Materials Compatibility

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June 9, 2010

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Overview

Timeline

- Project start date Oct. 2003
- Project end date Sept. 2015
- Percent complete 58%

Budget

- Total project funding (to date)
  - DOE share: $4.8M
- FY09 Funding: $1.0M
- FY10 Funding: $0.8M

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
  - FIBA Technologies, Plug Power
  - DOE Pipeline Working Group
  - HYDROGENIUS (AIST/Kyushu University, Japan)

Objectives/Relevance

- Enable *market transformation* through development and application of standards for H_2 components
  - Create materials reference guide ("Technical Reference") and identify material property data gaps
  - Execute materials testing following existing standards to meet immediate needs for data in technology deployment
    - Emphasis in FY09-FY10 on steel hydrogen storage tanks
  - Provide data that demonstrates how to improve efficiency and reliability of materials test methods in standards
    - Emphasis in FY09-10 on fatigue crack growth test methods
- Participate directly in standards development
  - Component/system design qualification standards
    - ASME Article KD-10, CSA HPIT1, SAE J2579
  - Materials testing standards
    - SAE/CSA

Approach

- Apply expertise and resources in materials compatibility to implement and improve standards for H_2 components
  - Sustain relationships with stakeholders (industry, SDOs) to identify needs in technology deployment and code development
  - Exploit unique laboratory capability for conducting material tests in high-pressure (>100 MPa) hydrogen gas
    - Generate data following existing standards (e.g., ASME Article KD-10 tank standard) to meet immediate needs for design qualification
    - Evaluate limitations of materials test standards (e.g., long test durations for fatigue crack growth) and develop improvements
  - Ensure data and technical perspectives are effectively communicated to stakeholders (industry, SDOs)
    - Materials guide ("Technical Reference") available on web site
    - Reports with material data distributed directly to stakeholders
    - Sandia technical staff serve on standards development committees

Approach: Milestones

- Compare cracking threshold to fracture toughness measurements for vessel steels in H_2 (complete)
- Draft of report on fatigue crack growth and cracking thresholds of SA372 Gr. J steel in H_2 (in progress)
- Quantify the effects of load cycle frequency on fatigue of steels in high-pressure H_2 (in progress)
- Add/update chapters on nickel-based alloys (complete) and ferritic steels (in progress) in “Technical Reference”

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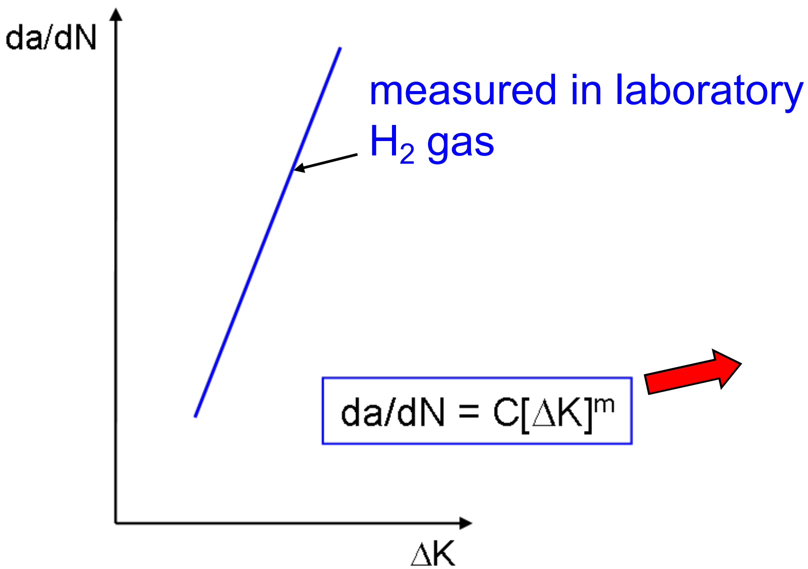
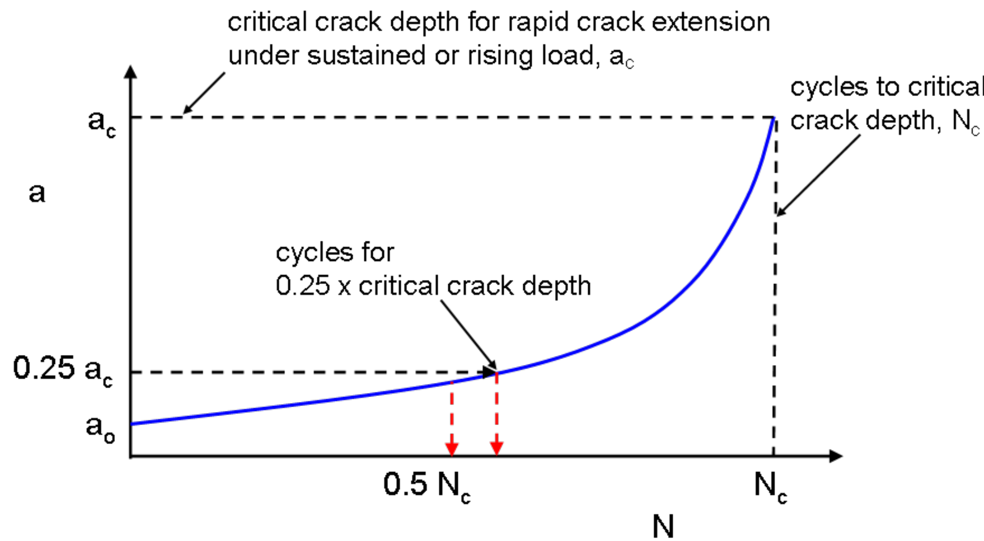
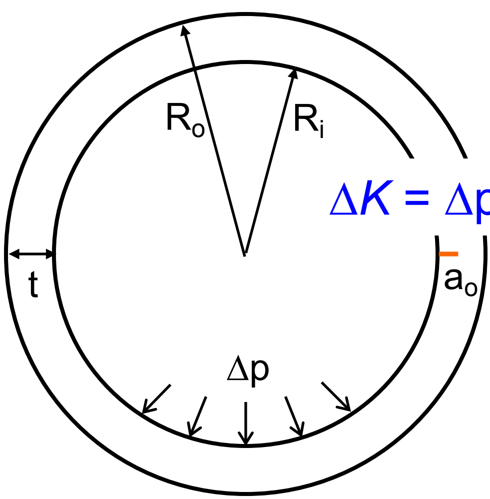
Approach: Enable application and revision of ASME Article KD-10







$\Delta K = \Delta p[f(a, t, R_o, R_i)]$

- Engineering analysis design method requires material fracture properties in H_2
  - Fracture threshold
  - Fatigue crack growth law
- Standard applies to metal hydrogen storage tanks

