

# Hydrogen Permeability and Pipeline Integrity/Fiber Reinforced Composite Pipeline

Dr. Thad M. Adams

Savannah River National Laboratory

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Project ID # PD20

# Overview

## Timeline

- Project start date:10/06
- Project end date:10/10
- Percent complete:45%

## Budget

- Total project funding
  - H<sub>2</sub> Permeation: \$175K
  - FRP Pipeline: \$300K
- Funding received in FY07
  - H<sub>2</sub> Permeation: \$100K
  - FRP Pipeline: \$100K
- Funding for FY08
  - H<sub>2</sub> Permeation: \$75K
  - FRP Pipeline: \$200K

## Barriers

- Barriers Addressed
  - Hydrogen Leakage: <0.5%
  - \$490K/mile and \$190K/mile Transmission and Distribution Costs
  - Safe delivery of hydrogen of hydrogen at a cost target of \$1.00/gge

## Partners

- ORNL
- Edison Welding Institute
- Praxair

# Objectives

## **Hydrogen Permeation & Integrity Project**

- Investigate the influence of weld fabrication microstructure (especially weld heat affected zones (HAZ)) on hydrogen compatibility.
- Measure hydrogen transport (diffusivity) in HAZ materials.
- Determine HAZ material susceptibility to hydrogen embrittlement

## **Fiber Reinforced Composite (FRP) Pipeline Project**

- Focused evaluation of fiber reinforced composite piping for hydrogen service applications.
- Assessment of the structural integrity of the FRP piping and leakage of existing commercial available FRP joint designs and joint components.

# Milestones

## Hydrogen Permeation & Integrity Project

- *FY08 Milestones:*
  - *Fabrication of GLEEBLE Weld HAZ Simulations Samples—0-1,3,5, Weld Pass Simulation—Complete*
  - *Hydrogen Transport Testing in Weld Simulation Samples-- 5-Pass Weld Samples—Complete—Defunded Before Additional Samples Completed*
  - *Characterization of Weld HAZ Simulation Microstructures—Initial Characterization Complete*
  - *Fabrication of ASTM G142—Hydrogen Susceptibility Test Samples—Unnotched/Notched Tensile Specimens--Complete*
  - *Unnotched/Notched Tensile Testing—Project Eliminated*

## Fiber Reinforced Composite Pipeline Project

- *FY08 Milestones:*
  - *Complete 1-year Hydrogen Exposures for Pipe Sections, Compression Samples, Tensile and DMA Samples—In-Progress—Completion in 7/08*
  - *Initiate Commercial FRP Joint Leakage Testing--In-Progress—Completion—10/08*

# Approach

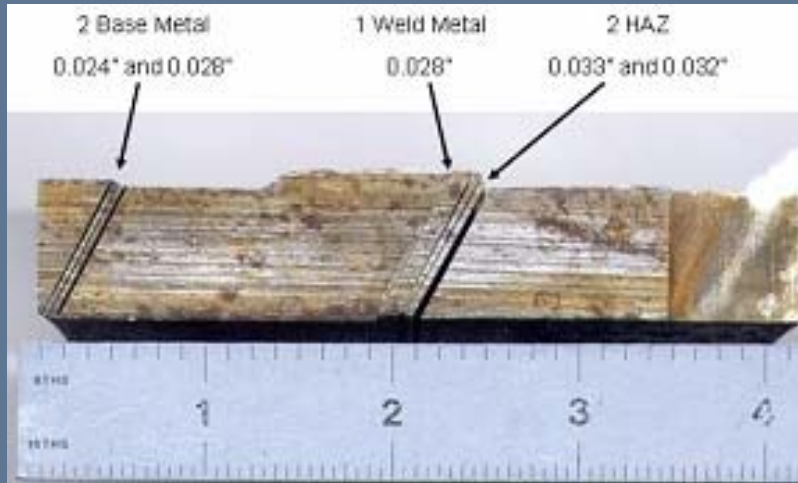
## Hydrogen Permeation & Integrity

- Fabricate Test Samples From Actual Welded Components and Using a GLEEBLE-Thermomechanical Weld Simulator
- Prepare Samples for Hydrogen Transport (Permeation/Diffusion) and Hydrogen Susceptibility (Smooth/Notched tensile tests—ASTM G142)
- Measure Gaseous Hydrogen Permeability, Diffusivity and Solubility at Sub-ambient Pressures and Moderate Temperatures
- Determine Tensile Properties at Nominal H<sub>2</sub> Pipeline Pressures and Temperature

