

H₂ Permeability and Integrity of Steel Welds

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Overview

Timeline

- Start – March 2004
- Finish – September 2011
- 40% Complete

Budget

- Total Project Funding
 - DOE share: \$815,000
 - Contractor share: N/A
- Funding received in FY08:
 - \$300,000
- Funding for FY09: \$0

Barriers Addressed

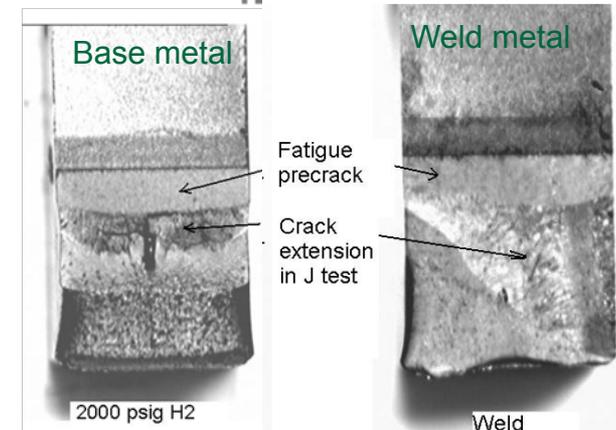
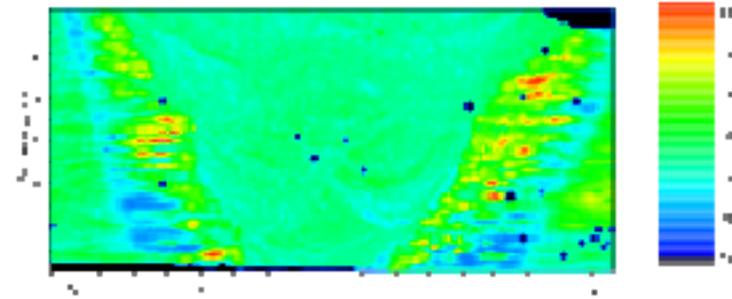
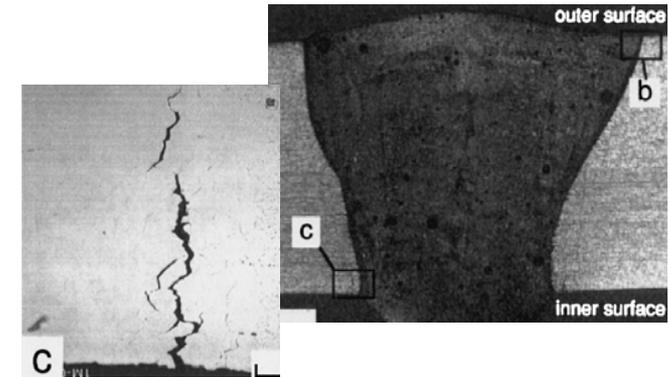
- High capital cost and hydrogen embrittlement (HE) of steel pipelines
 - Preventive measures for HE and permeation
 - Improved joining methods to reduce cost and mitigate HE
- Safety, codes and standards

Partners

- Oak Ridge National Laboratory
- Savannah River National Laboratory
- University of Illinois
- Praxair
- MegaStir Technologies
- Edison Welding Institute

Project Focus: Weld Property Assessment & Welding Technology Development

- Challenges:
 - Weld region is generally more vulnerable to hydrogen induced property degradation (sensitized microstructure, high weld residual stress, exposure to hydrogen)
 - Existing testing methods are not suitable to quantify the tolerance level to HE of weld
 - Lack of technical basis and guidelines for managing hydrogen, stresses, and microstructure in the weld region to ensure the structural integrity and safety of H₂ delivery infrastructure
- Goal: Improve resistance to hydrogen embrittlement (HE) in steel weldment and reduce welding related construction cost



Objectives

- Quantify the effects of high-pressure hydrogen on property degradation of weld in pipeline steels
- Develop the technical basis and guidelines for managing hydrogen, stresses, and microstructure in the weld region to ensure the structural integrity and safety of H₂ pipelines; &
- Develop welding/joining technology to safely and cost-effectively construct new pipelines and/or retrofit existing pipelines for hydrogen delivery.

Approach

- Understand hydrogen transport behavior in steels and weld region
 - High pressure (up to 5,000 psi) hydrogen permeation and diffusion measurement and modeling
 - Effect of steel composition and microstructure
 - Effect of surface conditions
- Determine mechanical property degradation in weld region
 - Effective testing methods for welds
 - Quick screening/comparative test
 - Weld property generation for fracture mechanics based pipeline design
 - Evaluation of weld microstructure effect in old and new pipeline steels
- Welding technology development for new construction, repair and retrofitting existing pipeline infrastructure for hydrogen delivery
 - Weld residual stress and microstructure management
 - Hydrogen management
- Develop technical basis and guidelines for welding construction and maintenance of hydrogen pipelines

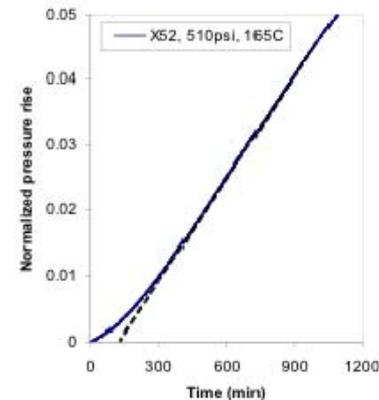
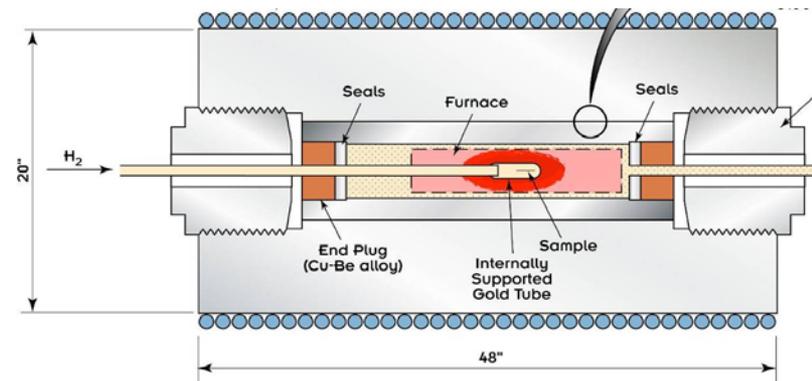
Technical Accomplishments: Previous Years

- High-pressure hydrogen permeation measurement system development and verification
- Baseline high-pressure hydrogen diffusion and permeation measurement with pure Iron
- Effect of weld microstructure on hydrogen trapping, diffusion and permeation
- Initial study on friction stir welding of pipeline steels
- Concept design and initial development of testing methods on weld mechanical property degradation

