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DOE Hydrogen Program

Materials Compatibility

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Overview

Timeline

- Project start date Oct. 2003
- Project end date Sept. 2015
- Percent complete 50%

Budget

- Total project funding (to date)
  - DOE share: $3.8M
- FY08 Funding: $1.1M
- FY09 Funding: $0.9M

(* R&D core, no IEA contracts)

Barriers & Targets

- Barriers and targets addressed
  - Materials reference guide for design and installation
  - Hydrogen storage tank standards for portable, stationary and vehicular use
  - Insufficient technical data to revise standards

Partners

- Interactions/Collaborations:
  - ASME, CSA, ISO
  - Swagelok, Fibatech
  - DOE Pipeline Working Group
  - HYDROGENIUS (AIST/Kyushu University, Japan)

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Objectives

- Enable development and implementation of codes and standards for H_2 containment components
  - Evaluate data on mechanical properties of materials in H_2 gas
    - "Technical Reference on Hydrogen Compatibility of Materials"
  - Generate new benchmark data on high-priority materials
    - Pressure vessel steels, stainless steels
  - Establish procedures for reliable materials testing
    - Sustained-load cracking, fatigue crack propagation
- Participate directly in standards development
  - Structural design standards
    - ASME Article KD-10
  - Materials testing standards
    - Automotive components

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Milestones

- Add/update chapters on stainless steels and precipitation-strengthened aluminum alloys in Technical Reference (FY08 Q4 and FY09 Q1; complete)
- Complete cracking threshold measurements on Ni-Cr-Mo pressure vessel steel (FY08 Q3; complete)
- Complete fatigue crack growth measurements on Ni-Cr-Mo vessel steel (FY08 Q4; in progress, expected FY09 Q3)
- Evaluate specimen designs for cracking threshold measurements of ferritic steels (FY09 Q2; complete)
- Compare fatigue crack growth data for stainless steels tested in H_2 gas *vs* H-precharged condition (FY09 Q2; in progress, expected FY09 Q4)
- Compare cracking threshold to fracture toughness measurements for vessel steels (FY09 Q3; complete)

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Approach

- Applied research
  - Conduct materials testing to address voids in data base
    - Emphasize high H_2 gas pressures (>100 MPa)
    - Apply test methods in ASME Article KD-10 (i.e., fracture mechanics)
    - Prioritize hydrogen-assisted fatigue crack growth
  - Critically review data to assess test methods
    - Ensure laboratory testing reveals material response for component
    - Evaluate variables that affect materials test methods
- Standards development activities
  - Provide feedback on ASME Article KD-10 based on results from materials testing
  - Develop ideas for materials testing standards applied to automotive components

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Technical Reference for Hydrogen Compatibility of Materials

- Additional/updated chapters in FY08-FY09
  - "Stabilized" austenitic stainless steels (i.e., 321 and 347)
  - Duplex stainless steels (updated with Sandia data)
  - Precipitation-strengthened aluminum (2xxx and 7xxx)
- Future chapters
  - Nickel alloys
  - Update chapters on ferritic steels with Sandia data
- www.ca.sandia.gov/matlsTechRef

Stakeholders (industry, SDOs, etc.) provide input on priority materials and receive completed products

