



SAND2011-2031C



DOE Hydrogen Program

# Hydrogen Embrittlement of Structural Steels

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Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under contract DE-AC04-94AL85000





# Overview

## Timeline

- Project start date Jan. 2007
- Project end date Oct. 2011\*
- Percent complete 50%

## Budget

- Total project funding (to date)
  - DOE share: \$900K
- FY10 Funding: \$150K
- FY11 Funding: \$200K

\*Project continuation and direction determined annually by DOE

## Barriers & Targets

- Pipeline Reliability/Integrity
- Safety, Codes and Standards, Permitting
- High Capital Cost and Hydrogen Embrittlement of Pipelines

## Partners

- DOE Pipeline Working Group
  - Federal Labs: Sandia, Oak Ridge, Savannah River, NIST
  - Universities: Univ. of Illinois
  - Industry: Secat, industrial gas companies, ExxonMobil
  - Standards Development Organizations: ASME



# Objectives/Relevance

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- Why steel hydrogen pipelines?
  - Safety of steel pipelines well understood (e.g., third-party damage tolerance, vulnerability of welds)
  - Hydrogen pipelines safely operated under *static pressure*
- Demonstrate reliability/integrity of steel hydrogen pipelines for *cyclic pressure*
  - Address potential fatigue crack growth aided by hydrogen embrittlement, *particularly in welds*
- Enable pipeline design that accommodates hydrogen embrittlement
  - Ensure relevance to pipeline design code ASME B31.12
- FY10-FY11: measure fracture thresholds and fatigue crack growth laws for X52 steel in H<sub>2</sub> gas, emphasizing welds

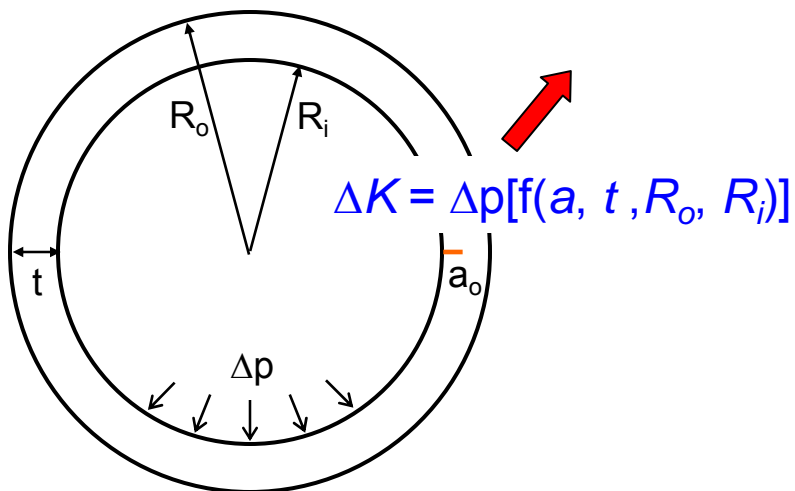
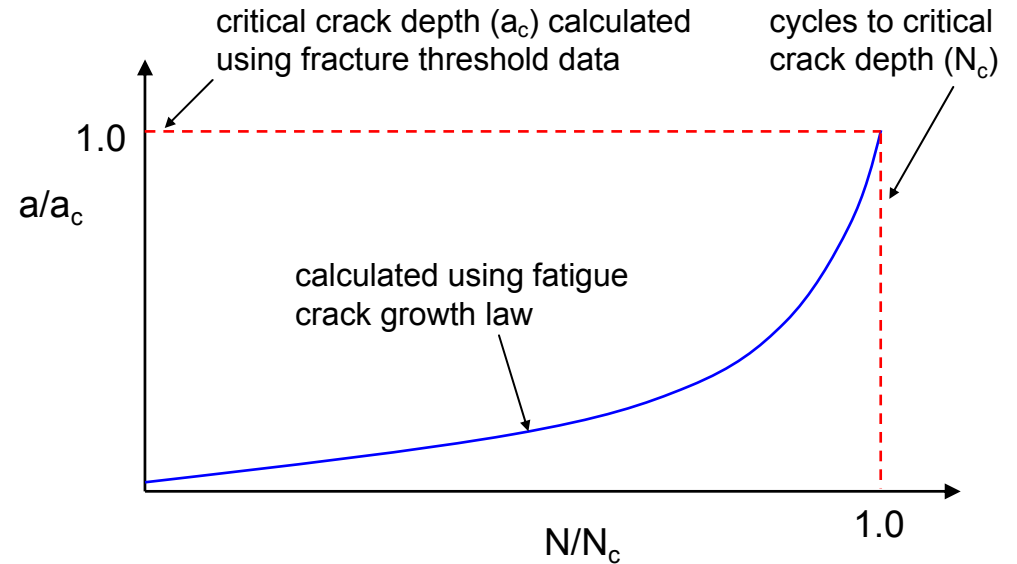
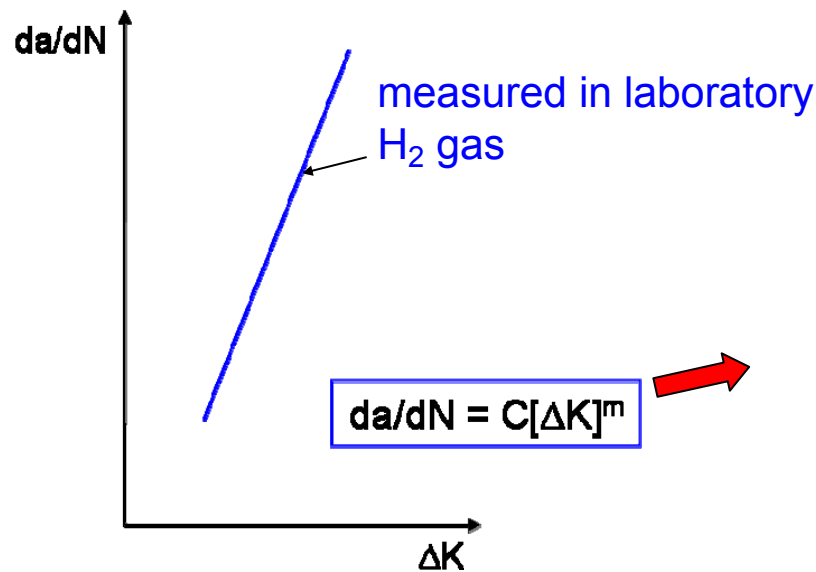


