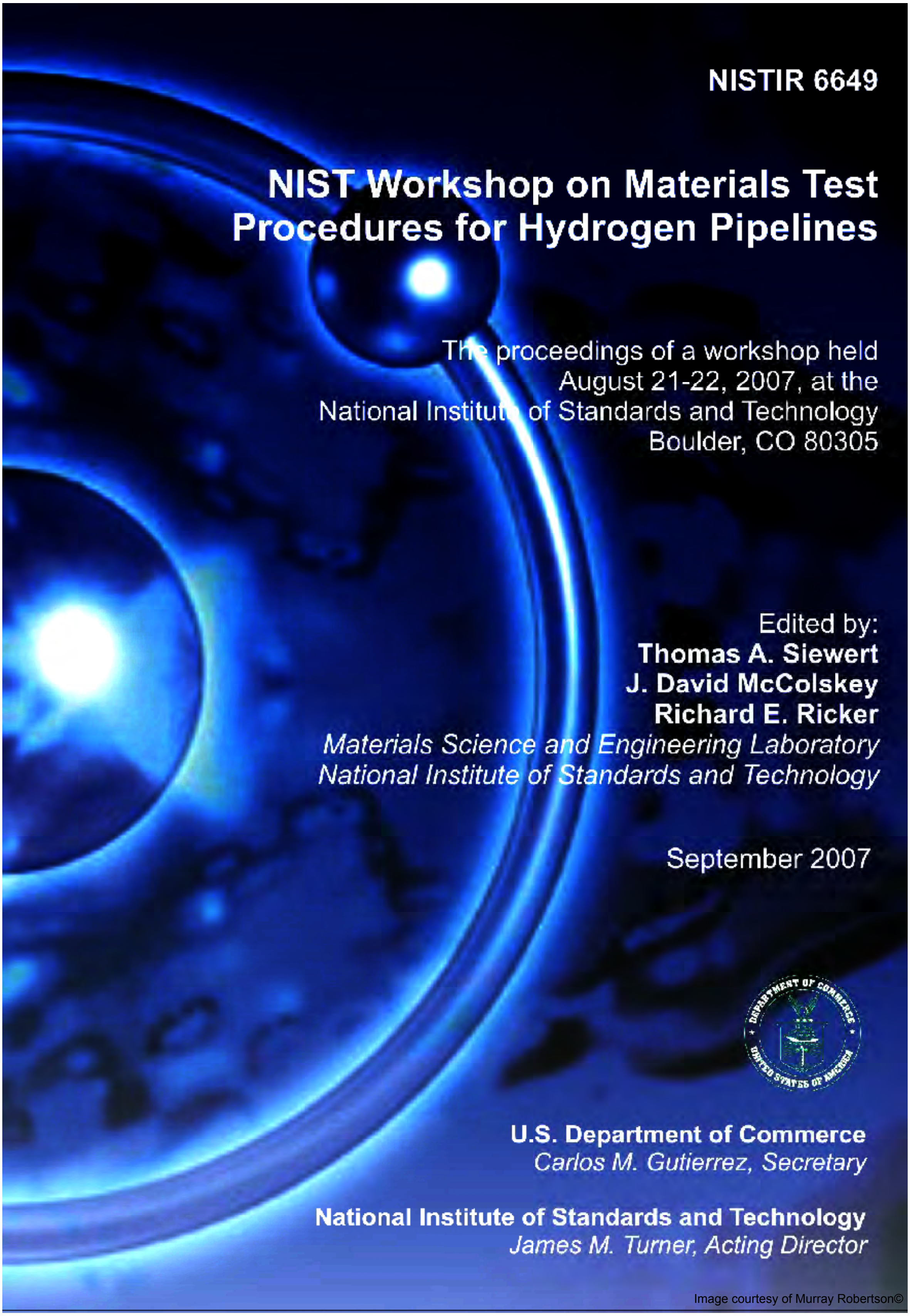


Image courtesy of Murray Robertson©

NISTIR 6649

NIST Workshop on Materials Test Procedures for Hydrogen Pipelines

The proceedings of a workshop held
August 21-22, 2007, at the
National Institute of Standards and Technology
Boulder, CO 80305

Edited by:
Thomas A. Siewert
J. David McColskey
Richard E. Ricker
Materials Science and Engineering Laboratory
National Institute of Standards and Technology

September 2007

U.S. Department of Commerce
Carlos M. Gutierrez, Secretary

National Institute of Standards and Technology
James M. Turner, Acting Director

Contents

Executive Summary

In 2007, the National Institute of Standards and Technology greatly expanded its efforts in support of the use of hydrogen as a fuel. To obtain user feedback on plans for a facility to evaluate and refine mechanical testing procedures for hydrogen pipelines, we held a workshop in Boulder, Colorado on August 21 and 22, 2007.

The workshop had 46 participants representing pipeline owners, industry and standards organizations, academic researchers, national laboratories, and government agencies. The workshop began with presentations on NIST (its mission and capabilities), the proposed NIST program on materials compatibility with hydrogen, activities in other government organizations (DOE and DOT), current standards activities and needs for supporting data (especially in ASME), and a description of the roadmap desired from the workshop. Next, the attendees divided into three working groups:

Materials – chaired by Brian Somerday, Sandia National Laboratory-Livermore,
Test Techniques and Methods – chaired by Andrew Duncan, Savannah River National Laboratory, and
Codes, Standards, and Safety – chaired by Lou Hayden, consultant.

At the end of the first day, we heard a short report from each group (to compare approaches and the standards and data needs being identified by each group). We continued the breakout sessions on the second day, and then met to summarize the findings and develop an overall list of needs. While detailed lists of all the needs are included in the reports of each group, the combined participants reviewed only the top three needs identified by each group and then ranked them in descending order of importance. These were:

Materials

- Develop advanced tools (measurement techniques, analytical methods, and models)
- Focus on current construction linepipe steels, with strengths under X70 (rather than other alloy types)
- Assess the performance of girth welds (and HAZ)

Test Techniques and Methods

- Complete the NIST Test Facility (following detailed guidance listed in the group report)
- Conduct a round robin (to assess repeatability between various hydrogen laboratories)
- Measure the performance of components (both fiber and matrix in composite linepipe materials as well as welds and their heat affected zones in welded linepipe steel)

Codes and Standards

- Measure the performance of current pipeline construction materials (especially those in current use such as API-X52 and SA106B)
- Study the effect of pressure

Evaluate the effect of microstructure

Evaluate non-metallic pipe (while just outside a top-three ranking, a topic the group felt could not be overlooked)

While most participants felt that 1.5 days for the workshop was too short to complete all tasks necessary for a thorough program plan, the recommendations made in the workshop sessions gives NIST a clear picture as to its necessary course of action with regards to pressurized hydrogen testing of linepipe steels, composite linepipes, and their associated components.