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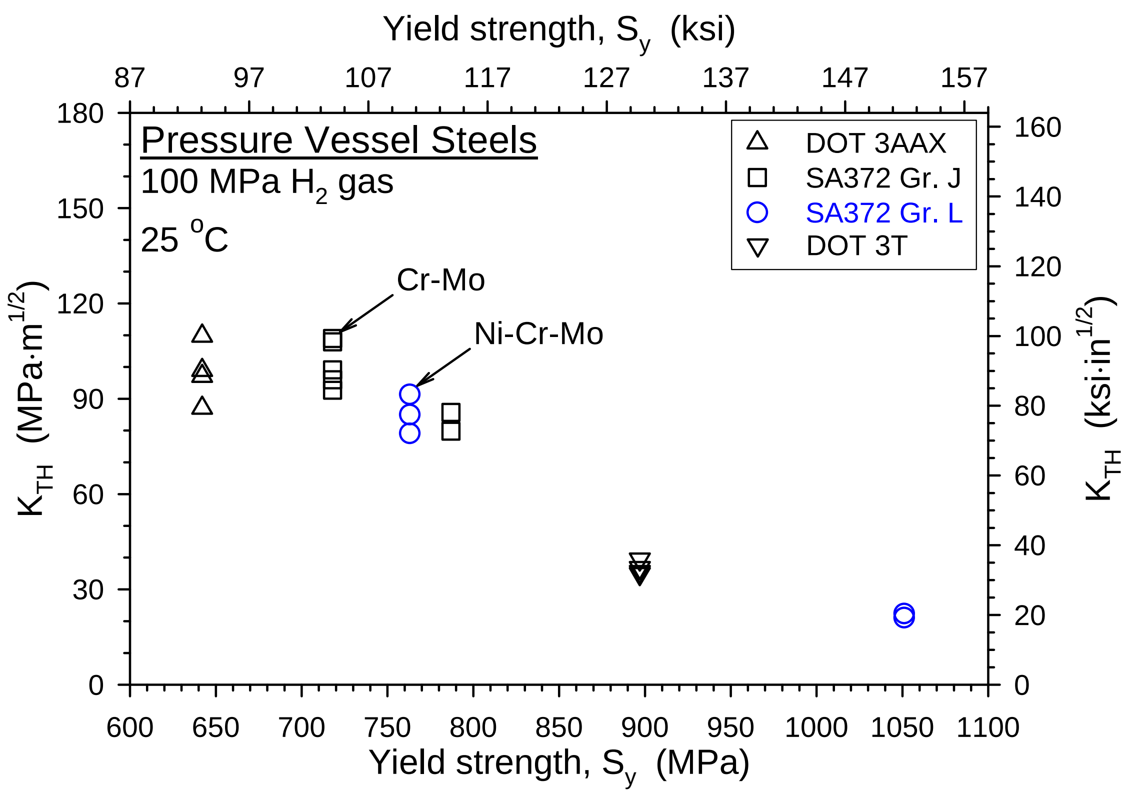
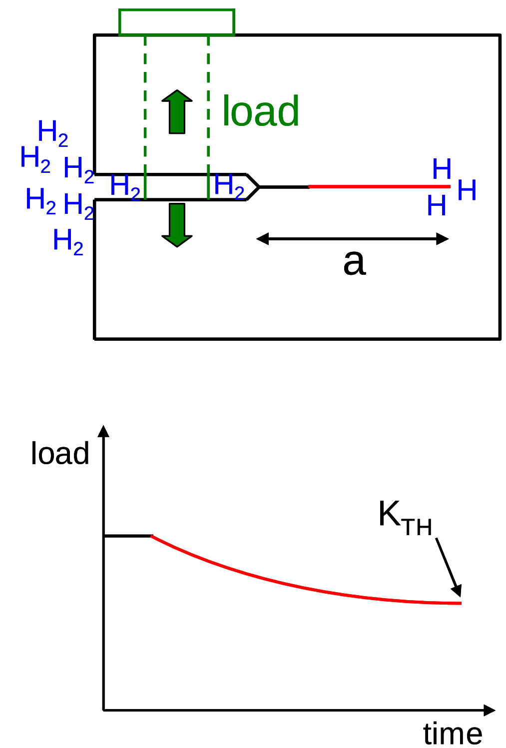
Measurement of thresholds (K_{TH}) for Ni-Cr-Mo steels completed