# Approach

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- Apply unique capability for measuring fracture properties of steels in high-pressure  $H_2$ 
  - Fracture properties serve as inputs into reliability/integrity assessment as specified in ASME B31.12 pipeline code
  - Milestone: Measure fracture thresholds for X52 steel base metal and seam weld (75% complete)
  - Milestone: Measure fatigue crack growth laws for X52 steel base metal and seam weld (75% complete)
    - Evaluate effect of load-cycle frequency on measurements
- Emphasize pipeline steels and their welds identified by stakeholders as high priority
  - Provide feedback to stakeholders through DOE Pipeline Working Group

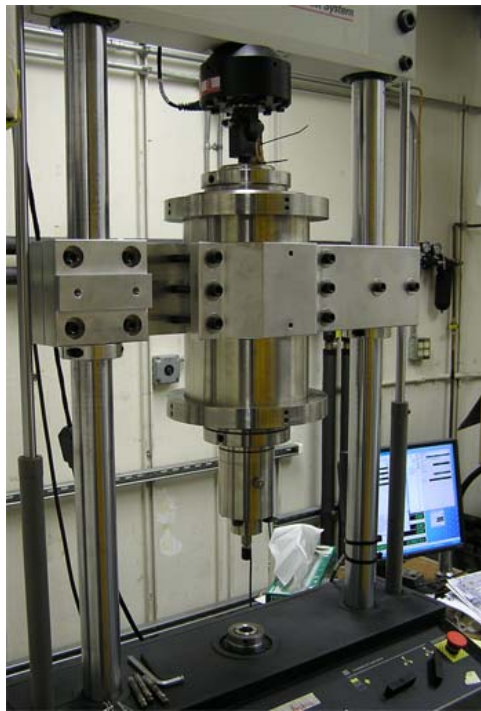
# Reliability/integrity assessment framework in ASME B31.12 requires fracture data in H<sub>2</sub>



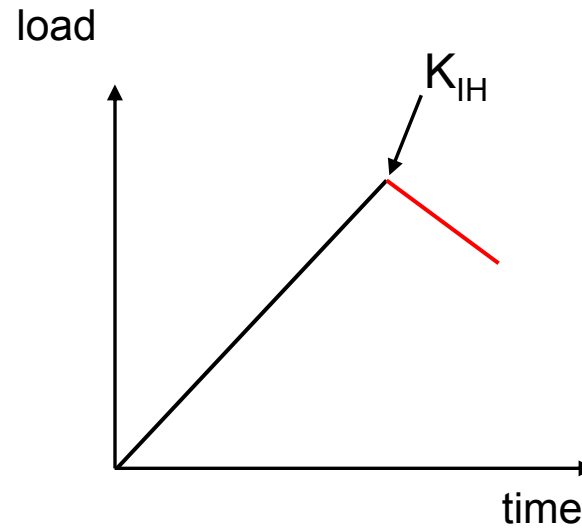
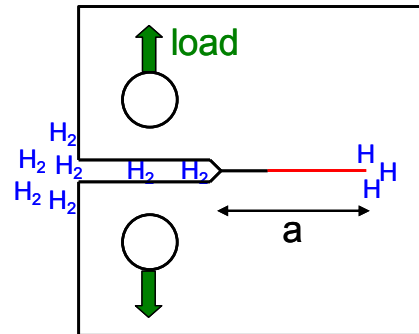
- Two fracture properties in H<sub>2</sub> needed
  - Fatigue crack growth law
  - Fracture threshold
- Reliability/assessment framework accommodates H<sub>2</sub> embrittlement



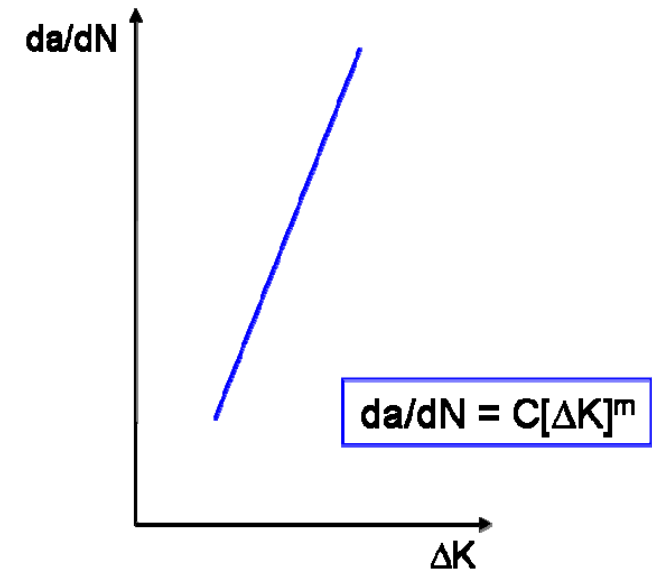
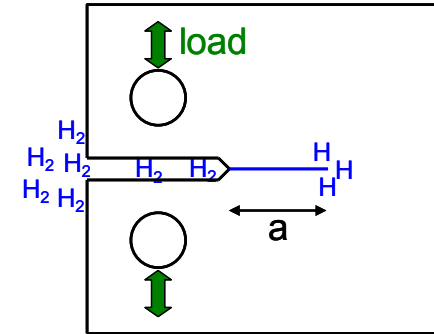
# Fracture data in $H_2$ measured using specialized laboratory capability



