Results… NIST Workshop on Materials Test Procedures for Hydrogen Pipelines

Held: August 21-22, 2007
Boulder, CO

NIST-Materials Science and Engineering Laboratory
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Codes ASME/SRNL Materials and Components for Hydrogen Infrastructure and Standards Workshop
Aiken, SC
September 24-25, 2007

Why NIST?

The Pipeline Safety Improvement Act (PL107-355) gave DOT, DOE, MMS and NIST responsibility for research on gas pipelines.

NIST has a long history in pipeline research and materials testing in harsh environments.

NIST MSEL Hydrogen Program (Boulder and Gaithersburg)

Fracture and fatigue resistance of pipeline materials in hydrogen gas

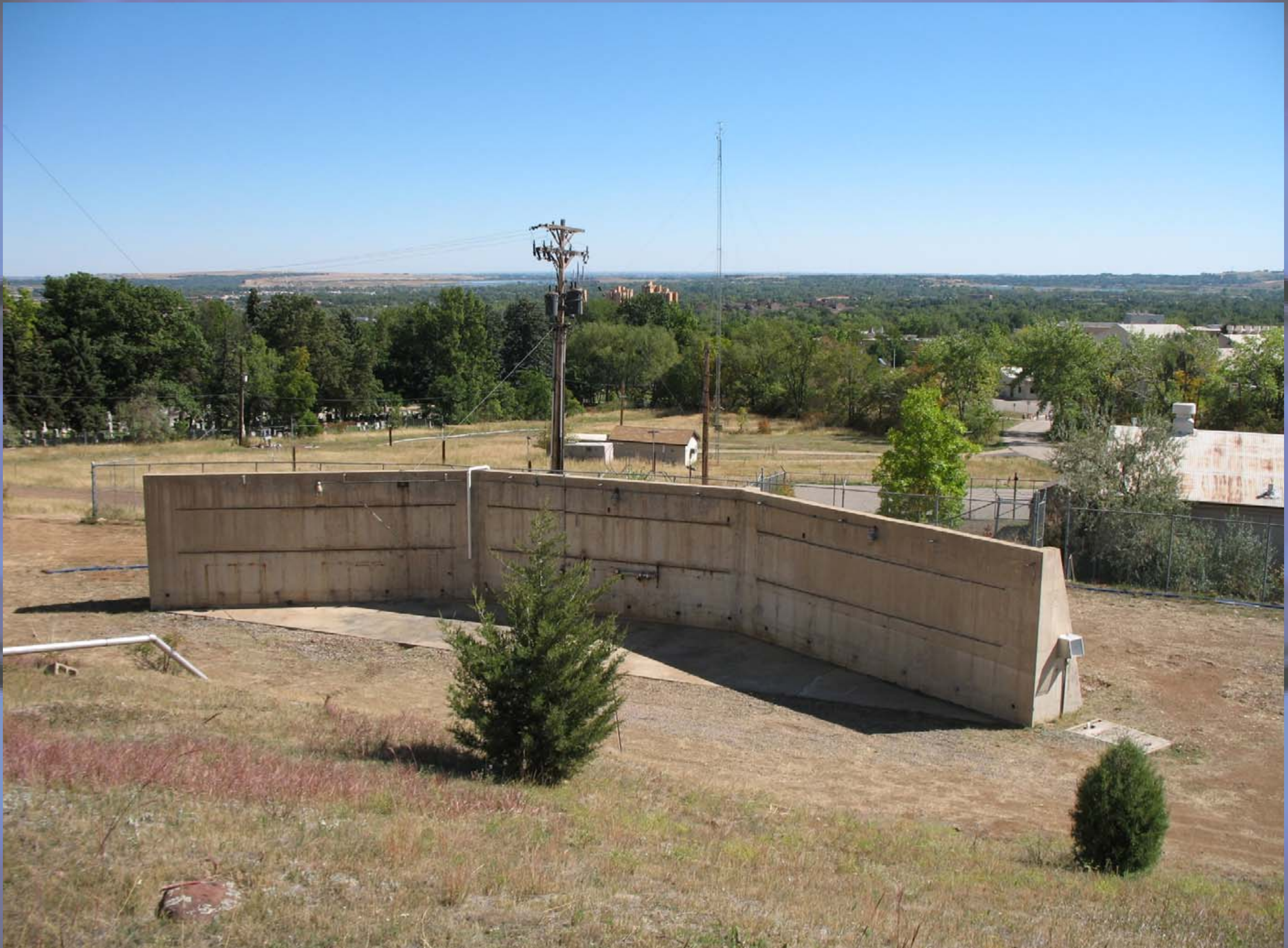
- Construct a pressurized gaseous hydrogen test facility in Boulder
- 2 Closed-loop servo-hydraulic testing systems
  - Tensile, fracture and fatigue properties
- In-situ permeation and solubility in pressurized hydrogen
- Related data and standards activities