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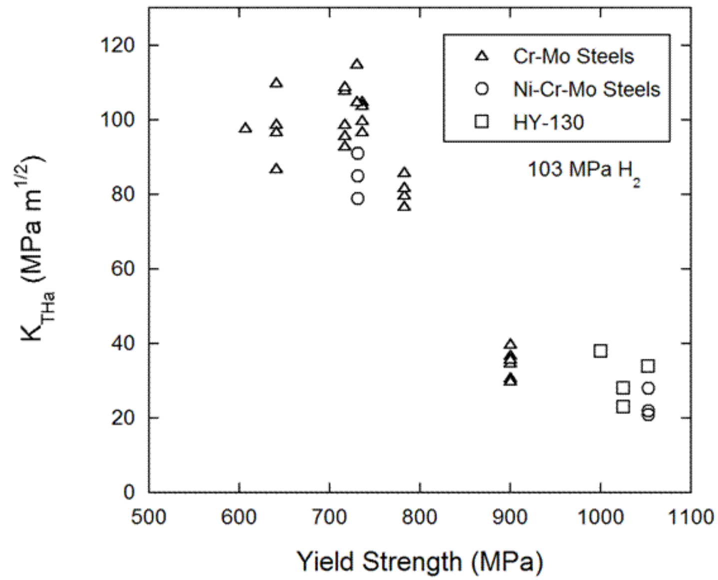
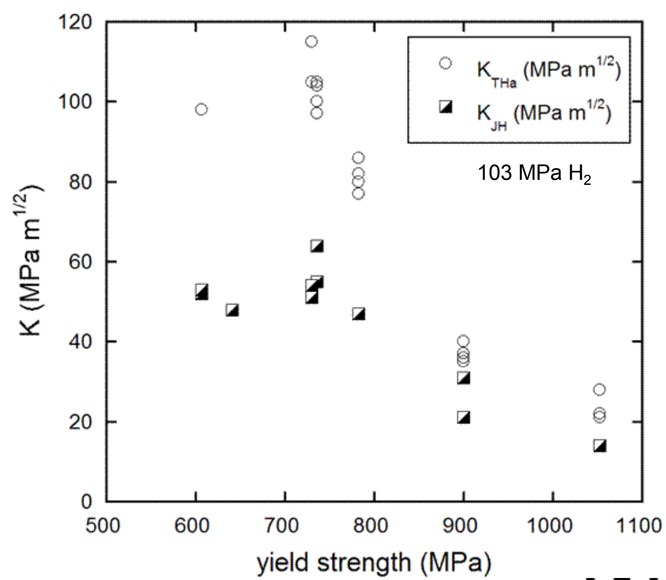
Accomplishment:

Completed report on fracture threshold measurements of tank steels in H_2

- Comprehensive summary of test methods and fracture data
- Steels provided by industry partner
- Only modern study of fracture thresholds for pressure vessel steels in high-pressure H_2
- Report distributed to stakeholders (industry, ASME)

Data have enabled revision of ASME KD-10 tank standard





Accomplishment:

Materials testing to meet immediate needs for market transformation: stationary tanks

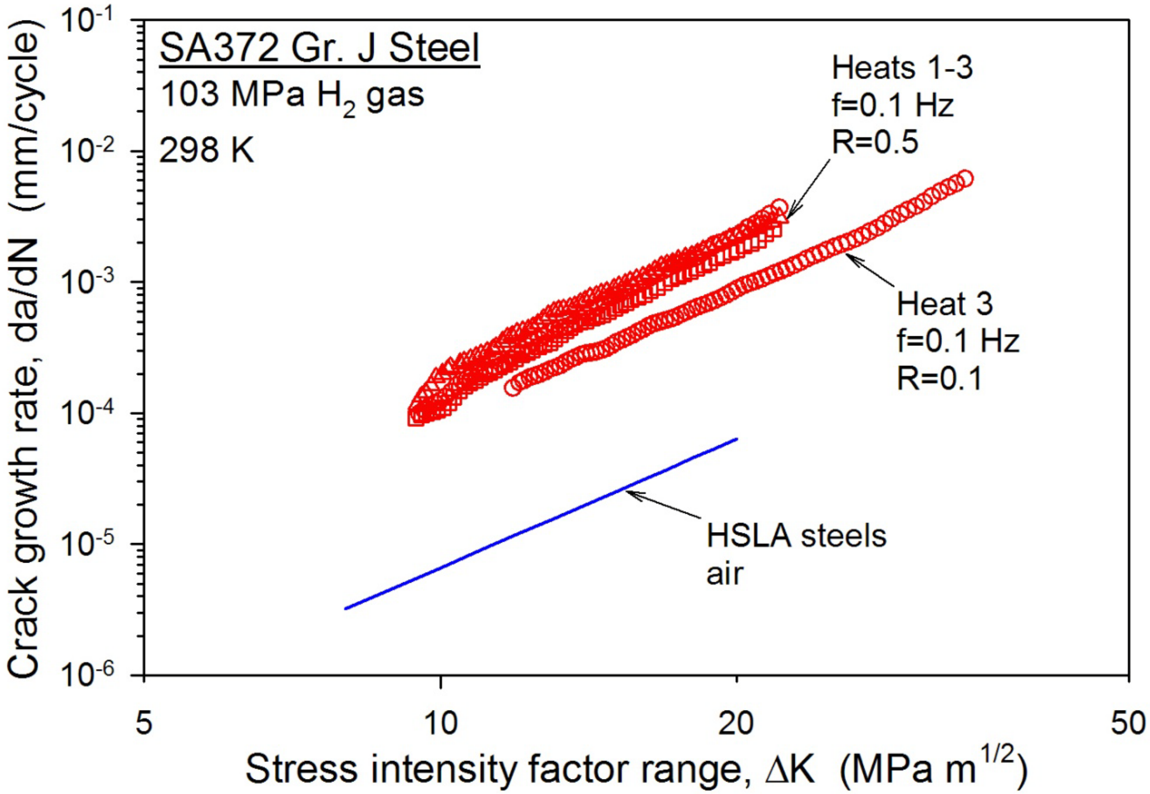
- Tank steels provided by industry partner

Steel	S_y (MPa)	H_2 pressure (MPa)	Test frequency (Hz)	Load ratio	Status
SA372 Gr. J Heat 1	642	103	0.1	0.2	Complete
		103	0.1	0.5	
		103	0.1	0.8	
SA372 Gr. J Heat 2	731	103	0.1	0.2	Complete
		103	0.1	0.5	
		103	0.1	0.8	
SA372 Gr. J Heat 3	784	103	0.1	0.2	Complete
		103	0.1	0.5	Complete
		103	0.1	0.8	

- Fatigue testing follows current ASME KD-10
- Test frequency of 0.1 Hz leads to long test durations

Accomplishment:

Results from completed fatigue tests provided to industry partner



- Data provides crack growth rate laws, $da/dN=C[\Delta K]^m$
- Data coupled with structural analysis allows quantification of cycle life