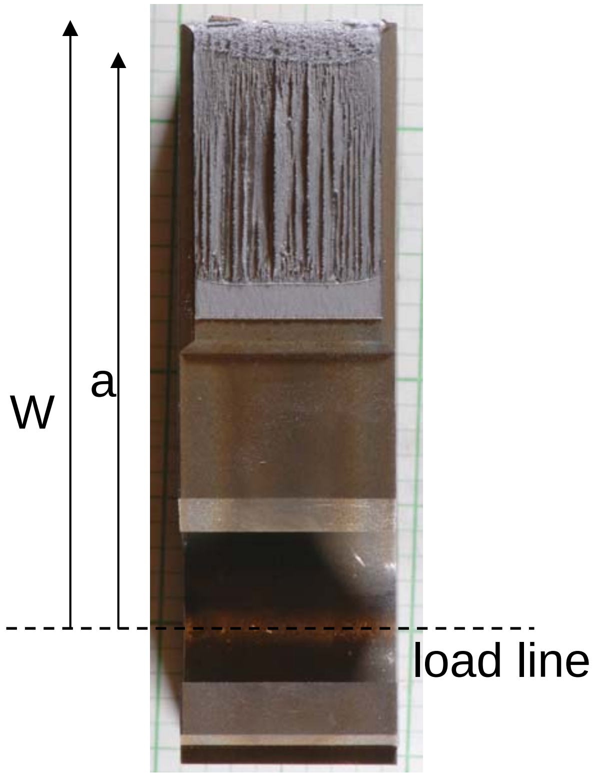




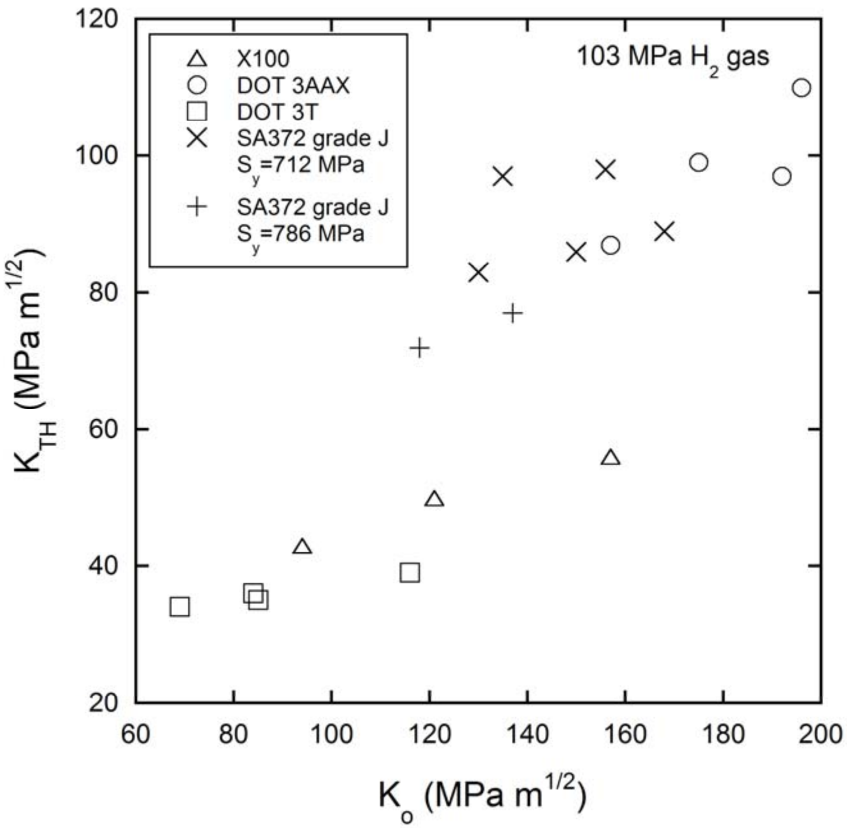
- K_{TH} measurements required in ASME Article KD-10
- Lower-strength variation of SA372 Gr. L may be attractive for thick-walled H_2 pressure vessels

Unexpected K_{TH} trend may be influenced by specimen features

Long cracks (large a/W) result from high initial load (K_o)