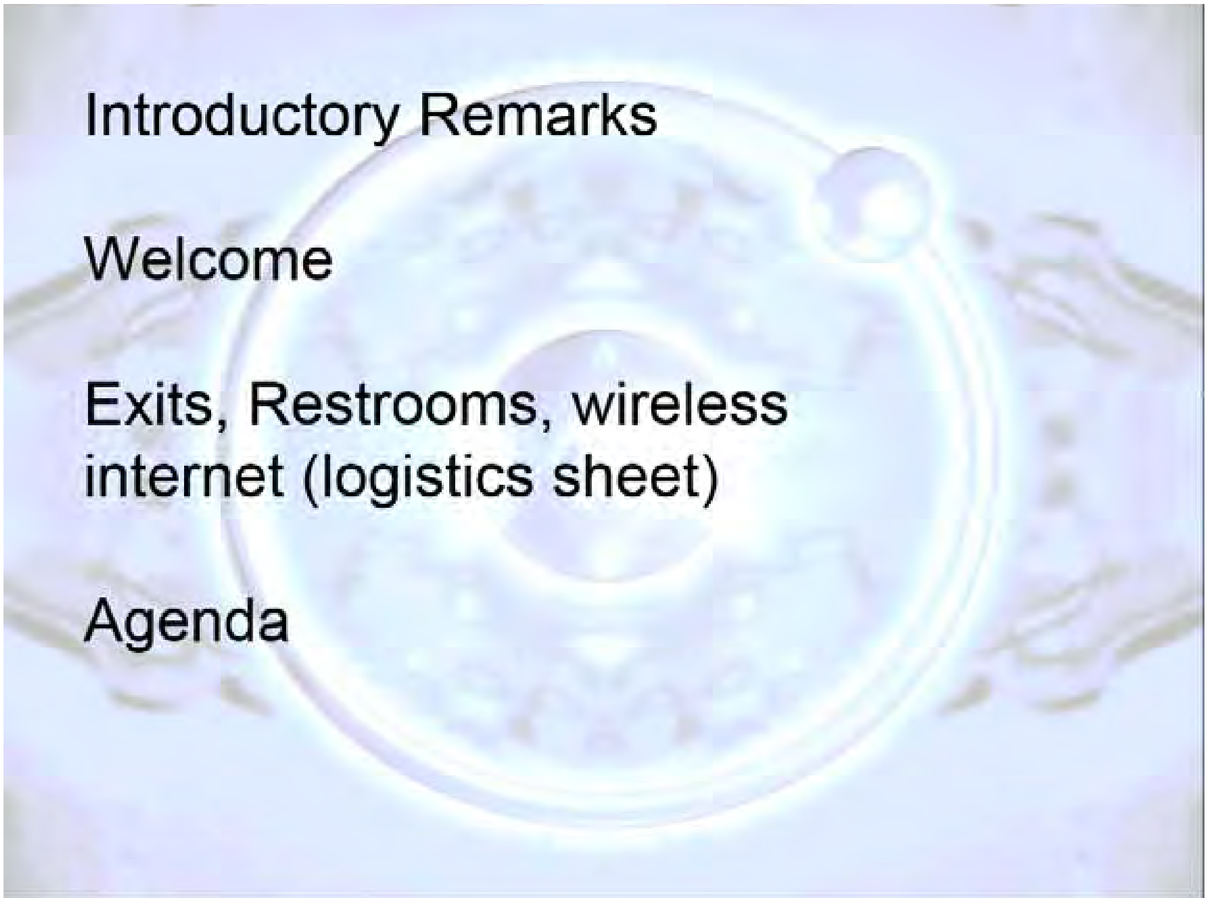
Acknowledgements

The Editors appreciate the contributions of the Presenters, the Working Group Leaders (Andrew Duncan, Lou Hayden, and Brian Somerday), the Recorders who keep the notes of the groups (Jenny Collins, Kamalu Koenig, Angelique Lasseigne, Andy Roubidoux, and Matt Treinen), and the Conference Support Staff (Wendy McBride, Marc Dvorak, and Ross Rentz).

NIST Workshop on Materials Test Procedures for Hydrogen Pipelines

August 21-22, 2007
Boulder, CO

U.S. Department of Commerce Boulder Laboratories


Introductory Remarks
Welcome
Exits, Restrooms, wireless internet (logistics sheet)
Agenda

NIST: Promoting U.S. Innovation & Industrial Competitiveness

Stephanie A. Hooker
Materials Reliability Division
National Institute of Standards and Technology (NIST)

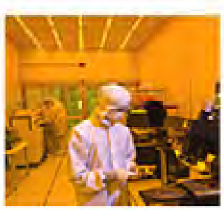
Materials Science and Engineering Laboratory
Materials Reliability Division
NIST
National Institute of Standards and Technology

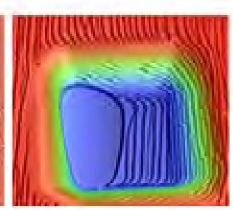
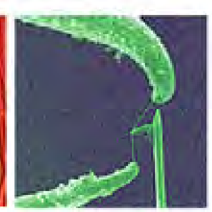
Promoting Industry Growth

NIST Mission: To promote U.S. innovation and industrial competitiveness by advancing

- *measurement science*
- *standards*, and
- *technology*

in ways that enhance economic security and improve our quality of life



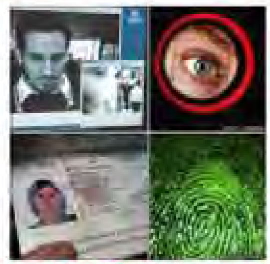
NIST Strategies for Success

- ➢ Focus on our unique mission & role
- ➢ Address national priorities
- ➢ Partner with others to achieve high impact

Making early automobiles safer

Providing medical testing standards

Enabling the first material classification



Improving identification security

Our Products

- ➢ **Measurement Research**
  - ➢ 2,100 publications/year
- ➢ **Standard Reference Data**
  - ➢ 5,000 units sold/ year
- ➢ **Standard Reference Materials**
  - ➢ >1,200 products available
  - ➢ 30,000 units sold/year
- ➢ **Calibrations and Tests**
  - ➢ 3,200 items calibrated/year
- ➢ **Laboratory Accreditation**
  - ➢ 826 accreditations
- ➢ **Standards Committees**
  - ➢ 450 committees

Setting Priorities

- Assess "health" of the U.S. measurement system
- Identify specific industry measurement needs that pose technical barriers to innovation
- Recommend actions to achieve solutions to priority measurement neeeds

What we need from you

- Advice on setting research priorities
  - Needs for data, test methods, inspection methods
  - Barriers/gaps/opportunities
  - Timelines
- Review of current plans
  - NIST-Boulder H2 Pipeline Test Facility
- Research collaborations
  - Technique validation
  - Inter-laboratory testing
  - Samples
  - Databases

NIST Program for Evaluation of Materials Compatibility with Hydrogen

Richard E. Ricker

Metallurgy Division
Materials Science and Engineering Laboratory
National Institute of Standards and Technology
Gaithersburg, MD 20899-8553
(richard.ricker@nist.gov)

NIST Workshop on
Materials Test Procedures for Hydrogen Pipelines
August 21-22, 2007

NIST Hydrogen Program

Overview

MSEL

Why NIST?

- Mission
- History (pipelines, hydrogen, fracture, standards, mandates, etc.)
- Capabilities, resources, and experience
- Congressional Act - ACI

What will NIST do?

- Four challenges for the hydrogen economy
- All materials that may be used to contain hydrogen
- Test in hydrogen gas to emulate service and establish allowable levels
- Laboratory characterization tests to understand mechanisms, rate determining processes and relationships to microstructure

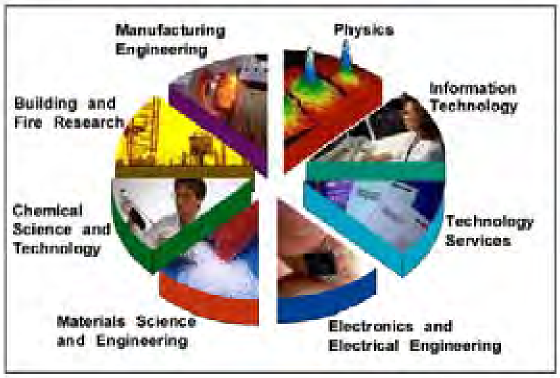
Measurement Issues?

