Project ID # SCS005

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

Sandia National Laboratories

Daniel Dedrick
Hydrogen Program Manager

Brian Somerday
(Presenting)
Principle Investigator

Chris San Marchi, Aaron Harris

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Overview

Timeline

- Project start date: Oct. 2003
- Project end date: Sept. 2015
- Percent complete: 80%

Budget

- Total project funding
  - DOE share: $6.0M
- Funding in FY11: $0.4 M
- Planned Funding for FY12: $0.8 M

Barriers (2012 MYRD&D)

- 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/CDOs: SAE, CSA, ASME, ISO

Industry: FIBA Technologies, Swagelok®, Plug Power, Nuvera FC, Norris Cylinder

Govt: DOE Pipeline Working Group

International: HYDROGENIUS, I^2CNER (Kyushu University, Japan), AIST

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

Harmonize Internationally

Regulations, Codes and Standards (RCS, GTR)
International Standards (eg. ISO)
International Agreements (IEA, IPHE)

- Metrics for Success
  - Number of codes, standards, regulations impacted
  - Degree of harmonization
  - Number of systems qualified based on developed standards

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Objectives/Relevance

- Enable technology deployment by providing science-based resources for standards and H_2 component development
  - Update materials reference guide (“Technical Reference”) and understanding of material property data gaps
  - Execute materials testing to address *targeted* data gaps in standards and critical technology development
    - FY12 examples: measure properties of H_2-exposed welds and Al alloys
  - Develop more efficient and reliable materials test methods in standards
    - FY12 example: optimize fatigue crack growth testing in ASME Article KD-10 tank standard

- Participate directly in formulating standards
  - Design and safety qualification standards for components
    - SAE J2579, CSA HPIT1, ASME Article KD-10
  - Materials testing standards
    - CSA CHMC1

Program Approach

- Apply specialized expertise and capabilities in H_2-materials interactions to enable standards and technology development
  - Sustain relationships with stakeholders (industry, SDOs) to ensure materials testing and standards participation is properly focused
  - Apply unique laboratory capabilities for conducting materials testing in hydrogen gas up to 100 MPa (15,000 psi) pressure
    - Generate *targeted* data to meet short-term needs for standards and critical technology development
    - Exercise proposed materials test methods and demonstrate improvements
  - Ensure R&D results and technical perspectives are effectively communicated to stakeholders (industry, SDOs)
    - Materials guide ("Technical Reference") available on website
    - Reports with R&D results distributed directly to stakeholders
    - Sandia technical staff serve on standards development committees

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Approach: Milestones

- Optimize fatigue crack growth rate measurements for pressure vessel steels in H_2 and report results to ASME
  - Presented update to ASME at quarterly meeting Nov. 2011
- Evaluate effects of load-cycle frequency on fatigue crack growth rates for 7XXX aluminum alloys in high-pressure H_2
  - Results presented at ASME PVP conference July 2011
- Measure H_2-affected fracture properties of technologically relevant welds in collaboration with industry partner
  - Conducted initial testing on tube welds supplied by partner
- Enable completion of standards through committee leadership and data evaluation
  - CSA CHMC1, CSA HPIT1, SAE J2579, ASME KD-10
- Develop capability for variable-temperature testing in high-pressure H_2 gas
  - Assembly of new fully automated gas manifold nearly complete

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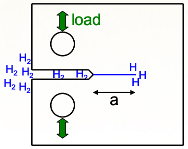
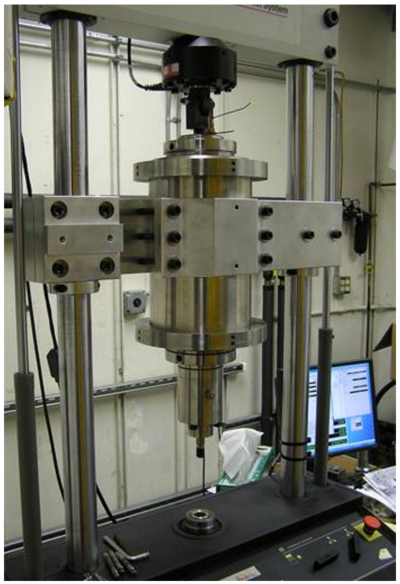
Materials Compatibility and Components project impacts multiple standards