- Industry partner using data to produce ASME-qualified stationary tank at H_2 refueling station
- Initial application of ASME Article KD-10 indicates path for optimization

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Accomplishment:

Materials testing to meet immediate needs for market transformation: forklift tanks

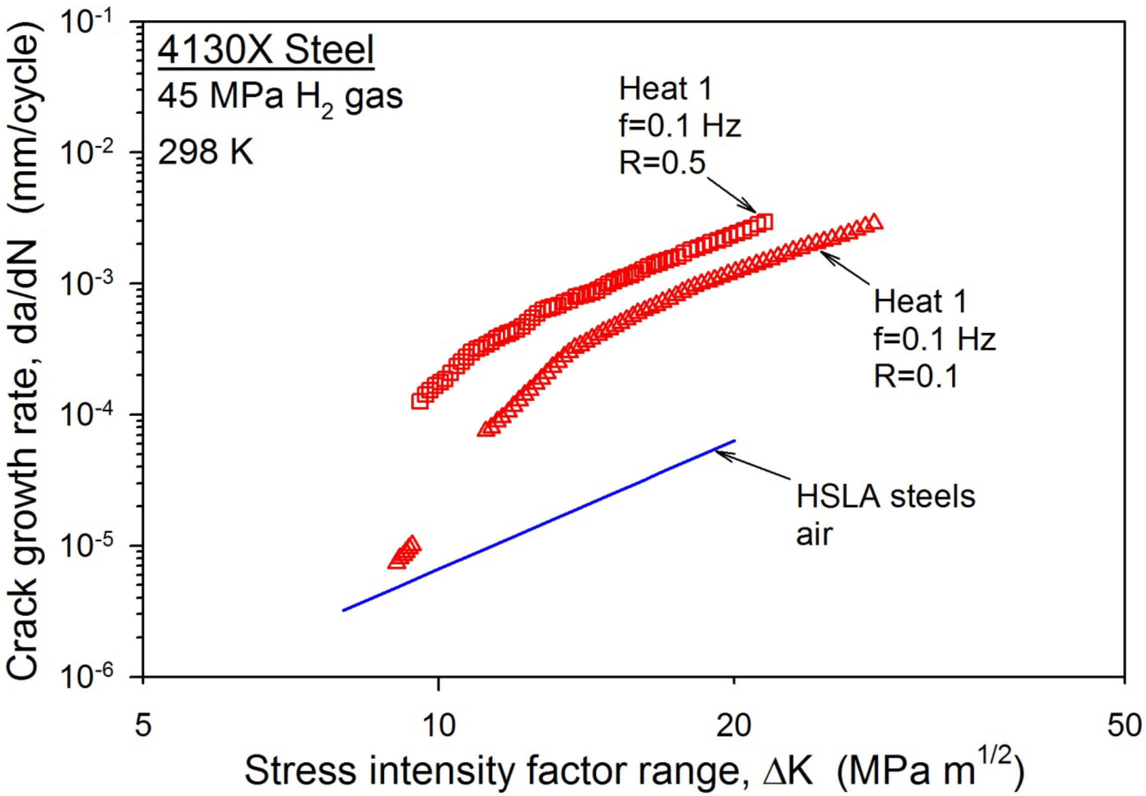
- Tank steels provided by 3 different industry partners

Steel	S_y (MPa)	H_2 pressure (MPa)	Test frequency (Hz)	Load ratio	Status
4130X Heat 1	600	45	0.1	0.1	Complete
		45	0.1	0.5	Complete
4130X Heat 2	768	45	0.1	0.1	Complete
		45	0.1	0.5	Complete
4130X Heat 3	543	45	0.1	0.1	
		45	0.1	0.5	

- Fatigue testing follows current ASME KD-10
- Test frequency of 0.1 Hz leads to long test durations

Accomplishment:

Results used in cycle-life analyses and code development for forklift tanks



- Data provides crack growth rate laws, $da/dN=C[\Delta K]^m$
- Data coupled with structural analysis allows quantification of cycle life

- Crack growth rate laws are critical input to analysis of results from DOE/Sandia tank testing activity
- Initial application of ASME Article KD-10 indicates path for optimization

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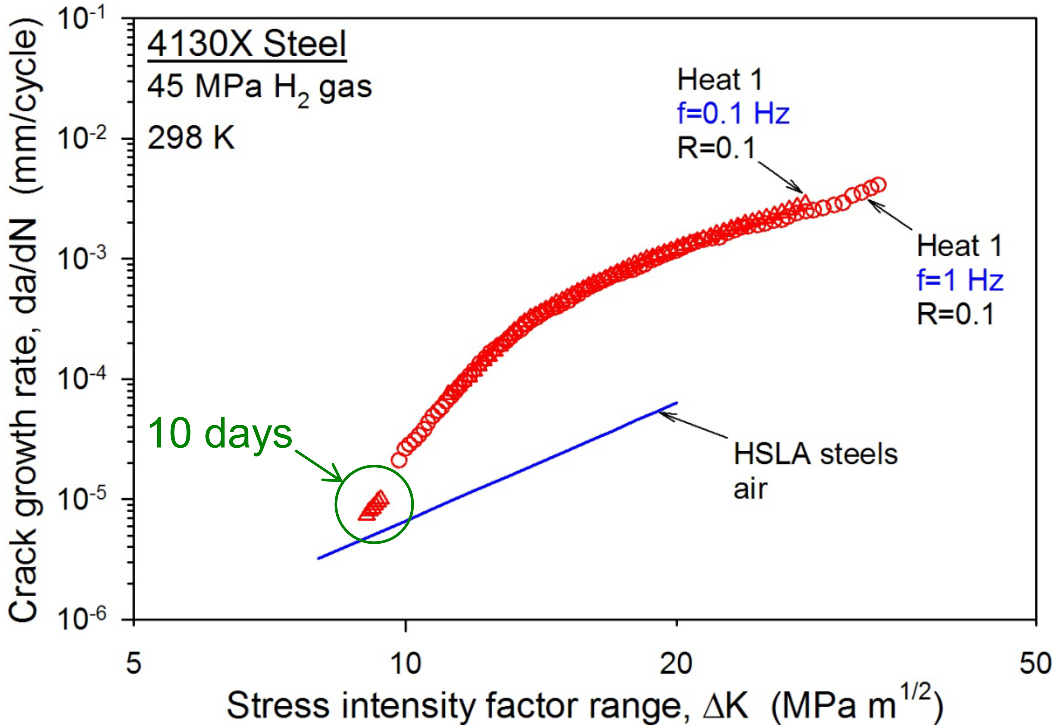
Test method improvements needed to enhance efficiency and reliability of data generation