Fracture threshold



Fatigue crack growth

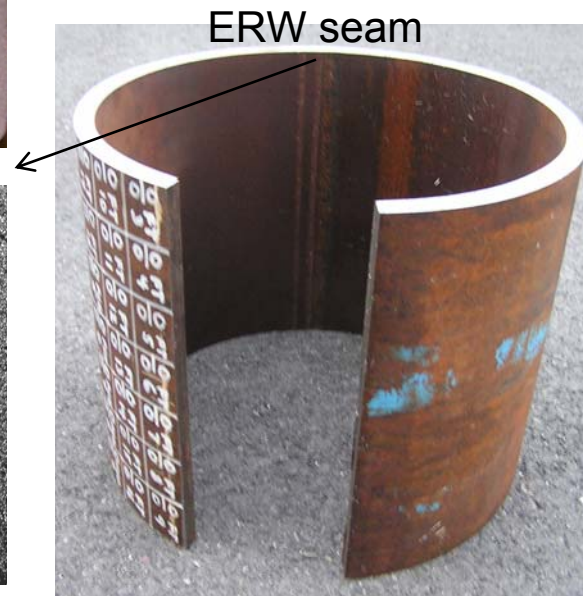
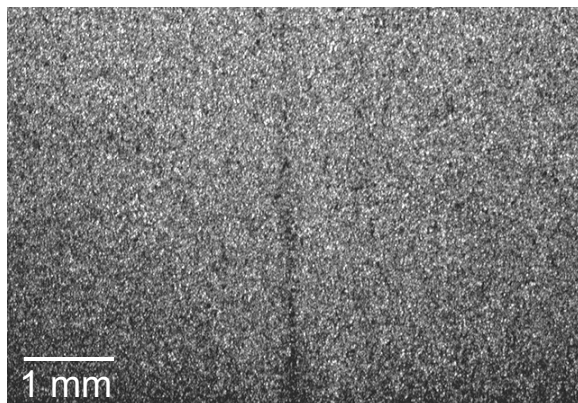




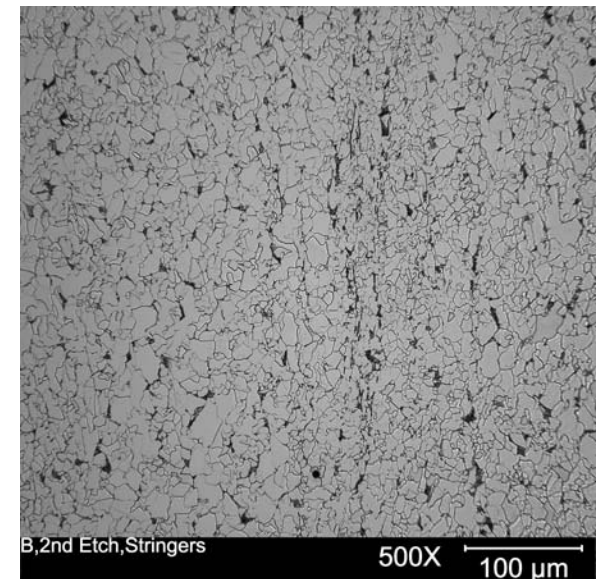


# Measured fracture properties of technologically relevant steel: API 5L X52

- Tested same X52 steel from DOE Pipeline Working Group tensile property round robin
  - Stakeholders expressed interest in X52 steel
- Tensile properties
  - Yield strength: 62 ksi (428 MPa)
  - Ultimate tensile strength: 70 ksi (483 MPa)

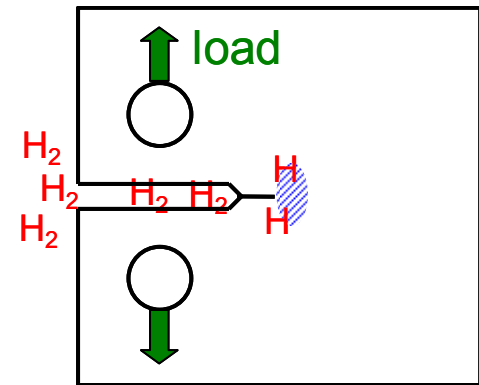
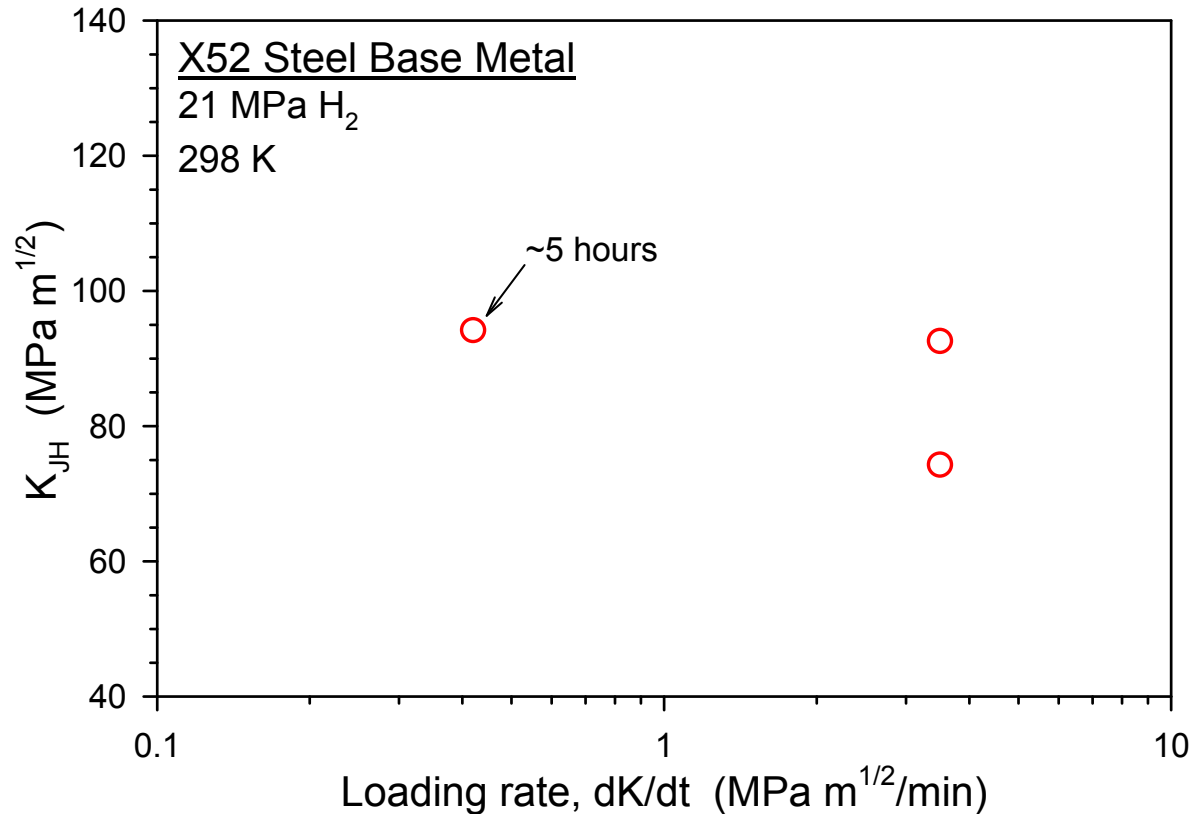


base metal



Accomplishment:

# Crack initiation thresholds measured for X52 in H<sub>2</sub> as function of loading rate

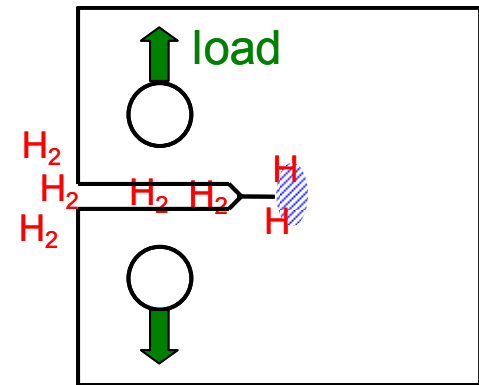
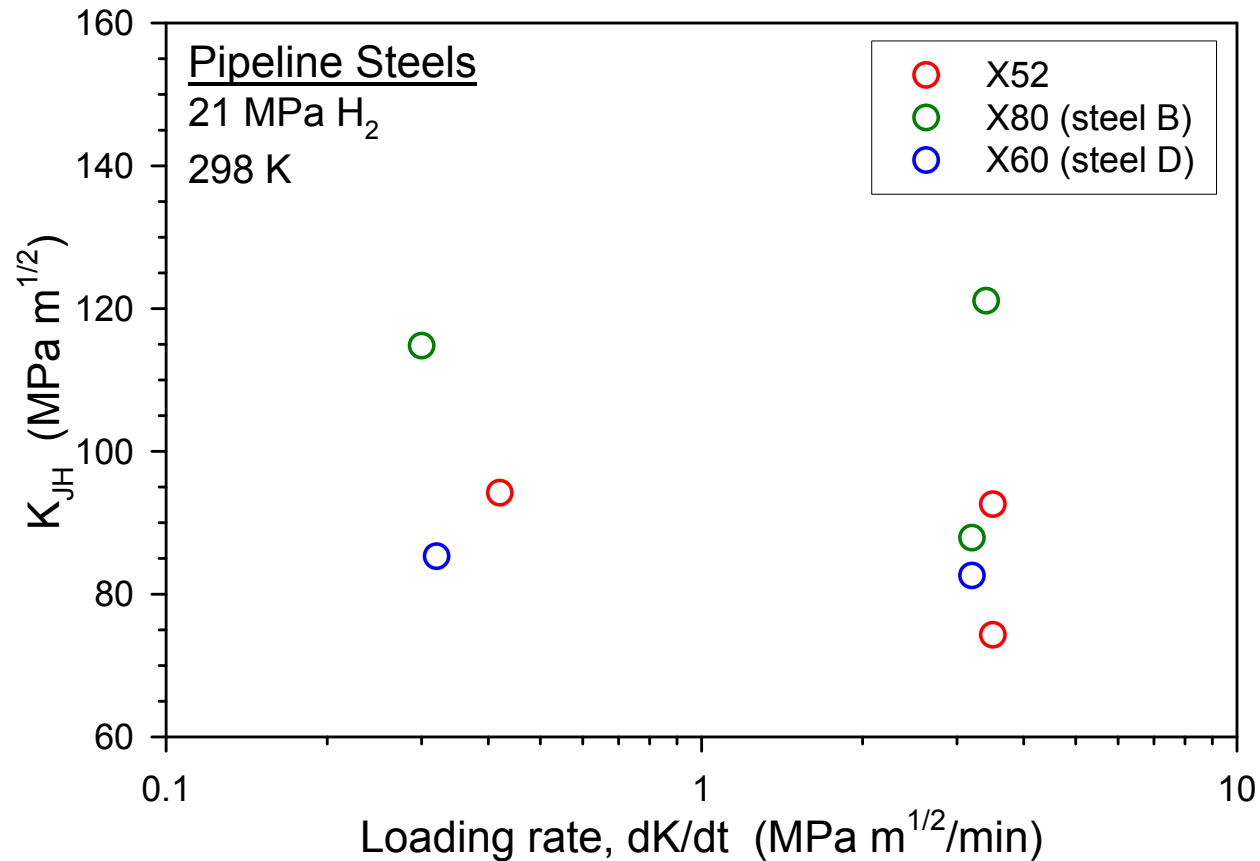


- Loading rate must be selected to balance test efficiency and data reliability
- Fracture threshold values ~80-100 MPa m<sup>1/2</sup> favorable for pipeline reliability/integrity



# Crack initiation thresholds similar for three different pipeline steels

X60 and X80 data: C. San Marchi et al., ASME PVP2010-25825, 2010

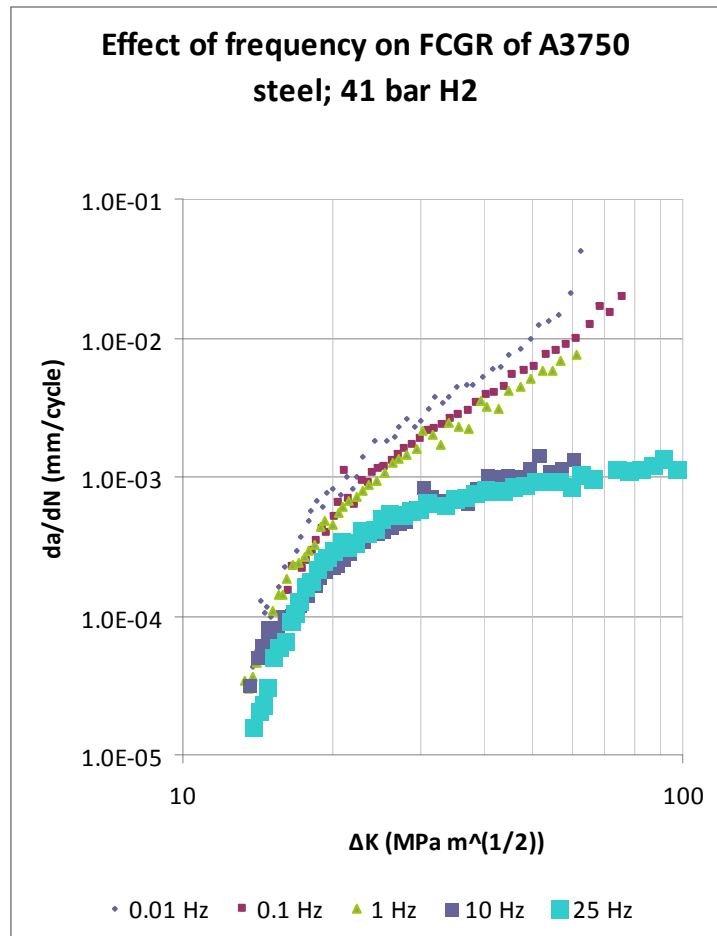


- Measurements for three steels conducted by participants in Pipeline Working Group