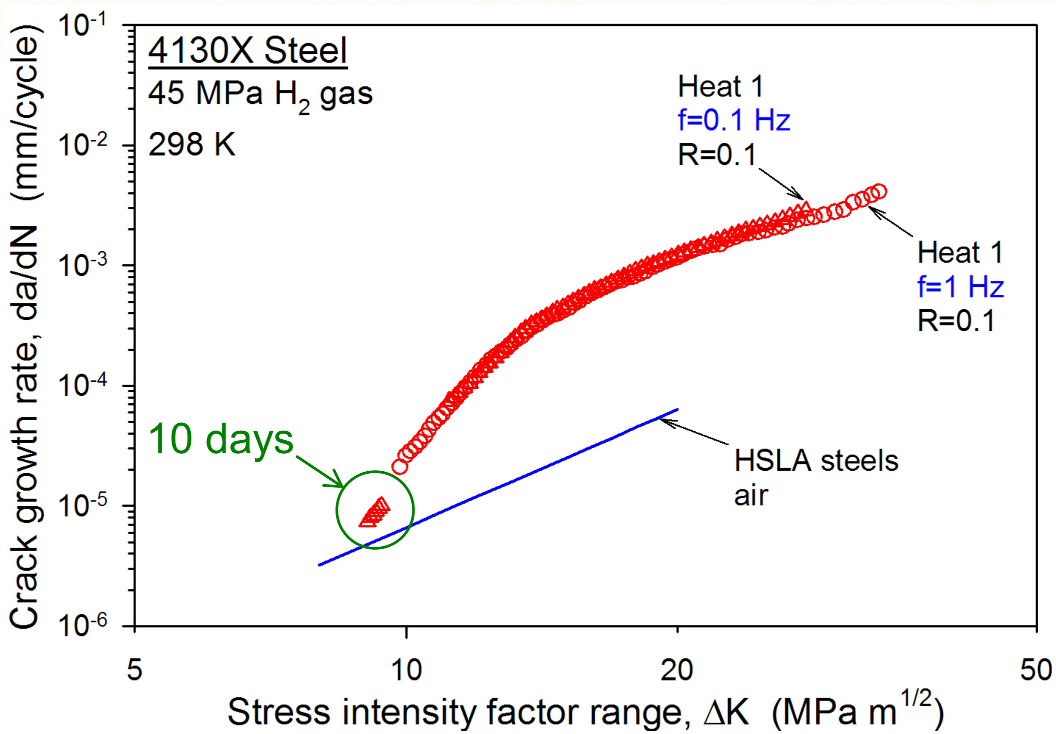
- CSA CHMC1
  - Materials testing and data application standard
  - Sandia provides leadership in technical committee and document preparation
  - Part 1 of standard published early 2012
- CSA HPIT1
  - Hydrogen-powered lift truck standard
  - Sandia provided tank-testing data and committee participation
  - Standard completed Sept. 2011
- SAE J2579
  - Hydrogen vehicle fuel system standard
  - Sandia serves as U.S. technical lead on addressing hydrogen embrittlement
  - Progress on new appendices addressing hydrogen embrittlement reported at International Conference on Hydrogen Safety in Sept. 2011
- ASME Article KD-10
  - Standard on high-pressure hydrogen tanks for transport and storage
  - Sandia provides data on exercising and improving materials test methods
  - Progress on optimizing fatigue crack growth testing reported at ASME Project Team meeting Nov. 2011

Previous Accomplishment:

Fatigue testing in H_2 must be optimized to balance efficiency and data reliability







- Load-cycle frequency currently in ASME KD-10 (0.1 Hz) leads to time-consuming testing
- Goal: *establish test procedure that shortens test duration without compromising data quality*

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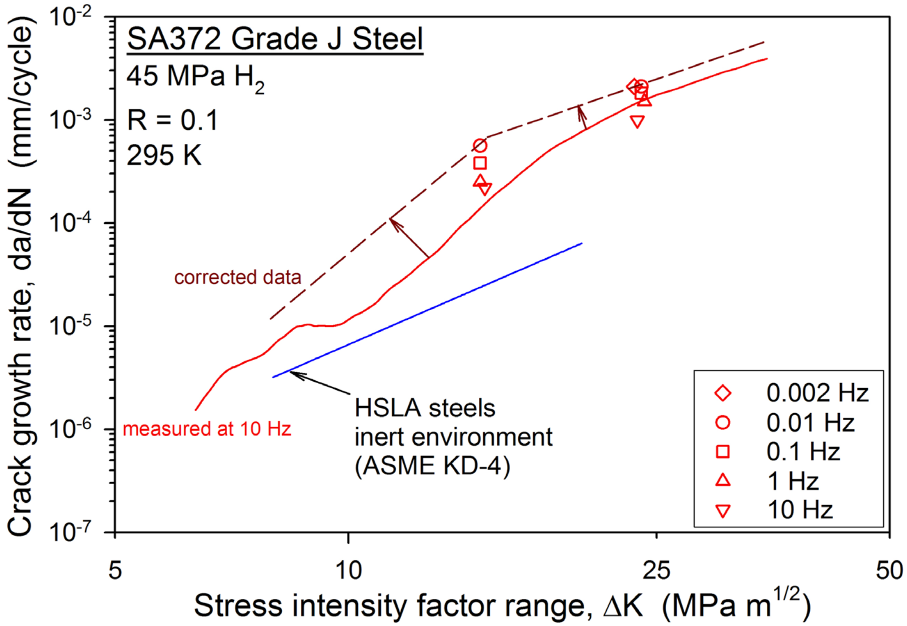
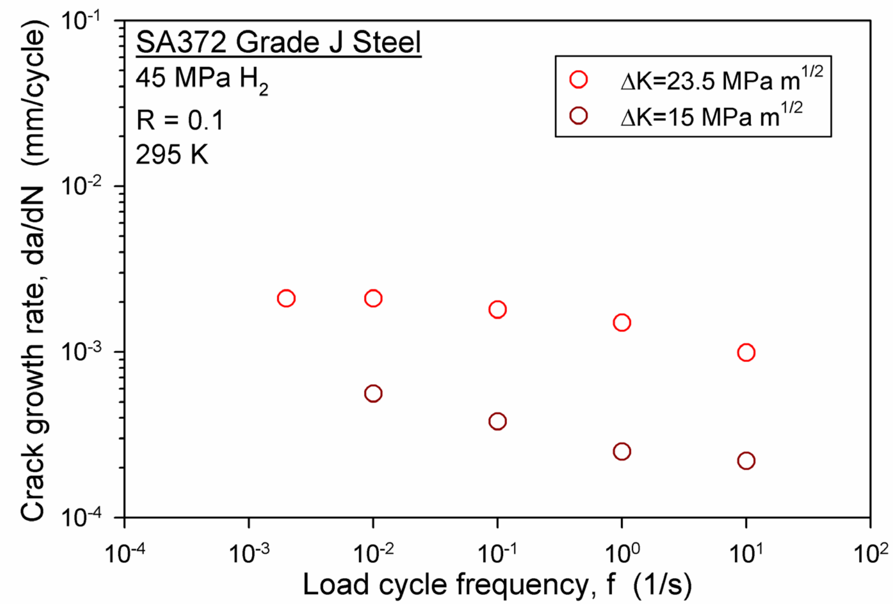
Accomplishment:

Possible approach: apply correction to da/dN *vs* ΔK measured at high frequency





- Measure da/dN *vs* ΔK relationship at high frequency
  - Data can be measured over wide ΔK range in reasonable time (40 hrs at 10 Hz)
- Determine correction for da/dN *vs* ΔK by measuring da/dN at selected constant ΔK levels under low frequency
  - Load-cycle frequency of 0.1 Hz may not provide upper-bound data
- Concept presented to ASME Project Team

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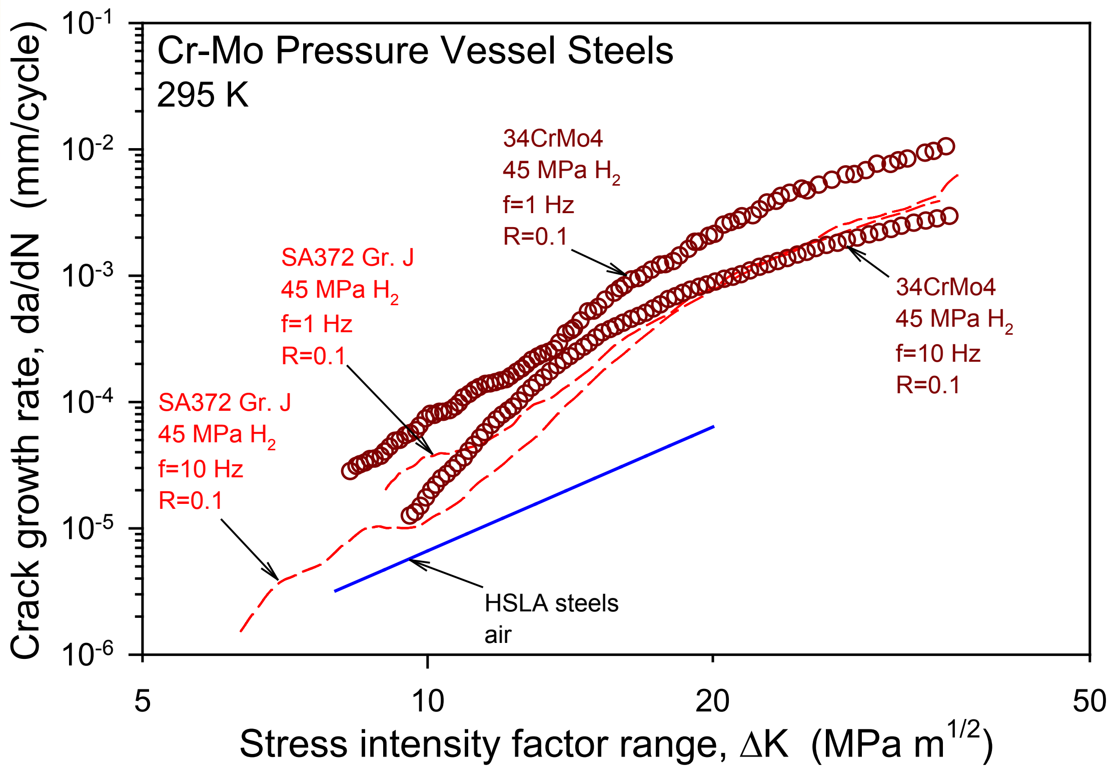
Accomplishment:

Modified fatigue testing methods must be demonstrated for range of steels



- Tensile strength of 34CrMo4 is 20% higher than SA372 Gr. J
- Data on 34CrMo4 important for optimizing test methods and providing insight into behavior of high-strength steels in H_2

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Accomplishment:

Test matrix includes multiple H_2 pressures for each pressure vessel steel

Steel	S_u (MPa)	H_2 pressure (MPa)	Test frequency (Hz)	Load ratio	Status
SA372 Gr. J	890	10	10	0.1	Attempted
		10	variable	0.1	
		45	10	0.1	Complete
		45	variable	0.1	Complete
		100	10	0.1	
		100	variable	0.1	In progress
34CrMo4	1045	10	10	0.1	
		10	variable	0.1	
		45	10	0.1	Complete
		45	variable	0.1	Attempted
		100	10	0.1	
		100	variable	0.1	

- Steels provided by two different industry partners: FIBA Tech and undisclosed steel cylinder manufacturer

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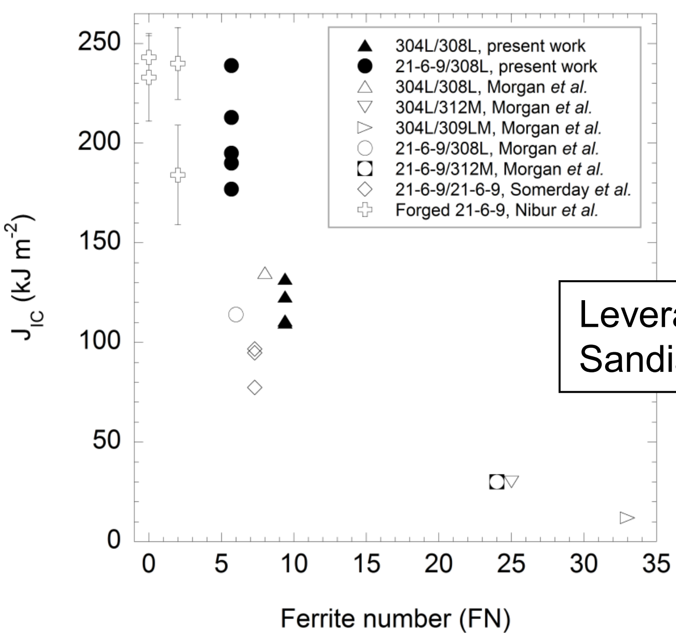
Previous Accomplishment:

Hydrogen compatible materials workshop: evaluation of welds is critical to technology deployment

- Welds are effective for joining metal components; reduce leaks in pressure systems compared to fittings
- Weld microstructures can be more susceptible to hydrogen embrittlement (HE), limiting performance of pressure systems
- Data gap: mechanical properties of technologically relevant, H_2-exposesd welds

H-affected fracture toughness vs. vol% ferrite

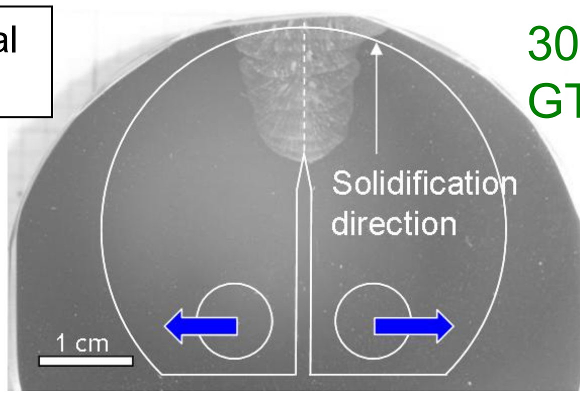


Leveraged internal Sandia programs

Jackson et al., *Corrosion Science*, *2012, in press*

Previous work

- HE sensitivity of welds depends on vol% ferrite
- geometry of tested welds not relevant to typical energy infrastructure

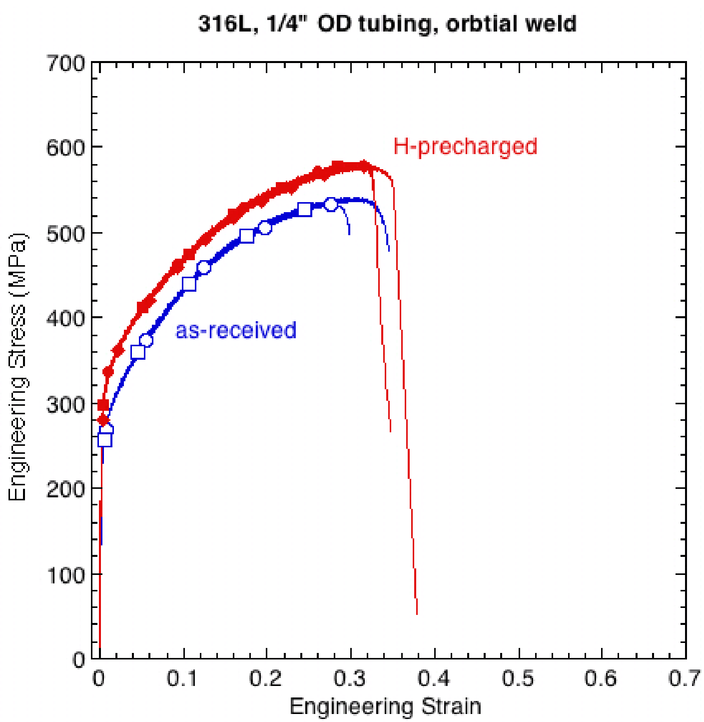


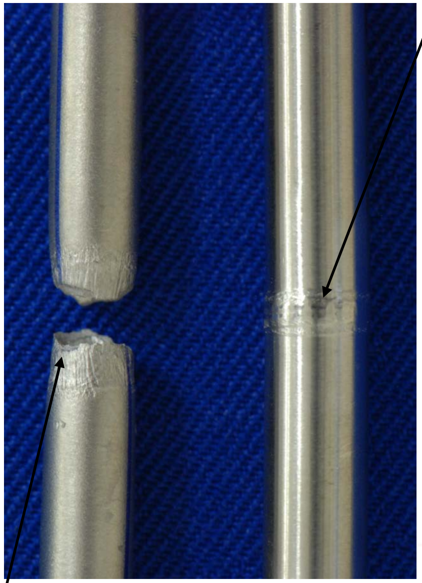
304L/308L GTA weld

Accomplishment:

Developed test methods for evaluating relevant stainless steel welds for hydrogen infrastructure

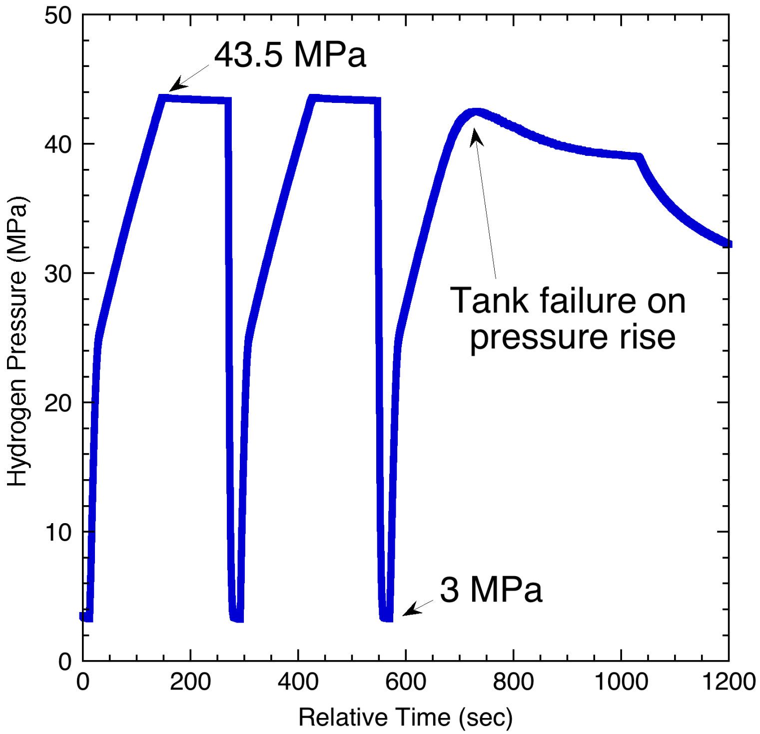
- Orbital tube weld chosen as relevant weld geometry
- Established industry partnership for production of 316L orbital tube weld specimens
- Preliminary tensile testing shows virtually nil effect of hydrogen on fracture resistance of weld, perhaps due to low vol% ferrite
- Fatigue testing protocol also being developed