Physics-based relationship may help optimize test frequency:

$$f \sim D_{eff} / (da/dN)^2$$

f = test frequency

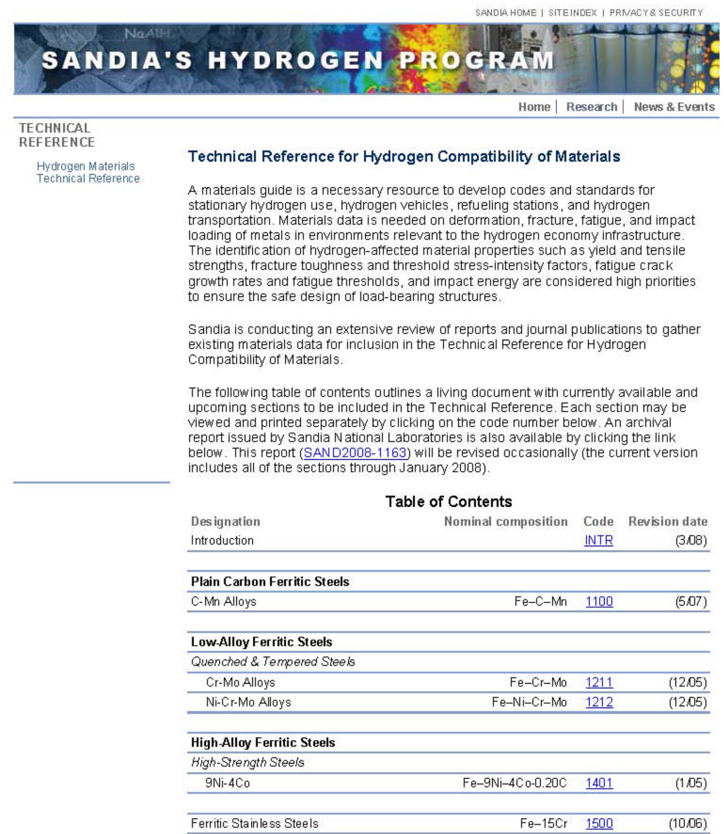
D_{eff} = effective hydrogen diffusivity

- Test frequency currently in ASME KD-10 (0.1 Hz) leads to impractical test durations
- Efforts in progress to establish test frequencies that shorten test duration without compromising data quality

Accomplishment:

Updated "Technical Reference for Hydrogen Compatibility of Materials"

- Data from technical literature and Sandia materials testing
  - 23 material-specific chapters
  - Content shaped by input from stakeholders (industry, SDOs)
- Additional chapters in FY09-FY10
  - Single-phase nickel alloys
  - Precipitation-strengthened nickel alloys
- Future chapters
  - Update ferritic steels with Sandia data
- www.ca.sandia.gov/matlsTechRef



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TECHNICAL REFERENCE

Hydrogen Materials Technical Reference

Technical Reference for Hydrogen Compatibility of Materials

A materials guide is a necessary resource to develop codes and standards for stationary hydrogen use, hydrogen vehicles, refueling stations, and hydrogen transportation. Materials data is needed on deformation, fracture, fatigue, and impact loading of metals in environments relevant to the hydrogen economy infrastructure. The identification of hydrogen-affected material properties such as yield and tensile strengths, fracture toughness and threshold stress-intensity factors, fatigue crack growth rates and fatigue thresholds, and impact energy are considered high priorities to ensure the safe design of load-bearing structures.

Sandia is conducting an extensive review of reports and journal publications to gather existing materials data for inclusion in the Technical Reference for Hydrogen Compatibility of Materials.

The following table of contents outlines a living document with currently available and upcoming sections to be included in the Technical Reference. Each section may be viewed and printed separately by clicking on the code number below. An archival report issued by Sandia National Laboratories is also available by clicking the link below. This report (SAND2008-1163) will be revised occasionally (the current version includes all of the sections through January 2008).

Table of Contents

Designation	Nominal composition	Code	Revision date
Introduction		INTR	(3/08)
Plain Carbon Ferritic Steels			
C-Mn Alloys	Fe–C–Mn	1100	(5/07)
Low-Alloy Ferritic Steels			
Quenched & Tempered Steels			
Cr-Mo Alloys	Fe–Cr–Mo	1211	(12/05)
Ni-Cr-Mo Alloys	Fe–Ni–Cr–Mo	1212	(12/05)
High-Alloy Ferritic Steels			
High-Strength Steels			
9Ni-4Co	Fe–9Ni–4Co-0.20C	1401	(1/05)
Ferritic Stainless Steels	Fe–15Cr	1500	(10/06)

Data used for materials selection in technology design and for standards development

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Collaborations

- Standards Development Organizations (SDOs)
  - Examples: ASME, CSA, ISO
  - Sandia technical staff serve on committees
  - SDOs communicate needs for materials test methods and data
  - SDOs receive data through “Technical Reference” and reports
- Tank manufacturers and forklift integrators
  - Examples: FIBA Technologies, Plug Power
  - Industry partners provide materials from market transformation technologies
  - Industry partners receive materials test data through reports
- DOE Pipeline Working Group (PWG)
  - Exchange data and ideas through participation in PWG meetings
- HYDROGENIUS (AIST/Kyushu University, Japan)
  - Exchange data and ideas for developing international standards

Future Work

Remainder of FY10

- Complete and distribute report on results from fatigue crack growth and cracking threshold testing of SA372 Gr. J tank steel in H_2
- Complete fatigue crack growth testing of forklift tank steels in H_2 to provide data for tank testing activity and enable standards development
- Establish optimum load cycle frequencies for fatigue crack growth testing of steels in high-pressure H_2 to enable standards revision
- Update "Technical Reference" chapters on ferritic steels with Sandia data
- Complete first phase of materials qualification standard development for vehicle components (i.e., fittings, regulators, etc.) in collaboration with international partners

FY11

- Conduct testing on additional materials (e.g., aluminum) that impact market transformation
- Complete materials qualification standard development for vehicle components (i.e., fittings, regulators, etc.)

Summary

- Completed report on fracture threshold measurements of tank steels in H_2 gas
  - Data have enabled revision of ASME KD-10 tank standard
- Conducting fatigue crack growth testing on tank steels in H_2 gas to meet immediate needs for market transformation
  - Two products: refueling station tanks and forklift tanks
  - Data for forklift tank steels also needed for DOE/Sandia tank testing activity
- Application of ASME Article KD-10 standard indicates path for optimization
  - Establishing fatigue crack growth test frequencies that shorten test duration without compromising data quality

Lifecycle Analysis of Forklift Tanks

Daniel Dedrick

Brian Somerday

Sandia National Laboratories

June 9, 2010

Project ID # SCS005

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Overview

Timeline

- Project start date Jan. 2010
- Project end date Sept. 2010
- Percent complete ~50%

