Hydrogen Delivery Pipeline Working Group Workshop
September 25-26, 2007 ♦ Center for Hydrogen Research, Aiken, GA

WORKSHOP SUMMARY

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1. Introduction

The DOE Hydrogen Pipeline Working Group (PWG) met on September 25-26, 2007, in conjunction with the September 24-25 Joint ASME/SRNL Materials and Components for Hydrogen Infrastructure Codes and Standards Workshop (see Appendices A & B). The PWG workshop was sponsored by the DOE Hydrogen, Fuel Cells and Infrastructure Technologies Program, and was hosted at the Center for Hydrogen Research, adjacent to Savannah River National Laboratory.

The PWG workshop served as a detailed review of the progress and results of pipeline research and development projects sponsored by DOE. More than 30 researchers and industry representatives attended, to share research results and discuss the current challenges and future goals for hydrogen pipeline research and development (R&D). One of the near-term goals of the PWG is to develop a set of standard materials test methods and procedures for the research program and to lay out a round-robin testing plan to assure that all participants are obtaining the same results. The draft testing plan was presented for review and discussion by workshop participants. The workshop also included presentations from each of the DOE-funded pipeline research projects, as well as facilitated discussion sessions. The research project presentations are provided at http://www1.eere.energy.gov/hydrogenandfuelcells/wkshp_pipeline_group.html.

2. Pipeline Working Group Plans for Round Robin Testing and Routine Research Testing

The outcomes from the workshop of the PWG Testing Subgroup, held on August 22, 2007 in Boulder, Colorado, were presented to the PWG for review and discussion. It was determined at the Boulder workshop that limited round robin testing was appropriate among the R&D program participants to assure proper function of new/upgraded experimental systems using high pressure hydrogen and to establish the reproducibility limits of fundamental test results. The group determined that the initial round robin efforts would focus on limited tensile testing (sub-size specimens) and permeation testing. It is not the intent of the round robin testing to replace the current R&D programs at the participating institutions. The round robin testing is designed to assure that all participants are using the same ASTM test procedures and obtain similar results (within standard error) when implemented. All delivery research projects should continue to follow the R&D plan described in their Annual Operating Plan.

3. Facilitated Discussion on Planned DOE Steel Pipeline Routine Research Testing: ASME and PWG Workshop Participants

Discussion Question 1: Are there additional tests that should be included for steel pipeline materials in DOE's test matrix?

- S-N fatigue curve
  - Measured under high pressure
    - Requires different equipment than the labs have now
- Flawed burst test
  - Measured on an actual pipe or vessel
  - Sandia has some promising data from the 80's
- **KD-10:** The ASME Boiler and Pressure Vessel Code Committee approved new fracture control rules for Section VIII, Division 3 vessels in 2006 (vessels to be used in high pressure gaseous hydrogen transport and storage). These rules have been incorporated into a new **Article KD-10** in Division 3.
  - Article KD-10 contains test methods for measuring the following fracture mechanics properties, and these methods could be used for the PWG round robin
    - Plane-strain fracture toughness, K_{IC}
    - Threshold stress intensity factor for hydrogen-assisted cracking, K_{IH}
    - Fatigue-crack-growth rate, da/dn
  - Article KD-10 specifies that specimens be tested from three locations: the base metal, the weld metal, and the HAZ of welded joints
- Need to test both base metal and weld metal (HAZ)
  - Test ERW weld seams (including submerged arc welds)
- Component testing should be included
- Is notched tensile testing needed?—it is not as important from a codes and standards vantage point

    - It is needed for understanding the loss of ductility
    - It is useful in R&D for materials screening, since it is not an expensive test
    - If it is useful, do not exclude it just because it is not needed to satisfy codes and standards requirements
- The Codes and Standards community has expressed a need for more data to satisfy codes and standards needs. Mark Paster suggested that they provide a *very specific* list of their data needs to DOE. The DOE Hydrogen Delivery subprogram *may* be able to generate some of this data through their research; the gaps will need to be filled by the DOE Hydrogen Codes and Standards subprogram.

Discussion Question 2: What should test conditions be?

