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

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DOE Hydrogen and Fuel Cells Program Annual Merit Review

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Overview

Timeline

- Project start date: Oct. 2003
- Project end date: Sept. 2015*

  * Project continuation and direction determined by DOE annually.

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

- FY14 DOE funding: $800K
- Planned FY15 DOE funding: $800K
- Total DOE project value: $8M

Partners

- **SDO/CDO participation:** CSA, ASME, SAE, ISO
- **Industry:** FIBA Technologies, Tenaris-Dalmine, Japan Steel Works (JSW), BMW, Opel
- **Academia:** Boise State
- **International engagement:** AIST-Tsukuba (Japan), I2CNER (Kyushu University, Japan), MPA Stuttgart (Germany), MATHRYCE (EC project), IPHE, TWI (UK), 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 material property data gaps
F. Enabling national and international markets requires consistent RCS	Develop more efficient and reliable materials test methods in standards Design and safety qualification standards for components (SAE J2579, ASME Article KD-10) and materials testing standards (CSA CHMC1)
G. Insufficient technical data to revise standards	Execute materials testing to address *targeted* data gaps in standards and critical technology development

Relevance:

Materials Compatibility and Components project impacts multiple standards

- ASME Article KD-10
  - Standard on high-pressure hydrogen tanks for transport and storage
  - Sandia and partners provide data on exercising and improving materials test methods
  - Reporting progress on optimizing fatigue crack growth testing and developing fracture threshold test method through ASME publications
- SAE J2579
  - Hydrogen vehicle fuel system standard
  - Sandia serves as U.S. technical lead on addressing hydrogen embrittlement, attends quarterly meetings of FC Safety Task Force
- CSA CHMC1
  - Materials testing and data application standard
  - Sandia provides leadership in technical committee and document preparation
  - Working on evaluation of methodology

Program Approach

The Safety, Codes and Standards program coordinates critical stakeholders and research to remove technology deployment barriers

Partnerships with industry, labs, academia

Identify R&D needs → *Perform High-Priority R&D* → *Impact Codes and Standards*

NFPA · CSA · SAE · ISO · UL · ASME SETTING THE STANDARD · ICC INTERNATIONAL CODE COUNCIL

Harmonize Internationally

Regulations, Codes and Standards [RCS]

International Standards (ISO)

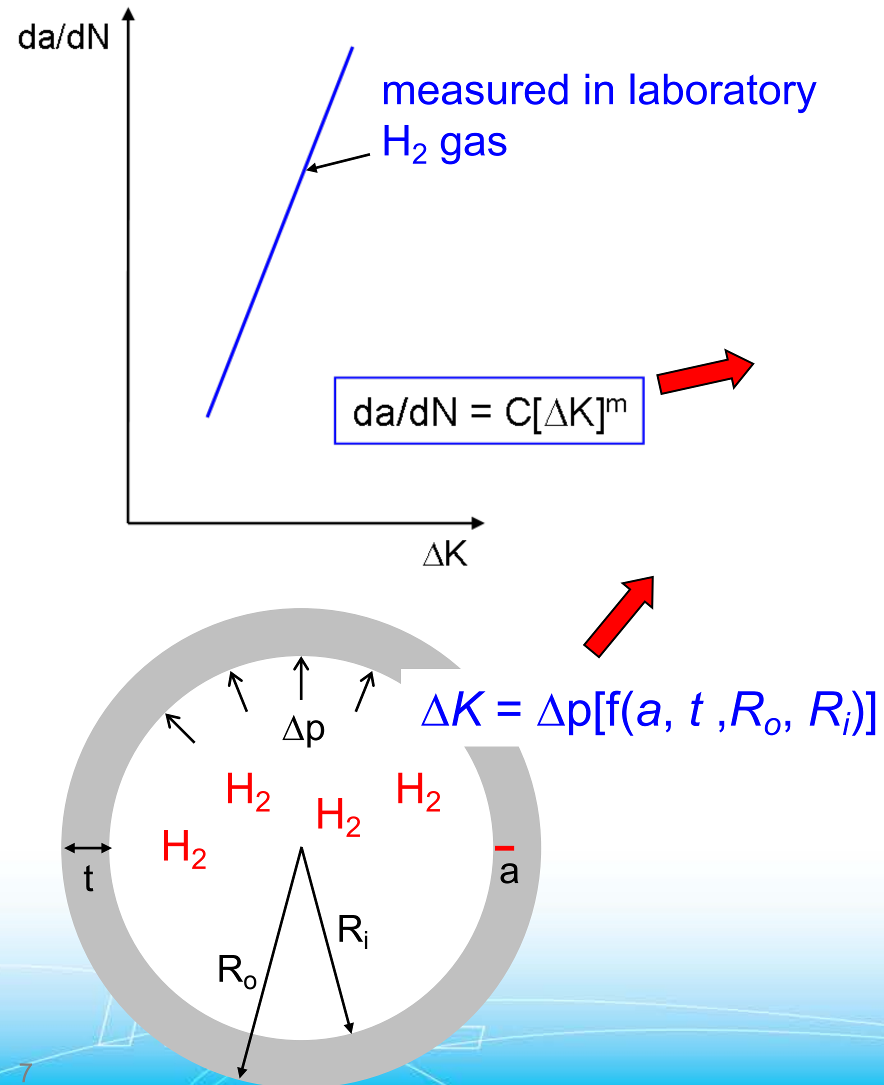
International Agreements (IEA, IPHE)

