10 – 103	0.1 – 0.5	ASME
	34CrMo4	950	10 – 103	0.1	European
	4130X	540	45	0.1	US DOT
	SCM 435	640 – 1200	45	0.1	JIS (Japanese)
	Cr-Mo	500 – 1200	<110	0.1 – 0.7	No international consistency
Ni-Cr-Mo pressure vessel steels	SA372 Gr. L	731 & 1053	103	0.1	ASME
	SA723	690 – 1240	103	0.1 – 0.7	ASME
	SNCM 439	800 – 1200	45	0.1	JIS (Japanese)
	Ni-Cr-Mo	690 – 1240	<110	0.1 – 0.7	No international consistency

Accomplishment: advanced storage

Partnership established for evaluating high-hardenability pressure vessel steels



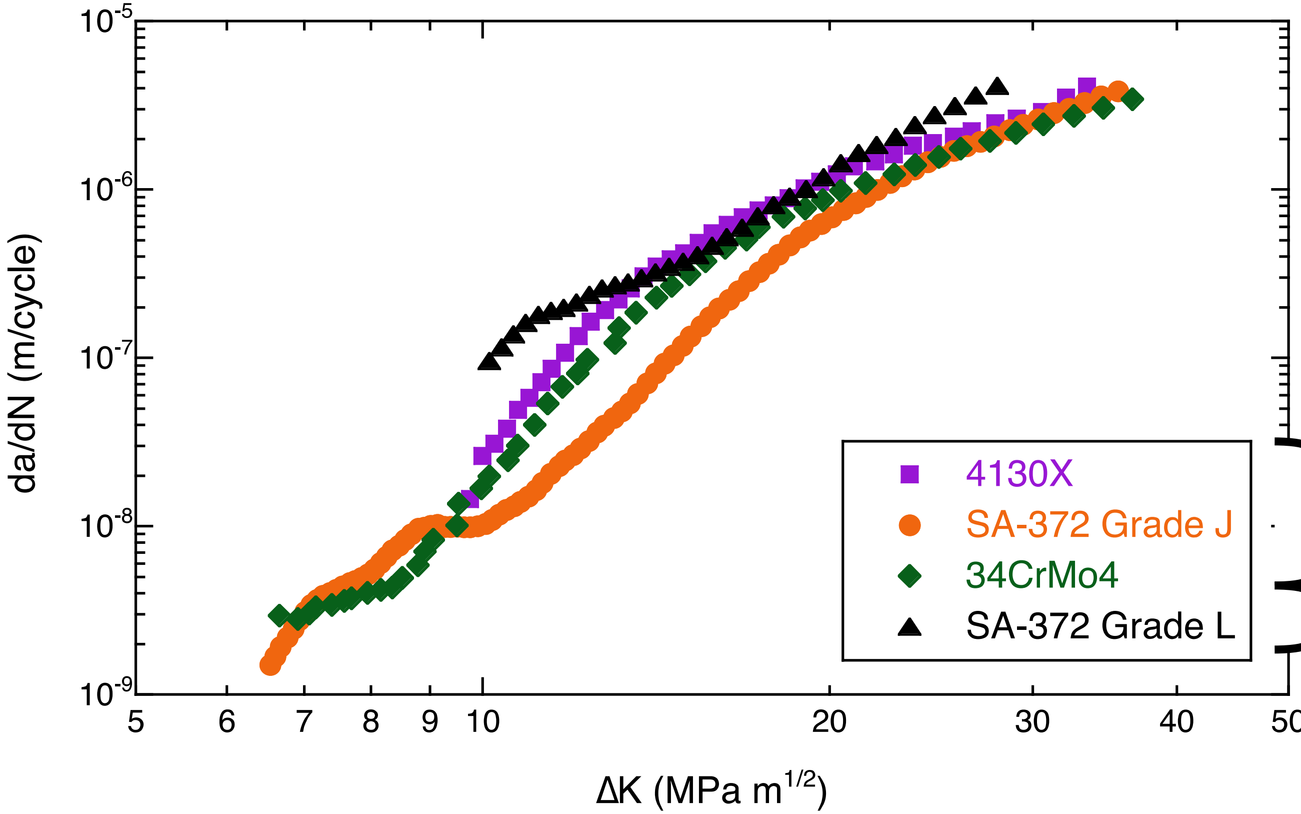
- Pressure vessel steels are quenched to achieve uniformity of desired properties through the wall thickness
- "Hardenability" of Cr-Mo steels limited to <38mm wall thickness