- What hydrogen concentration (purity), if any, should be specified for testing?
    - Agree that all participants in the round robin for a particular test/material should use the same hydrogen quality. Hydrogen quality should always be reported for all tests.
    - NIST participants suggested two possible experiments around hydrogen quality:
        - Test the hydrogen quality at the "inlet" and "exit" from the test fixture, to see what "sticks" inside the pipeline and to see what gets introduced by reactions within the pipeline
        - Start with high purity hydrogen and add contaminants in well known, measured ways to see effects of contaminants
    - There is literature that small amounts of oxygen, carbon monoxide, or other contaminants may inhibit embrittlement; other contaminants (e.g., H_2S) may increase embrittlement
        - ASME Article KD-10 has limits for the concentration of some contaminant species
        - Research is being conducted to determine what contaminants are bad for fuel cells and what hydrogen quality will be required to meet fuel cell specs; tests should be designed to account for these efforts
        - It would be useful to have a list of species that inhibit or exacerbate embrittlement
- What hydrogen pressure should be specified for testing?
    - Higher pressure increases embrittlement
    - All tests should be run at higher pressures than we plan to run in operating conditions
        - For 3,000 psi pipeline, conduct some testing up to 5,000 psi
        - For 15,000 psi pressure vessels, conduct some testing up to 20,000 psi
    - R&D to investigate degradation thresholds, etc., would be useful to better understand the relationship between hydrogen embrittlement and pressure
- Test different materials and different heat treatments within a grade (i.e. X52) to better understand heat-to-heat variations and why these occur (effects of microstructure)
    - Grades vary in composition and heat treatment.
    - Use statistical analysis to target uncertainty

- Where to take the sample from is an issue, especially for the heat affected zone—sampling methods must be specific for the HAZ.
    - Sour gas data from the petroleum industry may provide some guidance on test conditions and sampling methods: see reports published by the American Petroleum Institute (API) and Petroleum Research Council (PRC).
- K-Threshold test methods are specified in ASME E1681 and KD-10
    - K-Threshold test methods should specify a surface preparation method; this is covered in KD-10
- Fatigue Crack Growth: testing at 1 Hz is not sufficient; needs to go lower
    - Some testing should be done at 0.1 Hz
    - Some experiments at very low frequencies should also be done to understand the relationships (start at 0.1 Hz and reduce to the point of plateau)
        - In the future, accelerated testing techniques should be discussed and considered
- High temperature testing is not as important

Discussion Question 3: Does the round robin testing plan seem reasonable?

- What steels should be used?
    - One high strength steel should be used, e.g., X100
    - Steels to be used in round robin will be: X100 and X52
- The purpose of the round robin is to see if the different labs participating in materials R&D can run the same tests on the same materials and get the same results
- The round robin testing is anticipated to start in FY08, with tests that can be carried out with available equipment and expertise at the various participating institutions
- All round-robin-testing (RRT) members will be expected to carefully and fully document all test conditions

Discussion Question 4: How do the roles of DOE and NIST differ?

- The DOE Hydrogen Program addresses research aimed at making technologies possible; once these technologies become commercial possibilities, NIST focuses on addressing standards issues surrounding their commercial use.
- NIST may be able to help with filling some of the data gaps for the codes and standards community
- NIST may be able to play a role in component fatigue testing if they can develop that capability.

4. Facilitated Discussion on Next Steps for Hydrogen Delivery Research: ASME and PWG Workshop Participants

Discussion Question: What is needed in the area of R&D to lower hydrogen delivery costs to the target level of $1.00/kg?

- More testing is needed on FRP materials
    - Chemical tests during and after exposure to hydrogen to determine what is happening to the structure and where the hydrogen is going. Tests should be conducted both with and without water exposure
- Emphasize component testing and systems work to transcend between results in the lab and in field installations
    - In the short term, test key components under simulated service (e.g., include elbows and branches)
    - In the longer term, construct a hydrogen delivery "test loop" that includes all the delivery infrastructure (i.e., a few miles of pipeline with fittings, compressors, etc., for full scale testing)
- Gather more data on what chemistry current pipeline operators are using
- Be careful with setting chemistry limits—these may be difficult for manufacturers to comply with
- Conduct R&D into ways to modify surface chemistries of low-cost materials (rather than just focusing on mechanical barriers)

5. Facilitated Discussion of the Pipeline Working Group

Discussion Question 1: What is the pathway forward for steel pipelines for hydrogen?