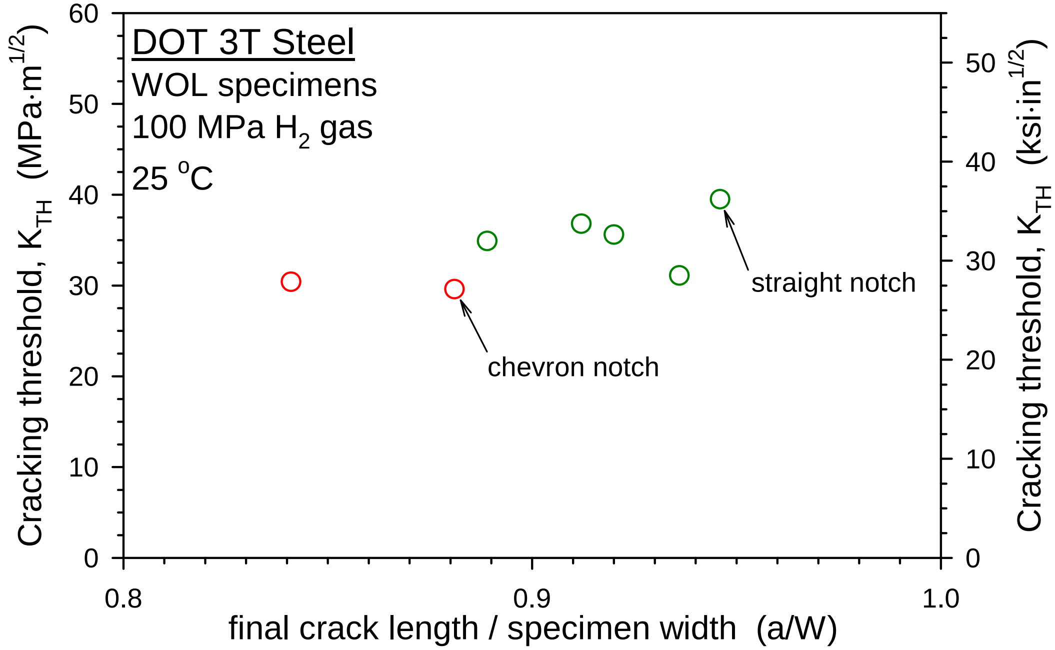


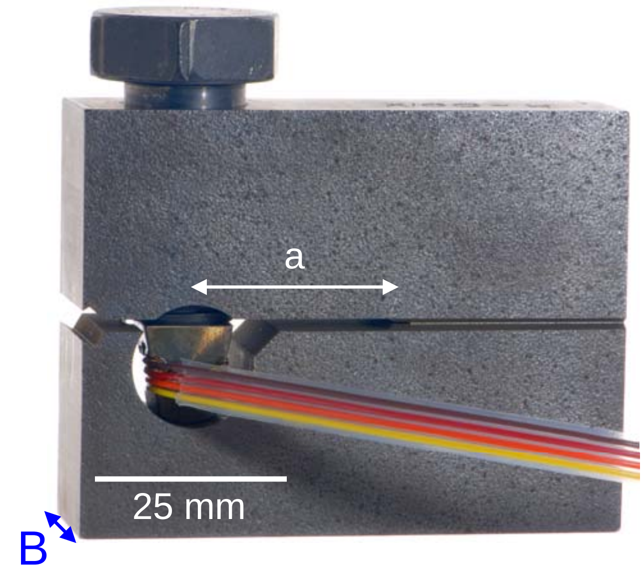
K_{TH} not independent of K_o

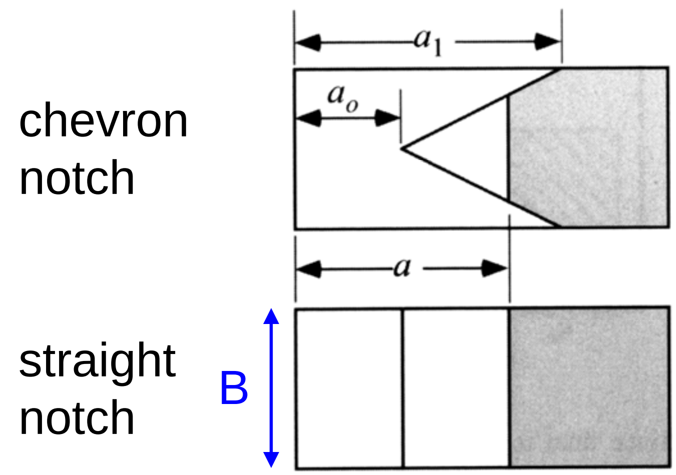


K_{TH} values must represent reliable lower bounds to ensure safe design of H_2 containment vessels