- 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 cooperative partnership established for evaluating Ni-Cr-Mo pressure vessels
  - Fiba Technologies (US)
  - Tenaris-Dalmine (Europe)
  - Japan Steel Works (Asia)

Objective: *evaluate fatigue crack growth and fracture thresholds of Ni-Cr-Mo pressure vessel steels*

Accomplishment: advanced storage

Initial results show consistency among pressure vessel steels



Fatigue crack growth in gaseous hydrogen

- $R = 0.1$
- $f = 1 – 10$ Hz
- $P = 45 – 103$ MPa

Cr-Mo steels

Ni-Cr-Mo

- Preliminary results show consistent fatigue crack growth at high ΔK
- Transition behavior associated with frequency and pressure differences

Accomplishment/Collaboration: advanced storage

High-pressure capabilities used to complement fatigue crack initiation methodology developed in Europe

H2 pressure (MPa)	waveform	Cycles for crack initiation	Test location
100	sinusodial	763	SNL
100	sinusodial	860	SNL
100	triangular	1017	SNL
30	triangular	2589	SNL
30	triangular	2764	MATHRYCE
10	triangular	7136	MATHRYCE
2	triangular	18292	MATHRYCE

MATHRYCE program (EC-sponsored) is developing crack initiation methodology to complement crack growth methods

- Crack initiation is sensitive to pressure

25CrMo4

- YS = 785 MPa
- $\Delta K \sim 19$ MPa $m^{1/2}$
- $R = 0.1$
- $f = 0.5$ Hz

Response to Previous Year Reviewers' Comments

- *FY15 Reviewer Comment:* "Weaknesses include the lack of coordination to turn pre-normative work into harmonized standards at the international level"
  - This project does not control international standardization activities. Every effort is made to generalize results so that they can be applied internationally.
  - Strong international collaborations exist and are exploited to establish international consensus (e.g., AIST coordination).
- *FY15 Reviewer Comment:* "The industry input is apparent, but perhaps more direct engagement is warranted."
  - The partnership to evaluate Ni-Cr-Mo pressure vessel steels includes industry participation from the US, Europe and Asia. All the partners have expressed willingness to provide steels for evaluation.
- *FY15 Reviewer Comment:* "The future work plans should align with the SAE Hydrogen Materials Round Robin… so that the plans for testing at SNL (and with IPHE) are aligned with the industry. There is a need to target and create an "open" materials database for automotive and stationary applications."
  - The goal of the project is to develop/evaluate test methods, not to generate materials design data for industry.
  - Fatigue life testing in coordination with SAE and IPHE will be the focus of activity with the new low-temperature, high-pressure testing apparatus. This activity (in collaboration with ST113) will also assess the value of fatigue testing at low temperature.
  - Database activities are a priority in FY16.

Collaborations

- Standards Development Organizations (SDOs)
  - Sandia technical staff participate on committees engaged in materials testing and selection for hydrogen service (e.g., CSA, SAE, ASME)
  - Low-temperature fatigue studies will inform existing methods from CSA and SAE
- Industry partners
  - Partners communicate materials testing gaps/needs and provide technology-relevant materials (FIBA Technologies, Tenaris-Dalmine, JSW, BMW, Opel, Swagelok)
  - Partnership for evaluation of Ni-Cr-Mo steels seeks international consensus to address need for advanced high-pressure storage
- International research institutions
  - Leverage specialized laboratories and expertise in international community to magnify impact of materials testing in high-pressure hydrogen gas (AIST, I^2CNER, MPA Stuttgart)
  - Fatigue testing at low temperature will be international focus in future

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
  - System design incorporates several unique innovations, thus timeline for full commissioning is uncertain
- Establish internationally harmonized fatigue life test methods
  - Europe and Asia embrace different test methods/parameter space
  - It may be a challenge to demonstrate data from these methods are self-consistent, despite different philosophy
- Formulate partnerships for effectively defining and performing high-impact R&D activities

Proposed Future Work

Remainder of FY16

- Expand Hydrogen Effects ***Database*** with focus on fatigue crack growth data
- Integrate sub-systems for high-pressure, ***low-temperature testing*** and demonstrate functionality
- Continue fatigue crack growth testing of high-hardenability steels (Ni-Cr-Mo pressure vessels steels) for ***advanced storage*** in partnership with international partners