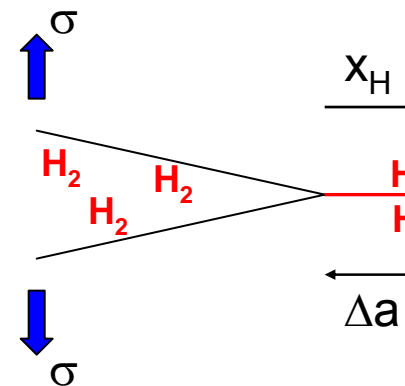


# Measurement of fatigue crack growth laws must consider effects of frequency

A.H. Priest, British Steel, EHC-(1)42-012-81UK(H), 1983



Condition for H penetration to affect crack growth:



$$x_H = (Dt)^{1/2}$$

$$\Delta a < x_H = (Dt)^{1/2}$$

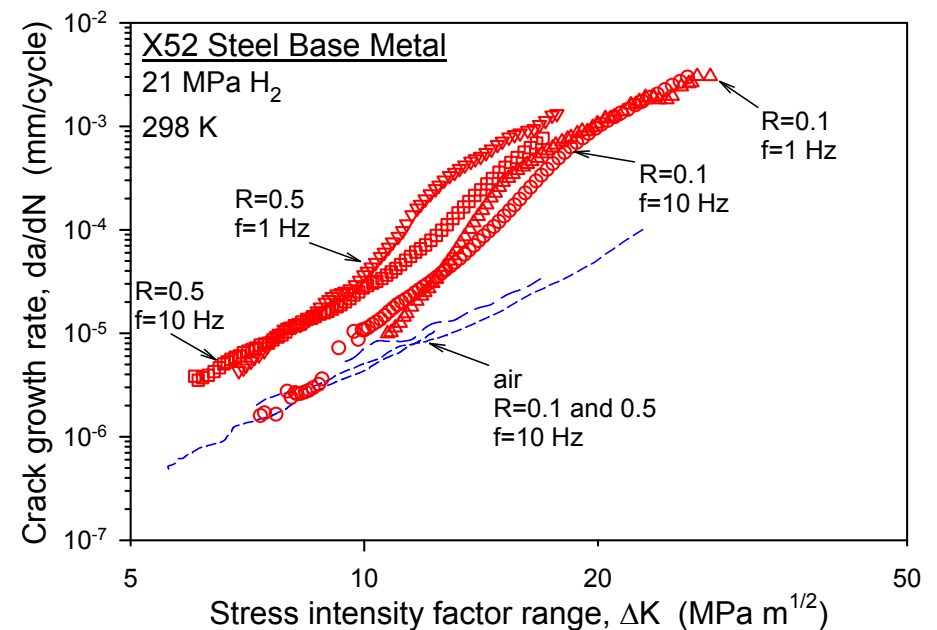
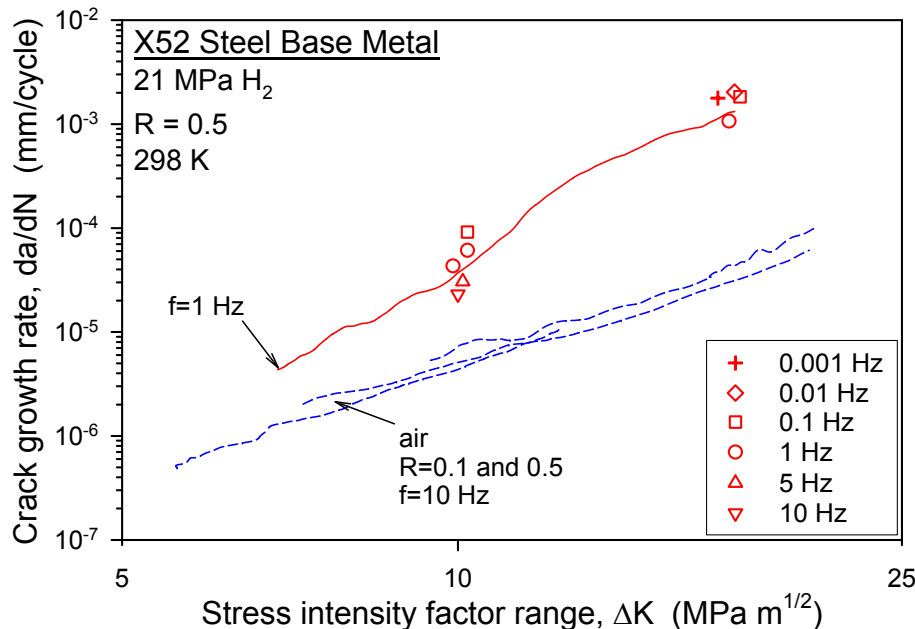
$$\Delta a < (D/f)^{1/2}$$

$$f \leq \frac{D}{(da/dN)^2}$$

- Frequency effects most pronounced at high da/dN

Accomplishment:

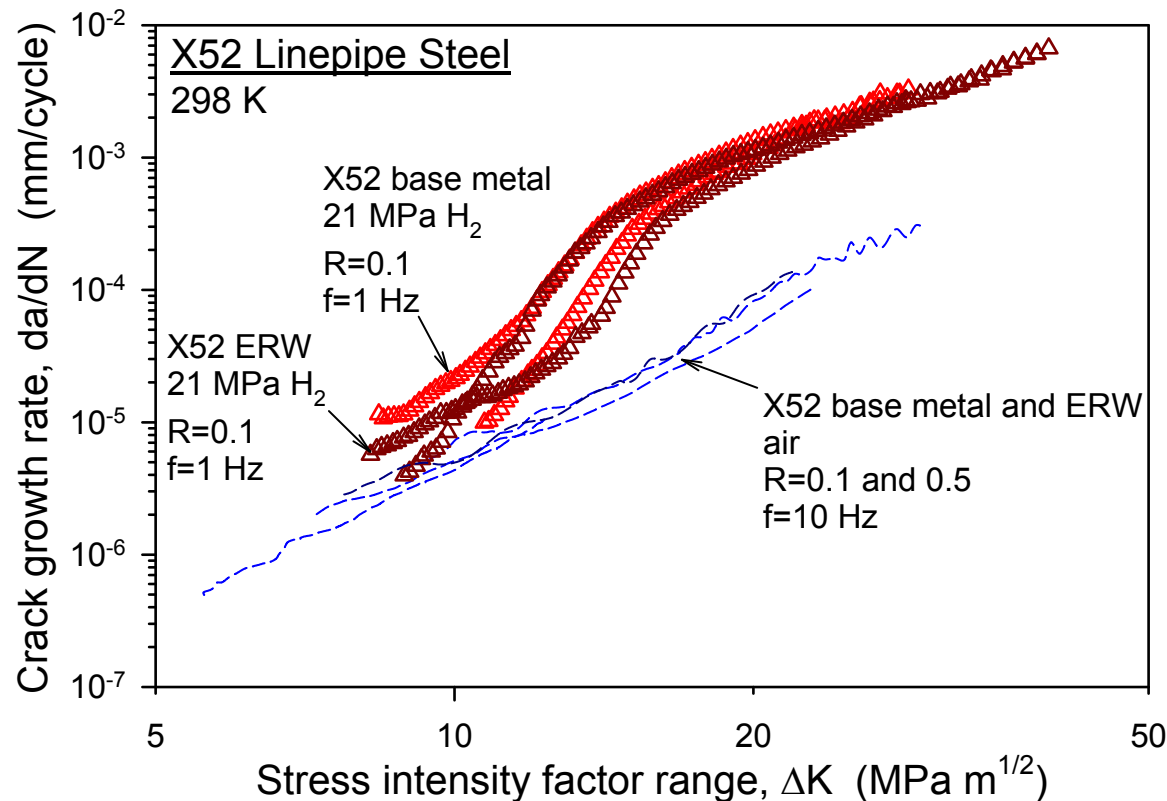
# Measured effects of frequency on fatigue crack growth laws for X52 base metal



- Tests at higher frequency (> 1 Hz) yield non-conservative data at high crack growth rates
- Frequency selected must balance test efficiency (i.e., duration) and data reliability
  - Tests for comparing different materials (e.g., base metal vs welds) conducted at 1 Hz

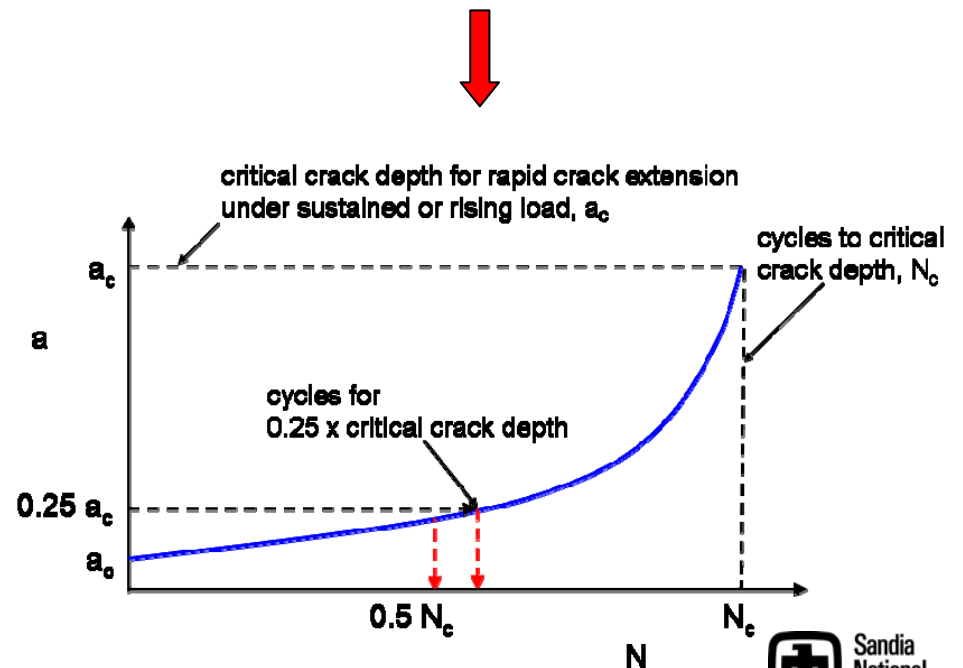
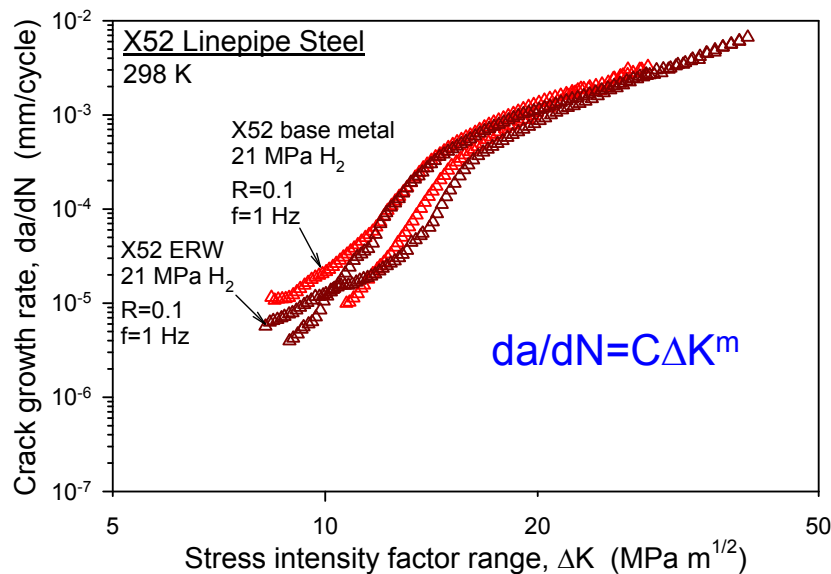
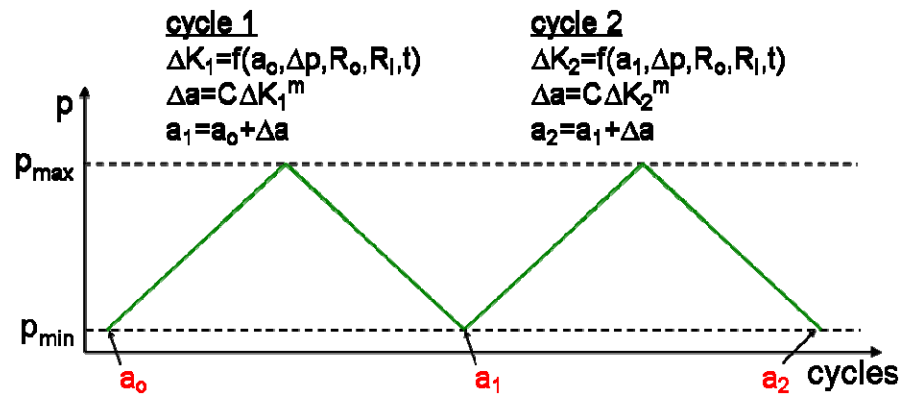
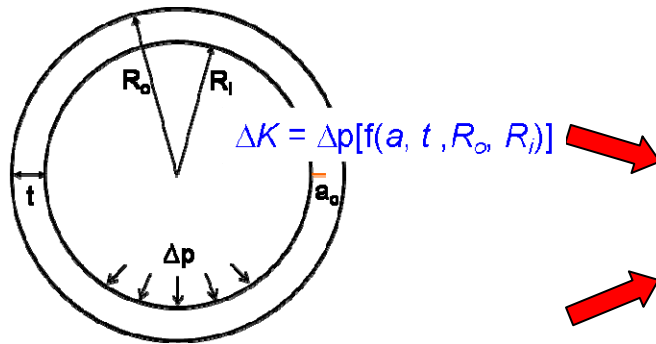
Accomplishment:

## Measured fatigue crack growth laws for X52 steel base metal and ERW (seam weld)



- Fatigue crack growth laws for X52 base metal and ERW similar in  $\text{H}_2$
- Notable variability in data from replicate tests for both base metal and ERW in  $\text{H}_2$

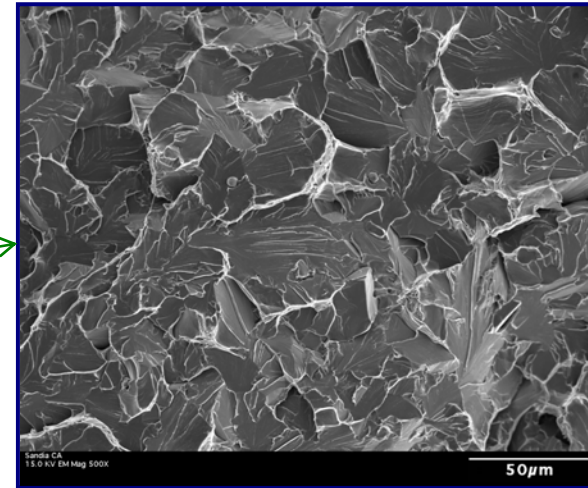
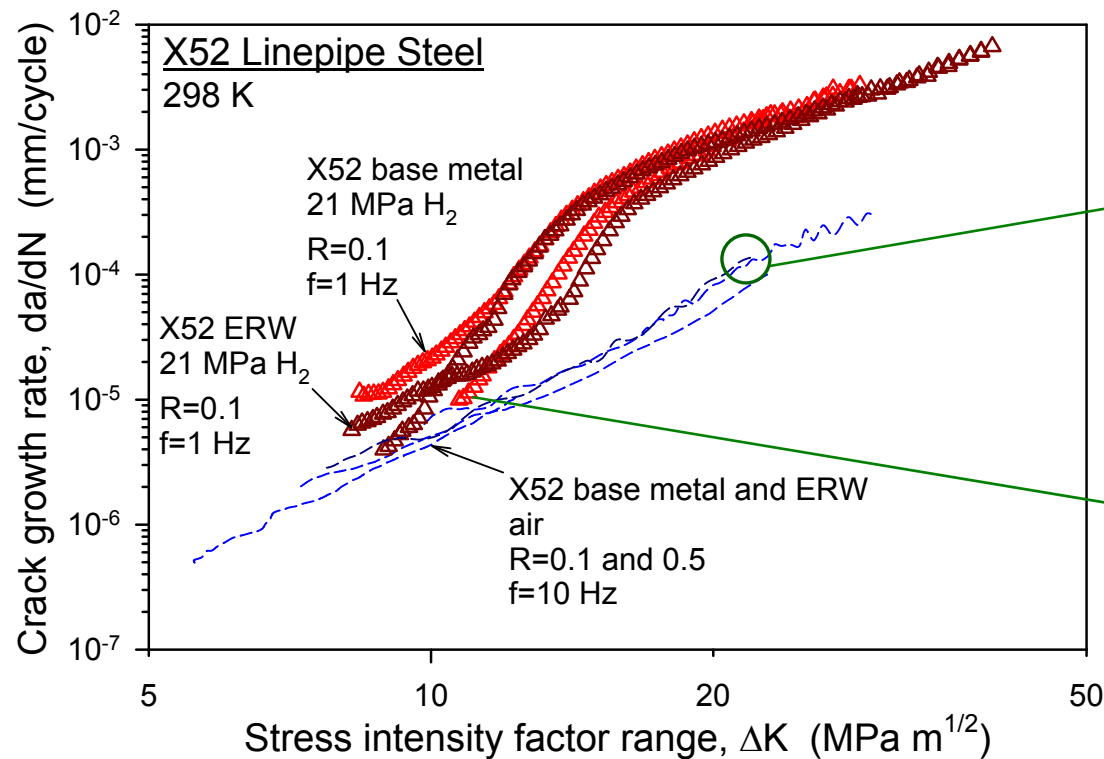
# Fatigue crack growth laws can be used to evaluate reliability/integrity of X52 H<sub>2</sub> pipelines



