Testing Subgroup Workshop on Critical Property Needs

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Presented at AMSE/PWG Joint Meeting

Participants

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Paul Bakke - DOE

David McColskey, Richard Ricker - NIST

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Brian Somerday - SNL

Andrew Duncan - SRNL

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Objectives

Develop an action plan that:

- details the necessary tests to measure and compare the physical properties of metallic materials relevant to high pressure hydrogen service,
- prioritizes these tests based on need,
- determines the standard test procedure and range of critical variables all participants will use,
- selects the initial materials for testing, and
- establishes a round robin test procedure for one or more properties to assure all participants are obtaining similar results.

Critical Properties

- Fatigue Crack Growth
- Critical (K_{ic}) and Integral Fracture (J_{ic}) Toughness and Tearing Modulus
- Fatigue (ΔK_{th})
- Constrained Fracture Mechanics
- K Threshold
- Time Dependant Rate of Change of Crack Length
- Permeation (for solubility and diffusivity)
- Surface exchange kinetics
- Thermal Desorption Spectroscopy
- Hydrogen Concentration Analysis
- Residual Stress
- Component Testing
- Burst Test
- NACE Hydrogen Induced Cracking (HIC) Testing
- In-situ Neutron Analysis
- Tensile testing/Notched Tensile Testing
- Fracture Mechanisms/Modeling

Critical Properties-Ranked

- Fatigue Crack Growth
- Critical (K_{ic}) and Integral Fracture (J_{ic}) Toughness and Tearing Modulus
- Permeation
- K Threshold
- Tensile testing/Notched Tensile Testing
- Fatigue (ΔK_{th})
- Surface exchange kinetics
- Hydrogen Concentration Analysis
- Constrained Fracture Mechanics
- Thermal Desorption Spectroscopy
- Burst Test
- Time Dependant Rate of Change of Crack Length
- Residual Stress

Critical Test, Standard Test Method and Test Conditions

Property	Test	Conditions
Fatigue Crack Growth	ASTM 647	1 Hz; R ratio TBD
K_{ic} & J_{ic}	E1820	$<3x10^{-4}$ mm/sec
Permeation	Gas Flux Measurement	-40 to 300°C; Up to 10 ksi
K Threshold	E1681	TBD
Tensile Test (smooth & notched)	E8 (smooth), A370, E602	10^{-6} sec (smooth), 10^{-3} sec (fixed notch)
Fatigue	E647	1 Hz, R Ratio TBD
Surface Exchange Kinetics	TBD	-40 to 300°C; Up to 10 ksi

Critical Test, Standard Test Method and Test Conditions

Property	Test	Conditions
Hydrogen Concentration Analysis	LECO-H, Si-oil, Thermoelectric	
Constrained Fracture Mechanics	TBD	
Thermal Desorption Spectroscopy		1 mm thick Span TBD
Burst Test	Rupture Disk	TBD
Time Dependant Rate of Change of Crack Length	E1681	TBD
Residual Stress	E1426 (xrd), E837 (hole drilling)	

Materials

- Air Products, El Paso, and TransCanada recommend lower strength more ductile materials were a appropriate choice of starting materials.
  - More easily accommodate third party damage
  - higher strength steels are expected to have a service stress penalty (conservative allowable value) in the piping code to account for hydrogen sensitivity unknowns
  - X52

- NIST Workshop conclusion: consider X70 and below as well as >X70.

Initial Test Conditions

- Gas quality: 99.9999 (analyze post-test)
- Temperature:
  - -40 to 150°C (mechanical property testing),
  - -40 to 300°C (permeation testing)
- Pressure: ambient to 10ksi

- Will be evaluated after first year
- Impurities will be selected and introduced after consultation with gas and pipeline companies

Communication/Reporting

- Semi annual meetings to discuss programs and testing
  - 1st TDB
  - 2nd to coincide with 2nd NIST Workshop
- Bimonthly conference call

Round Robin Testing

- Testing will be coordinated by CTC
  - Procure stock material
  - Coordinate specimen machining
    - Track where samples were taken from stock
  - Coordinate and track all testing
  - Report findings