Budget

- Total project funding (to date)
  - DOE share: $1.2M
- FY09 Funding: none

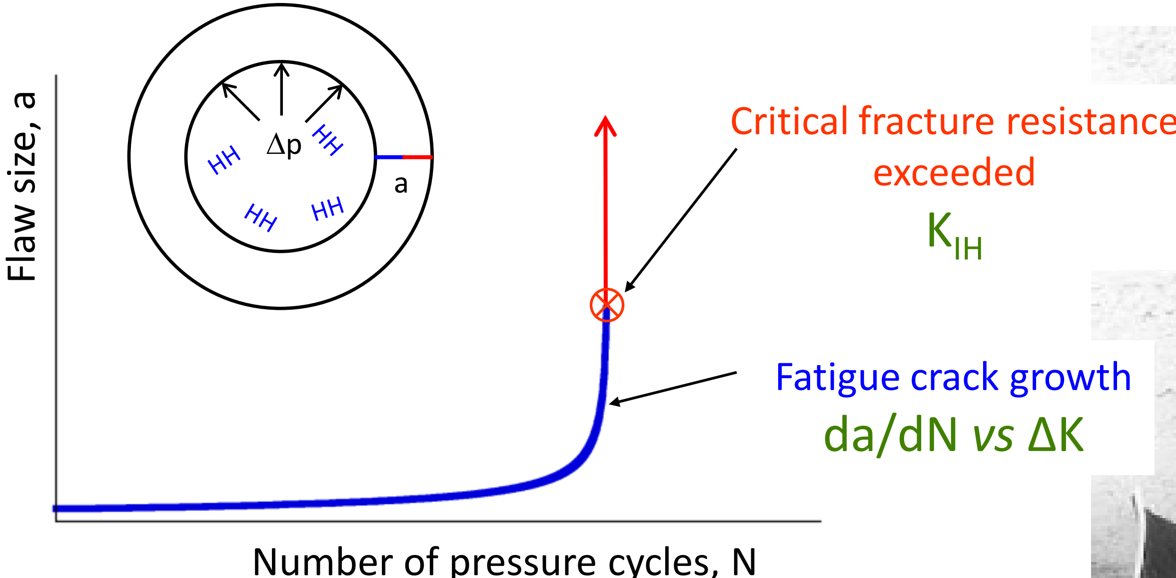
Barriers & Targets

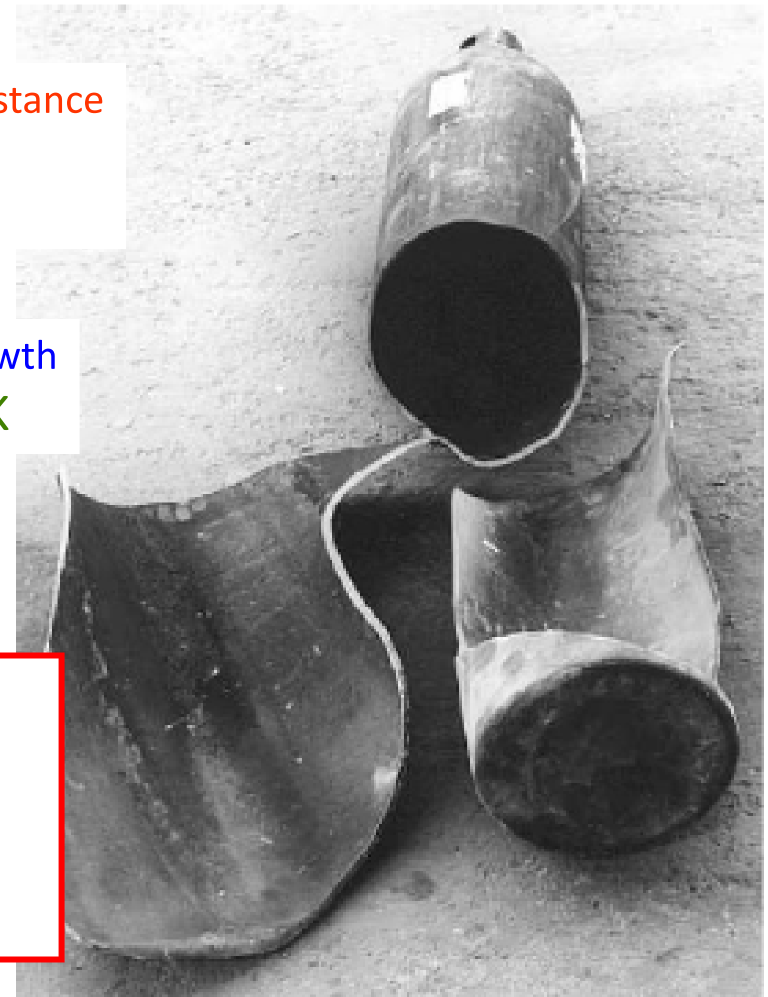
- Barriers and targets addressed
  - Materials reference guide for design and installation
  - Hydrogen storage tank standards for portable, stationary and vehicular use
  - Insufficient technical data to revise standards

Partners

- Collaborators:
  - CSA (HPIT1 working group)
  - Nuvera
  - Plug Power
  - Norris Cylinder

Problem: hydrogen accelerates fatigue crack growth *of existing defects*





Two modes of crack growth facilitated by gaseous hydrogen:

- Crack growth under cyclic stress
- Critical crack propagation

Ref.: *Barthélémy, 1st ESSHS, 2006*

Objectives & Relevance

- Provide technical basis for the development of standards defining the use of steel (type 1) storage tanks with existing defects
  - **Engineering Analysis Method**: validate fracture mechanics-based design approach in ASME BPVC Sec VIII, Div 3, Article KD-10
  - **Performance Evaluation Method**: provide data to help determine if time for crack initiation can be reliably credited in design qualification process
  - Quantify failure characteristics, such as *leak-before-break*
- Participate directly in standards development
  - Component design standards
    - ASME BPVC Sec VIII, Div 3, Article KD-10
    - CSA Hydrogen-Powered Industrial Trucks (HPIT1) working group

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Approach

- Applied research
  - Build infrastructure for accelerated pressure cycling of steel hydrogen storage tanks
  - Characterize crack initiation and growth in ***two tank designs*** that are currently in use
  - Manufacture tanks with ***engineered defects*** to quantify effects of existing flaws
  - Validate structural analysis tools and existing design methodologies using ***tank testing and materials testing***
- Standards development activities
  - Provide technical basis for defining the use of steel tanks in CSA HPIT1 working group

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Approach: Testing will characterize crack initiation and growth

Tank condition	*Objective*	*Tank Design*	*Max # of tanks*
As-manufactured	Baseline Crack initiation	A	4
		B	3
Engineered Defects	Crack initiation	A	4
		B	3
Engineered Defects	Validation of Engineering Analysis	A	3
		B	2
Additional testing / Engineered Defects	Effect of pressure	A	1
		B	1