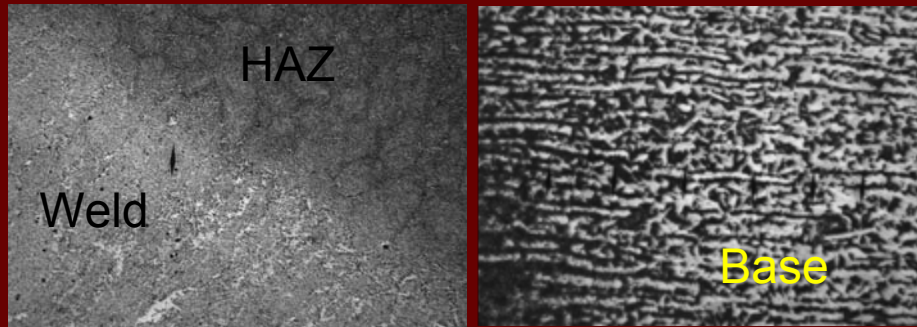
## Fiber Reinforced Composite Pipeline

- Complete Hydrogen Exposure of FRP Pipe Sections to Support Material Testing
- Investigate FRP Joint Types—Threaded Compression & Hydraulically Crimped-- to Determine Leakage Rates in Hydrogen Service

# Hydrogen Permeation & Integrity

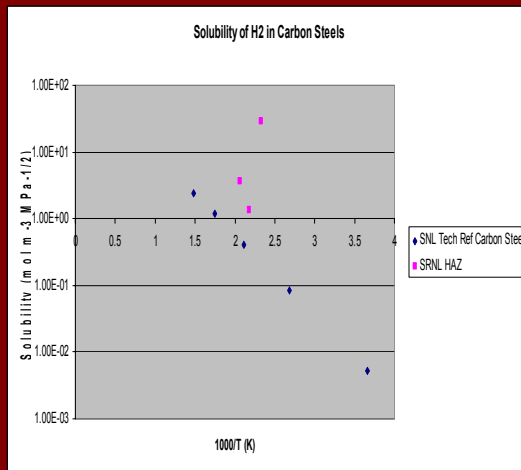
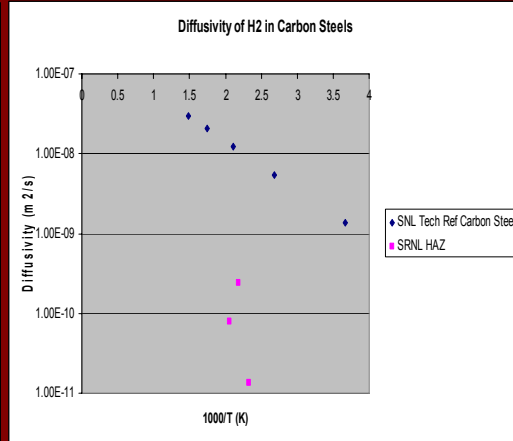
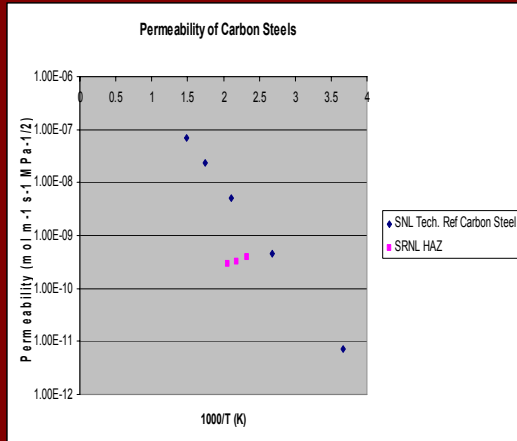