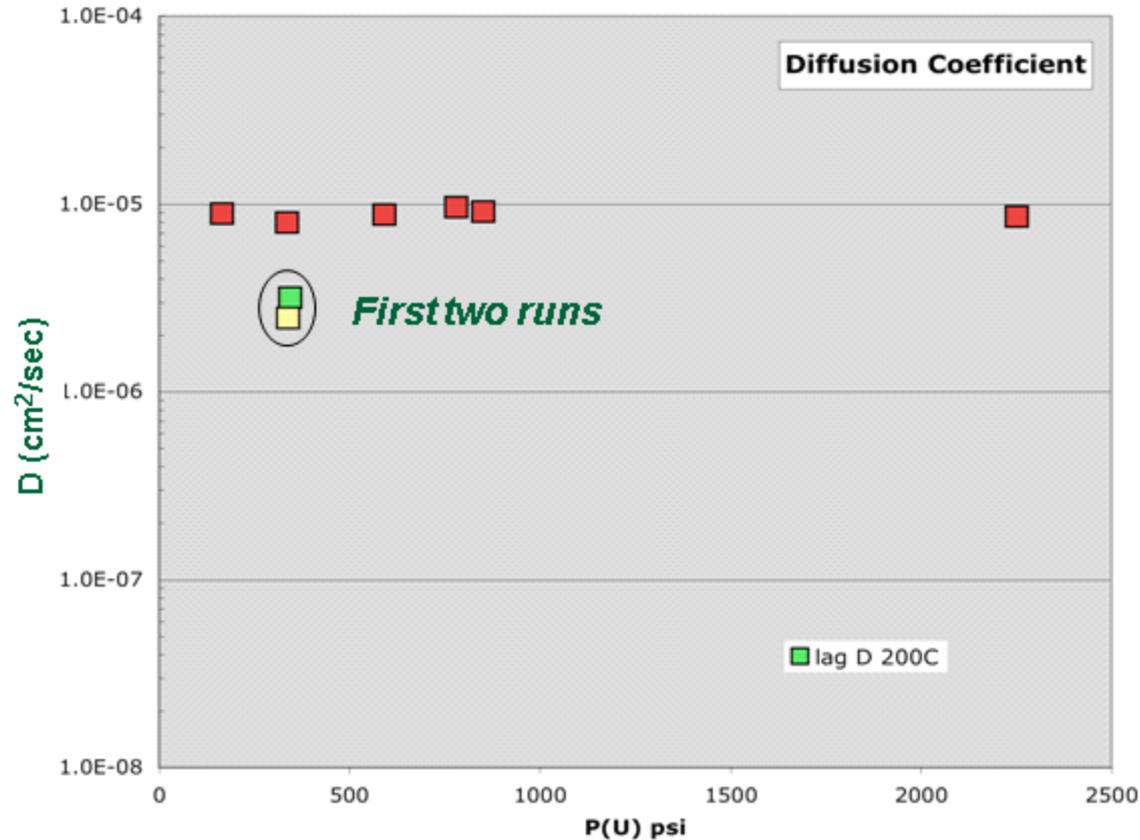
Technical Accomplishments: H Permeation & Diffusion Measurements

- Investigate the hydrogen transport behavior
 - Hydrogen absorption/surface effect
 - Influences amount and rate of hydrogen entering steel
 - Hydrogen diffusion
 - Influences crack propagation rate
 - Hydrogen solubility/concentration
 - Influences the degree of mechanical property degradation
- Under conditions relevant to hydrogen delivery infrastructure
 - Gaseous hydrogen: composition and purity level
 - Pressure range: up to 5,000 psi H₂
 - Temperature range: -40 to 150°C
 - Material: Pipeline steels and their welds; Polymer/composite materials
 - Surface condition: Naturally formed surface oxide layer; Surface coating/modification; Others

- Utilizing ORNL's unique high-pressure permeation measurement system
 - Charging pressure up to 150,000psi
 - Temperature range: 0 to 1000°C
 - Small disk specimen