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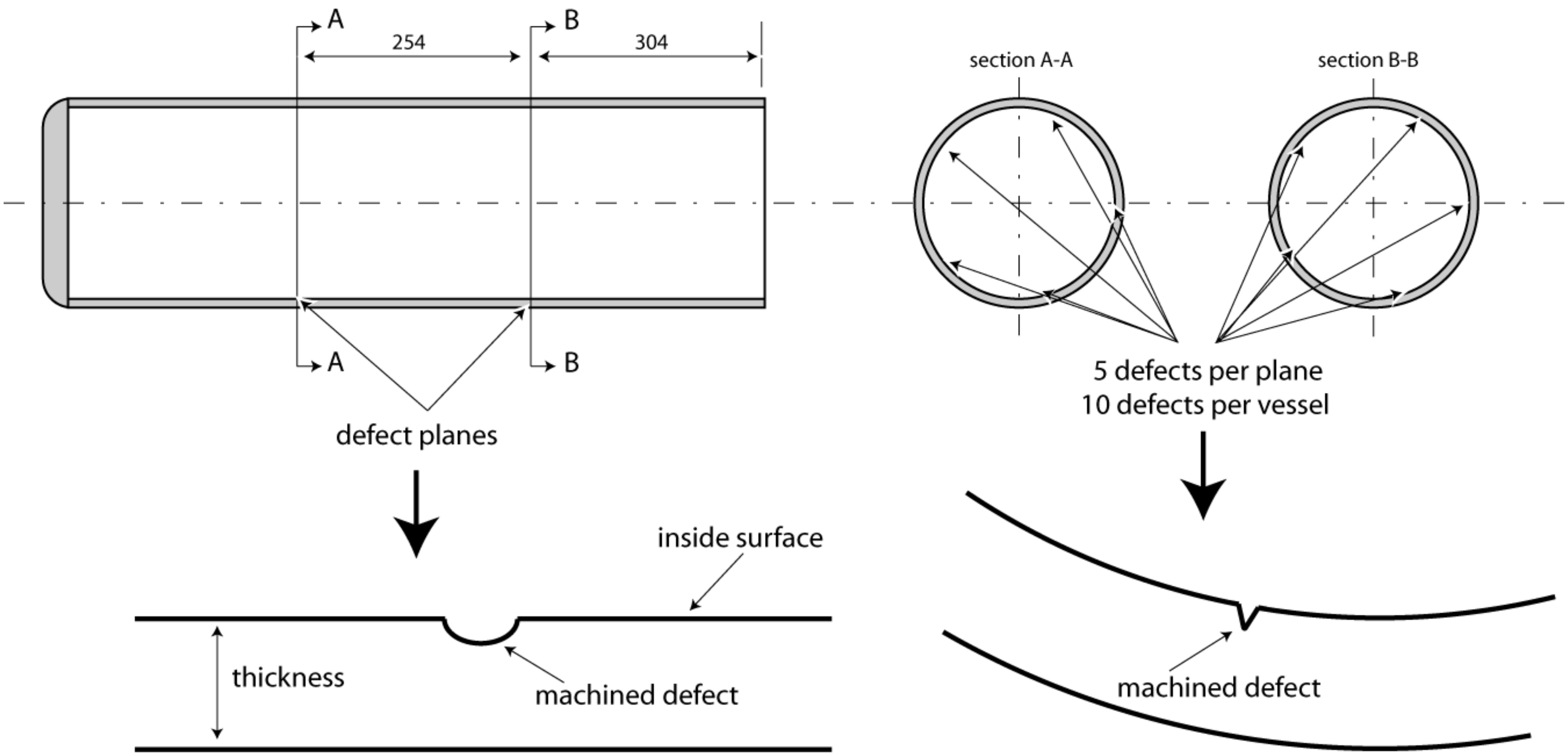
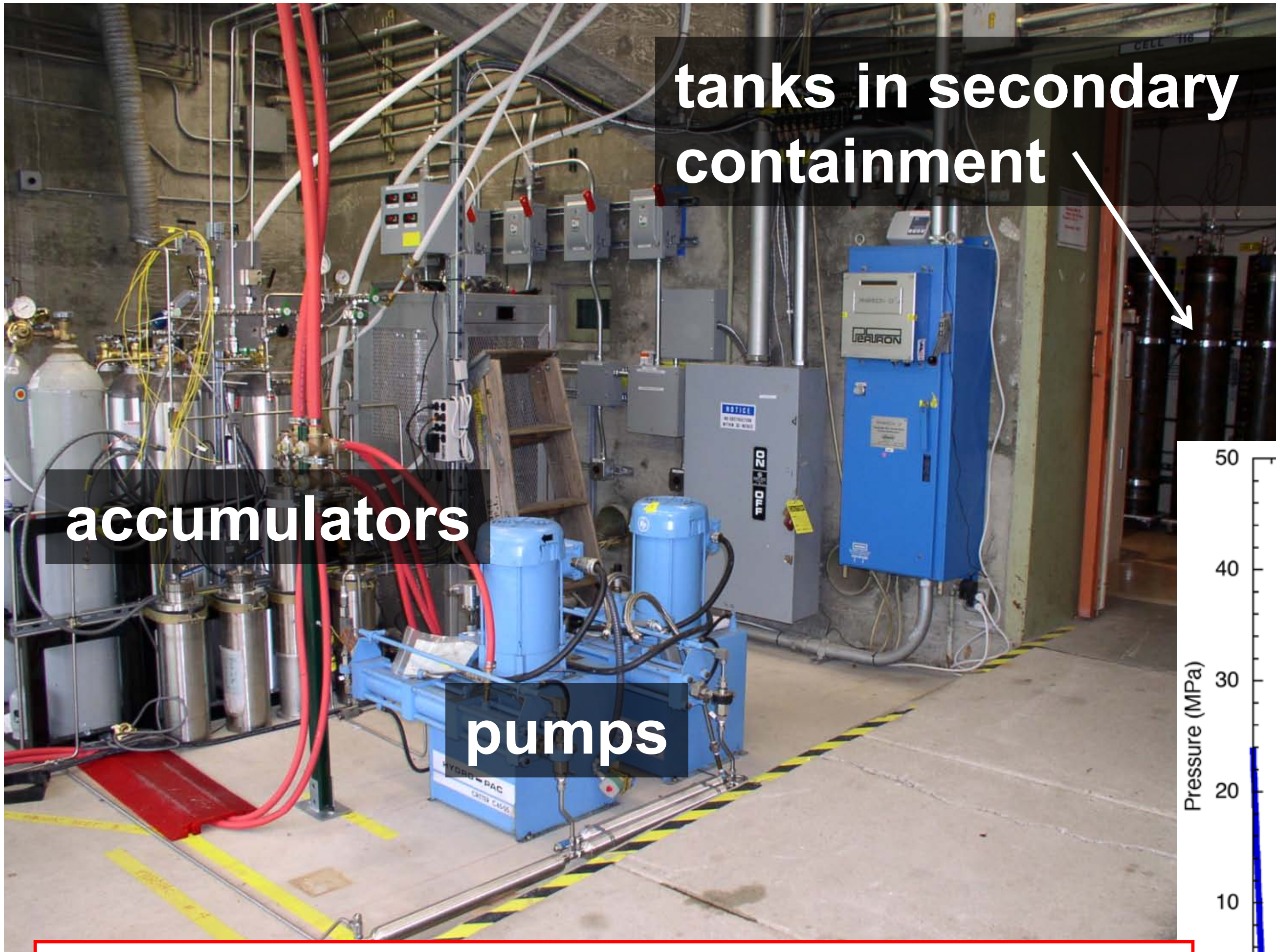
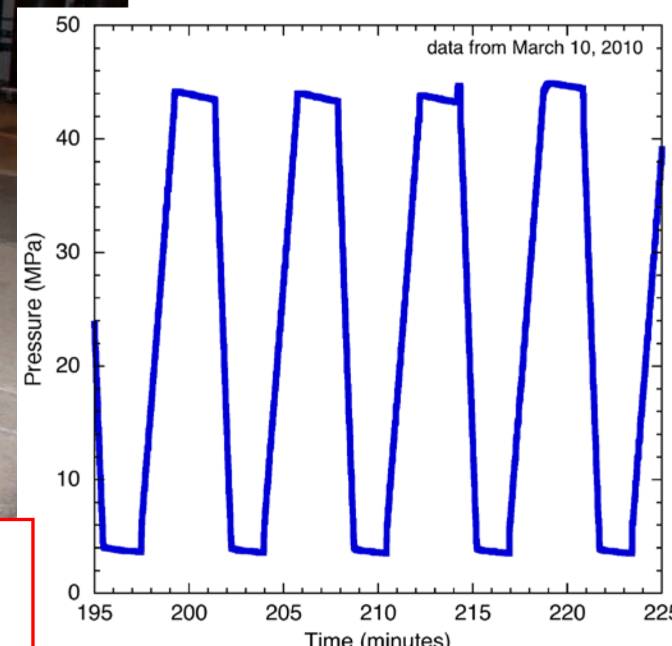
Approach: Engineered defects allow quantitative evaluation of design methods