- H charging conditions
- Strain rate effects (or frequency effects in fatigue)
- Permeation, diffusivity, and solubility issues
- Microstructure, trapping, and cracking
- Susceptibility

Why NIST?
Mission

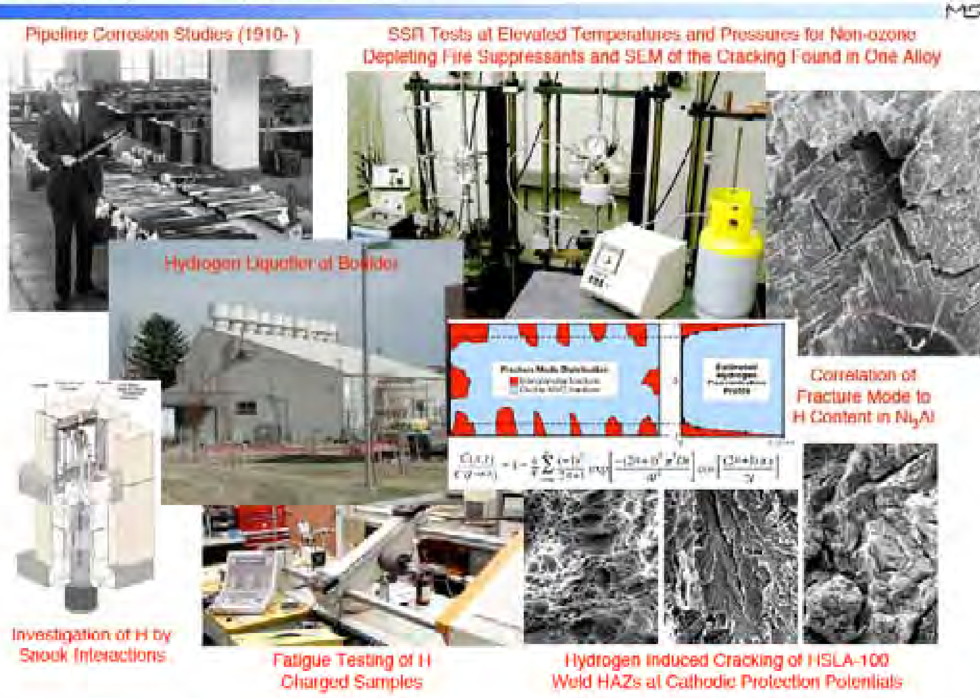
MSEL

NIST's mission is to promote U.S. innovation and industrial competitiveness by advancing measurement science, standards, and technology in ways that enhance economic security and improve our quality of life.



Why NIST?
History, Capabilities, and Resources

MSEL



Why NIST?
American Competitiveness Initiative

MSEL

Overcoming Technical Barriers to the Hydrogen Economy

DOE, NSF, and NIST challenged to work together to overcome the technical barriers that could inhibit the development and growth of hydrogen fuels


OVERCOMING TECHNOLOGICAL BARRIERS TO THE HYDROGEN ECONOMY

The "hydrogen economy" would use domestic sources of energy to create hydrogen gas, which in turn could be used as a transportation fuel. Other possibilities include using hydrogen-bearing fuels such as alcohol or natural gas as potentially economical and long lasting sources of electrical power for portable electronics such as cell phones and laptop computers, or even for powering buildings that are remote from power lines.

Many technical and economic hurdles remain before these technologies can be made widely available. Reducing costs, improving efficiencies, and making the technology reliable enough for everyday use will all be important. If these technical goals are achieved, the broad use of hydrogen as a fuel may prove to have environmental advantages as well. The Department of Energy is leading the President's Hydrogen Fuel Initiative to achieve these goals.

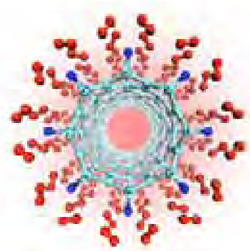
NIST research helps support this developing technology in many ways. Projects currently under way at NIST are providing measurements, data, and technologies needed to develop and test the performance of hydrogen-based power sources and to improve the efficiency of hydrogen production methods.

A new imaging facility at the NIST Center for Neutron Research provides a rare portal for visualizing water and hydrogen transport in fuel cells. Neutrons reveal how water forms and moves while a fuel cell is operating. Mastering the combined challenge of managing incoming humidity, proper hydration of fuel cell membranes, and the handling of water byproducts is essential to the development of fuel cells that are practical for automotive and residential applications as well as portable devices.

NIST Enabling the Hydrogen Economy Initiative
Four Challenges

MSEL

1. Making Better Fuel Cells



2. Storing More Hydrogen

3. Creating Consensus Standards

4. Ensuring Fair Trade

What?
NIST Hydrogen Program at a Glance

MSEL

Fracture resistance in hydrogen gas
- Construct a testing facility at Boulder
- Closed-loop servo-hydraulic testing
  - Tensile
  - Fracture toughness, (K_{th}, da/dt, etc.)
  - Fatigue (ΔK_{th}, da/dN, etc.)
- Related data and standards activities

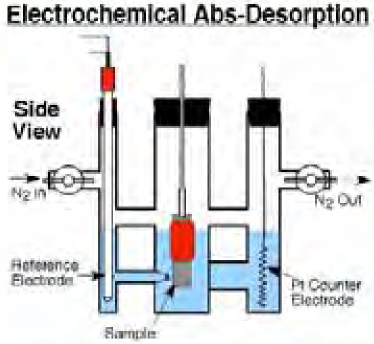
Metrology for hydrogen resistant materials
- Slow strain rate tensile testing facility (cathodic charging)
- Electrochemical permeation, solubility, and diffusion rate measurements
- Relationships to chemistry and microstructure
- Measurement methods research
  - Modulus, CTE, and other measures of interatomic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

Other hydrogen economy structural materials issues
- Vehicle storage containers, tubing, and fuel cell materials (?)
- Compressor materials (high strength wear resistant materials)

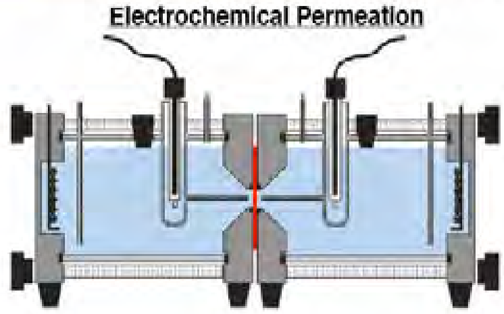
Examples of Experiments
Correlation of H diffusion and embrittlement