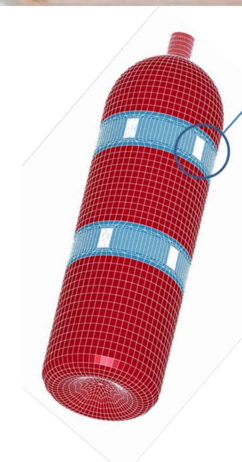




Previous Accomplishment:

Typical transport cylinders exhibited leak-before-break after failure due to cyclic pressure in H_2

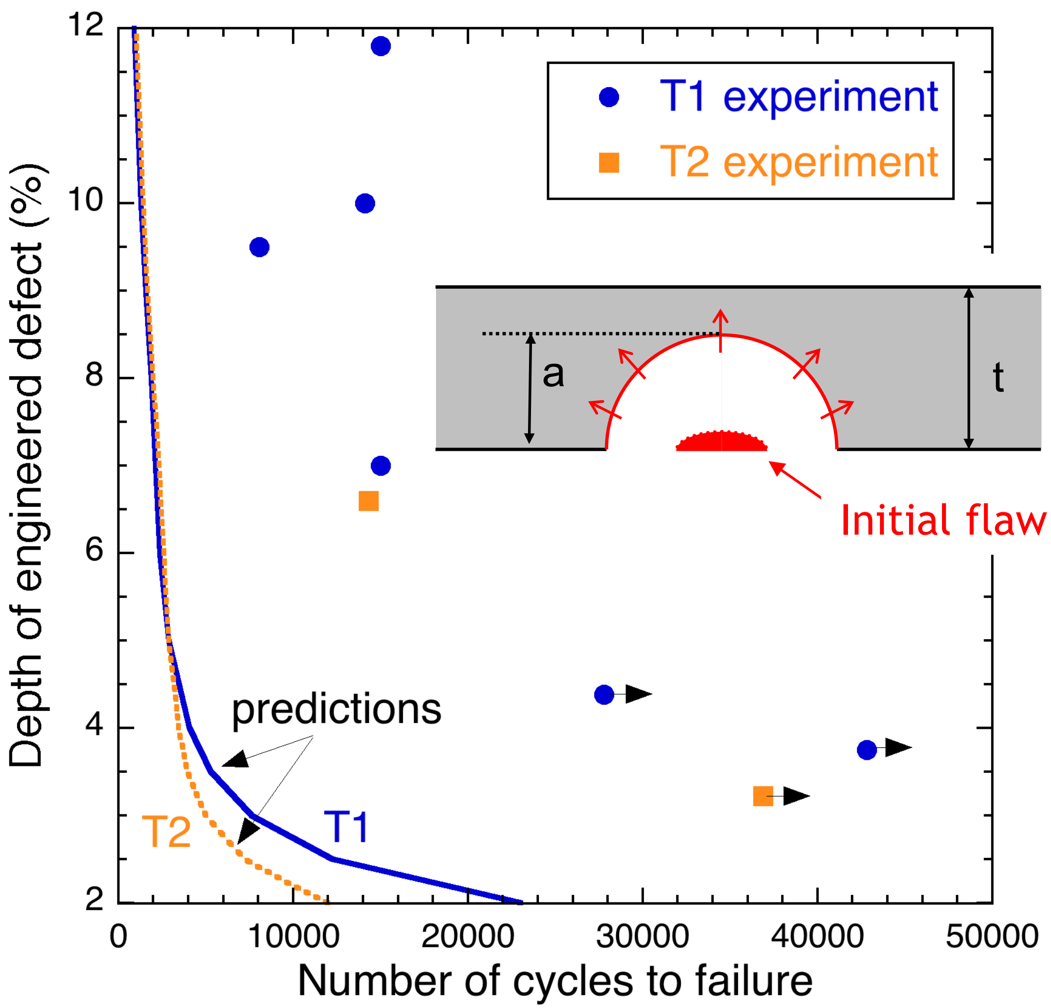




- At failure, pressure vessel “slowly” leaks gas into secondary containment
- Through-wall fatigue crack cannot be detected visually

Accomplishment:

ASME design qualification (BPVC VIII.3 KD-10) demonstrated to be very conservative



- ASME predictions based on *crack growth only*
- Symbols are full-scale pressure vessel experiments
- Arrows indicate vessels that did not fail