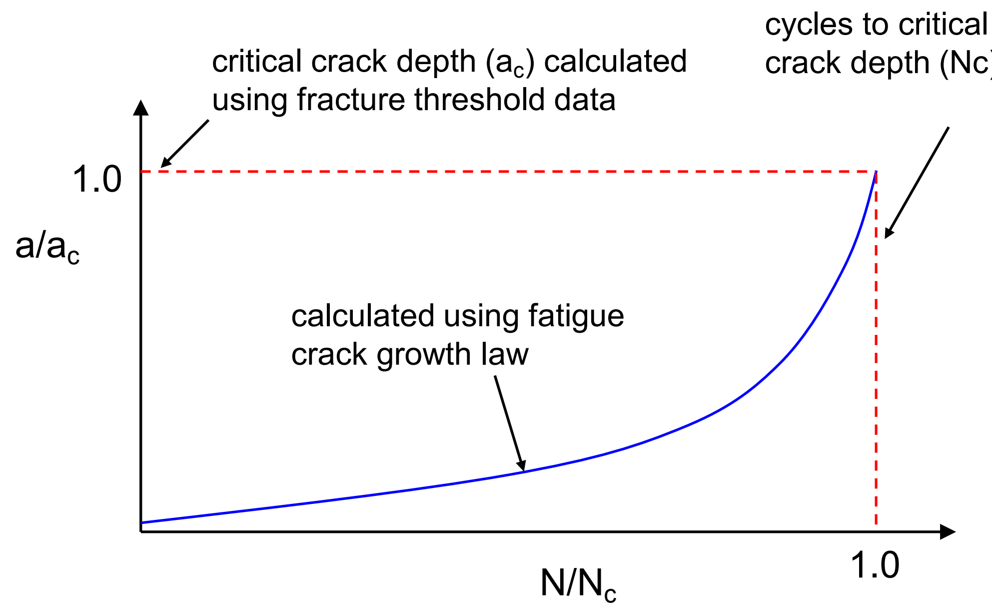
Project Approach and Milestones

MYRD&D 2013 Barrier	FY15 Milestone	Status
A. Safety Data and Information: Limited Access and Availability	Develop material property database	Working with MDMC and Granta to build schema for hydrogen effects in materials database in Granta MI
F. Enabling national and international markets requires consistent RCS	Lead development of international consortium to evaluate suitability of high-hardenability steels for stationary high-pressure H_2 vessels	Preliminary consortium consists of 4 partners: SNL, FIBA Technologies, Tenaris-Dalmine, and Japan Steel Works (JSW)
G. Insufficient technical data to revise standards	Document H_2-assisted fatigue crack growth measurements for Cr-Mo steels	ASME PVP paper complete, yielded insights for additional measurements to optimize fatigue crack growth testing
	Complete fracture threshold testing of Cr-Mo steels in high-pressure H_2 gas in collaboration with AIST	Testing completed, results documented in ASME PVP paper

Approach:

Deployment of H_2 fueling stations focusing attention on components such as stationary storage vessels

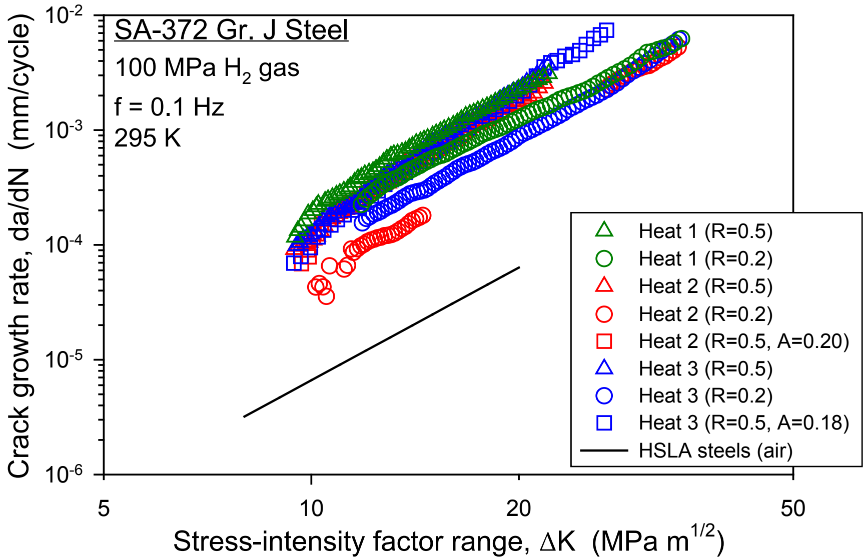


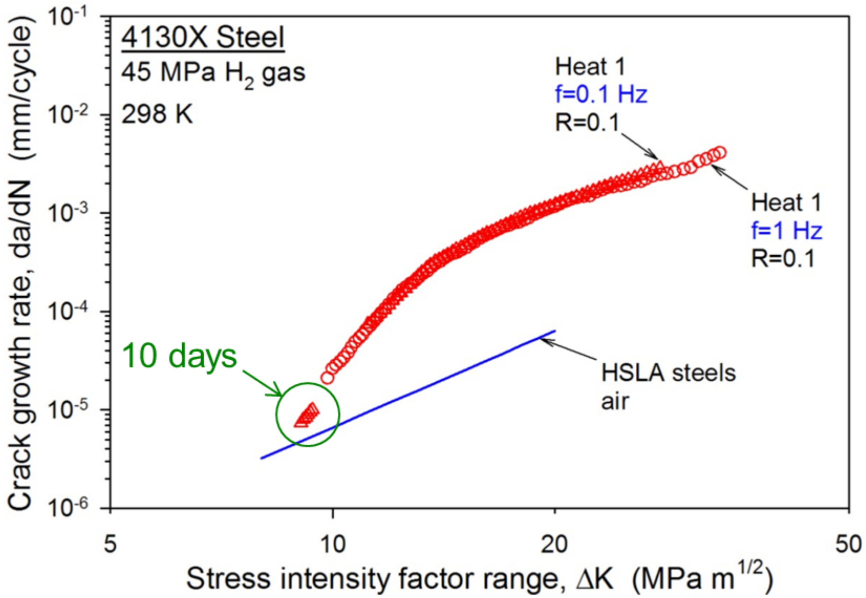


- Industry partners determining fatigue life of vessels with fracture mechanics approach (e.g., ASME Article KD-10)
- Two fracture properties in H_2 needed
  - Fatigue crack growth law
  - Fracture threshold
- Fatigue life analysis framework accommodates H_2 embrittlement