- **Top priority:** determine whether cyclic fatigue is a problem
- Suspend work on coatings until/unless it is determined that liners are needed
- Develop a better understanding of the effects of hydrogen on welds and heat affected zones (HAZ); verify no problems with sustained load cracking.
- Generate some data between 3,000 and 5,000 psi, and up to 20,000 psi for tanks
- Understand the benefits (or penalties) of trace contaminants
- Identify the best steel for hydrogen service, in terms of both performance and cost (include consideration of new/alternative alloys)
- Explore options for reducing steel pipeline installation cost
- Increase fundamental understanding of hydrogen embrittlement, including microstructure, etc., for safety research
- Develop a database for use in structural analysis of pipelines (pipeline dimensions, etc.)

Discussion Question 2: What are the top priorities for FRP research in the near term?

- Study axial loading due to winding direction (and potential impacts of geotechnical phenomena), third-party damage, etc.
- Study environmental effects on polymers (e.g., water)
- Apply the same rigor to FRP testing as we have to testing of metals
- Tap polymer experts from industry to join the PWG?

Discussion Question 3: Additional thoughts and comments on the round robin testing plan?

- Cyclic Fatigue test conditions:
  - 1 Hz
  - R = 0.1
  - 2,000 psi
  - 3 samples
- Add S-N tests on "good" and "bad" materials; this is a lower priority
- Add flawed burst test of a welded pipeline segment (rising load? cyclic?); this is also a lower priority
- The testing subgroup will get together to decide and record detailed testing parameters

6. Next Meetings

- Pipeline Working Group
  - Will meet twice yearly in face-to-face workshops with one mid-term conference call. The 2008 workshops are as follows:
    - February 20-21, 2008 at Sandia National Lab
    - September 2008 before/after the materials conference in Jackson Hole, WY
- Testing Subgroup
  - Will meet monthly or bimonthly via conference call
- Mark Paster is retiring from DOE and Tim Armstrong (on detail from Oak Ridge National Laboratory) will be taking over management of the Pipeline Working Group. For questions on upcoming meetings or next steps, please contact Tim at armstront@ornl.gov.

7. Summary of Action Items

Name	Action Item
All PIs in the DOE Pipeline Working Group	• Complete and provide to Tim Armstrong a 1-page slide that summarizes their R&D project. **Tim** will provide a template and deadline.
Permeability R&D (Zhili Feng)	• Provide GLEEBLE samples to NIST in ¼ inch size • Consider conducting permeability measurements at higher temperatures
CTC Pipeline R&D (CTC – Dave Moyer)	• Follow up with NIST on the problem of fabricating and working with test coupons (CTC described problem gripping coupons and expressed issue with flat coupon representing filament-wound structure)
Steel Pipeline R&D (Secat – Subodh Das)	• Conduct all testing *in-situ* • Suspend work on coatings until it is determined that liners are needed • Consider using a different testing method than ABI; the ABI

	process has not generally been accepted by industry and cannot be relied upon for fracture toughness data • In future presentations, present all the conditions under which data was taken so that data is more meaningful and useful
Angelique Lasseigne, NIST	• Will provide information (including a presentation) to the group on high C-N steel
Composite Pipeline R&D Project (Barton Smith and Thad Adams)	• Conduct testing *in-situ* in the presence of hydrogen • Consider alternative stress tests: e.g., crack the pipe perpendicular to the fiber direction, expose to hydrogen, and examine the effects on the fiber interfaces • A leakage rate of 0.4% may be too high – evaluate decreasing the permeation by 10 times (to levels similar to steel)
All	• Think about what questions need to be answered related to use of FRP pipelines for hydrogen service, what kinds of tests will be needed, and which questions should have priority
DOE	• Consider inviting more surface chemistry, modeling, and sensor development experts to the next PWG workshop; consider recruiting more industry members.
DOE and Round Robin Test participants	• Put together document outlining tests and testing procedures for the first round of the Round Robin Test
CTC	• Fabricate test samples for the labs involved in the round robin test

Appendix A – Attendees

Name	Organization	Email	Phone
Thad Adams	Savannah River National Lab	thad.adams@srnl.doe.gov	803-725-5510
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Appendix B – Agenda