Permeation Samples A106 Pipe Weld



- Received Welded Pipe Materials—Praxair—API 5L Grade B/A106 Grade B—SMAW Girth Weld w/ TIG Root Pass
- Sectioned Weld and Identified Base, HAZ, and Weld Regions (Approx. 0.030" thick)
- Low Pressure Permeation Testing
  - Pressure up to 700Torr
  - Temperature up to 200°C
- Measure Hydrogen Permeability and Diffusivity
  - Solubility Determined from Permeation Test Data

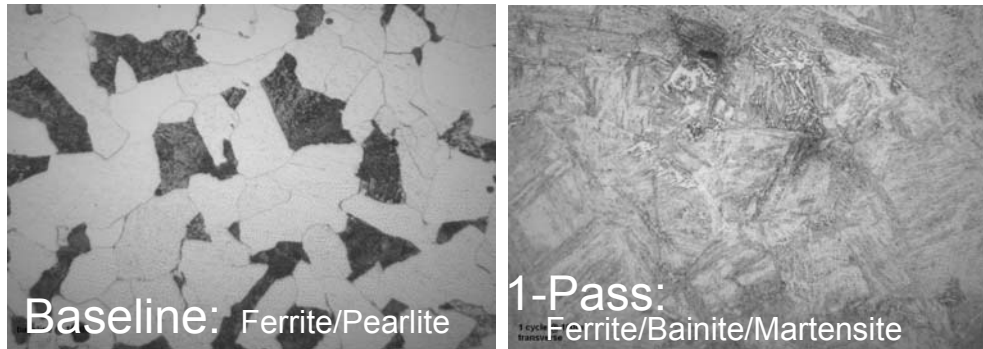
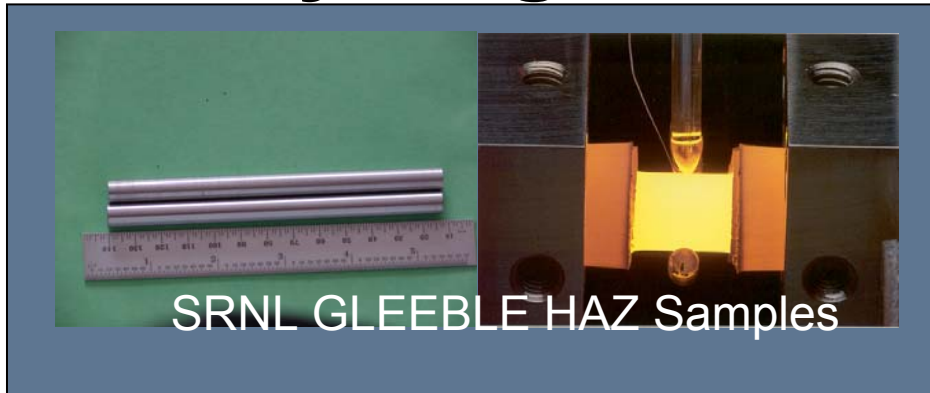
# Hydrogen Permeability, Diffusivity, Solubility Measurements of HAZ



- Permeability , Diffusivity, Solubility Measurements of HAZ—700T @150, 175, and 200°C
- Permeability
  - 150°C =  $3.92 \times 10^{-10}$  mol/m s MPa<sup>1/2</sup>
  - 175°C =  $3.23 \times 10^{-10}$  mol/m s MPa<sup>1/2</sup>
  - 200°C =  $2.87 \times 10^{-10}$  mol/m s MPa<sup>1/2</sup>
- Diffusivity
  - 150°C =  $1.34 \times 10^{-11}$  m<sup>2</sup>/s
  - 175°C =  $2.36 \times 10^{-10}$  m<sup>2</sup>/s
  - 200°C =  $7.87 \times 10^{-11}$  m<sup>2</sup>/s
- Solubility
  - 150°C =  $29.25$  mols/m<sup>3</sup> MPa<sup>1/2</sup>
  - 175°C =  $1.36$  mols/m<sup>3</sup> MPa<sup>1/2</sup>
  - 200°C =  $3.64$  mols/m<sup>3</sup> MPa<sup>1/2</sup>
- Preliminary Measured Data from Actual Weld Samples Questioned—Challenge in Sample Extraction