Previous Accomplishment:
Qualification of SA-372 Gr. J steel for FIBA Tech H_2 storage vessel revealed non-optimum test procedures

B. Somerday, C. San Marchi, K. Nibur, *ASME PVP*, 2013





- Load-cycle frequency specified for fatigue crack growth rate testing in ASME KD-10 (0.1 Hz) can lead to impractical test durations
- Curtailing test duration by initiating tests at higher ΔK compromises value of fatigue crack growth rate data
- ***Goal: explore modified procedure that shortens test duration while maintaining data value***

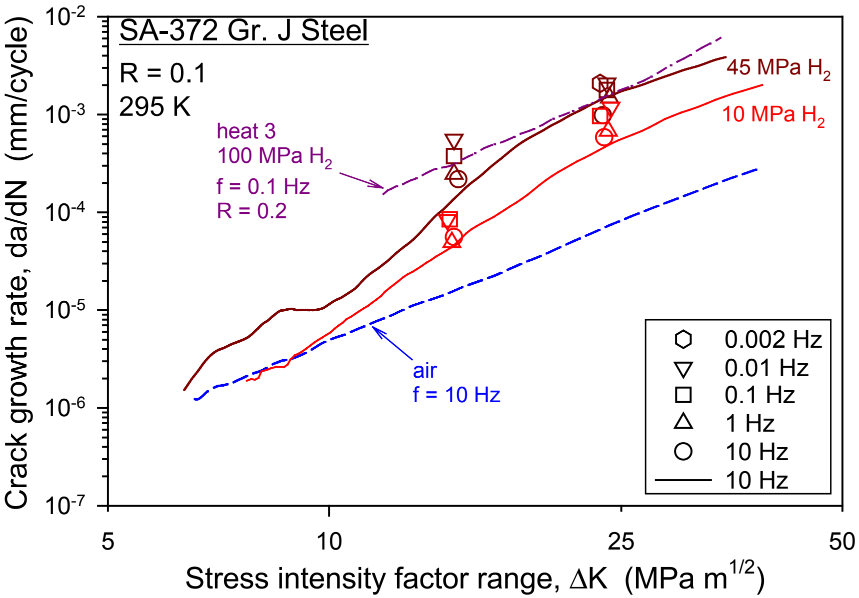
Approach:
Research activity aimed at modifying test procedure in ASME KD-10 focused on two commercial steels

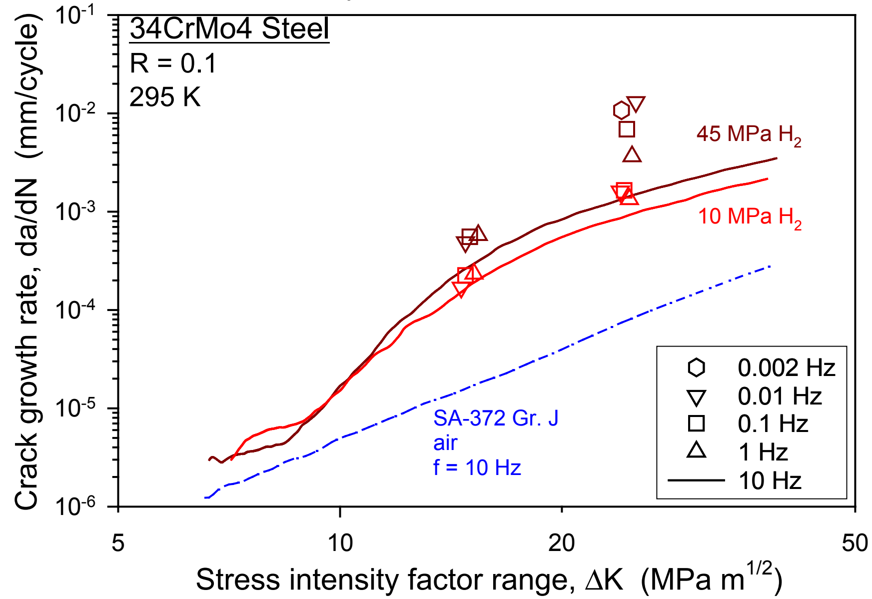
- Steels provided by two partners: FIBA Technologies and Tenaris-Dalmine

Steel	S_y (MPa)	H_2 pressure (MPa)	Test frequency (Hz)	Load ratio	Status
SA372 Gr. J	760	10	10	0.1	Complete
		10	variable	0.1	Need low ΔK data
		45	10	0.1	Complete
		45	variable	0.1	Low ΔK completed
		100	10	0.1	Pending
		100	variable	0.1	Need low ΔK data
34CrMo4	950	10	10	0.1	Complete
		10	variable	0.1	Need low ΔK data
		45	10	0.1	Complete
		45	variable	0.1	Low ΔK completed
		100	10	0.1	Complete
		100	variable	0.1	Withdrawn

Accomplishment:
Possible modified approach: measure da/dN vs. ΔK at 10 Hz, correct data based on da/dN vs. frequency