Modified specimen may yield lower K_{TH} values



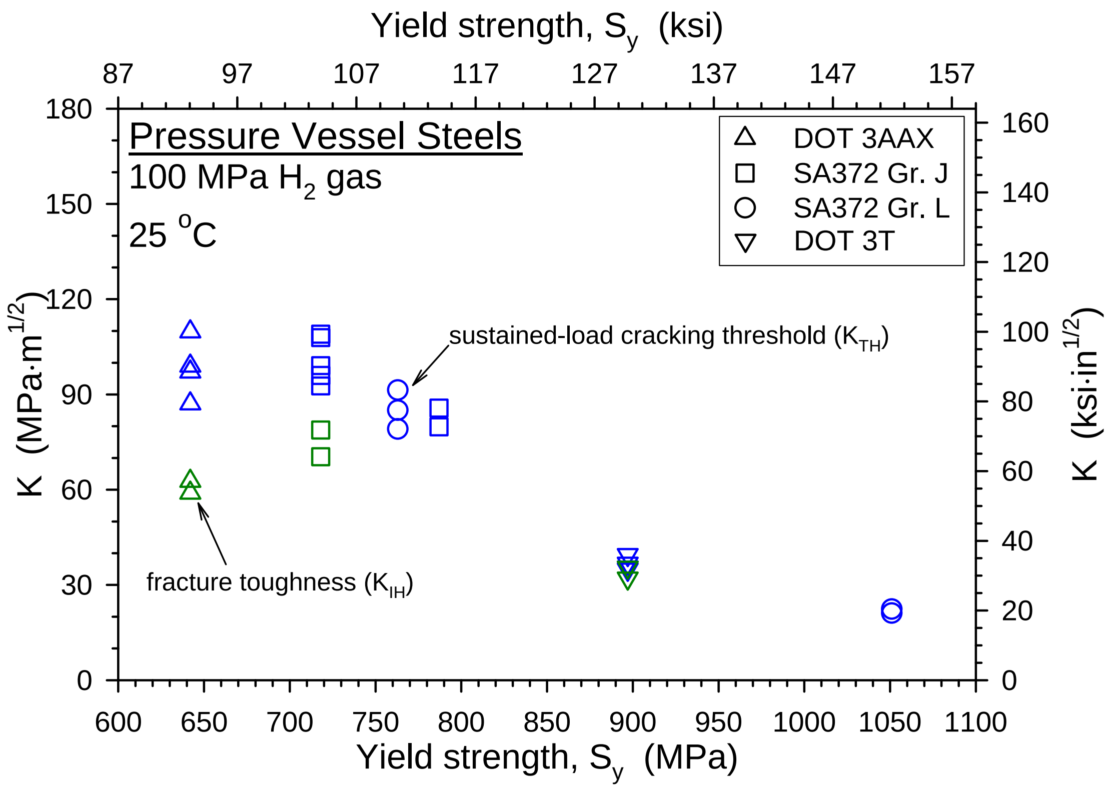




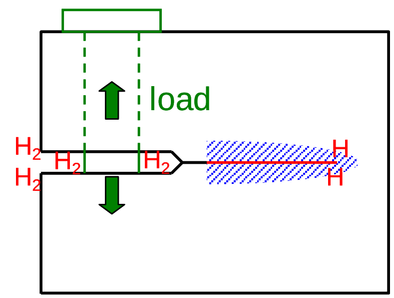
Chevron notch allows shorter initial crack lengths (a_o), leading to crack arrest further from back face

From: Anderson, T.L. 2005, *Fracture Mechanics*, 3rd edition, Taylor and Francis p.379

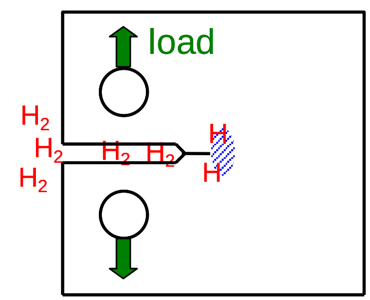
Two test methods compared: "crack arrest" *vs* "crack initiation"