MSEL

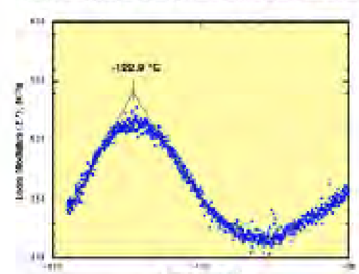
Electrochemical Abs-Desorption



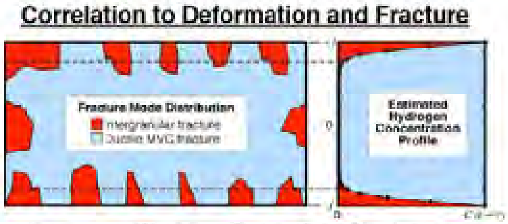
Electrochemical Permeation



Internal Friction - Snoek Peaks



Correlation to Deformation and Fracture

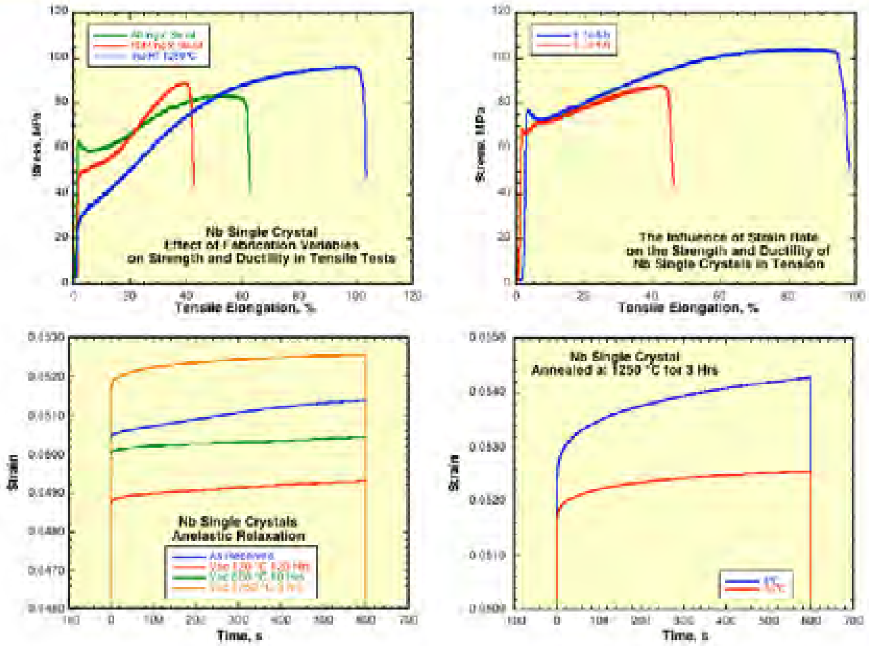


$$\frac{C(x,t)}{C(t\to\infty)} = 1 - \frac{4}{\pi}\sum_{n=0}^{\infty}\frac{(-1)^n}{2n+1}\exp\left[\frac{-(2n+1)^2\pi^2 Dt}{4l^2}\right]\cos\left[\frac{(2n+1)\pi x}{2l}\right]$$

Ultra High Purity Nb Single Crystals

Evidence for H uptake during fabrication and polishing

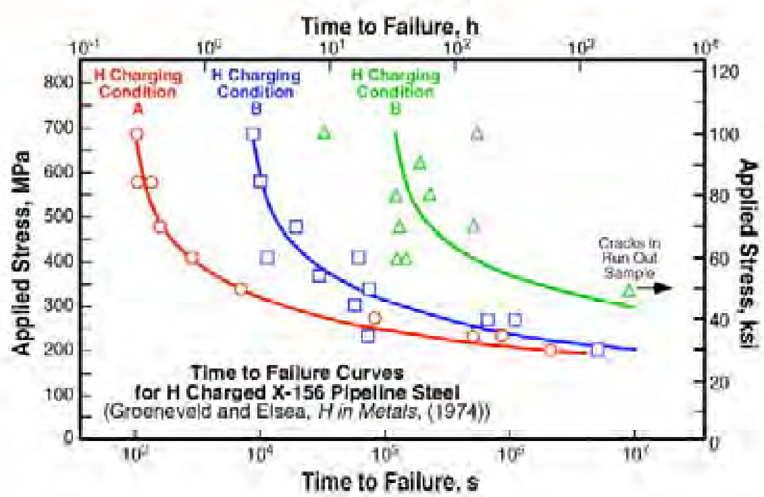
MSEL



Measurement Issues

H charging conditions

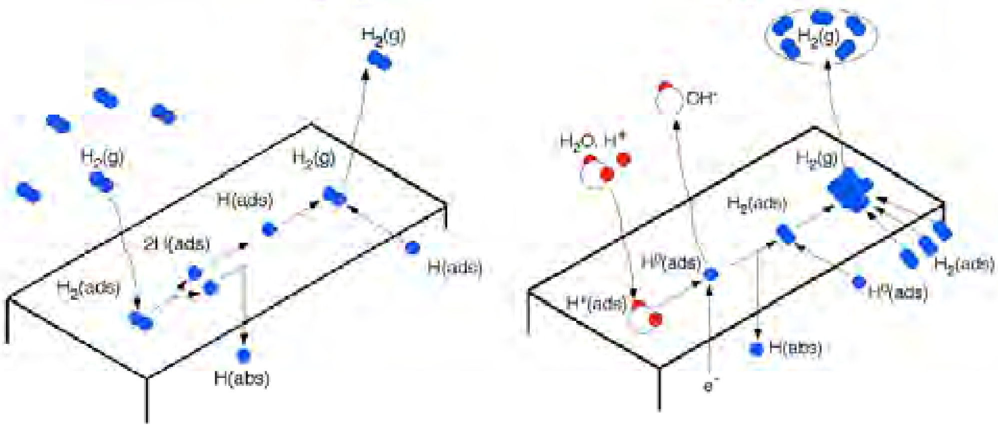
MSEL



Measurement Issues

H charging conditions

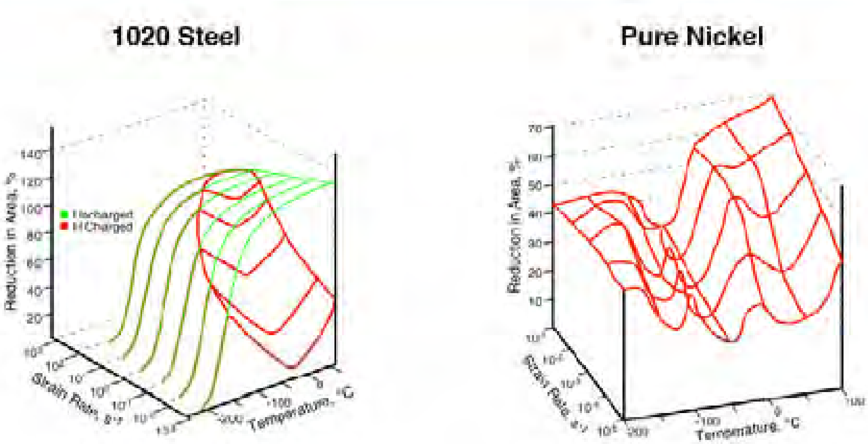
MSEL



Measurement Issues

Temperature and strain rate effects

MSEL

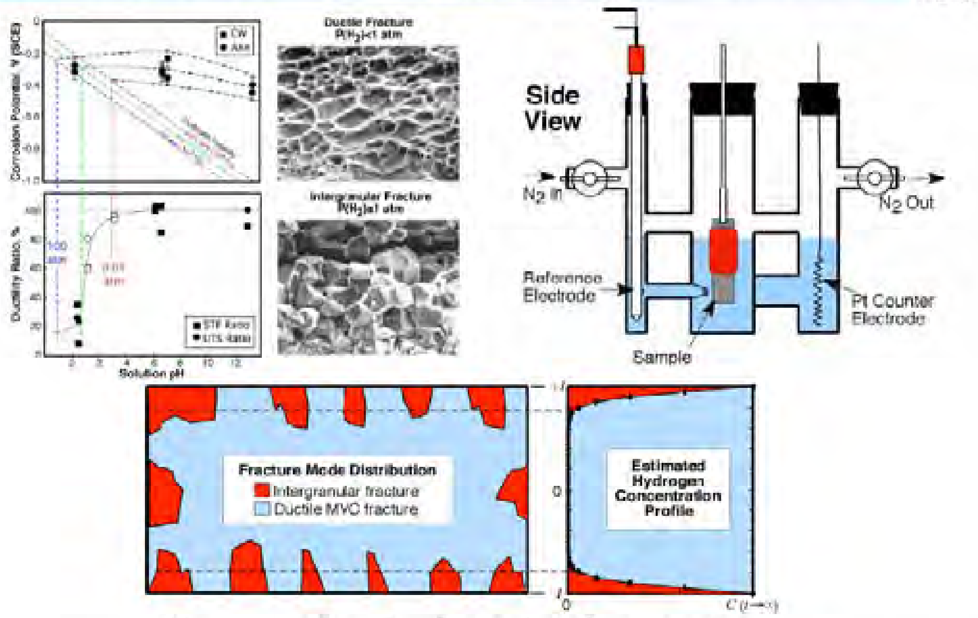


T. Toh and W. M. Baldwin, *SCC and Embrittlement*, W. D. Robertson edt. 176, (1956).

A. H. Windle and G. C. Smith, *Metal Sci. J.*, **4**, 136 (1970)
G. C. Smith, *Hydrogen in Metals*, ASM, 485 (1974)

Measurement Issues
Relationships between diffusion, solubility, trapping, and ductility measurements