<u>Significance</u>

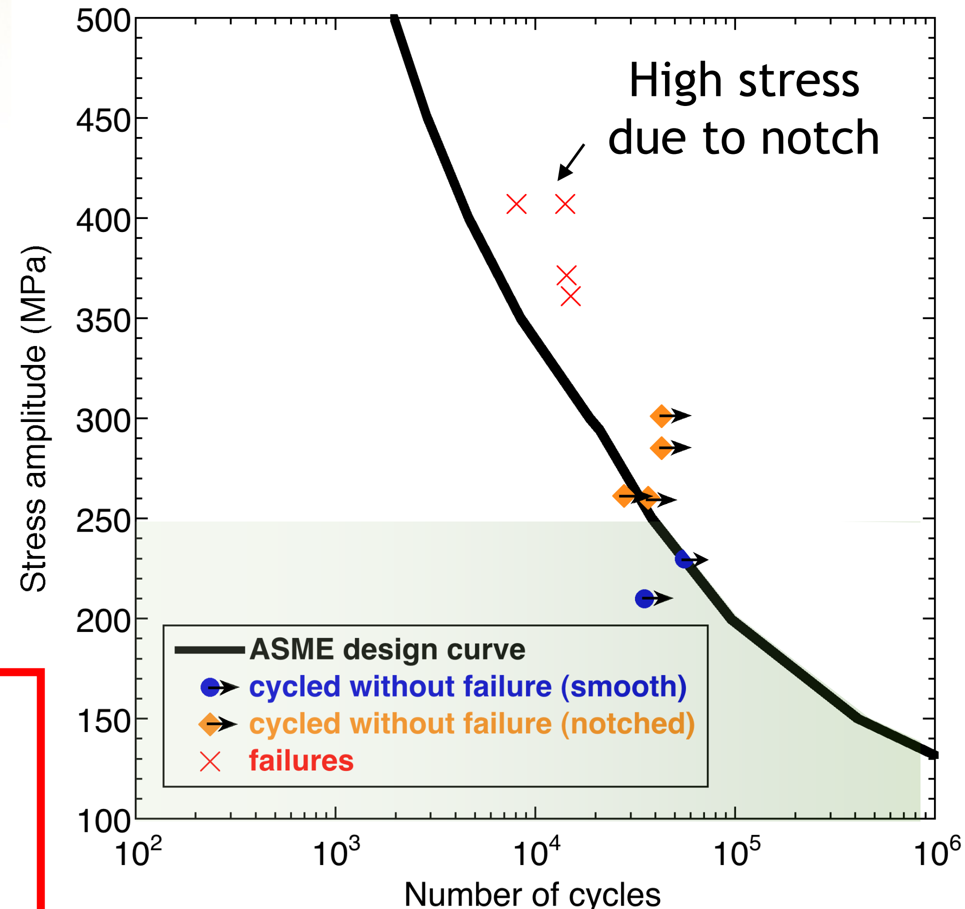
- Fatigue life calculation is conservative by factor of 4 or more
- For small initial defects, effective safety factor approaches 10

Accomplishment:

Results used to establish design rules for Type 1 pressure vessels in CSA HPIT1 standard

Requirements for Type 1 tanks

- Quench and tempered Cr-Mo steels
- $S_u \leq 890$ MPa
- hoop stress $\leq 0.4\ S_u$
- ASME design curves from BPVC VIII.3 Article KD-3



ASME design curve (BPVC VIII.3 Article KD-3): carbon and low alloy steels with UTS = 620 MPa

Engineering Significance of these requirements

- Maximum fatigue stress amplitude ~250 MPa
- Design life > 40,000 cycles

Collaborations

- Standards Development Organizations (SDOs)
  - Examples: SAE, CSA, ASME, ISO
  - Sandia technical staff lead and serve on committees
- Industry partners
  - Examples: FIBA Technologies, Swagelok, undisclosed steel cylinder manufacturer
  - Provide technology-relevant materials for Sandia testing activities
- DOE Pipeline Working Group (PWG)
- International research institutions
  - HYDROGENIUS/AIST (Tsukuba, Japan)
  - International Institute for Carbon-Neutral Energy Research (I^2CNER), Dr. Brian Somerday (Sandia) serving as Lead PI for Hydrogen Structural Materials Division

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Proposed Future Work

Remainder of FY12

- Complete test matrix on SA372 Gr. J and 34CrMo4 steels to enable optimization of methods for measuring fatigue crack growth in H_2
- Complete tensile fracture measurements of H_2-exposed stainless steel tube welds
- Complete SAE J2579 appendices and CSA CHMC1 Part 2
- Integrate automated gas handling manifold into existing H_2 test system
- Exercise test method in CHMC1 for measuring fatigue crack initiation in H_2 on pressure vessel steels
- Issue Sandia report reflecting updated content from Technical Reference website

FY13

- Measure fatigue crack initiation resistance of H_2-exposed stainless steel tube welds
- Develop validated methodology to account for fatigue crack initiation life in steel H_2 pressure vessels for consideration in ASME Article KD-10
- Develop R&D program with industry partner to improve resistance of high-strength pressure vessel steel to H_2-assisted fatigue crack growth
- Procure pressure vessel to complete variable-temperature testing in H_2 gas system
- Leverage results on fatigue crack growth of steels in H_2 to advance international coordination with AIST and I^2CNER on materials testing and basic science

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Summary

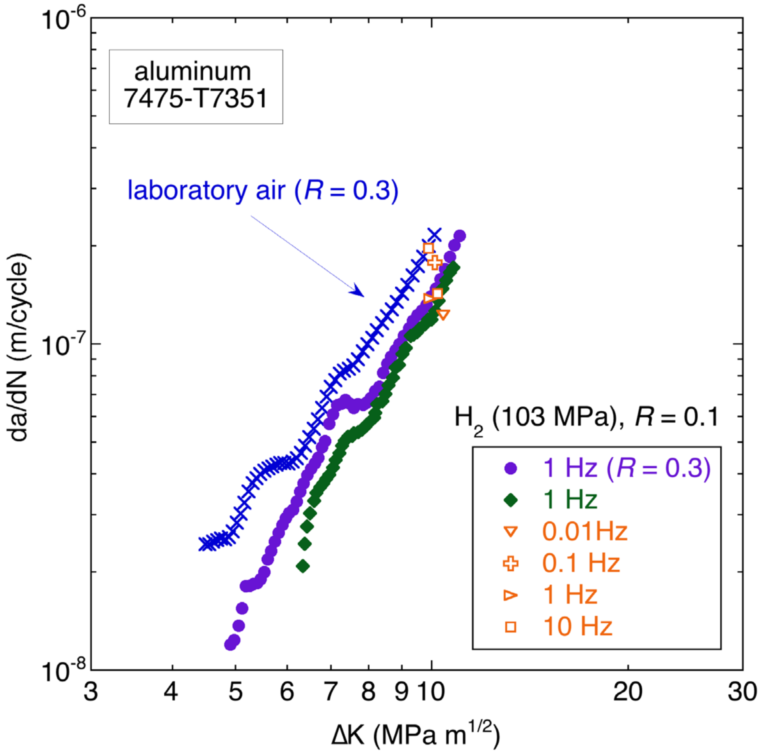
- Materials testing motivated by standards development and technology needs
  - Optimizing fatigue crack growth test method in ASME KD-10 to balance efficiency and data reliability
  - Measuring tensile and fatigue properties of H_2-exposed tube welds in collaboration with industry partner
  - Demonstrated resistance of 7XXX aluminum alloys to H_2-assisted fracture and fatigue
- Concrete progress in developing standards that address hydrogen compatibility of components
  - Part 1 of CSA CHMC1 published early 2012
  - CSA HPIT1 completed Sept. 2011
  - SAE J2579 completion expected 2012
- International collaborations enhanced
  - HYDROGENIUS/AIST (Tsukuba, Japan)
  - I^{2}CNER (Kyushu University, Japan)