- Initial testing will include smooth tensile and permeation

RRT – Material Selection & Procurement

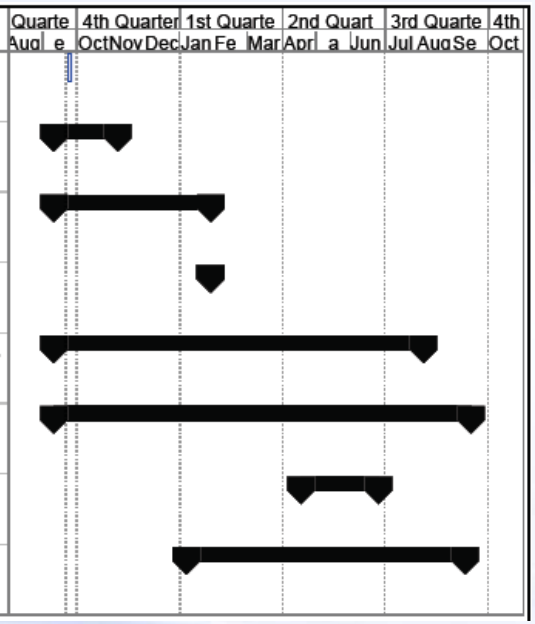
- Different options considered A106 Grade B, X42, X52
  - New material vs. material removed from service
  - Plate or pipe
  - Specimen extraction orientation
- Resolution
  - X42 or X52 (per DOE direction)
  - New material consistent w/current pipeline practice
  - Welded pipe large and thick enough for current and future extraction of specimens
  - Extraction orientations TBD, based on finalized specimen geometries
- *CTC* currently exploring suppliers and will procure pipe

Round Robin Tests

- Tensile testing
  - Participants: all
  - Conditions:
    - Temperature - 25°C;
    - Environment -Air, He and H_2 (99.999%)
  - Samples - 3 @ each condition
- Permeation testing
  - Participants: ORNL, NIST & ?
  - Samples - 1 HAZ & 1 base material
  - Conditions:
    - Temperature - 25°C to 300°C;
    - Environment - H_2 (99.999%)
    - Pressures - 1 to 10 ksi

Round Robin Schedule

ID	Task Name	Start	Finish
1	ASME/SRNL/DOE Workshop - Aiken, SC	Mon 9/24/07	Wed 9/26/07
2	Material Procurement	Tue 9/11/07	Tue 11/6/07
21	Specimen Machining	Tue 9/11/07	Mon 1/28/08
73	Specimen Distribution	Mon 1/28/08	Mon 1/28/08
84	Tensile Testing	Tue 9/11/07	Mon 8/4/08
128	Permeation Testing	Tue 9/11/07	Mon 9/15/08
164	Report Writing	Fri 4/18/08	Wed 6/25/08
185	Present Results	Mon 1/7/08	Wed 9/10/08



Property	NIST (Future)	ORNL	SNL	SRNL	U. Illinois
Fatigue Crack Growth	✓	✓	✓		✓
Critical and Integral Fracture Toughness (K_{ic}, J_{ic}) and Tearing Modulus	✓	✓	✓	✓	✓
Permeation*	LP & HP	LP & HP	LP	LP	LP
K Threshold	✓	✓	✓	✓	
Tensile Test (smooth & notched)	✓	✓	✓	✓	✓
Fatigue (ΔK_{th})	✓		✓		
Surface Exchange Kinetics	✓	✓		✓•	✓
Hydrogen Concentration Analysis	✓			✓	✓
Constrained Fracture Mechanics	✓	✓	✓	✓	✓
Thermal Desorption			✓	✓••	✓
Spectroscopy					
Burst Test				✓	
Time Dependant Rate of Change of Crack Length			✓	✓	✓
Residual Stress	✓	✓		✓	

* Low Pressure (LP) is defined as <2000 psi and High Pressure is defined as >2000 psi.

Questions?

Materials

API 5L (X42 and/or X52)

- steels are really represented by a strength requirement and composition can vary as long as strength minimums are maintained.
- Nb and V can be added at the discretion of the manufacturer; the sum of Nb+V+Ti cannot exceed 0.15, and the sum of Nb+V cannot exceed 0.06 unless the purchaser and manufacturer agree on another number.
- Si is not limited in these specs as it is considered a deoxidizer, but the steel will no doubt have anywhere from 0.1% Si up to maybe 1%.
- analysis for Cr, Ni, Mo, and Cu is required, but these have no limits except as required by a carbon equivalent (hardenability) calculation.

Weight %	X42	X52
Max C	0.28	0.28
Max Mn	1.30	1.40
Max P	0.030	0.03
Max S	0.030	0.030
Max Ti	0.04	0.04

PSL 1 for seamless pipe, for welded pipe % C decreases to 0.26