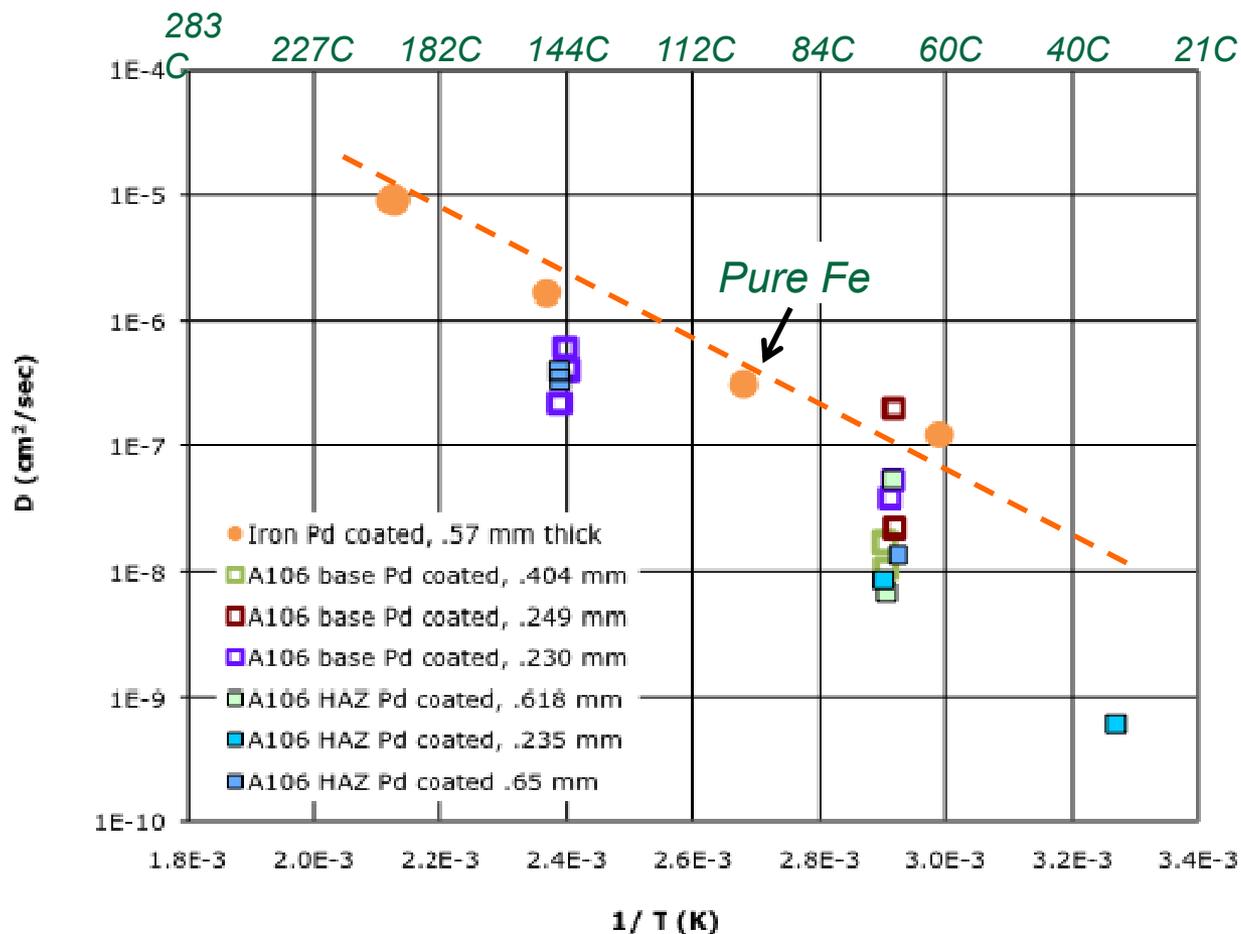


Technical Accomplishments: Pressure Effects on Hydrogen Diffusivity

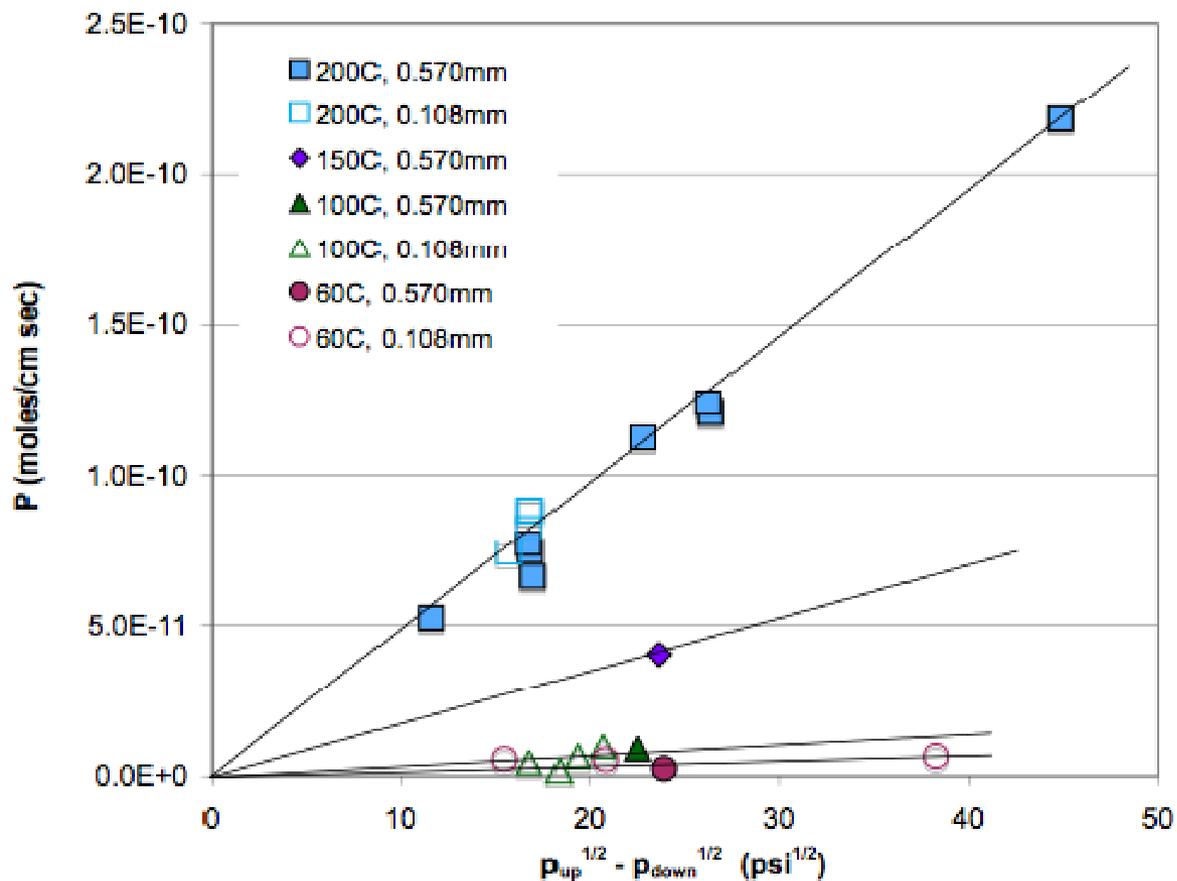


- Pd Coated Pure Iron at 200°C
- Lower “effective” diffusivity of first two runs were due to hydrogen traps and/or surface conditions

Technical Accomplishments: Diffusivity of Pure Iron and Steel A106 Gr B of Different Microstructures



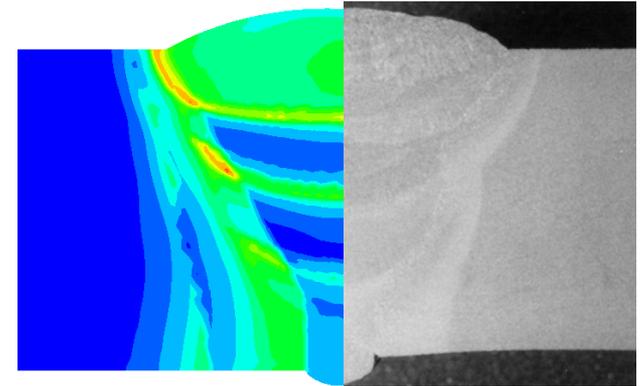
Technical Accomplishments: Pressure Effects on Permeability (Pd Coated Pure Iron)



- Permeation rate has square root dependency on pressure at given temperature

Mechanical Testing of Weld Hydrogen Embrittlement

- Existing mechanical testing methods are generally designed for homogenous materials (base metal) and difficult to reliably test the weld region due to the complex microstructural and property gradients
- Two types of test methods are developed
 - Multi-notch tensile specimen as a simple way for screening and comparative test of different regions of weld and HAZ relative to the base metal
 - Spiral notch torsion test (SNTT) for sustained-load threshold value (K_{th}) of weld and HAZ
 - Determine the tolerance level to hydrogen of different weld microstructures
 - Both methods are under patent application
- Features of test methods
 - Miniature specimen geometry
 - Miniature self-loading rig (sustained load) inside the autoclave
 - Continuous load monitoring
 - Sampling various regions of a weld in a single test to determine the most susceptible region to HE in a weld
 - Low cost and time effective



Technical Accomplishments: Multi-Notch Tensile Comparative Test

Designed for cost-effective evaluation of weld microstructure in long-time exposure to high-pressure H₂. Hydrogen induced crack will initiate and grow in the most sensitive (weakest) microstructure region



Miniature notch tensile specimen (3mm dia.) with notches to sample different microstructures in weld, HAZ and base metal for comparative evaluation