Measures K_{TH} at crack arrest

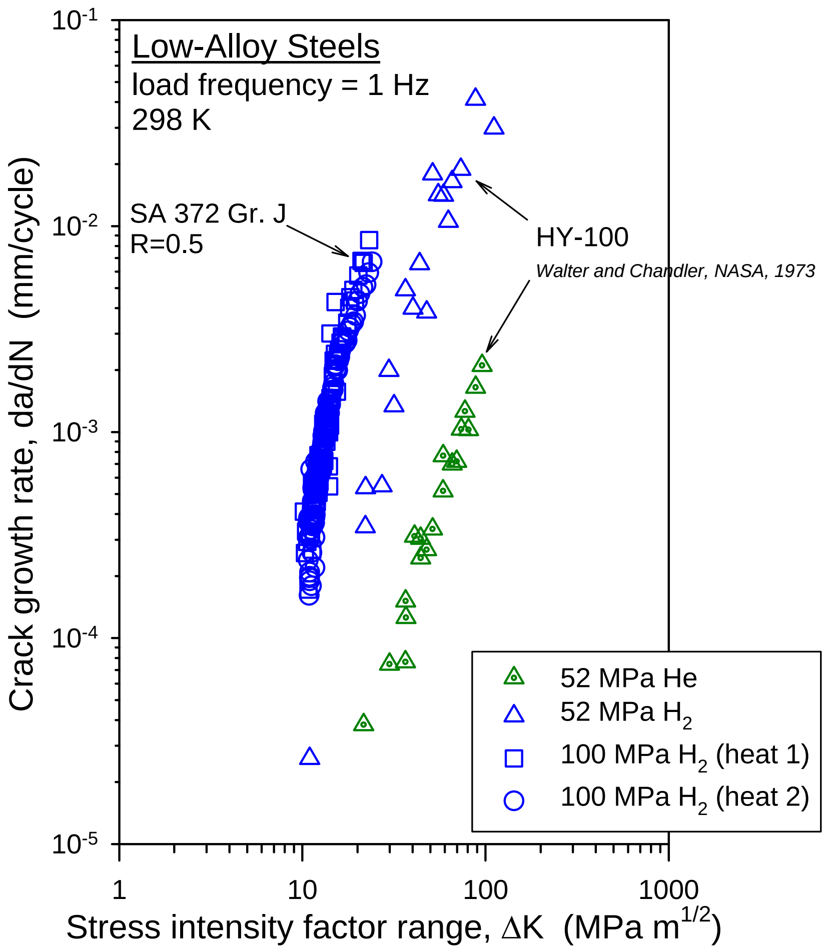


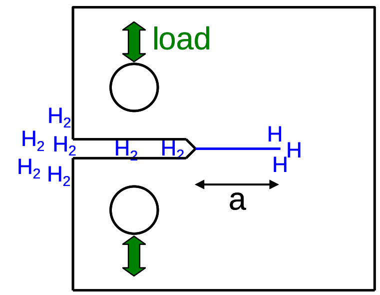
Measures K_{IH} at crack initiation

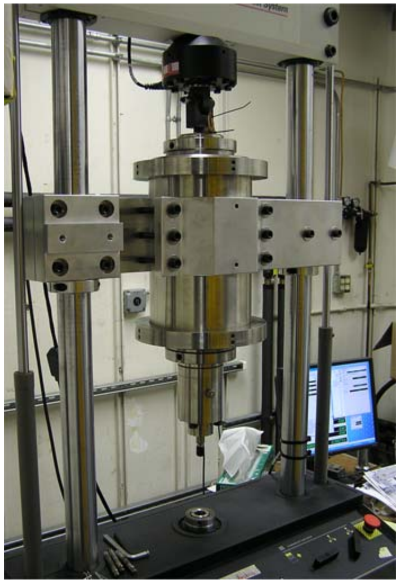


- Materials testing prescribed in ASME Article KD-10 (i.e., K_{TH} measurements) appears non-conservative
- Results will be presented to ASME Project Team on Hydrogen Tanks