MSEL



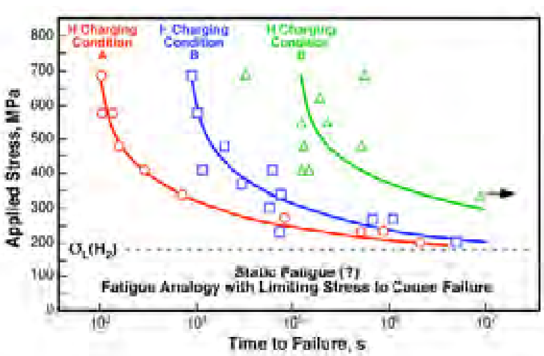
$$\frac{C(x,t)}{C(t\to\infty)} = 1 - \frac{4}{\pi}\sum_{n=0}^{\infty}\frac{(-1)^n}{2n+1}\exp\left[\frac{-(2n+1)^2\pi^2 Dt}{4l^2}\right]\cos\left[\frac{(2n+1)\pi x}{2l}\right]$$

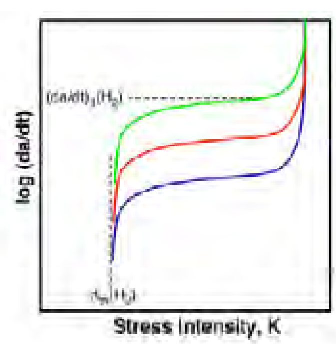
Measurement Issues
Susceptibility

MSEL

Everyone knows what it means, but how do we quantify it?

- Ductility ratios STF(H/no H), RA(H/no H)
- Threshold stress intensities as a function of P(H) or [H]
- Crack propagation rates as a function of P(H) or [H]
- Other (?)





Conclusions

1. **Workshop objective**
2. **The fundamental scientific understanding must be sufficient to insure that the rate determining processes have been correctly identified.**

Hydrogen Effects in Materials, Jackson Hole, WY Sept. 2008
http://www.mechse.uiuc.edu/conferences/hydrogen08/

U.S. Department of Energy Hydrogen Delivery Program

Tim Armstrong

August, 2007

Mission Statement

The Hydrogen Program mission is to research, develop, and validate hydrogen production, storage, and fuel cell technologies to reduce dependence on oil in the transportation sector, and to enable clean, reliable energy for stationary and portable power generation.

Hydrogen Fuel Initiative
BUDGET by Participant Organization

Activity	Funding ($ in thousands)			
	FY2005 Approp	FY2006 Approp	FY2007 Approp	FY2008 Request
Hydrogen Fuel Initiative				
EERE Hydrogen (HFCIT)	166,772	153,451	193,551	213,000
Fossil Energy (FE)	16,518	21,036	23,611[1]	12,450
Nuclear Energy (NE)	8,682	24,057	19,265	22,600
Science (SC)	29,183	32,500	36,388	59,500
DOE Hydrogen TOTAL	**221,155**	**231,044**	**272,815**	**307,550**
Department of Transportation	549	1,411	1,420	1,425
Hydrogen Fuel Initiative TOTAL	**221,704**	**232,455**	**274,235**	**308,975**

[1] FY07 Request

Hydrogen Fuel Initiative
Total Budget

- **President Bush committed $1.2 billion over 5 years (FY04 – FY08) to accelerate R&D to enable technology readiness in 2015.**

Hydrogen Fuel Initiative Funding[1] ($ in millions)				
FY2004 Approp.	**FY2005 Approp.**	**FY2006 Approp.**	**FY2007 Approp.**	**FY 2008 Request**
157	222	232	274	309

- **President's cumulative request has been consistent with the commitment: $1.2 B (FY04 – FY08).**

[1] Includes EERE, FE, NE, SC and Department of Transportation

EERE Hydrogen Budget

Activity	Funding ($ in thousands)			
	FY 2005 Approp	FY 2006 Approp	FY 2007 Actual	FY 2008 Request
Hydrogen Production & Delivery	13,303	8,391	34,594	40,000
Hydrogen Storage R&D	22,418	26,040	34,620	43,900
Fuel Cell Stack Component R&D	31,702	30,710	38,082	44,000
Technology Validation	26,098	33,301	39,566	30,000
Transportation Fuel Cell Systems	7,300	1,050	7,518	8,000
Distributed Energy Fuel Cell Sys.	6,753	939	7,419	7,700
Fuel Processor R&D	9,469	637	4,056	3,000
Safety, Codes & Standards	5,801	4,595	13,848	16,000
Education	0	481	1,978	3,900
Systems Analysis	3,157	4,787	9,892	11,500
Manufacturing R&D	0	0	1,978	5,000
Technical/Program Mgt. Support	535	0	0	0
Congressionally Directed Activities	40,236	42,520	0	0
TOTAL	166,772	153,451	193,551	213,000

Hydrogen Delivery

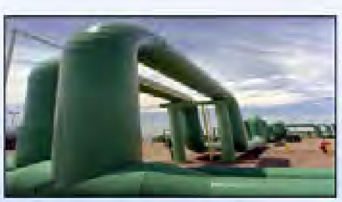
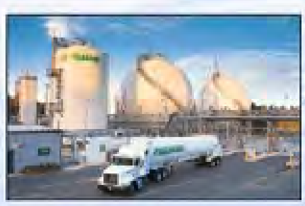
Goal

- **Develop hydrogen delivery technologies that enable the introduction and long-term viability of hydrogen as an energy carrier for transportation and stationary power**

Scope

- **From the end point of central or distributed production (2 MPa H2) to and including the dispenser at a refueling station or stationary power site**
  - **GH2 Pipelines and Trucks, LH2 Trucks, Carriers**

<$1.00/kg of Hydrogen by 2017


Delivery Targets

Category	Units	2005 Status	2015	2017
Pipelines				
Tranmission Capital	$k/mile	$700		$490
Distribution Capital	$k/mile	$320		$190
Reliability (Embrittlement)		Acceptable for current service		Acceptable for H2 as a major energy carrier
Compression				
Large: Reliability		Low		High
Large: Capital Cost	$M (200k kg/day)	$15		$9
Forecourt: Reliability		Low	High	
Forecourt: Capital Cost	$k/(kg/hr)	$4.60	$3	
Forecourt Fill Pressure	psi	5,000	10,000	
Tube Trailer				
Delivery Capacity	kg of H2	280		1,100
Capital cost	$	$165,000		<$300,000
Storage Tanks				
Capital Cost	$/kg of H2	$820	$300	
Liquefaction				
Small: Capital Cost	$M (30,000 kg/d)	$50		$30
Small: Energy Efficiency	%	70%		85%
Large: Capital Cost	$M (300,000 kg/d)	$170		$100
Large: Energy Efficiency	%	80%		87%
Carriers				
Carrier H2 Content	% by weight	6%		13%
Carrier H2 Content	kg H2/liter	0.05		>0.027
Energy Efficiency	%	Undefined		85%
System Cost Contribution	$/kg H2	Undefined		<$1