Miniature self-loading fixture with strain gage load sensor



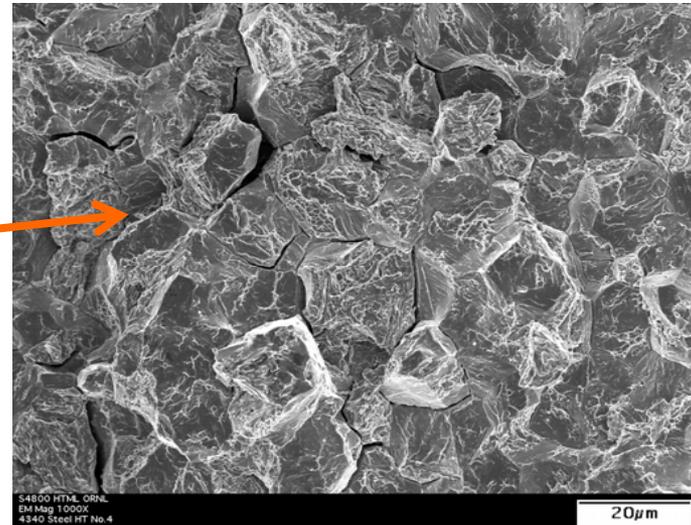
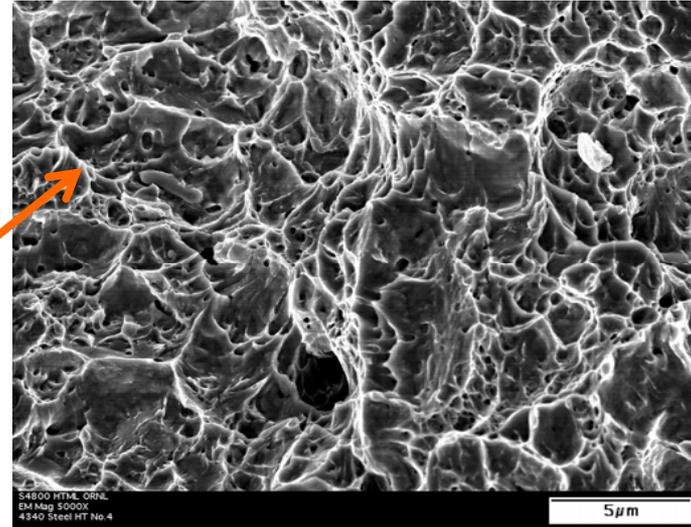
Pressure vessel and instrumentation capable of multiple tests at a time

Technical Accomplishments: Baseline Test with AISI 4340 Steel

- AISI 4340 steel
 - Fully hardened to ~50 Rc, Sult=285ksi
 - Expected to be sensitive to HE
- Testing procedure development
 - Overall design of testing device
 - Effectiveness of self-loading and strain gage load cell
 - Sensitivity to quantify degree of HE
 - Hydrogen update rate
 - Testing protocol

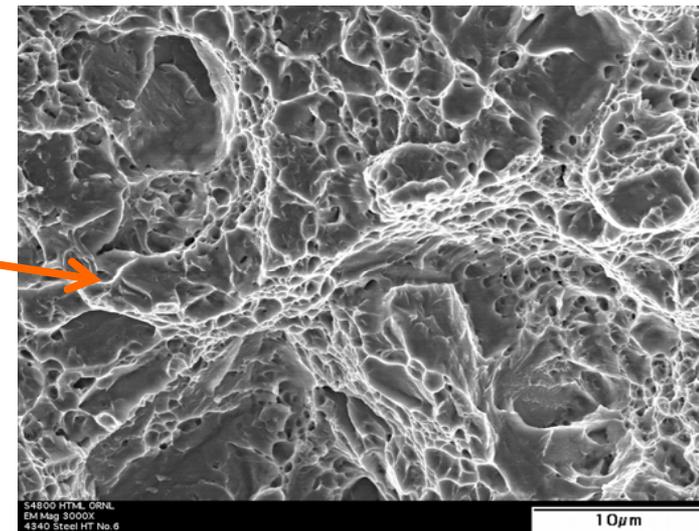
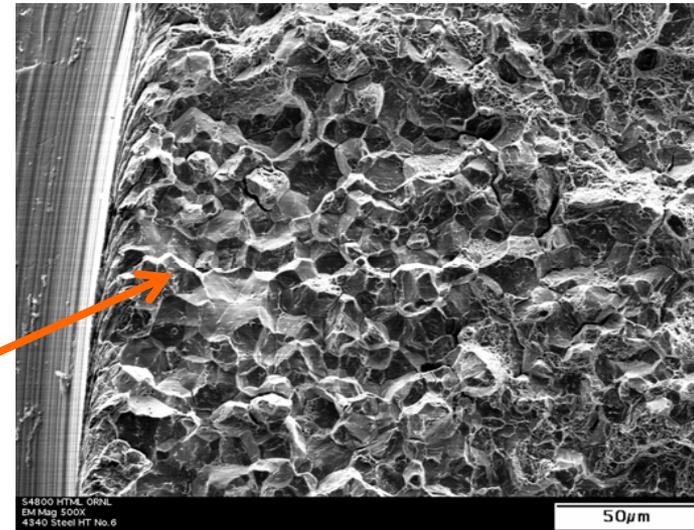
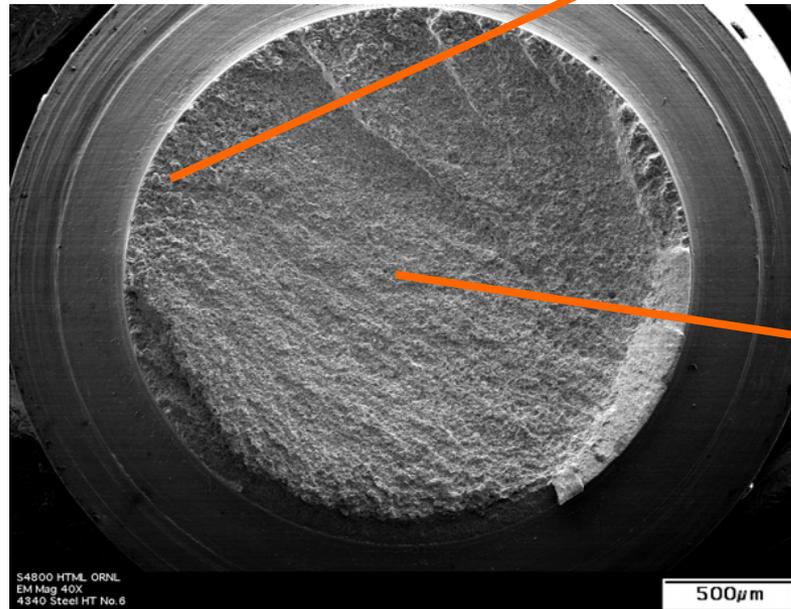
Technical Accomplishments: Multi-Notch 4340 Sample in 2000psi H₂