B. Somerday, P. Bortot, J. Felbaum, *ASME PVP*, 2015

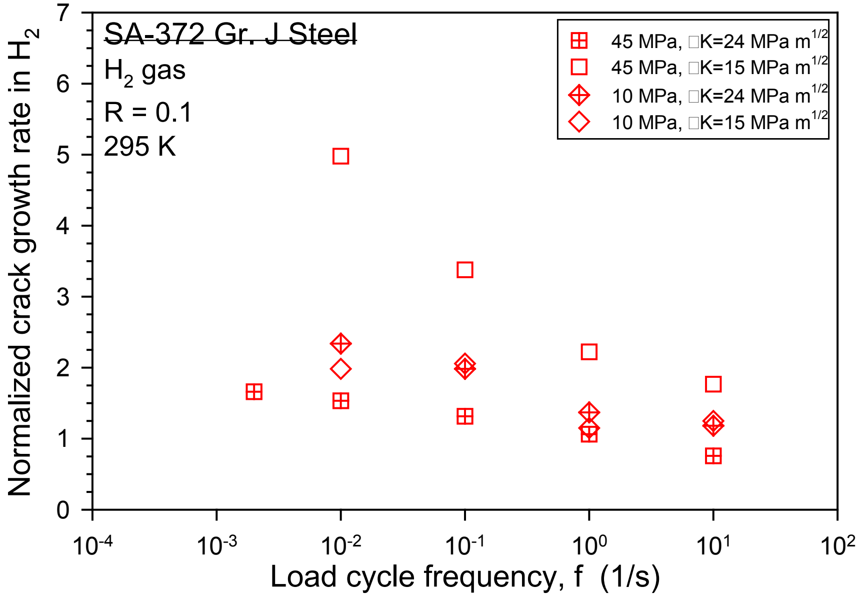


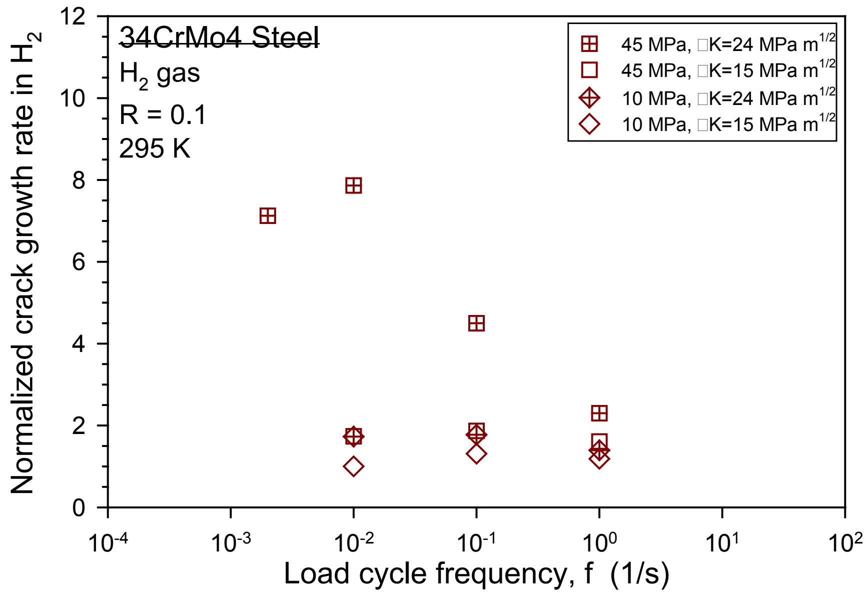


- Reliability of approach depends on adequacy of crack growth rate (da/dN) vs. frequency data
  - Data must include upper-bound crack growth rates
  - Data must be measured at appropriate constant-ΔK levels
- ***Needs:***
  - ***Confirm upper-bound da/dN included in da/dN vs. frequency data sets***
  - ***Measure da/dN vs. frequency at lower ΔK***

Accomplishment:
Assessing attainment of upper-bound crack growth rates facilitated by "normalized" da/dN vs. f plots