*Initial SRNL Weld HAZ Data from Field Pipe Weld Consistent with Some Low Temperature Literature Values—Reflects Greater Impact of Surface and Internal Trapping Effects@ Lower Temperature—Sections from Actual Welds Presents Challenge of Ensuring Samples Extracted Exclusively from HAZ Materials*

# Hydrogen Permeation & Integrity



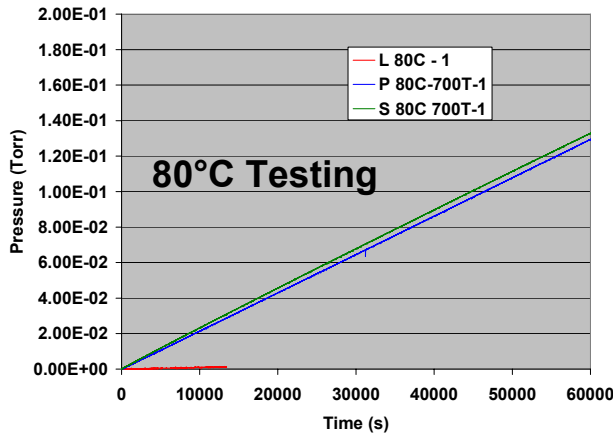
- *Critical Issue Identified by PWG is Relation of Materials Properties to Microstructural Features Not Simply Materials Chemistry*
  - *SRNL Approach Allow for Tailoring of the Microstructure and Fundamental Understanding Effects on Properties*
  - *Used to Aid the Development of New Welding Procedures and Technologies*
- *Developed E-Weld Predictor® Simulations for Weld HAZ Microstructures*
  - *Developed Sub-routine to Predict Peak Weld and HAZ Temperature During Welding*
  - *Peak Temperature Profiles Helps Predict HAZ Microstructures*
- *Fabricated and Treated 20 GLEEBLE—HAZ Simulation Samples*
  - *Controlling/Tailoring Microstructures Allows for Development of Fundamental Relationship Between Structure and Properties*
- *Documented GLEEBLE Microstructures Consistent with Literature Data for HAZ in Pipeline Steels*

*Effect of Microstructure on Diffusivity of Hydrogen in Pipeline Steels is Critical to Aiding Understanding Hydrogen Embrittlement—Fundamental Materials Science Link Between Structure-Properties-Processing—Management of Hydrogen Embrittlement Key to Hydrogen Pipeline Structural Integrity*

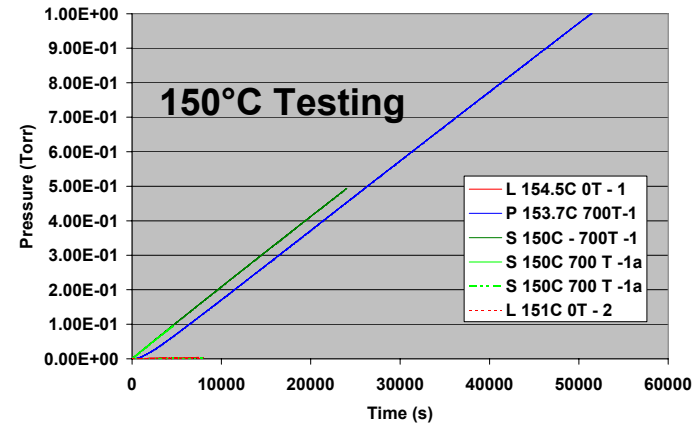


# Hydrogen Permeation & Integrity

Gleeble Sample 5 Cycles



Gleeble Sample 5 Cycles



## Permeability Determination from Permeation Experiments

- Permeability<sub>150</sub> : 150°C and 5-pass Weld Simulation:  $2.2-3.2 \times 10^{-10} \text{ Mol H}_2/\text{m/s/Pa}^{1/2}$
- Permeability<sub>80</sub> : 80°C and 5-pass Weld Simulation:  $3.0-4.25 \times 10^{-11} \text{ Mol H}_2/\text{m/s/Pa}^{1/2}$