Accomplishment: Infrastructure for tank cycling has been operating for 3 months



- Periodic pressure profile
- Monitored high-purity gaseous hydrogen
- Closed system pressurizing up to 10 tanks in parallel



Tests are accelerated, but remain relatively long: 10,000 cycles ~ 45 days

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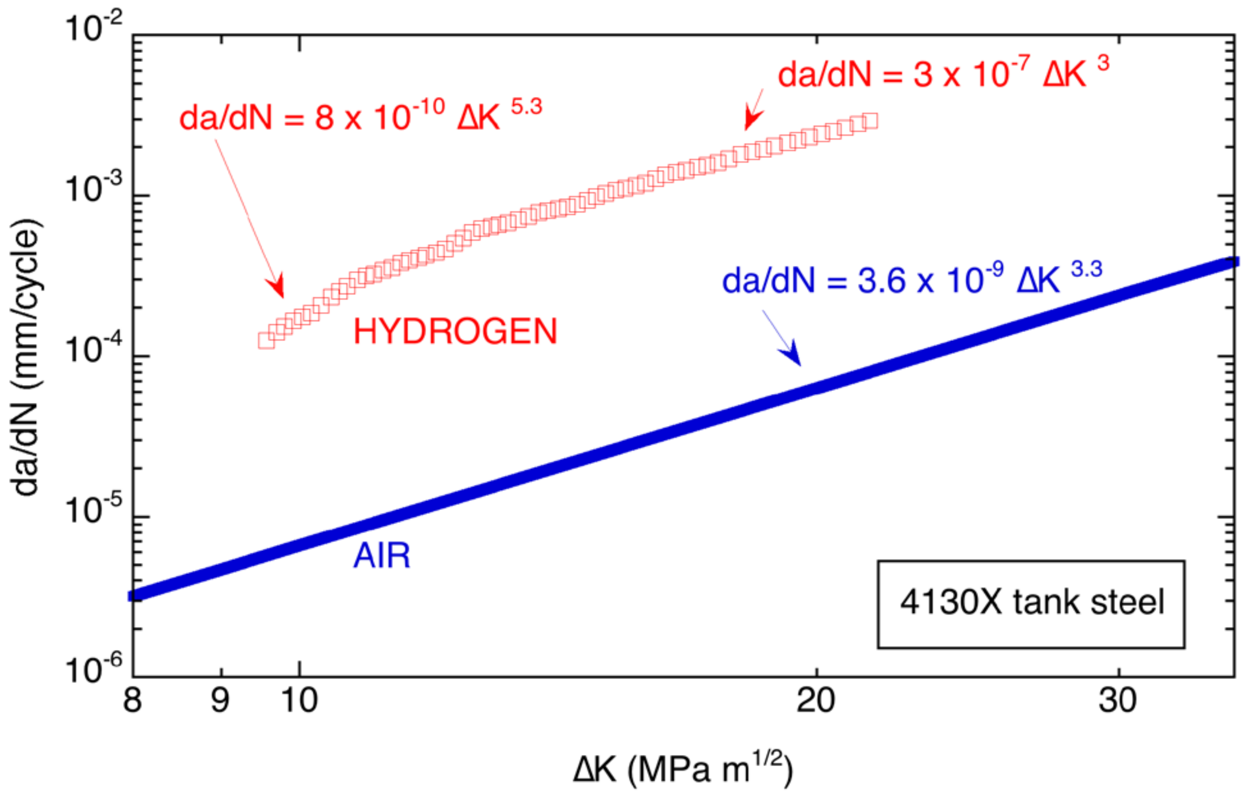
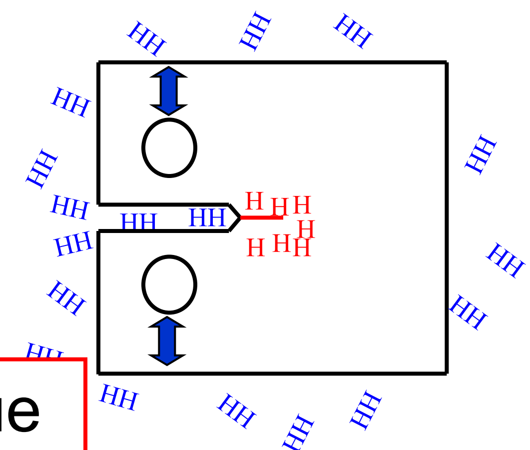
Current status of tank testing program

- Test matrix defined with aid of HPIT1 working group
- As-manufactured tanks
  - 3 tanks have been exposed to 2,500 cycles as of April 9th (tank A design)
  - Expect 220 cycles per day, >10,000 cycles by AMR
- Tanks with engineered defects
  - Tank A: Sixteen (16) tanks have been machined; replicate tanks for each test condition
  - Tank B: Six (6) tanks have been machined
  - Significantly fewer cycles are expected for tanks with engineering defects
- Materials testing
  - Fatigue crack growth rates have been measured for tank B material