B. Somerday, P. Bortot, J. Felbaum, *ASME PVP*, 2015



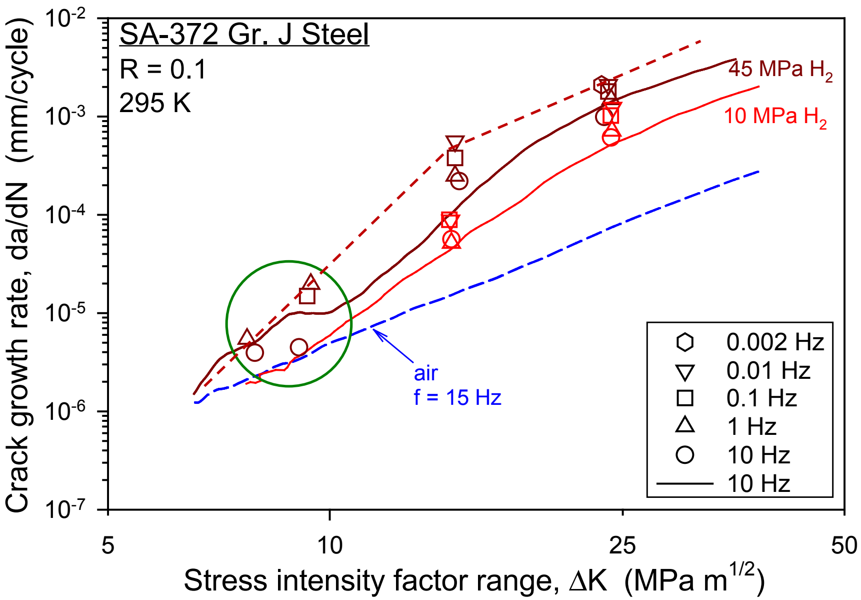


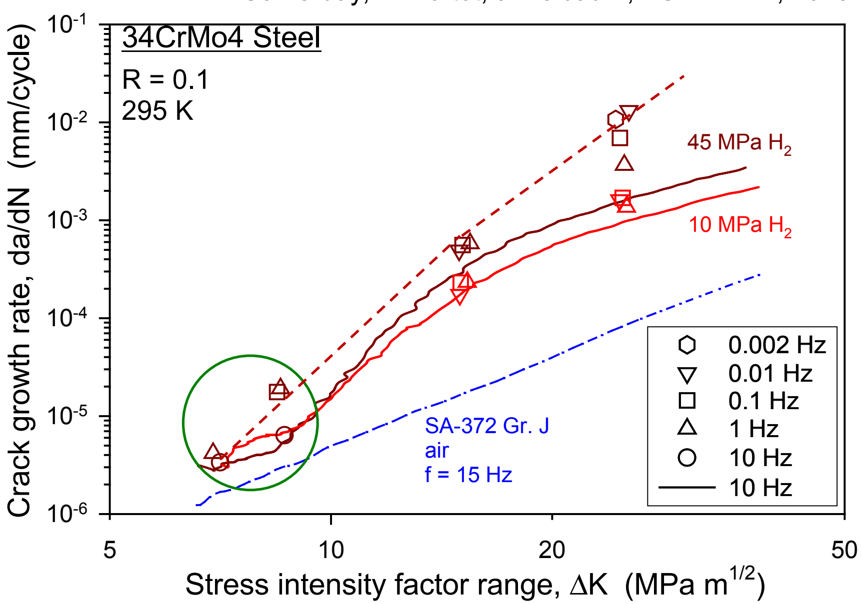
- Normalized crack growth rate: ratio of da/dN measured in constant ΔK test and da/dN measured at 10 Hz (from baseline da/dN vs. ΔK plot)
  - Accounts for modestly varying ΔK during da/dN vs. frequency measurements at nominally constant ΔK
- Normalized da/dN vs. frequency plots confirm that crack growth rates approach upper-bound values as frequency decreases for most conditions
  - Exception: SA-372 Gr. J steel at ΔK = 15 MPa $m^{1/2}$ and 45 MPa H_2 pressure

Accomplishment:

Additional da/dN vs. frequency measurements at lower ΔK clarify onset of H_2-accelerated crack growth

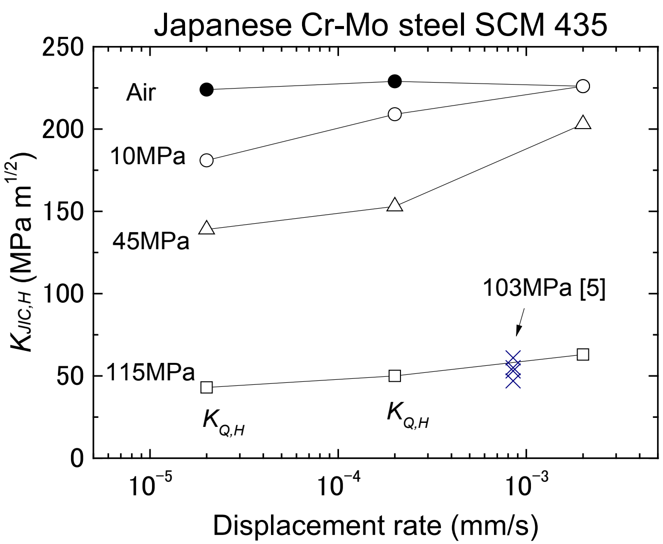
B. Somerday, P. Bortot, J. Felbaum, *ASME PVP*, 2015

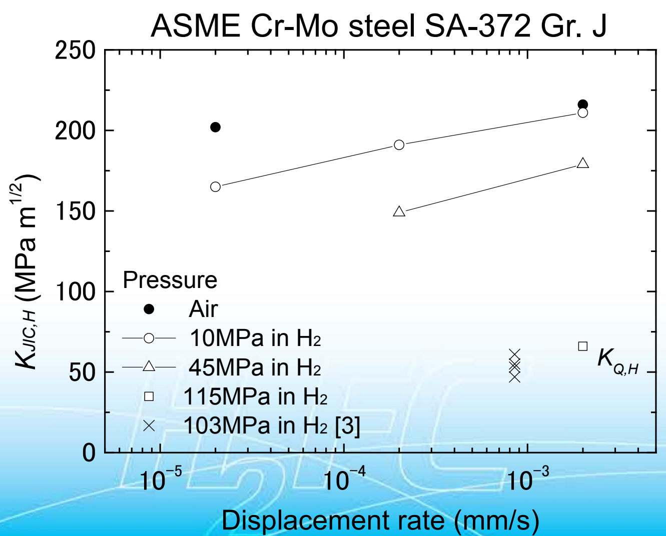




- Crack growth rate (da/dN) vs. frequency data at lower ΔK indicate that da/dN measurements at 10 Hz may not represent upper-bound values
  - Onset of H_2-accelerated crack growth not reliably represented by baseline da/dN vs. ΔK relationship measured at 10 Hz
- Reliable correction to da/dN vs. ΔK relationship measured at 10 Hz must include da/dN vs. frequency data sets measured at lower ΔK

Accomplishment/Collaboration:
AIST-SNL qualifying test methods for measuring fracture thresholds of pressure vessel steels in H_2 gas