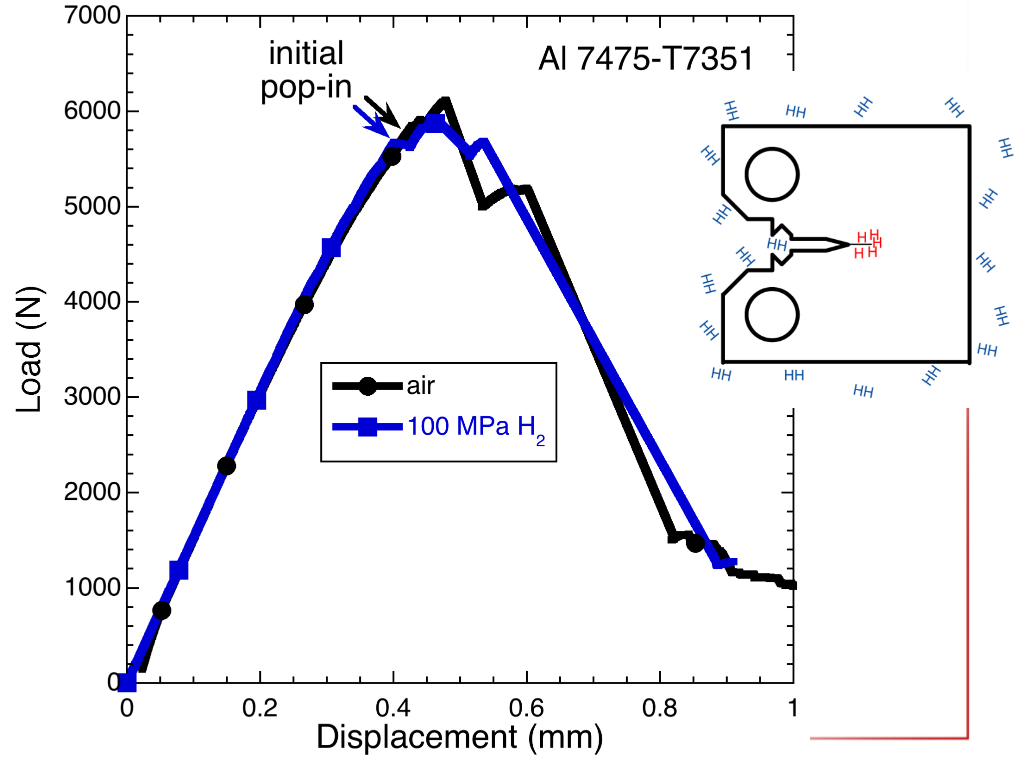
Technical Back-Up Slides

Accomplishment (leveraged with internal Sandia programs):

Fracture and fatigue crack growth in 7475-T7351 are unaffected by gaseous hydrogen

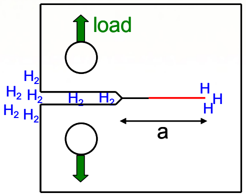
- Preliminary fatigue measurements suggest that the effects of H_2 on aluminum are not dependent on frequency or R ratio
- Cracking threshold is ~44 MPa $m^{1/2}$ in both air and H_2

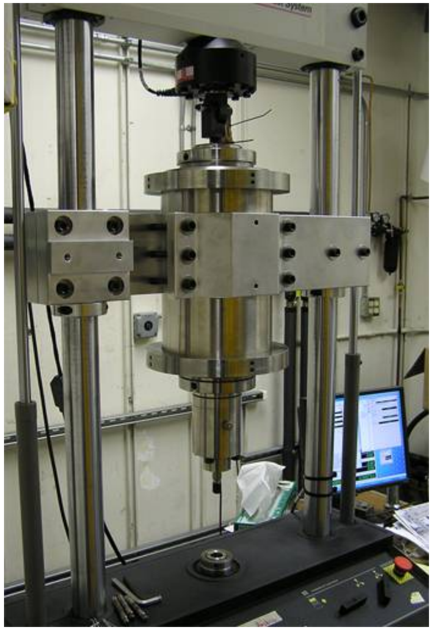




Collaborations:

AIST-SNL collaboration established to harmonize test methods and standards





- Two joint activities identified from AIST-SNL meeting (Livermore, CA) in Sept. 2011
  - Validate and promote method for measuring “initiation” threshold of ferritic steels in H_2
  - Explore basic mechanisms of H_2-assisted fracture in stainless steels
- Detailed project plan document developed in Jan. 2012
- Sandia hosted AIST visiting researcher in Feb. 2012
- Pressure vessel steel test specimen exchange expected during summer 2012