Fatigue crack growth measurements on vessel steel

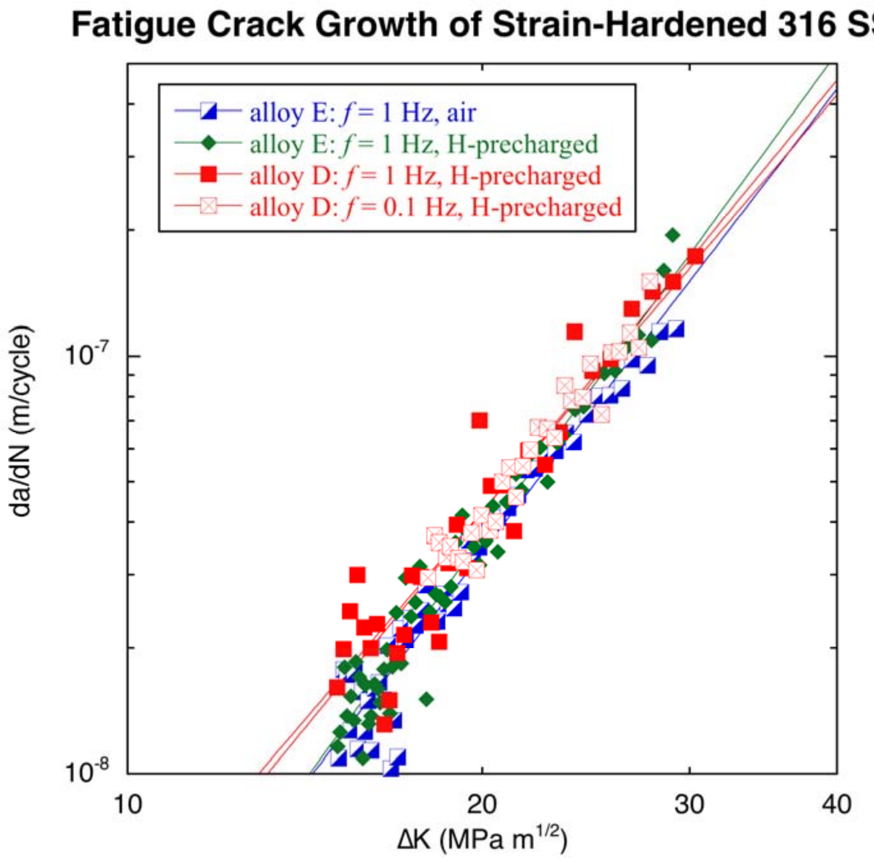




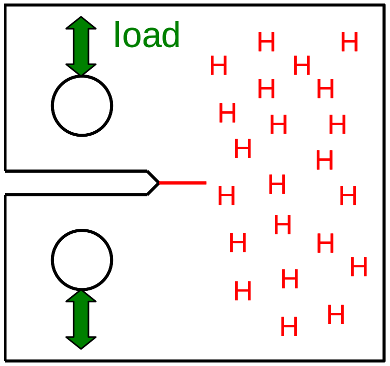


Industry partner using fatigue data to design H_2 pressure vessel according to ASME Article KD-10

Fatigue crack growth measurements on stainless steel



Alloy D: 12% Ni
Alloy E: 13% Ni



~140 wppm hydrogen

Materials testing standards must address effect of load cycle frequency on fatigue cracking rates

Future Work

Remainder of FY09

- Add/update Technical Reference chapters on nickel-based alloys and ferritic steels (Q3 and Q4 milestones)
- Evaluate the effects of load cycle frequency on fatigue crack growth rate of ferritic steels in H_2 gas (Q3 milestone)
- Compare fatigue crack growth data for stainless steels tested in H_2 gas *vs* H-precharged condition (Q2 milestone in progress)
- Develop reliable methods for measuring fracture response of aluminum alloys in H_2 gas (Q4 milestone)
- Engage domestic and international stakeholders to develop standards for pressure manifold components (i.e., fittings, regulators, etc.)

FY10

- Update Technical Reference chapters with Sandia data
- Evaluate effects of load cycle wave form on fatigue testing in H_2 gas
- Develop high- and low-cycle fatigue test methods for manifold component materials in H_2 gas

Summary

- Completed measurement of cracking thresholds (K_{TH}) for Ni-Cr-Mo pressure vessel steels in high-pressure H_2 gas
  - K_{TH} measurements required in ASME Article KD-10
- “Crack arrest” test methods appear to yield non-conservative results compared to “crack initiation” test methods
  - Proposal to insert “crack initiation” test methods in Article KD-10 will be presented to ASME Project Team on Hydrogen Tanks
  - “Crack initiation” methods require test apparatus designed for dynamic loading of specimens in H_2 gas
- Demonstrated ability to measure fatigue crack growth of pressure vessel steels in high-pressure H_2 gas
  - Fatigue crack growth data in H_2 required in ASME Article KD-10
  - Test apparatus is one of few in U.S. or abroad for measuring fatigue crack growth in >100 MPa H_2 gas