- Pd surface coating
- Critical fracture load: ~ 45% of failure load in air
- Incubation time to failure: ~ 30 min



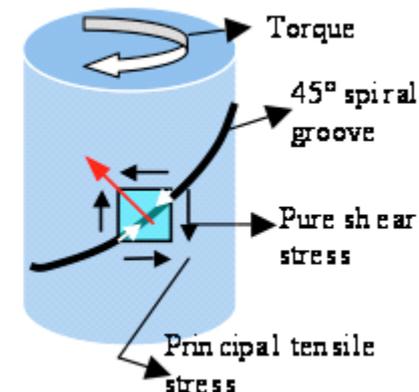
Technical Accomplishments: Multi-Notch 4340 Sample in 2000psi H₂

- Pd surface coating
- Pre-load to 1700N (26% of failure load in air)
- No noticeable HE initiation/growth in hydrogen for 6 days at 23°C
- Sample was pulled broken within 5 min after removing from 2000psi H₂ to check hydrogen level in sample
- **Hydrogen was not saturated to specimen center (~1.5 mm from surface) after 6 days H₂ for charging**

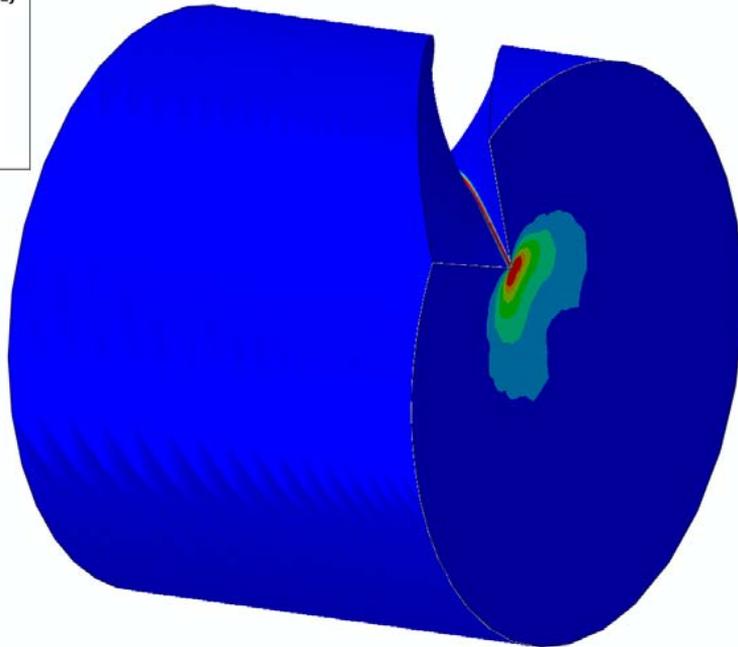
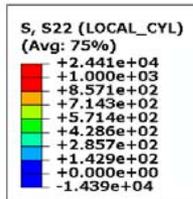


Technical Accomplishments: SNTT for K_{th} Measurement in high- pressure H_2

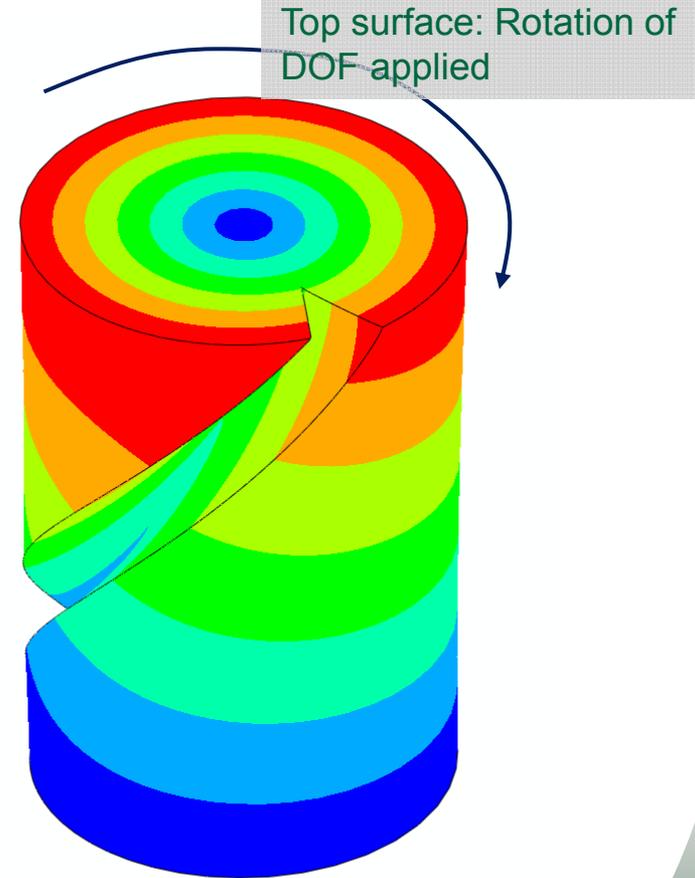
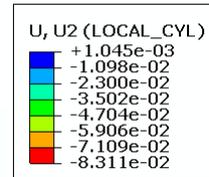
- Based on a R&D100 award-winning method invented by ORNL
- 45 deg spiral notch and twist loading result in Mode I (opening mode) of the spiral notch
- Development for H_2 testing
 - Miniature load cell
 - Finite element analysis was used to determine the stress intensity factor
 - AISI 4340 steel was used for baseline testing procedure development



Technical Accomplishments: FEM analysis of SNTT



Circumferential stress
(crack opening stress)



Circumferential displacements

Technical Accomplishments: SNTT Testing of AISI 4340 Steel

- In air : $K_{IC} = 67.4 \text{ ksi-in}^{1/2}$
 - Consistent with reported 65 - 75 $\text{ksi-in}^{1/2}$ by standard CT specimen of the same steel (Bandyopadhyay et al, Metallurgical Transactions A, 1983)
- Under 2000psi H_2
 - $K_{th} = 36 - 39 \text{ ksi-in}^{1/2}$ (multiple samples) $\sim 55\%$ of K_{IC} in air.