AIST NATIONAL INSTITUTE OF ADVANCED INDUSTRIAL SCIENCE AND TECHNOLOGY (AIST)

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Collaborations:

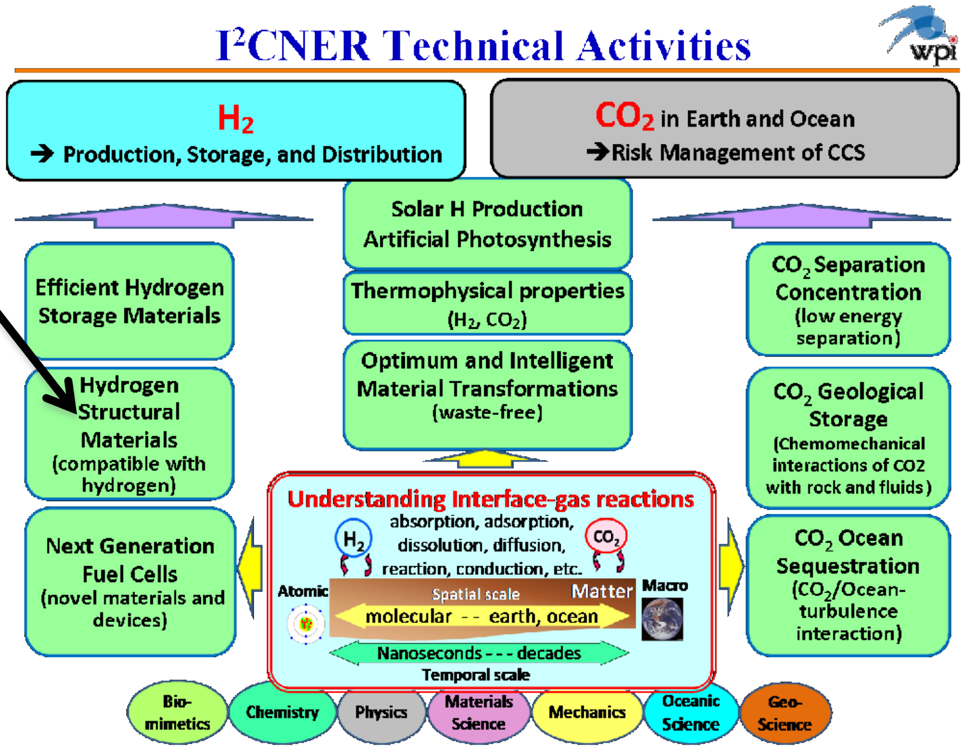
DOE investment in materials compatibility lead to Sandia leadership role in I²CNER

International Institute for Carbon-Neutral Energy Research
$10M/year Director P. Sofronis

KYUSHU UNIVERSITY ILLINOIS

SNL LVOC-H_2 objective: connect international community of H_2-energy stakeholders (research, industry, code development)

B. Somerday



- DOE encouraging collaborations with Japan
- Existing collaborations enabled SNL role in I²CNER
  - SNL/Illinois relationship since 2002
- Activities at SNL and I²CNER are complementary
  - Emphasis in I²CNER on basic research

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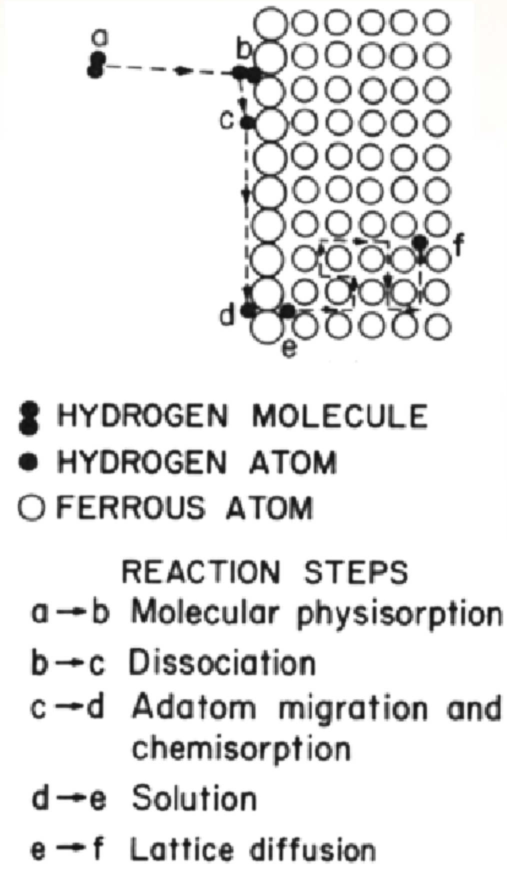
Accomplishment:

CSA CHMC1: provide confidence in hydrogen-material compatibility

- Part 1:
  - Describes mechanical tests to characterize strength, ductility, fracture resistance and fatigue resistance of metals in H_2
  - Specific test parameters provided in CHMC1 consider the kinetic processes required to transport molecular hydrogen from the gaseous environment to atomic hydrogen at critical sites within the metal.
  - Published early 2012
- Physics of hydrogen embrittlement makes quantitative evaluation of component compatibility challenging
  - Part 2 applies specific criteria and procedures to Part 1 to provide guidance on quantifying component compatibility
  - Part 2 scheduled for completion late 2012



Accomplishment:

Progress in augmenting laboratory testing capabilities

- Sandia is establishing new capability for conducting fatigue testing of materials in H_2 at low temperature
  - Hydrogen embrittlement can be enhanced at low temperature in certain metals (e.g., stainless steels)
  - Low temperatures can be attained in H_2 containment components (e.g., tanks, dispensers)
- Fully automated gas-handling manifold for new testing capability nearly complete