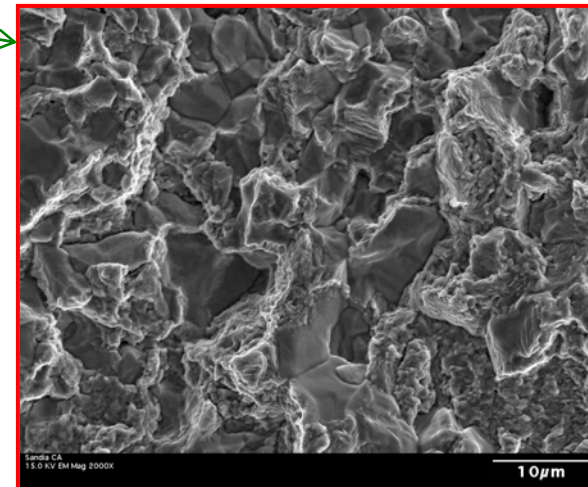


Accomplishment:

# Examined fracture surfaces from fatigue crack growth specimens



ERW  
air



base  
metal  
H<sub>2</sub>

- Base metal in H<sub>2</sub> exhibits intergranular fracture at low  $\Delta K$
- ERW in air (R=0.5) exhibits unstable fracture at  $K_{\max} \sim 40 \text{ MPa m}^{1/2} \rightarrow$  cleavage





# Collaborations

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- DOE Pipeline Working Group (PWG)
  - Participants funded by DOE FCT Program
    - Federal Labs: Sandia, Oak Ridge, Savannah River
    - Universities: Univ. of Illinois
    - Industry: Secat
  - Participants not funded by DOE FCT Program
    - Federal Labs: NIST
    - Industry: industrial gas companies, ExxonMobil
    - Standards Development Organizations: ASME
  - Extent of collaborations include:
    - PWG meetings (up to 2 times/year) for participants to report results and receive feedback
    - Leveraging resources for testing (e.g., Secat-Sandia)
    - Supplying materials (e.g., ExxonMobil-Sandia)
    - Coordinating testing (e.g., NIST-Sandia)



# Proposed Future Work

## Remainder of FY11

- Expand evaluation of X52 seam weld to understand implication of cleavage fracture
- Determine threshold level of  $O_2$  to inhibit accelerated fatigue crack growth of X52 steel in 21 MPa  $H_2$  gas
- Measure fatigue crack growth law of girth weld fusion zone in  $H_2$  gas



## FY12

- Measure fatigue crack growth law of girth weld heat-affected zone (HAZ) in  $H_2$  gas
- Evaluate effects of load-cycle frequency on  $O_2$  inhibition of  $H_2$ -accelerated fatigue crack growth



# Summary

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- Measured fracture thresholds and fatigue crack growth laws allow evaluation of reliability/integrity of steel H<sub>2</sub> pipelines
  - Hydrogen embrittlement accommodated by measuring fracture properties in H<sub>2</sub> following ASME B31.12 design standard
- Measurements on X52 steel reveal the following trends:
  - Fracture thresholds of base metal in H<sub>2</sub> (~80-100 MPa m<sup>1/2</sup>) are favorable for pipeline reliability/integrity
  - Fatigue crack growth laws for base metal and seam weld are similar in H<sub>2</sub>
  - Unstable cleavage fracture observed in seam weld at  $K_{\max} \sim 40$  MPa m<sup>1/2</sup> during fatigue crack growth testing in air



# Reviewer-Only Slides



# Responses to Previous Year Reviewers' Comments

1. *“Testing of the fatigue growth rates of welds is an appropriate next step. Future testing should be focused toward understanding the effects of pressure cycling on steel pipelines in hydrogen operation”; “Plans are clear for the scope of work and the overall program objectives. There is a need to focus more on newer high-strength pipeline steels that are common today and not what was common 40 years ago. Focus on X-70 thru X-100 type steels.”*

Materials included in this testing effort are determined based on input from hydrogen pipeline stakeholders, e.g., the industrial gas companies and ASME. The current emphasis on X52 resulted from input provided by these stakeholders. Sandia has conducted some testing on X100 in this project during past years. In addition, over the past 2 years, Sandia has tested X80 steels in collaboration with Secat. Results from the latter testing on X80 are reported in the Pipeline Working Group meetings and in the Secat presentation at the AMR.

2. *“The project investigator needs to coordinate his research and development efforts with members of the oil and gas pipeline industry.”*

Collaborations with the oil and gas industry, e.g., ExxonMobil, have been established over the past year. The girth welds included in the testing plans for FY11 and FY12 were supplied by ExxonMobil.

3. *“The amount of reliable data that can be obtained in this project is limited because there is only one test apparatus.”*

Sandia has recognized the need for more testing capacity and is currently developing an additional test apparatus for conducting fatigue crack growth tests in hydrogen gas. This effort is enabled by leveraging funds from the Fuel Cell Technologies program and National Nuclear Security Administration, but the status of these funds in FY11 and FY12 is uncertain.



# Critical Assumptions and Issues

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1. Activities are focused on measuring 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 in hydrogen must probe lower load-cycle frequencies, since non-conservative fatigue crack growth rates may be measured at higher frequencies. Consequently, test durations in hydrogen gas can be rather extended, e.g., as much as 7 days at 1 Hz. 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 members of the PWG such as NIST, which operates one of the other specialized laboratories for measuring fracture properties of materials in hydrogen gas.
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 some materials through our interactions with stakeholders, e.g., ExxonMobil. We must maintain and expand relationships with stakeholders so that we continue to have access to materials. These interactions are fostered through participation of stakeholders in the DOE Pipeline Working Group.





# Publications and Presentations

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1. “Microstructure and Mechanical Property Performance of Commercial Grade API Pipeline Steels in High Pressure Gaseous Hydrogen”, D. Stalheim, T. Boggess, C. San Marchi, S. Jansto, B. Somerday, G. Muralidharan, and P. Sofronis, *Proceedings of IPC 2010 8th International Pipeline Conference*, Calgary, Alberta, 2010, Paper No. IPC2010-31301
2. “Fracture Toughness and Fatigue Crack Growth of X80 Pipeline Steel in Gaseous Hydrogen”, C. San Marchi, B. Somerday, K. Nibur, D. Stalheim, T. Boggess, and S. Jansto, *Proceedings of the ASME 2011 Pressure Vessels & Piping Division / K-PVP Conference (PVP11)*, Baltimore, MD, 2011, Paper No. PVP2011-57684
3. (invited) “Improving the Fatigue Resistance of Ferritic Steels in Hydrogen Gas”, B. Somerday, I<sup>2</sup>CNER Kick-off Symposium, Fukuoka, Japan, Feb. 2011