ASME/SRNL Materials and Components for Hydrogen Infrastructure Codes and Standards Workshop

and the

DOE Hydrogen Pipeline Working Group Workshop

Sponsored by SRNL, ASME, and DOE
Center for Hydrogen Research, Aiken, SC
Garden Conference Center
September 23-26, 2007

Sunday, September 23, 2007

6:00 – 7:30 pm	Registration
6:15 – 7:30 pm	Opening Reception ▪ *Sponsored by ASME*

Monday, September 24, 2007

Joint Materials and Components for Hydrogen Infrastructure Codes and Standards Workshop

8:00 am	Continental Breakfast
8:30 am	Welcome ▪ **Natraj Iyer**, Director, Material Science & Technology, Savannah River National Laboratory (SRNL)
8:45 am	Opening Remarks ▪ **Cheryl Cabbil**, Acting Laboratory Director, SRNL
9:00 am	Overview of ASME Hydrogen Codes and Standards Development ▪ **John Koehr**, ASME
9:30 am	Material and Component Issues for Gaseous Hydrogen Service ▪ **George Rawls**, SRNL
10:15 am	Break

10:30 am — DOT Hydrogen Program
- **William Chernicoff**, U.S. Department of Transportation, Research and Innovative Technology Administration

11:30 – 1:00 pm — Lunch
SC Hydrogen Coalition Presentation
- **Fred Humes**

1:00 pm — Development of a 15,000 psig Fitting for Hydrogen Service
- **Shelly Tang**, Swagelok

1:45 pm — ASME B31.12 Hydrogen Piping Code
- **Lou Hayden**, Chairman B31.12

2:30 pm — Break

2:50 pm — Development of Code Rules for 15,000 psi H_2 Vessels
- **Mahendra Rana**, Praxair, Chairman H_2 High Pressure Vessels

3:35 pm — Composite Vessels for Hydrogen Service
- **Norm Newhouse**, Lincoln Composites

4:20 pm — Adjourn
Dinner on Own

Tuesday, September 25, 2007

ASME/SRNL Materials and Components for Hydrogen Infrastructure Codes and Standards Workshop

7:30 am — Continental Breakfast

8:00 am — Meeting Announcements

8:10 am — Opening Remarks
- **Mark Paster**, U.S. Department of Energy (DOE)

8:30 am — DOE Hydrogen Delivery Program Overview
- **Tim Armstrong**, Oak Ridge National Laboratory (ORNL)

9:00 am — Material Testing for Hydrogen Pipelines (and Storage Vessels)
- Results… NIST Workshop on Materials Test Procedures for Hydrogen Pipelines: **Dave McColskey**, NIST
- Testing Subgroup Workshop on Critical Property Needs: **Tim Armstrong**, ORNL
- ASME Material Testing Priorities for H2 Infrastructure: **Lou Hayden**
- Discussion: **All**

11:30 am — Hydrogen Delivery Analysis Models
- **Mark Paster**, DOE

12:00 – 1:30 pm Lunch

Tuesday, September 25, 2007

DOE Hydrogen Pipeline Working Group Workshop

1:30 pm — Mechanical Properties of Structural Steels in Hydrogen
- **Brian Somerday**, Sandia National Laboratories

2:30 pm — Hydrogen Embrittlement: Fundamentals, Modeling, and Experiments
- **Petros Sofronis**, University of Illinois, Materials Research Laboratory

3:30 pm — Break

3:45 pm — Permeation, Diffusion, Solubility Measurements: Results and Issues
- **Zhili Feng**, ORNL
- **Thad Adams**, SRNL

4:45 pm — Pipeline and Pressure Vessel R&D Under the Hydrogen Regional Infrastructure Program in Pennsylvania
- **Kevin Klug,** Concurrent Technologies Corporation

5:30 pm — Adjourn

Wednesday, September 26, 2007

DOE Hydrogen Pipeline Working Group Workshop

8:00 am — Materials Solutions for Hydrogen Delivery in Pipelines
- **Subodh Das**, Secat

9:00 am — Composite Technology for Hydrogen Pipelines
- **Bart Smith**, ORNL
- **Thad Adams**, SRNL

10:00 am — Break

10:30 am — Pipeline Working Group Discussion and Path Forward

11:30 am — Adjourn