Proposed Future Work

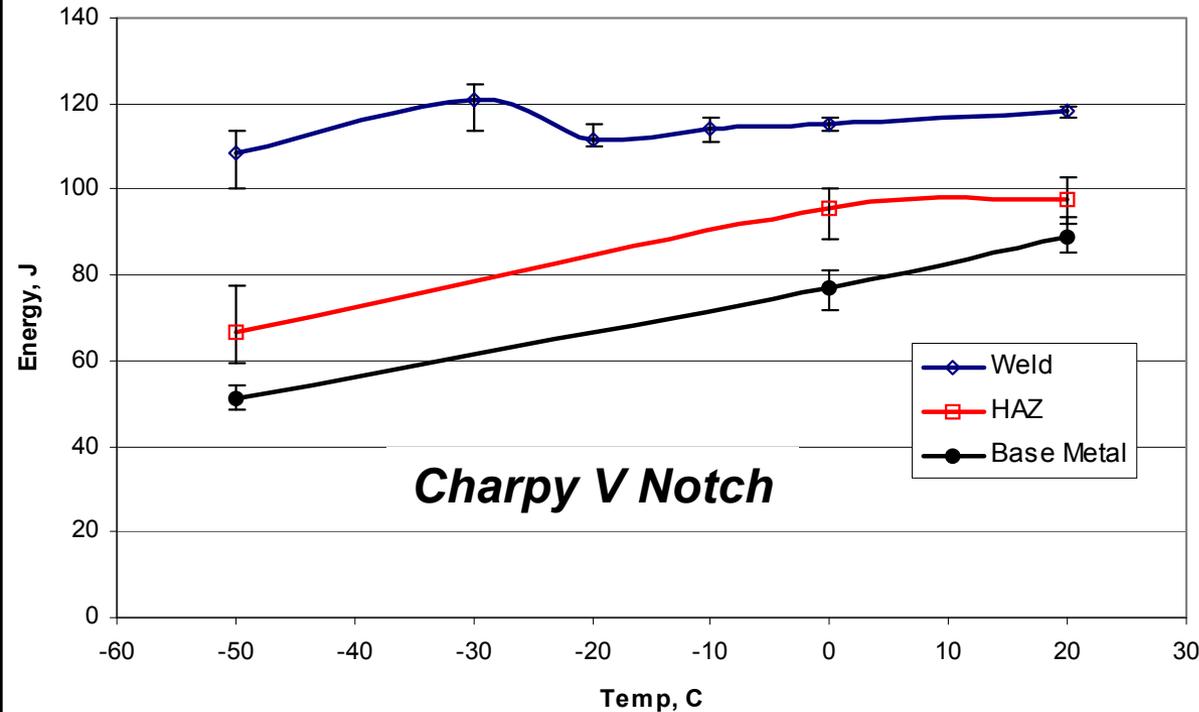
- Permeation/Diffusion in High Pressure Hydrogen
 - Complete comparative measurement on different grades of pipeline steels both weld and base metal (A106, X52, and X100)
 - Study of effect of surface conditions and hydrogen purity (experiment and modeling)
- Mechanical Property Test of Weld
 - Comparative test of X52 welds and X100 welds with multi-notch tensile delay cracking test
 - Kth test with SNTT
 - Cost effective fatigue life test
- Welding technology development
 - Friction stir welding
 - Weld residual stress and microstructure management
 - Cost-effective hydrogen management
- All depending on DOE funding priority and level

Welding Technology Development: Friction stir welding

- A solid-state joining process, no melting
- Extensive thermomechanical deformation during FSW results in wrought weld microstructure with improved properties
 - Conventional fusion welds have cast microstructure
- Eliminate/reduce the coarse grain HAZ (the hard spot) that is generally associated with HE in steel welds
- ORNL is working with major energy and welding equipment companies for natural gas and oil pipeline applications
 - Expect 15-30% cost saving compared to today's pipeline welding construction technology
 - Superior weld property (better than base steel) has been demonstrated
 - http://www1.eere.energy.gov/industry/intensiveprocesses/pdfs/flexible_hybrid_friction.pdf

Superior Weld Properties – Better Than Base Metal

API 5L grade X-65 steel tested *in air*.



ERW Line Pipe
OD: 12.75", 0.25" t
Base metal properties :
Yield: 67 ksi, Tensile: 77 ksi,
Elongation: 33%



Friction Stir Welding of Steel Pipe (Prototype Pipe Welding System by MegaStir/ESAB)



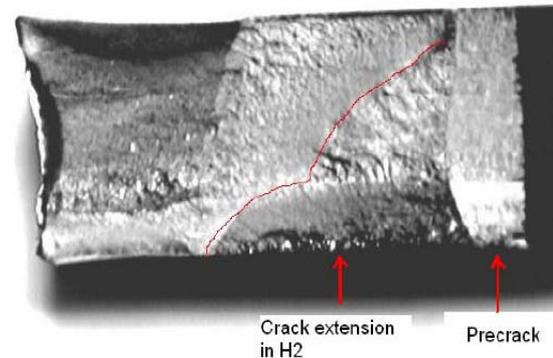
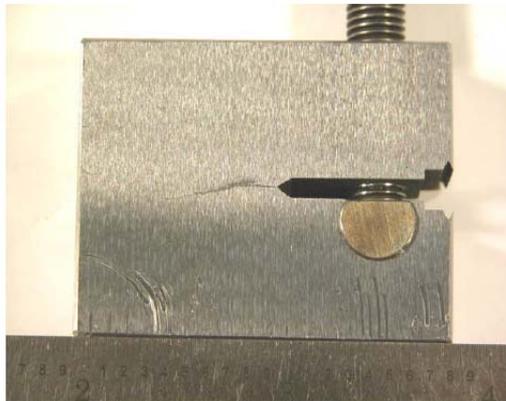
Summary

- Project Focus: Integrity of weldment in steel hydrogen delivery pipeline infrastructure
 - Goal: improve tolerance to HE in steel weldment and reduce construction and retrofitting cost associated with welding
- Technology Development
 - Testing methods suitable for weld property measurement with the complex microstructure and property gradients
 - Two testing methods are under patent application
 - Welding technologies for weld microstructure improvement, residual stress control and hydrogen mitigation
 - Friction stir welding can be a cost-effective construction technology
- Close interactions with other related projects on pipeline steel development and material property testing

Backup Slides

Recent Evidence

- The weld region exhibit less resistance to HE than the baseline pipe steels
 - Xu: X80 with high C_{eq} (0.5)
 - SRNL: A106 Grade B Carbon Steel
 - BM specimens did not crack after 2000 hrs exposure.
 - All weld specimens cracked on one side of the specimen (root pass side)