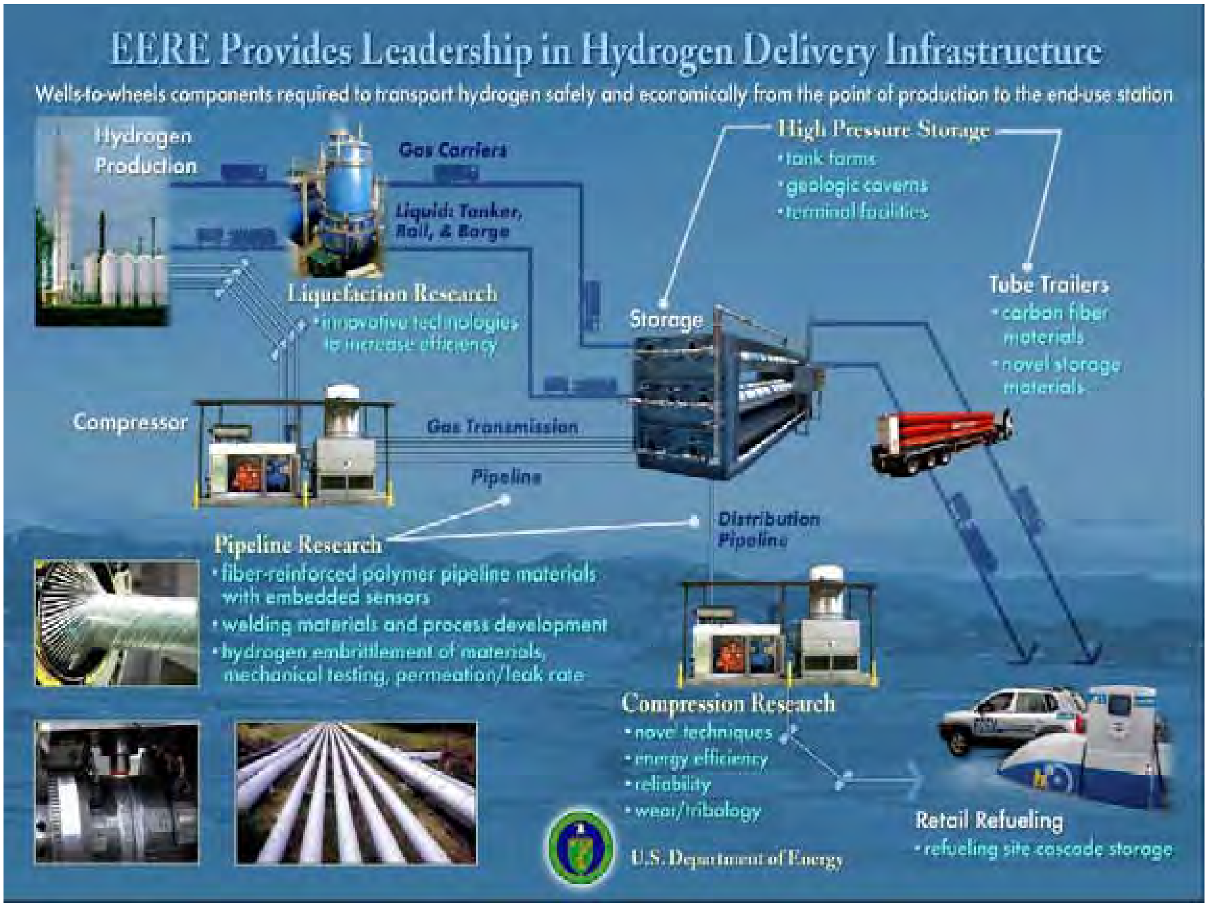
Research Areas

Pathways

- Gaseous Hydrogen Delivery
- Liquid Hydrogen Delivery
- Carriers

Components

Pipelines
Compression
Liquefaction
Carriers & Transformations
Gaseous Storage Tanks
Geologic Storage
GH2 Tube Trailers
Purification
Terminals
Dispensers
Liquid Storage Tanks
Mobile Fuelers
Liquid Trucks, Rail, Ships



Carriers

Liquid Carriers

- ***Ethanol, Methanol, Bio-oils, Ammonia, etc. (Distributed Reforming Production)***
- ***Liquid Hydrocarbons:*** A liquid hydrocarbon is catalytically dehydrogenated at a station or on a vehicle ("dehydrided") and is then returned to a central plant or terminal for "rehydriding":

$$C_nH_2n \leftrightarrow C_nH_n + n/2\ H_2$$

Solid Carriers

- ***Metal Hydrides***
- ***Nanostructures:*** carbon structures, MOF's, etc.
- ***Flowable Powders, Slurries, "Bricks":*** Stable solid carriers might be delivered in many different ways.

H_2 Delivery Current Status

- Technology
  - GH_2 Tube Trailers: ~340 kg, ~2650 psi
  - LH_2 Trucks: ~3900 kg
  - Pipelines: ~ 1000 psi (~630 miles in the U.S.)
  - Refueling Site Operations (compression, storage dispensing): Demonstration projects
- Cost (Does NOT include refueling Site Operations)
  - Trucks: \$4-\$12/kg
  - Pipeline: <\$2/kg

Composite Technology for Hydrogen Pipelines

Fiber-reinforced polymer pipe has excellent burst and collapse pressure ratings, large tensile and compression strengths, and superior chemical and corrosion resistance. Long lengths can be spooled for delivery, and a few workers can install thousands of feet of pipeline per day.

Fiber optic sensors, copper wires and power cables can be embedded a composite pipeline, enabling it to function as a *smart structure*.

Project Overview: Investigate application of composite, fiber-reinforced polymer pipeline technology for hydrogen transmission and distribution.

Technical Targets (2017):
- \$490k/mile capital cost for transmission pipelines
- \$190k/mile capital cost for distribution pipelines
- Hydrogen delivery cost below \$1.00/gge
- High reliability
- Low hydrogen permeation

Technical Approach:
- Evaluate H_2 compatibility of pipeline materials
- Identify advantages and challenges of various manufacturing methods
- Identify polymeric liners with acceptably low hydrogen permeability
- Evaluate options for pipeline joining technologies
- Implement composite pipeline codes & standards
- Determine requirements for structural health monitoring and real-time measurements of H_2 parameters

Managed by UT-Battelle for the Department of Energy

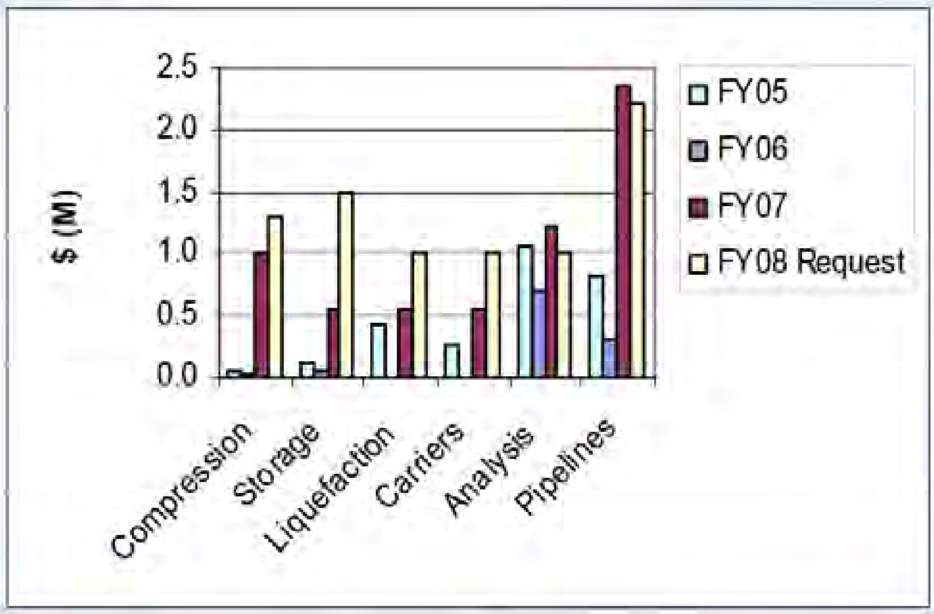
Impact:
- Composite pipeline technology has the potential to reduce installation costs, improve reliability and provide safer operation of hydrogen pipelines