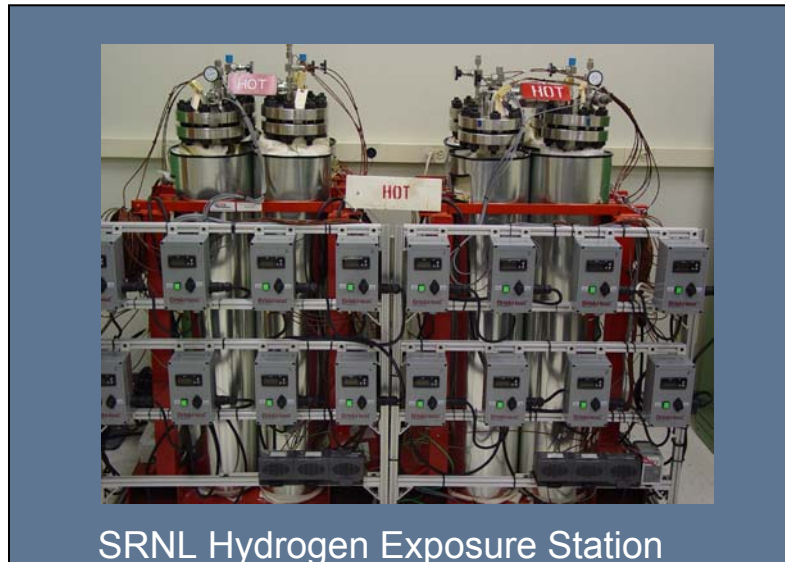
## Diffusivity Determination from Permeation Experiments

- Diffusivity<sub>150</sub> : 150°C and 5-pass Weld Simulation:  $3.5 \times 10^{-6} - 3.0 \times 10^{-7} \text{ cm}^2/\text{s}$
- Diffusivity<sub>80</sub> : 80°C and 5-pass Weld Simulation:  $1.25 \times 10^{-6} \text{ cm}^2/\text{s}$

**Permeability & Diffusivity Data Consistent w/ Literature—Surface/Trapping Values**

*Preliminary 5-Pass Weld HAZ Microstructure (Ferrite/Acicular Ferrite/Sm. Pearlite) Diffusivity Data Comparative to Previous Measured Actual Field Weld HAZ Microstructure (Ferrite/Pearlite)—Measurement of Both Dominated by Ferrite (BCC) Permeation/Diffusivity*

# Fiber Reinforced Composite Pipeline



- Hydrogen Exposure Test Matrix
  - 1-Month & 1-year Exposures
    - 2-FRP Pipe Section for Hydrostatic Burst
    - 2-FRP Pipe Sections for Radius Bend Test
    - Glass Fiber, Resin, HDPE Liner Samples
    - 1000 psig @140°F
- Control Sample Thermal Exposures
  - 1-month and 1-year Exposures@ Temperature in Air
- Pipe Sections, Compression Samples, Tensile Dogbones, and DMA Samples
- 1-month Exposure Testing Completed—  
1 Year Exposures In Progress—  
Complete 7/08



*Critical Issue for Demonstrating Feasibility of FRP Pipeline for Hydrogen Service is Development of the Technical Basis to Demonstrate Structural Integrity by Defining FRP Susceptibility to Hydrogen Embrittlement* 10

# Fiber Reinforced Composite Pipeline

Fiberspar™ Threaded  
Compression Joints Components



Polyflow™ Hydraulically  
Crimped Joints Components



- DOT Gap Analysis Report Identifies 4 Major Needs for Composite FRP Piping
  - Lack of Design Specifications
  - **Qualified Joints/Joining**
  - Permeation
  - Robustness to External Damage
- Performed Preliminary Leak Rate Measurements Using He Leak Check System with a Sensitivity of  $10^{-9}$  cc/sec:
  - Results for Fiberspar Pipeline Compression-Thread Joint Fitting:  $> 10^{-4}$  stdcc/sec @ 1atm test pressure
  - Results for Polyflow Crimp End Connection:  $> 10^{-5}$  stdcc/sec
  - Rough Correlation to 0.5stdcc/sec @1000psig
- Preliminary Short Duration Pressure Decay Test of Crimped Polyflow End Fittings and Fiberspar Compression-Thread Fittings Completed
  - Both Joint Design Measures  $> 10^{-2}$  std cc/sec for a 1 hr Duration Test