Metrology for hydrogen resistant materials

- Slow strain rate tensile testing facility (cathodic charging)
- Electrochemical permeation, solubility, and diffusion rate measurements
- Relationships to chemistry and microstructure
- Measurement methods research
  - Modulus, CTE, and other measures of interatomic forces
  - Measures of microstructural changes with hydrogen
- Related data and standards activities

Other projects within Chemical Science and Engineering Lab and Weights and Measures Division

U.S. Department of Commerce Boulder Laboratories



Boulder Hydrogen Test Facility

- 750 sq ft test laboratory (additional area for control room, hydraulics and gas supply)
  - 2 servo-hydraulic fatigue test machines
    - 20 kip load frame
    - 220 kip load frame
  - 2 high-pressure hydrogen test chambers
    - Max pressure capability??
    - Temperature capability???
  - Mechanical Test Capabilities??
    - Tensile (small size to full pipe-wall thickness)
    - Fatigue C(T), M(T), (da/dN) full pipe-wall thickness
    - Fracture toughness (CTOD, CTOA…)
    - Residual strength (SCT specimen with fatigue pre-crack)
  - Other Capabilities???
  - What's needed to support the Hydrogen Effort???

NIST workshop held in Boulder on Aug 21 and 22, 2007

We had 41 registered attendees from national labs, government, industry, academia

"Thank you" to all participants

Expectations from the Boulder workshop….

Develop roadmap for materials, test procedures, mechanical properties data and standards for future hydrogen pipelines. This data will be used as input into the research plan for the new hydrogen test facility being constructed in Boulder.

Breakout Sessions:

Materials:

Session Chair: Brian Somerday

Codes and Standards:

Session chair: Lou Hayden

Test Methods:

Session Chair: Andrew Duncan

Breakout Session Results…

Materials…..

1) <u>Test relevant materials in hydrogen transportation infrastructure</u>

- **Best practice, industry standard steels, (low and high strength steels X70 and below and X70 and above)**
- **Steels currently in the ground**
- **Materials in components for pipelines (valves, fittings, etc)**
- **Linepipe composites**
- **Storage vessel material**
- **Pressure manifold component mat'l (e.g., stainless steels)**

Materials…

2) Consider important variations in materials

- Welds (fusion zone, heat-affected zone)
- Base metal: assess allowable range of variables
- Hard spots
- Microalloying
- Heat treating
- Strength range within specification
- Chemical banding
- Impurity elements such as phosphorus and sulfur

  (Note: the above list depends on variations created by best practices
- Residual stresses

Materials…

3) Develop advanced tools

- Develop physical models to understand important phenomena for materials in hydrogen transportation infrastructure (e.g., hydrogen transport in materials with gradients, structure-property relationships, behavior of coatings)

- Convene workshop to foster interdisciplinary approach

- Collect information on line pipe steel failures related to hydrogen

Codes and Standards and Safety Results…

(Goals: How to process and evaluate data and techniques, develop design allowables, and safety considerations for test facility and personnel)

Establishment of Goals:

1) <u>Testing commonly used (API 5LX52, SA106B) linepipe steel base metal and weldments</u>:

-For loss of ductility, loss of toughness, fatigue properties (low cycle and high cycle da/dN) at varying ΔK and R values.
-Test materials over a range of temperatures to determine the scope of the embrittlement range.
-Support the prescriptive design method currently planned for B31.12.
-Document and archive test results in a database.

Codes and Standards and Safety Results…

2) <u>Verify the effect of pressure on embrittlement of commonly used (API-5LX52, SA106B) linepipe steel base metal and weldments</u>.

- -Test up to 3000 psi
- -Test up to 15000 psi to determine the maximum pressure limit (if any) for carbon steels.

3) a. <u>Evaluation of microstructure of commonly used pipeline material (API 5LX52, SA106B) base metal and weldments for performance in H_2</u>

b. Based on (a) above, determine what changes to microstructure would improve performance in H_2 .

c. Based on (a) and (b) above, determine what new alloys of C-Mn, C-Mn-Microalloy, and C-low alloy can be developed to improve H_2 performance.

Codes and Standards and Safety Results…

4) Mitigation of hydrogen embrittlement through hydrogen additives or internal coatings.