Additional Slides

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Responses to Previous Year Reviewers' Comments

1. *"Additional materials could enhance the usability of the project data"; "It is important to also check "legacy" materials where modern data is not available, such as existing pipeline materials and existing valve, pressure vessel, and appurtenance material where data is lacking"; "It is important to test materials that are candidate materials for advanced systems. This should be done by working with advanced tank and system designers in the Hydrogen Program."; "The project could be enhanced by polling the entire Hydrogen Program for candidate materials of interest"*

   Our materials testing priorities are established through interactions with various stakeholders, including industrial partners and standards development organizations. We also depend upon these stakeholders to provide material samples that are relevant to commercial applications. We are engaged with other Hydrogen Program activities, such as the DOE Pipeline Working Group. Collaborating in this particular activity ensures that materials like legacy pipeline steels are receiving attention. We are certainly open to interacting with other participants in the Hydrogen Program having interests in materials compatibility, for example, those working on compressors. We have tangible plans to expand the range of materials in our testing activities. For example, we intend to have some focused efforts on aluminum alloys.

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Publications and Presentations

1. *2008 International Hydrogen Conference: Effects of Hydrogen on Materials*, B. Somerday and P. Sofronis, co-organizers, Moran, WY, Sept. 2008
2. (invited) "Hydrogen Effects in Materials", B. Somerday, *3rd European Summer School on Hydrogen Safety*, Belfast, UK, July 2008
3. (invited) "Hydrogen-Assisted Fracture of Austenitic Stainless Steels", C. San Marchi et al., *2008 International Hydrogen Conference*, Moran, WY, Sept. 2008
4. (invited) "Variables Affecting Measurement of Sustained Load Cracking Thresholds in Hydrogen Gas", K. Nibur and B. Somerday, *ISO/TC 58/WG 7 Workshop on H_2 Test Methods,* Atlanta, GA, Sept. 2008
5. (invited) "Fracture Control of Hydrogen Containment Components", B. Somerday, *International Hydrogen Energy Development Forum 2009*, Fukuoka, Japan, Feb. 2009
6. "Sustained Load Cracking of Steels for Hydrogen Storage and Delivery", K. Nibur, B. Somerday, D. Balch, and C. San Marchi, in *Proc. 2008 International Hydrogen Conference*, 2009, in press
7. "Hydrogen-Assisted Fracture of Welded AISI 316 Austenitic Stainless Steel", X. Tang, G. Schiroky, C. San Marchi, and B. Somerday, in *Proc. 2008 International Hydrogen Conference*, 2009, in press
8. "Characterization of Hydrogen-Assisted Fracture Mechanism by Fracture Surface Topography Analysis (FRASTA)", T. Kobayashi, B. Somerday, C. San Marchi, and K. Nibur, in *Proc. 2008 International Hydrogen Conference, 2009*, in press
9. "Effect of Internal Hydrogen on Fatigue Strength of Type 316 Stainless Steel", C. Skipper, G. Leisk, A. Saigal, D. Matson, and C. San Marchi, in *Proc. 2008 International Hydrogen Conference*, 2009, in press

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Critical Assumptions and Issues

1. The intended focus of our testing activities for FY09 and FY10 is on development of test methods and benchmark data for fatigue properties of materials in high-pressure hydrogen gas. It is known that the fatigue behavior of materials in hydrogen gas is sensitive to testing variables such as the load cycle frequency. If low test frequencies (i.e., less than 0.1 Hz) are needed to develop test methods and material data, then the duration for individual tests can be rather extended. For example, the duration of a test that requires 10^5 cycles at 0.1 Hz is nearly 12 days. The realities of test duration must be considered in materials testing activities related to fatigue.

2. We are dependent on stakeholders to supply 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 component manufacturers. It is important that we maintain and expand our individual relationships with contacts at the SDOs and manufacturers so that we continue to have access to materials.