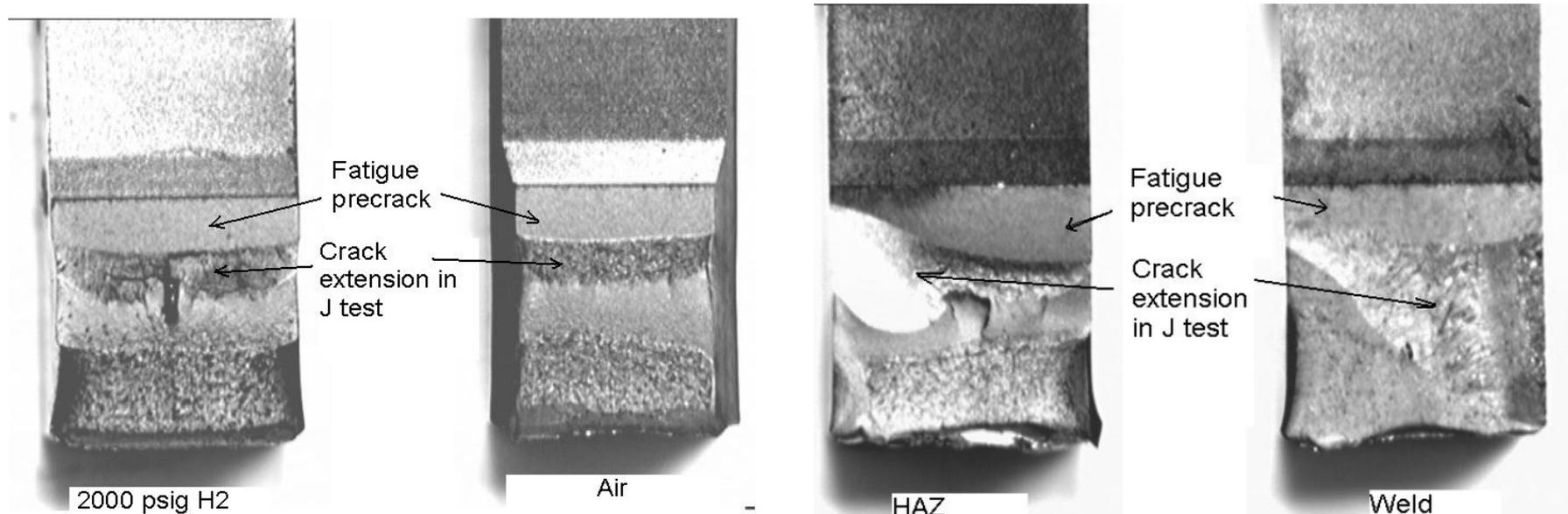


(Xu, 2005, ASTM HE Workshop, X80 steel)

Complications of Inhomogeneous Weld Property – Inadequacy of Current Testing Methods

Valid J_{1c} test of base metal

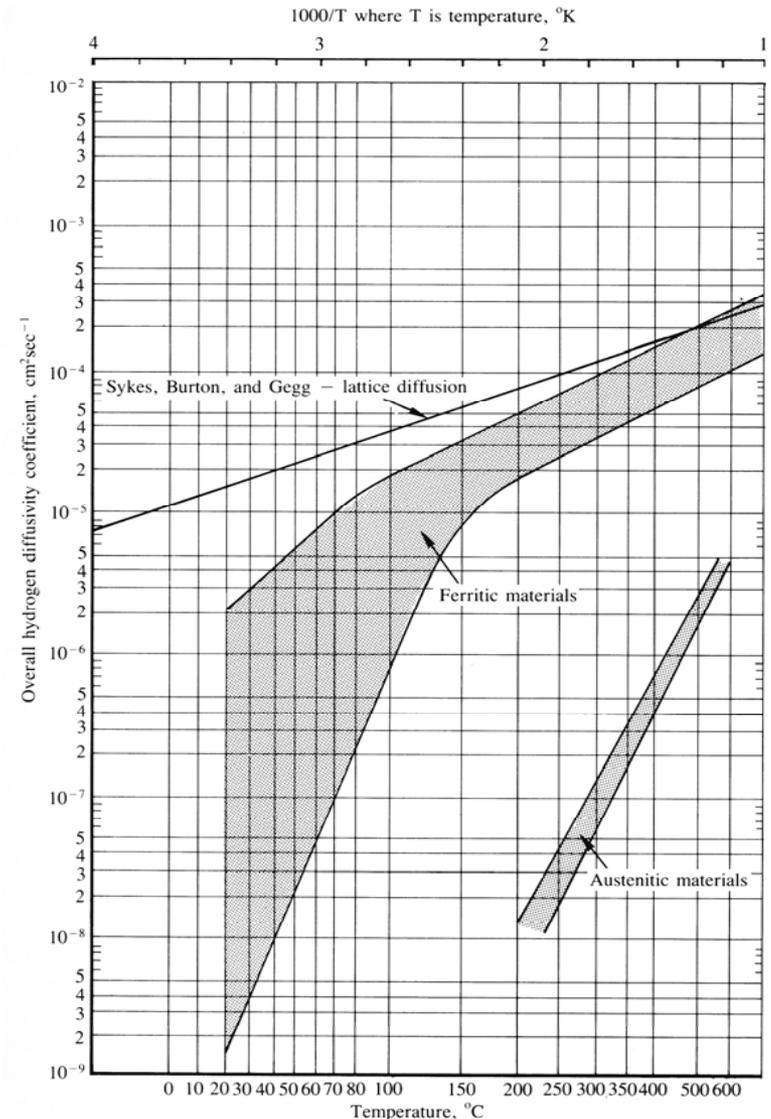
Invalid J_{1c} test of weld and HAZ due to uneven crack front



(Xu, 2005, ASTM HE Workshop)

Hydrogen Diffusivity Data in Literature

- Extensive data available from electrochemical charging at low gaseous pressure ($< 1\text{atm}$), mostly under “controlled” laboratory surface conditions
 - Clean, polished surface
 - Surface coating (Pd) to eliminate surface effects
- Very limited data for high-pressure gaseous hydrogen in “real-world” pipeline environment
 - Surface effects
 - Microstructure effects
 - Hydrogen purity
- Literature data indicates that hydrogen will permeate through pipeline steel during long-term (>20 years) service



Bailey et al, in *Welding without Hydrogen Cracking*

What Really Happens In Permeation Test

- Several major processes operate simultaneously

- On entrance surface:

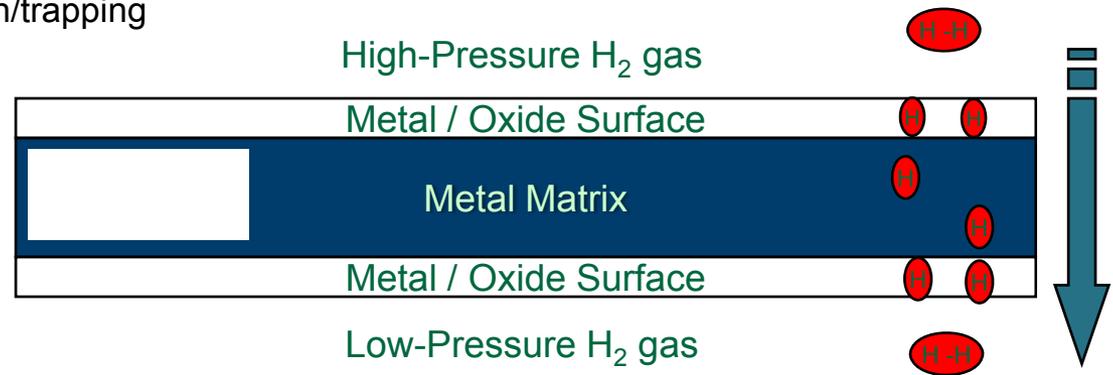
- Hydrogen molecule adsorption/trapping
- Hydrogen dissociation
- Hydrogen dissolution