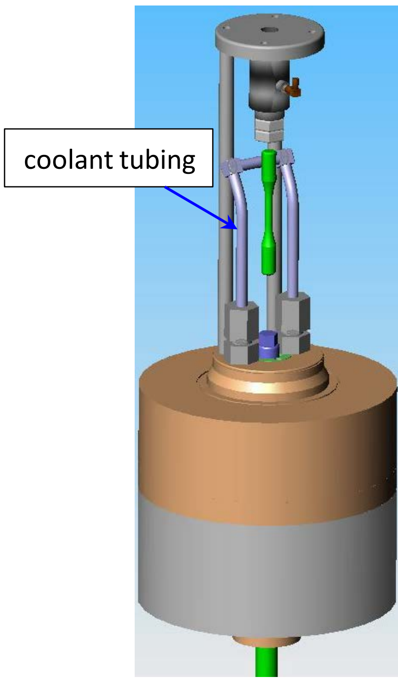




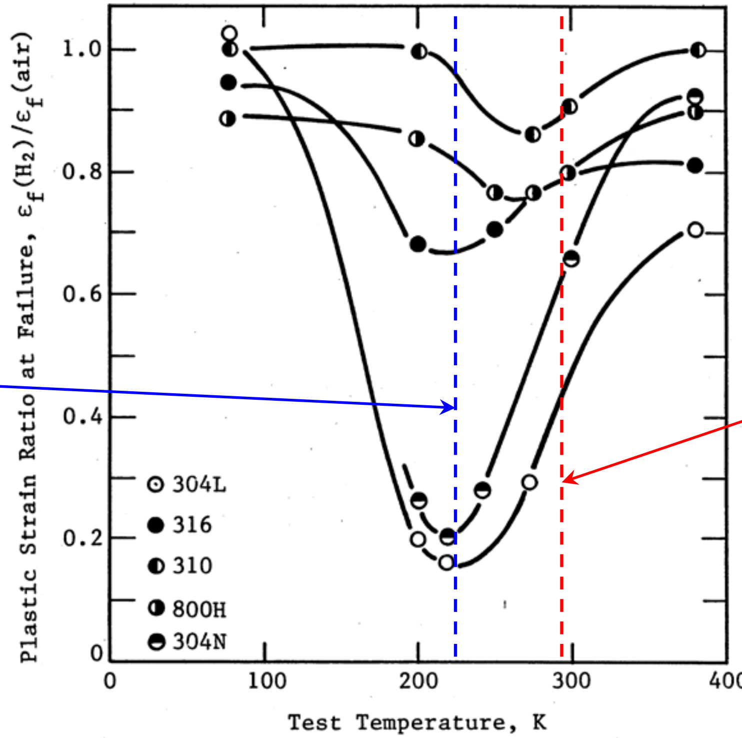
- Collaboration resulted in most comprehensive “rising displacement” fracture threshold measurements in high-pressure hydrogen gas
  - Thresholds measured as function of H_2 pressure and displacement rate for two Cr-Mo steels
- Results contribute to technical basis for developing “rising displacement” threshold testing standard
  - AIST presented results at ASTM subcommittee meeting (E08.06.02 Environmentally Assisted Cracking) in Nov. 2014
- Results documented in AIST-SNL joint publications (ASME PVP-2015 and ASME PVP-2014)

Accomplishment:
Final sub-system in low-temperature testing capability specified: pressure vessel with internal cooling

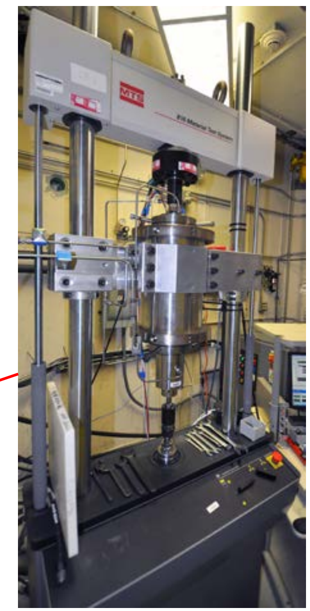
Low-temperature pressure vessel internal structure

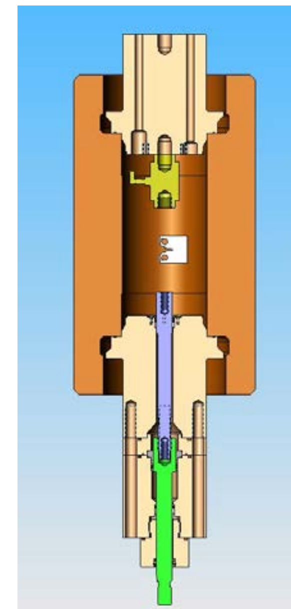


Ductility of stainless steels in H_2 gas normalized by ductility in air



Current capability for fatigue testing in high-pressure H_2: room temperature only





- New capability essential since hydrogen embrittlement of technology-critical stainless steels maximum at sub-ambient temperature
- SNL working collaboratively with vendor to finalize design and engineering drawings

Collaboration/Future Work:

Immediate demands for low-temperature testing in high-pressure hydrogen capability

- BMW and Opel coordinating activity to provide fatigue data to industry
  - MPA Stuttgart visited SNL (March 2015) to discuss low-temperature capabilities and potential round-robin testing of stainless steels at low temperature

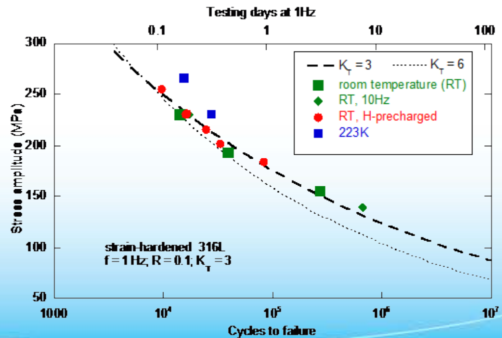
parameter	*3. option:* comparison with the new high pressure test equipment and proof of comparatibility of test results
test method	1st SSRT & 2nd Fatigue Life Test
Indicator for HEE	RRA
test numbers per medium	3-5 (Depending on Scattering)
material (*)	1.4435 (12,5-12,7% Ni) (***) TBC, PREFERENCE LOW 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
  - Next steps: comment from IPHE members