OAK RIDGE

Delivery Challenges

- **Pipelines:** hydrogen embrittlement, capital cost, urban distribution
- **Compression -Transmission and Refueling Stations**: reliability, capital cost, energy efficiency, new technologies
- **Liquefaction:** capital cost, energy efficiency
- **Off-Board Storage Vessels:** capital cost
- **Geologic Storage:** sufficient suitable sites and capacity?
- **Gaseous Tube Trailers**: cost - is 1000 kg capacity possible?
- **H2 Quality:** must meet stringent quality requirements for PEM FC
- **Carriers (Leverages the On-Board Storage Program)**
  - Liquid two-way carriers: low cost and efficient hydrogenation and dehydrogenation, high (~100%) yields and selectivity
  - Solid carriers: high volumetric and gravimetric hydrogen density, energy efficiency and cost

DOE Delivery Budget



FY04: $.4M FY05: $2.5 FY06: $1M FY07: $6.3M FY08 Request: $8.0M

H2A Analysis

- Consistent, comparable, transparent approach to hydrogen production and delivery cost analysis
- Excel spreadsheet tools with common economic parameters, feedstock and utility costs, and approach
- Project Team
  - Production: DTI, TIAX, Parsons, Technology Insights, PNNL, NREL,
  - Delivery: U.C. Davis, ANL, PNNL, NREL, Nexant, Chevron, Air Liquide, GTI
- Other Industrial Collaborators

Eastman Chemical
AEP
Entergy
Framatome
APCi
Exxonmobil
BOC
Ferco
Thermochem
GE
Stuart Energy
Praxair
BP

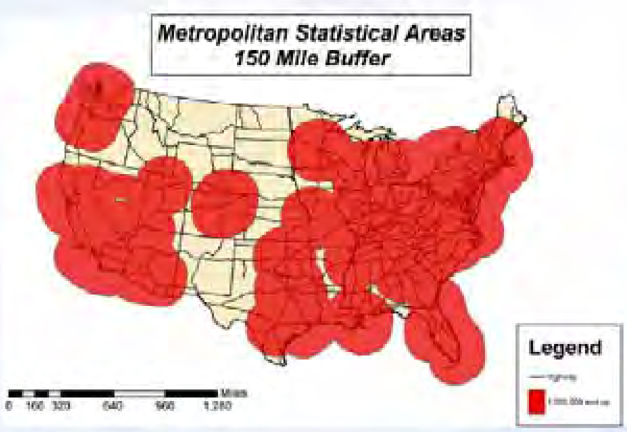
H2A Delivery Goals

- Develop spreadsheet database on delivery system component costs and performance: Component Model
- Develop delivery scenarios for set of well defined major markets and demand levels. Scenario Model
- Estimate the cost of H_2 delivery for base cases with current (2005 costs)