Materials studies are necessary for quantifying tank testing results



- Procedures ≈ ASTM E647
- Constant load amplitude (ΔK increasing)
- triangle waveform
- P_{H2} = 45 MPa
- Yield strength = 600 MPa



Materials compatibility studies provide fatigue crack growth laws for engineering analysis

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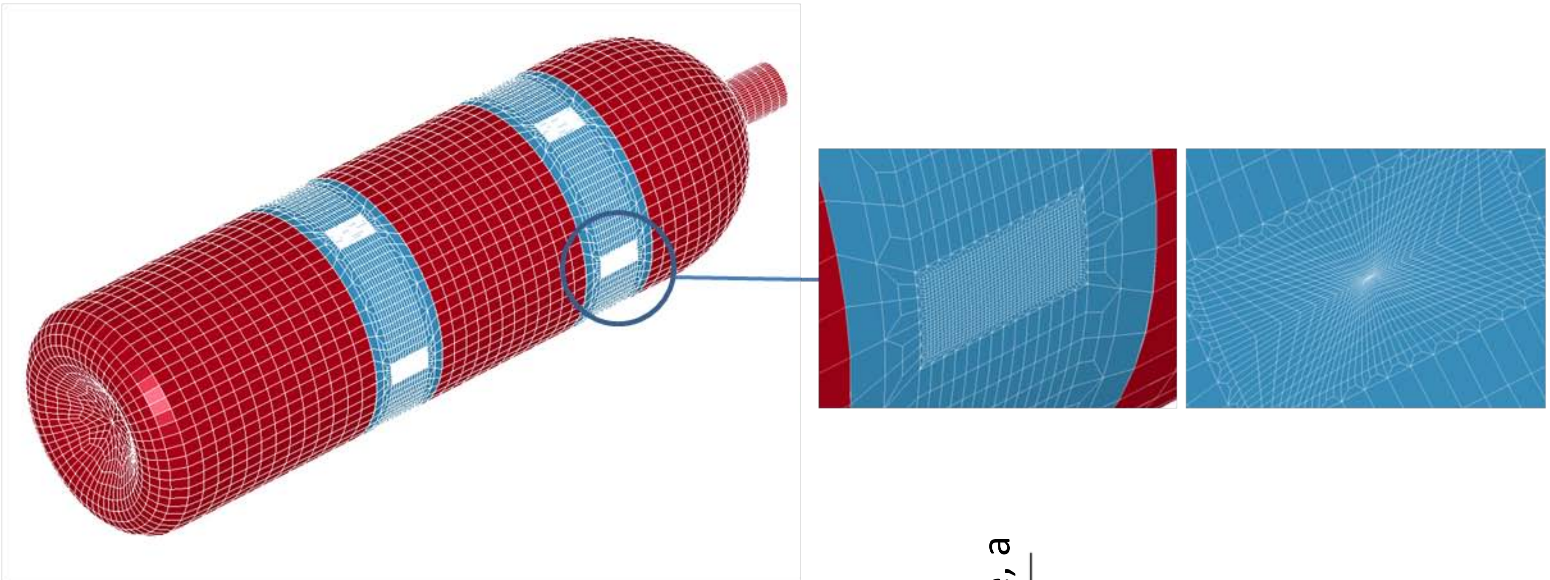
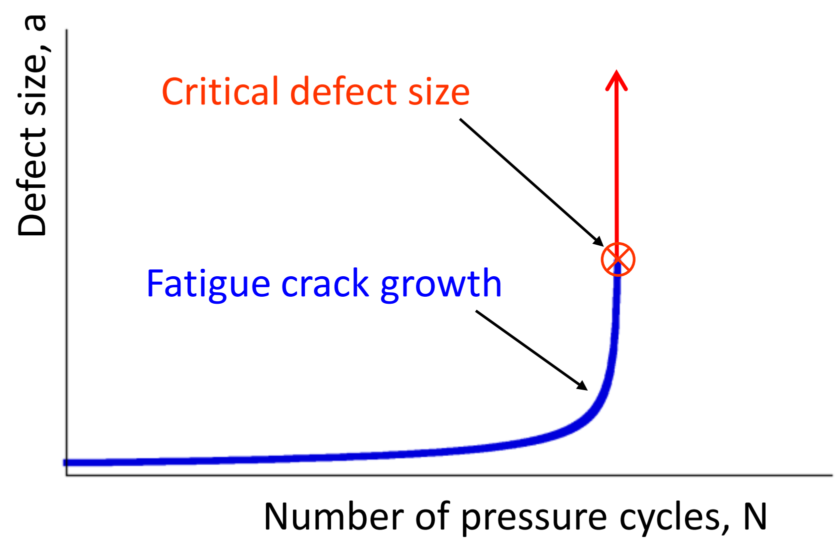
Structural engineering analysis provides predictive capability



- Defects of a specified geometry are assumed to exist in the cylinder
- The stress intensity factor associated with the defect is determined based on fracture mechanics, $K_I = f(a, c, t, \text{pressure})$
- Crack growth is calculated using materials data



Collaborators

- CSA working group: Compressed Hydrogen Powered Industrial Truck On-board Fuel Storage and Handling Components (HPIT1)
  - Peer-review of testing plan and guidance on relevance
  - Coordinated with UL activities
- US Fuel Cell Council: Fork Lift Task Force
  - Peer-review of testing plan and guidance on relevance
- Tank manufacturers
  - Production of tanks
  - Coordination of production/machining of engineered defects
- Two system integrators for lift trucks: Nuvera & Plug Power
  - Peer-review of testing plan and guidance on relevance to lift truck application as well as standards development

Future Work

Remainder of FY10

- Complete cycling of as-manufactured tanks and tanks with engineered flaws until failure
- Complete benchmark fatigue crack growth testing of 3 heats of 4130X (shared activity with materials compatibility effort)
- Quantify number of cycles for initiation and growth as well as size and distribution of engineered and “natural” defects
- Validate structural engineering tools and existing design methodology, in particular ASME VIII.3.KD-10 and *leak-before-break* criteria
- Communicate results to CSA HPIT1 working group

FY11

- Current program ends Nov 2010
- Follow-on testing will be identified and prioritized based on results and input from CSA HPIT1 working group

Summary

Standards for steel hydrogen tanks that experience a large number of cycles is currently missing,
e.g. Hydrogen-Powered Industrial Trucks

- This program will provide the technical basis addressing this need within the scope of CSA HPIT1
  - Infrastructure for accelerated pressure cycling of tanks has been constructed
  - Targeted pressure cycles have been demonstrated for 3 tanks in parallel
  - Tanks with engineered defects are being machined
  - Materials testing to support validation of design methods is nearing completion as part of ***materials compatibility*** effort
  - Structural analysis tools for both tank designs have been generated

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