*Qualification of Existing Commercial FRP Joint Components With Respect to Leakage Identified in DOT Gap Analysis Report as 1 of 4 Major Needs for Demonstrating Composite FRP Piping Feasibility  
SRNL Focused on Evaluation of Commercial FRP Joints—for Leakage/Structural Integrity*

# Future Work

## Hydrogen Permeation & Integrity

- Program Eliminated 3/12/08

## Fiber Reinforced Composite Pipeline

- Complete Hydrogen Exposures and Return Samples to ORNL—July 2008
- Complete Commercial FRP Joint/Joint Component Leak Testing
  - Complete Hydrogen Pressure Decay Testing
    - Pressure up to 1000 psig and Temperatures up to 140°F
- Initiate Testing to Assess Impact of Joint Loading and Flexure on Commercial FRP Joint/joint Component Leakage
  - Soil/Ground Movement/Relocation has Been Shown the Place Load on Pipeline Joint /Fittings and Can Exacerbate Leakage
  - SRNL will Fabrication a Simple Loading Rig to Place the Joint/Joint Components Under a Measured Flexural Load and Assess Changes in Leakage Rate
- Initiate Testing to Evaluate Effect of Pressure Cycling on Leakage of Polyflow Crimp-Type Joint/Joint Component Leakage
  - Fatigue Due to Pressure Cycling in Pipeline System Has Been Identified by PWG as an Issue for Overall Structural Integrity—Additionally, this Pressure Cycling Could Affect the Integrity of the FRP Joint/Joint Components Especially the Crimped Connections Used by Polyflow
  - SRNL will Pressure Cycle a Section of Pipe with the Polyflow Crimp Joint Connectors for a Prescribed # of Cycles and then Evaluate any Change in Crimp-Joint Connection Using Gas Leakage Testing—He-Leakage/Hydrogen Pressure Decay
- *FY09 Effort*
  - Continued Leakage Evaluation of FRP Commercial Joint Components
  - Hydrogen Absorption/Solubility Testing—Evaluation of the Long-Term Absorption/Solvation of Gaseous Hydrogen in Typical FRP Liner Materials—HDPE—Pressure Absorption and Thermal Desorption Experiments
  - Structural Integrity—Hydrogen Embrittlement Susceptibility Evaluation (ASTM G142) of Metallic Commercial FRP Joint/Joint Component Materials

# Summary

## Hydrogen Permeation & Integrity

- *Effect of Microstructure on Diffusivity of Hydrogen in Pipeline Steels is Critical to Aiding Understanding Hydrogen Embrittlement—Fundamental Materials Science Link Between Structure-Properties-Processing—Management of Hydrogen Embrittlement Key to Hydrogen Pipeline Structural Integrity*
- *Preliminary 5-Pass Weld HAZ Microstructure (Ferrite/Acicular Ferrite/Sm. Pearlite) Diffusivity Data Comparative to Previous Measured Actual Field Weld HAZ Microstructure (Ferrite/Pearlite)—Measurement of Both Dominated by Ferrite (BCC) Permeation/Diffusivity*
- *Project Eliminated March 2008*

## Fiber Reinforced Composite Pipeline

- *Critical Issue for Demonstrating Feasibility of FRP Pipeline for Hydrogen Service is Development of the Technical Basis to Demonstrate Structural Integrity by Defining FRP Susceptibility to Hydrogen Embrittlement*
- *Qualification of Existing Commercial FRP Joint Components With Respect to Leakage Identified in DOT Gap Analysis Report as 1 of 4 Major Needs for Demonstrating Composite FRP Piping Feasibility*
- *Initial Leakage Results Indicate Higher Leak Rates Than Anticipated—SRNL and ORNL Meeting to Review Results and Review Leak Testing Protocols for FRP*