5) Non-metallic linepipe characterization:

- Permeation – Rates need to be stated for these general types of pipes
- FRP, FRP-Lined (metallic and plastic liners), Plastic, Plastic Fiber Reinforced
- Joints
  Mechanical (Metallic joints, Non-metallic joints), Bonded – Fiber Overwrap, Heat Fusion Welded, Cement Welded
- Composition
  - FRP (Fiber Glass Reinforced, Carbon Fiber Reinforced, Other Fibers, Vinyl Ester, Epoxy)
  - Plastics: HDPE, PEX, Fluoro-plastics, Others ????

Test Techniques and Methods Results……

- Test capabilities…
  - Mat'l: Steels (focus on lower strength) and composites.
  - Gas: Quantify hydrogen concentration, 6-9's purity?, consistent evac and purge procedures
  - Loading rate down to slow strain rate (i.e., 10^{-4}/s to 10^{-7}/s), fatigue rates 0.5 to 1 hz
  - Pressure: overlapping pressures between labs a benefit, up to 15ksi in small chamber and 3000 in large chamber
  - Temperature: -40 to 300F (-80 C? for arctic service), RT to 300F in large chamber ?
  - Data needs: Tensile, K_{th}, LEFM, EPFM, Fatigue, $Fatigue_{th}$, test capabilities should support the validation and further development of consensus codes and standards (e.g., ASME B31.12)

Test Techniques and Methods Results……

Inter-laboratory cooperation/ test program

- Cross-compare test results/ methods (purging, machining, gas purity)
- Understand test methods/results
- Compare laboratory abilities (i.e., choose same sample type, compare different test methods…what needs to be identical and what can remain the same with no change in results?)
- Evaluate hydrogen concentration in materials
- Test hydrogen purity (before, during, after testing???)

Test Techniques and Methods Results……

•Better understanding of hydrogen transport phenomena

•Baseline test procedures (gas purity, sample geometry, pressure range, surface condition, strain rates, temperature, etc.)

•Test Method standardization needed (improved instrumentation and techniques inside chamber…strain gages, LVDT's, clip gages, heating techniques, etc.)

•Collaborative effort between labs in hydrogen purity measurement

•RR effort needed

•Component testing capabilities needed

Test Techniques and Methods Results……

Observations…

•Composite testing will lead to component (fibers and resins) testing

•Develop test methods that will benefit standard design methodology

•Two chambers recommended at NIST:

3 liter capacity at 15000 psi

100 liter capacity at 3000-5000 psi (excess volume filled with solid)

•Fatigue threshold testing very desired but time consuming

•Important to conduct RR

•Duplication of efforts between labs is beneficial for establishment of national capability

Summary

	Materials Session	C&S Session	Test Methods Session
Materials	Steel (X70 and below as well as above X70), composites, Welds, HAZ, FL, valves, vessel mat'l,	Steel (X52, SA-106B), composites, plastics, componenets, BM, HAZ, FL	Steel (focus on lower strength), composites, welds, HAZ
Test methods	Advanced modeling tools	E647, support B31.12,	E8, E338, E647, E1681, E1820, E399, G129, support C&S
Capabilities		2 chambers	2 chambers, Duplication of capabilities an asset
Inter-lab testing			Cross-compare data, RR
Pressure		3000, 15000 psi	3000, 15000 psi
Temperature		Embrittlement range?	-40 to 300F
Gas purity		Effect of additives?,	Quantify amt, purity (6-9's?), purge technique?, sample before and after test
Other	Structure-property relationships	Structure-property relationships	Hydrogen transport phenomena, instrumentation issues

Workshop Report will be available following final editing and will be published as NISTIR 6649

Draft session handouts are available today

Summary

Materials: Steel and composite pipeline and vessel materials and their welds and/or joints, consider material variations created by best practices, advanced tools, consider X70 and below as well as >X70. Materials in components.

Codes and Stds: Commonly used pipeline steels (X52) and composites for associated material properties to support prescriptive design method proposed for B31.12. Test at wide temp and pressure (3 kpsi-15 kpsi) ranges. Evaluate pressure effects on embrittlement. Structure-property relationships. Non-metallic linepipe characterization.

Test Techniques and Methods: Small to large dia pipeline material (steel and composite) up to 1" wall thickness (start with X52 steel), hydrogen 6-9's purity, low strain rate capability, 3 kpsi-20kpsi psi chamber capability, wide temp range capability, test methods and data to support consensus codes and standards (ASME B31.12), interlaboratory test program (RR), duplication of test efforts beneficial to all labs.