- H2 Storage project on materials for BOP
  - Fatigue test matrix established with industry partners
  - Next step: low-T tests in low-pressure H_2 at Hy-Performance Materials Testing



Response to Previous Year Reviewers' Comments

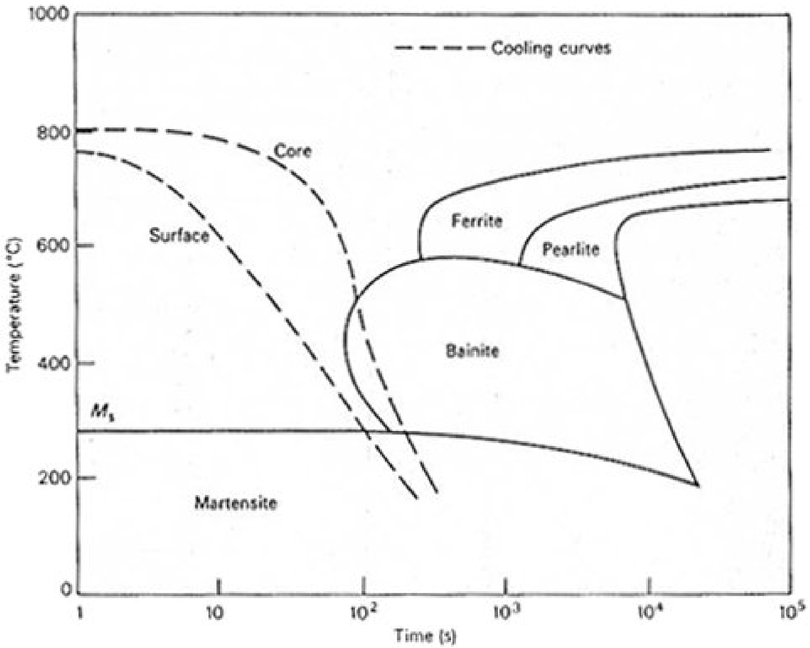
- *FY14 Reviewer Comment: "…relatively little progress…made in the execution of mechanical tests. This impression is strengthened by the facts that: (1) no information is provided on…activities identified as proposed further work in the 2013 AMR, and (2) the fiscal year (FY) 2013 funding is double that expected in 2012…"*
  - The project budget is not exclusively dedicated to mechanical testing. In addition to mechanical testing, expectations for this project include providing leadership in codes and standards committees, partnership development, documenting and communicating research results, and development of specialized laboratory capabilities, such as low-temperature testing in high-pressure hydrogen gas.
  - Mechanical testing in high-pressure hydrogen gas, particularly fatigue testing, is not routine and requires significant investments in labor and time. For example, the crack growth rate vs. frequency measurements at lower ΔK on slide 12 required over 1 month to complete.
  - This project is guided by longer-term objectives and planning but tries to be responsive to high-priority needs. For example, this project pivoted back to fatigue crack growth testing of pressure vessel steels in hydrogen gas based on interactions with industry partners after the 2014 AMR.
- *FY14 Reviewer Comment: "Reaching out to specialized non-US testing houses is recommended to increase density of experimental data and to include materials extensively used for hydrogen applications in other parts of the world"; "…the project needs to develop further international relationships."*
  - The collaboration with AIST (slide 13) is an excellent example of leveraging capabilities in non-US testing facilities to increase the density of experimental data and include materials (e.g., SCM 435) from other regions.
  - The project placed particular emphasis on developing additional international partnerships, e.g., MPA Stuttgart and IPHE.

Collaborations

- Standards Development Organizations (SDOs)
  - Sandia technical staff lead and serve on committees (CSA, SAE, ASME)
- Industry partners
  - Partners communicate materials testing gaps/needs and provide technology-relevant materials (FIBA Technologies, Tenaris-Dalmine, JSW, BMW, Opel)
- Academia
  - Student intern led prototyping and analysis for internally cooled pressure vessel in low-temperature testing in H_2 gas capability (Boise State)
- 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)

Multiple industry stakeholders advocate evaluating high-hardenability steels for H_2 vessels

High hardenability steel concept:



- Need to quench both surface and core of steel vessel wall to 100% martensite
- Phase transformation (e.g., bainite) start extended to longer times for "high hardenability" steels
  - Ni-Cr-Mo steels higher hardenability than Cr-Mo steels

- Hardenability of current Cr-Mo steels in H_2 pressure vessels (e.g., SA-372 Gr. J) limits wall thickness to ~1.5 in (38 mm)
- Higher-hardenability steels favorable for high-pressure H_2 vessels
  - Reduce heat treatment variability in thick-walled steel vessels
  - Reliably increased wall thickness allows design of steel vessels with increased inner diameter (greater volume or reduced length)
- Assembling international consortium of industry stakeholders to characterize H_2 compatibility of high-hardenability steels
  - Preliminary consortium partners: FIBA Technologies, Tenaris-Dalmine, JSW
  - Fatigue crack growth and fracture threshold testing of high-hardenability steels in Hydrogen Effects on Materials Lab at SNL

Remaining Challenges and Barriers

- Demonstrate low-temperature testing in H_2 gas capability that functions according to specifications
- Formulate partnerships for effectively defining and performing high-impact R&D activities