FY17

- Develop long-term strategy for ***database*** distribution
- Determine the effect of composition on the appropriate ***test temperature*** for fatigue life testing of austenitic stainless steels in high-pressure hydrogen
  - Coordinate activity with international partners (e.g., SAE, IPHE)
- Quantify the role of strength and frequency on fatigue crack growth of high-hardenability steels in high-pressure gaseous hydrogen for ***advanced storage*** in collaboration with international partners

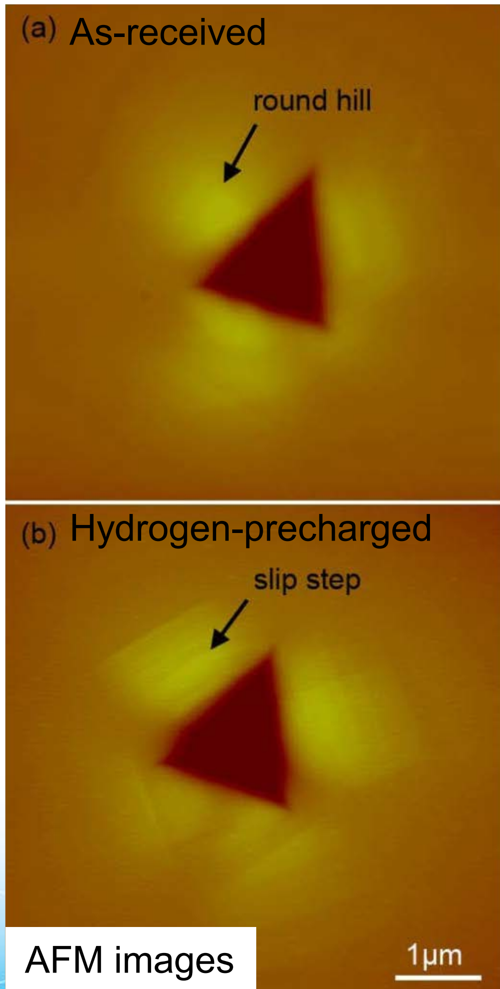
Summary

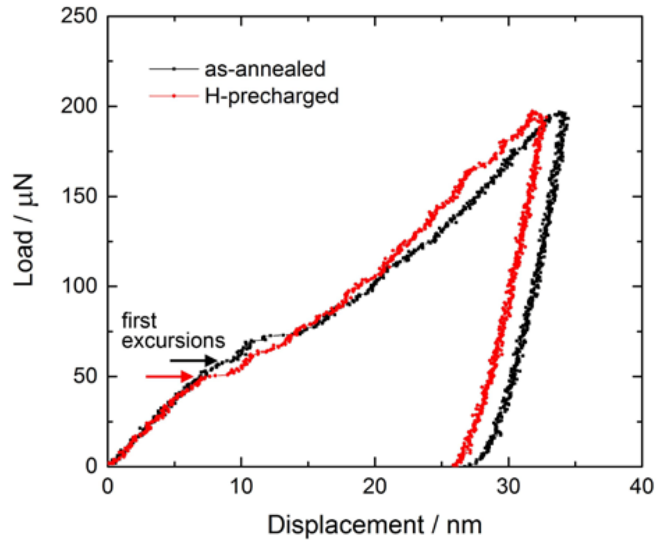
- Definitive ***database tools*** for materials selection
  - Formulating materials property database of hydrogen effects using state-of-the-art data management tools
  - Identifying metrics for materials selection
- Advancements in ***testing at low temperature*** in high pressure
  - Innovative new platform for testing
  - Deeper understanding of temperature effects on hydrogen-assisted fatigue to inform development of fatigue life test methodology
- Materials selection for ***advanced high-pressure hydrogen storage***
  - Harmonizing fatigue crack growth test method for pressure vessel steels
  - Evaluating suitability of high-hardenability steels for stationary storage with international partners
- Extensive international partnerships
  - Asia: AIST (Japan) , I^2CNER (Kyushu University, Japan), KRISS (Korea)
  - Europe: MATHRYCE (EC-supported project), MPA Stuttgart (Germany)
  - International Partnership for Hydrogen and Fuel Cells in the Economy (IPHE)

Technical Back-Up Slides

Accomplishment/Collaboration:

AIST-SNL collaboration on fundamental mechanisms of hydrogen embrittlment





Nanoindentation and atomic force microscopy (AFM) of SUS304 after hydrogen-precharging to evaluate effects of hydrogen on deformation character

- Deformation pile-up around nanoindent demonstrates changes in deformation mechanisms
- Load excursions during loading suggest that the onset of dislocation nucleation is enabled by hydrogen