- Within metal

- Hydrogen diffusion
- Hydrogen trapping

- On exit surface

- Hydrogen recombination
- Hydrogen desorption



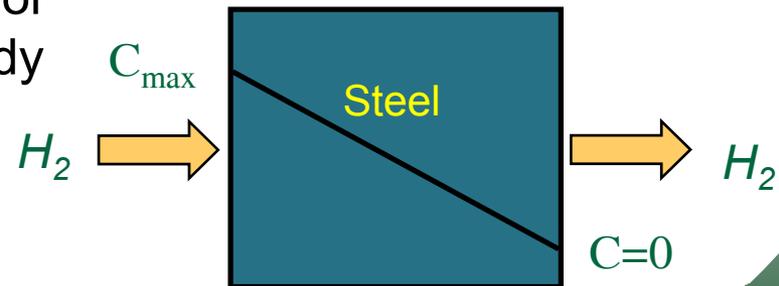
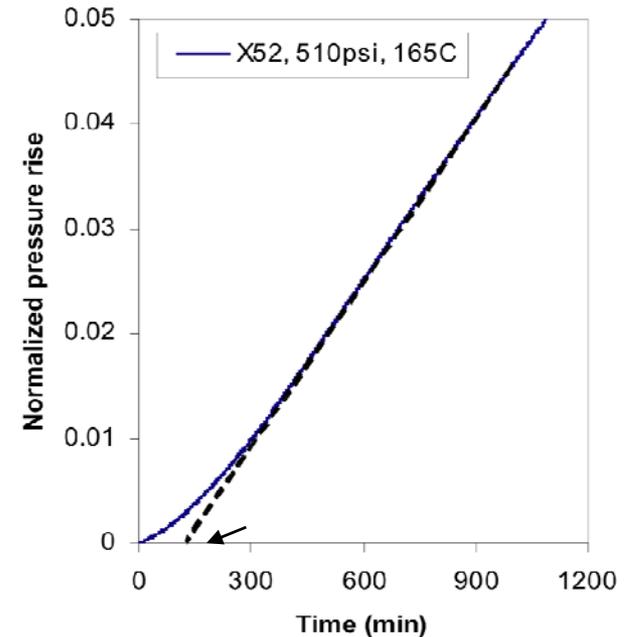
- In order to determine hydrogen diffusion in bulk metal, the surface processes must be controlled and their influence on the kinetics (rate of permeation) must be minimized or separated

- If $J_{\text{surface}} \ll J_{\text{bulk}}$ (i.e. rate at surface dominate), then $J_{\text{measure}} = J_{\text{surface}}$ and diffusivity of metal cannot be determined reliably

- Once the bulk diffusivity is understood, separate tests can be performed to specifically study the surface effects on hydrogen transport in metal.

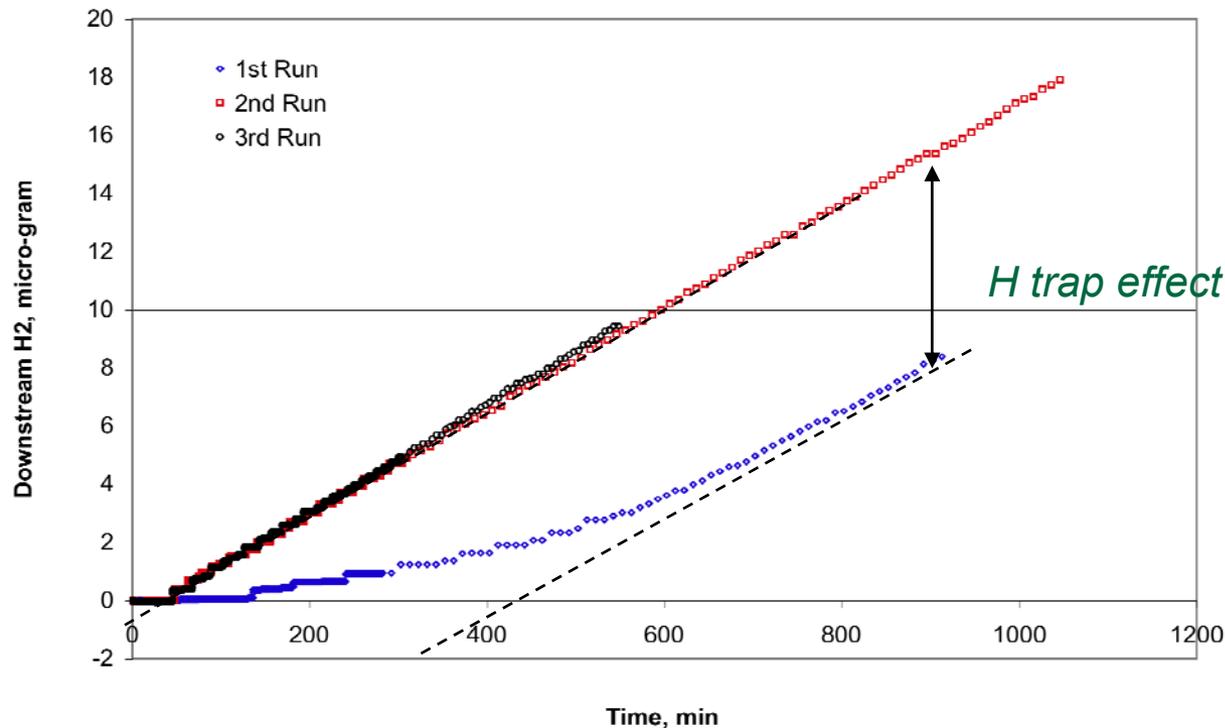
Determination of “Effective” Diffusivity and Solubility from Permeation Test

- Basic assumptions:
 - Diffusivity is independent of H concentration
 - Surface processes are so fast that the permeation rate is control by the bulk diffusion process in metal
- “Effective” diffusivity is determined from the accumulated pressure vs time curve using the asymptotic slope method
- Atomic hydrogen concentration on the upstream surface (max concentration or solubility) is determined from the steady state permeation rate and diffusivity:



Permeation Curves of Pipeline Steel A106 Grade B (Coarse Grain Heat Affected Zone)

- Multiple runs on one sample at 300psi H₂ and 150°C reveals the effect of hydrogen trapping on diffusion
- Hydrogen traps contributes to the differences between 1st and 2nd runs
 - Can be used to estimate the trapped hydrogen concentration
- Nearly identical 2nd and 3rd runs indicate high repeatability of measurement
- Same permeability at steady-state



Weld Hydrogen Embrittlement Test Device



Pressure vessel and instrumentation for HE test



Miniature self-loading device



Miniature notch tensile specimen