Proposed Future Work

Remainder of FY15

- Complete fatigue crack growth rate (da/dN) vs. frequency measurements for SA-372 Gr. J and 34CrMo4 steels at lower ΔK levels
- In collaboration with EC-supported MATHRYCE project, perform fatigue testing on notched specimen of Cr-Mo steel in high-pressure H_2 gas to evaluate methods for assessing the fatigue life of pressure vessels
- Work with vendor to complete manufacturing of pressure vessel for low-temperature testing in H_2 gas capability

FY16

- Evaluate suitability of high-hardenability steels for stationary high-pressure hydrogen vessels in partnership with industry stakeholder consortium
- Integrate sub-systems in low-temperature testing in H_2 gas capability and demonstrate functionality
- 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 H_2 gas
- Identify needs of community for more comprehensive use for Technical Reference (TR); for example, add stress-based fatigue data to TR

Technology Transfer Activities

- Evaluating hydrogen compatibility of high-hardenability steels in consortium with industry stakeholder foundation enhances probability of technology impact from applied R&D

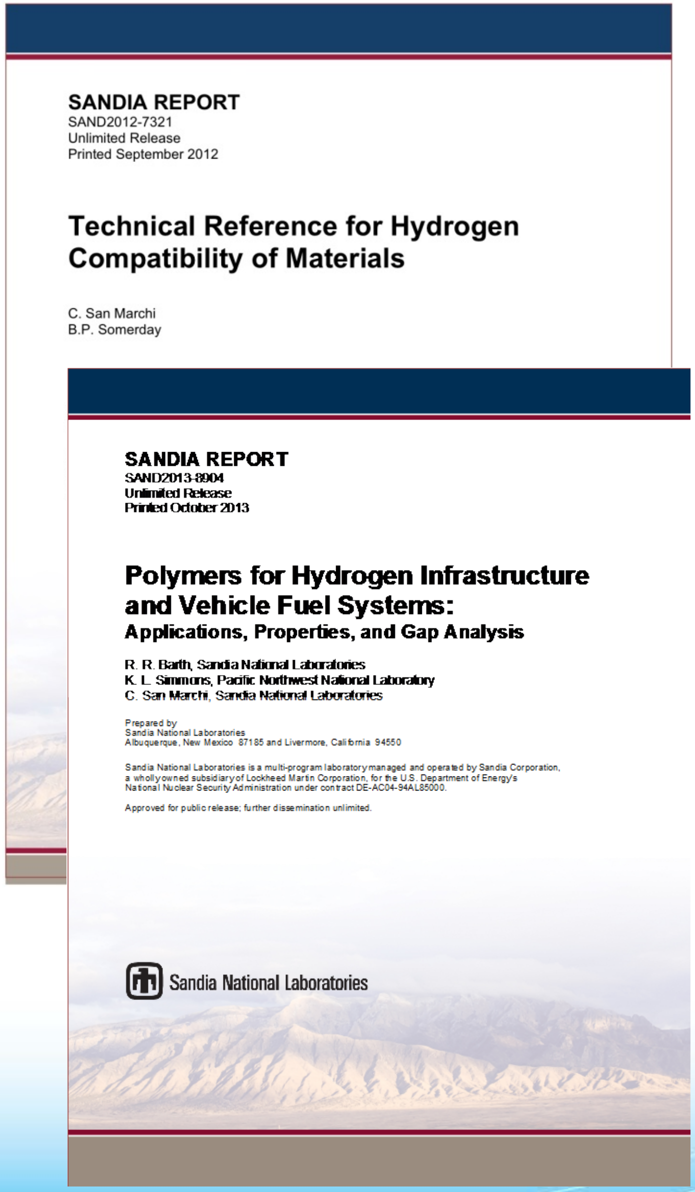
Summary

- Materials testing motivated by standards development and technology needs
  - Optimizing fatigue crack growth test method for pressure vessel steels in H_2 gas
  - Evaluating fracture threshold test method for pressure vessel steels in H_2 gas
  - Leading international consortium to evaluate suitability of high-hardenability steels for stationary high-pressure hydrogen vessels
- Providing leadership in materials testing
  - Completing new low-temperature testing in H_2 gas capability
  - Developing international partnerships to define test methods and augment database for stainless steels in high-pressure H_2 gas
- Contributing to technical basis for modifying or developing standards
  - Fatigue crack growth measurements for SA-372 Gr. J and 34CrMo4 steels in H_2 gas may impact test method in ASME Article KD-10
  - Results from fracture threshold testing of SA-372 Gr. J and SCM 435 steels in H_2 gas may inform ASTM standard
- Sustaining and initiating international partnerships
  - AIST (Tsukuba, Japan)
  - I^2CNER (Kyushu University, Japan)
  - MATHRYCE (EC-supported project)
  - MPA Stuttgart (Germany)
  - IPHE

Technical Back-Up Slides

Accomplishment:
Update information resources

- Developing informational hydrogen resource pages to augment Technical Reference website
  - includes resources for other hydrogen programs at Sandia as well
- Working with the Materials Data Management Consortium (MDMC) and Granta to build schema for hydrogen effects in materials database in Granta MI



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Polymers for Hydrogen Infrastructure and Vehicle Fuel Systems:
Applications, Properties, and Gap Analysis

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Collaborations:

SNL and I^{2}CNER leverage applied and basic research for common goal

Friction and Wear


Sugimura


Sawae


Tanaka


Somerday (Lead PI)


Kirchheim

Materials Processing


Takaki


Tsuchiyama


Nakada


Macadre

Optimize cost, performance, and safety of H_2 components

- **Predictive models based on physics of gas-surface interactions, H migration, and material degradation**
- **Advanced methods for characterizing hydrogen-induced degradation in materials**

- **Next-generation H_2 compatible materials having lower cost and higher strength levels**

Fatigue and Fracture


Sofronis


Robertson


Kubota


Matsunaga


Yamabe


Ritchie


Xu


Nagao


Aravas