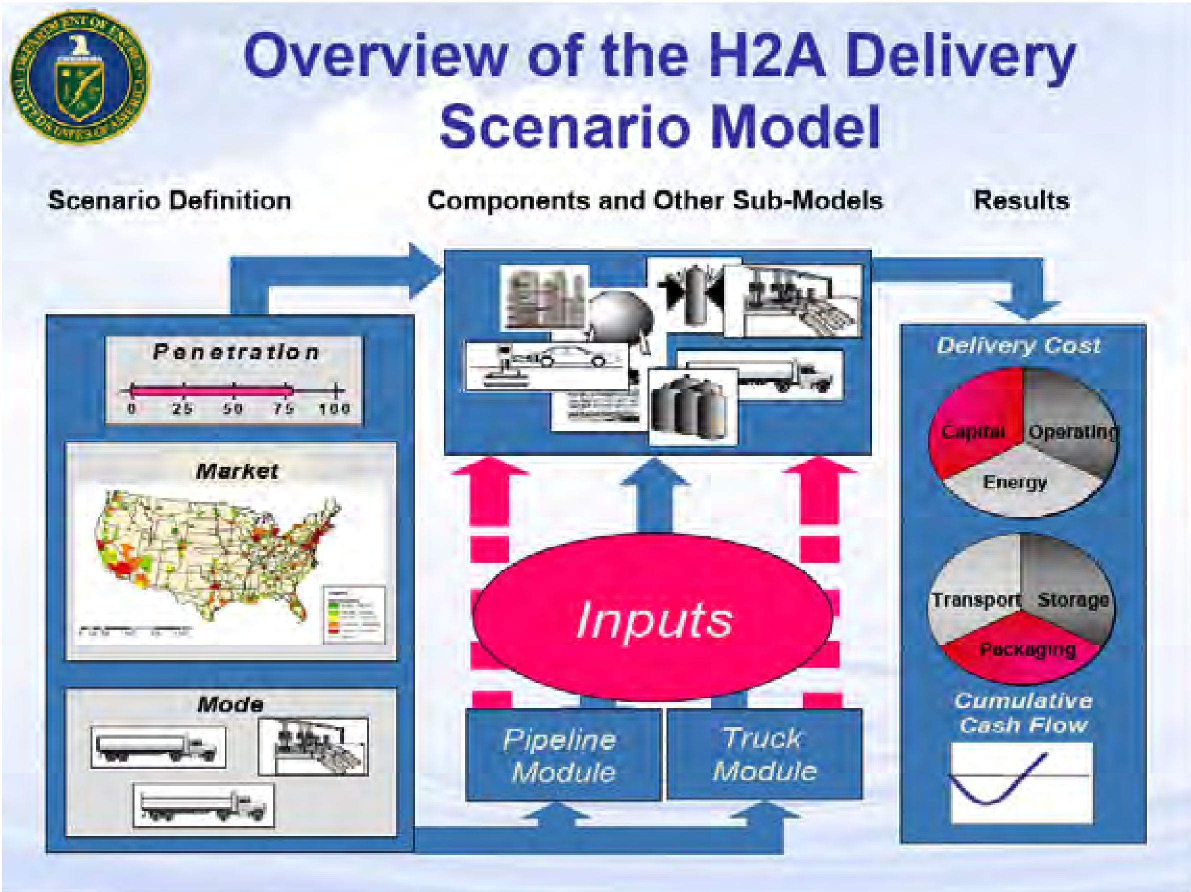
List of Delivery Components

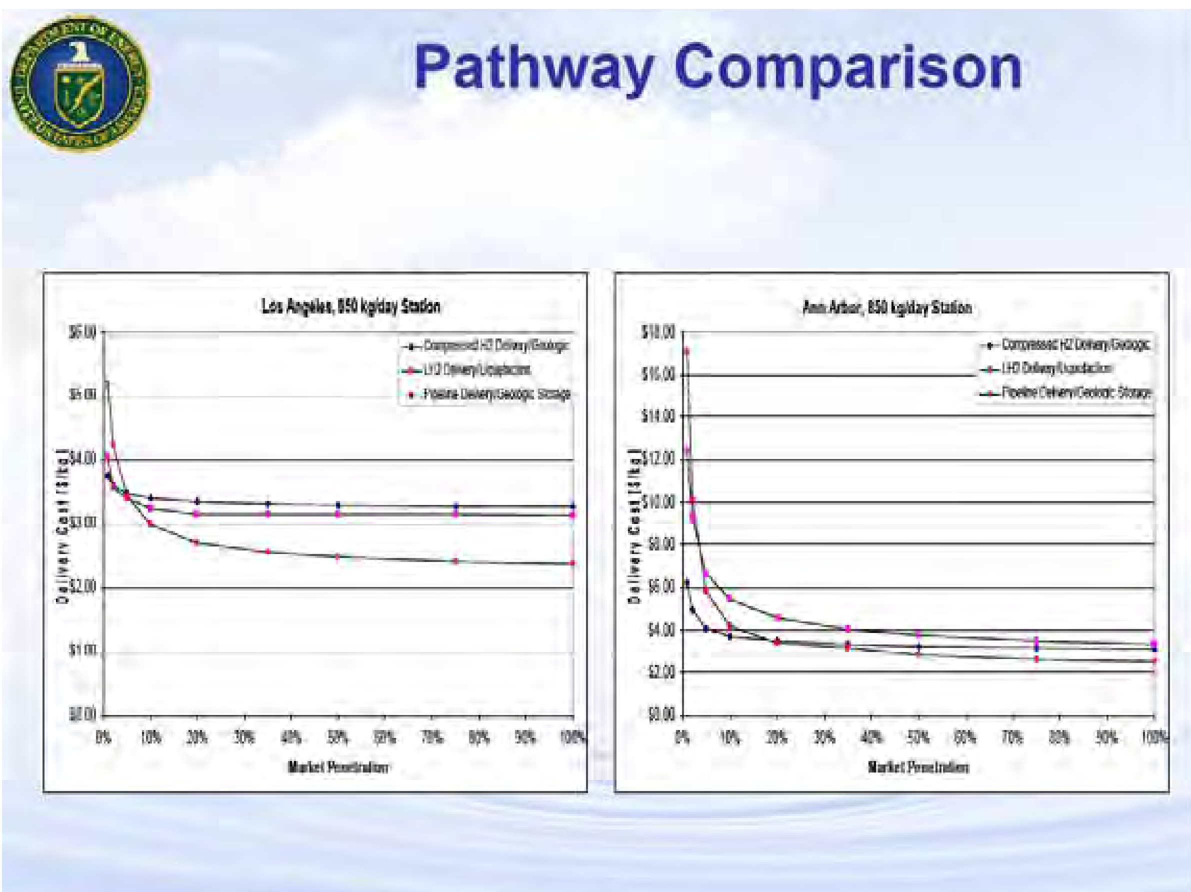
- Compressed Hydrogen Gas Truck (Tube trailer)
- Compressed Hydrogen Gas Truck Terminal
- Liquid Hydrogen Truck
- Liquid Hydrogen Truck Terminal
- H2 Transmission Compressor
- H2 Forecourt Compressor
- Hydrogen pipelines
- H2 Liquefier
- LH2 Storage Tank
- Gaseous H2 Storage "Tank"
- Gaseous H2 Geologic Storage
- Dispenser
- Forecourt: GH2
- Forecourt: LH2

Hydrogen Plants can be Located Near the Market Demand

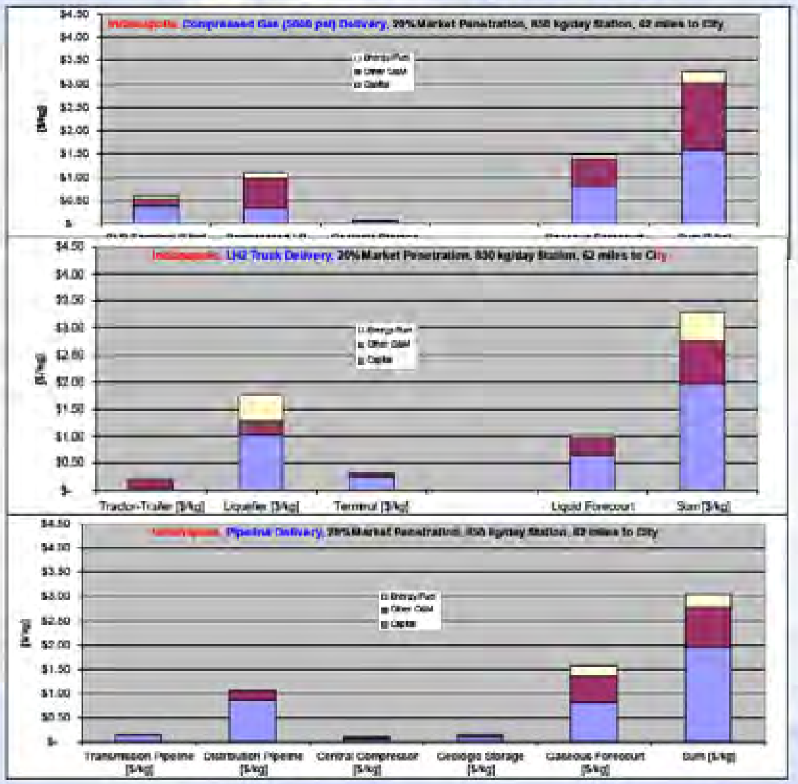


Nearly all areas East of the Mississippi and West of the Rockies are within 200 highway miles (320 km) of large urbanized areas


Overview of the H2A Delivery Scenario Model
Scenario Definition
Components and Other Sub-Models
Results
Penetration
0 25 50 75 100
Market
Mode
Inputs
Pipeline Module
Truck Module
Delivery Cost
Capital
Operating
Energy
Transport
Storage
Packaging
Cumulative Cash Flow


Pathway Comparison
Los Angeles, 850 kg/day Station
Compressed H2 Delivery/Geologic
LH2 Delivery/Liquefaction
Pipeline Delivery/Geologic Storage
Delivery Cost [$/kg]
Market Penetration
Ann Arbor, 850 kg/day Station
Compressed H2 Delivery/Geologic
LH2 Delivery/Liquefaction
Pipeline Delivery/Geologic Storage
Delivery Cost [$/kg]
Market Penetration

Pathway Comparison



Improvements to Scenario Model

- ✓ Mixed delivery pathway (e.g., pipeline to GH2 Terminal with Tube Trailer or LH2 Truck distribution)
- ✓ Variable sized Forecourts
- Energy efficiencies, CO_2 emissions and other emissions of entire pathways
- Carriers
- Mixed demands/markets (combining urban area with interstate demand)

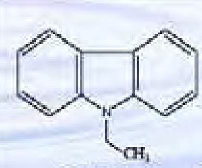
Programmatic Accomplishments

- Roadmap (V2) completed and posted
- All Component Targets revised
- Delivery Section of the DOE HFCIT Multi-Year R&D Plan completely revised
- Established a portfolio of Research Projects
- Established a Pipeline Working Group
  - National Labs (ORNL, SRNL, SNL), industry (CTC, APCi, RDC, SECAT, Chemical Composite Coatings Intl., Columbia Gas of KY, Oregon Steel Mills, Hatch Moss MacDonald, AME Stds., etc.), and universities (U. of Illinois)

Technical Accomplishments

- Compression (MITI)
  - Centrifugal pipeline compression: Feasible unit scoped and designed
  - Unique air foil bearings and seals are the key enabler (very high rotational speed)

- Carriers
  - Liquid hydrocarbon with 6 wt. % H_2 identified (Air Products)
  - Leveraging On-Board Storage R&D



N ethyl carbazole

Lessons Learned/Challenges

- Forecourt costs are significant and need to be reduced
  - Compression reliability needs to be improved
  - Storage: Need a breakthrough in high pressure storage or carrier system
  - Larger and fewer forecourts is very beneficial
- Pipelines are the current low cost pathway for the long term, but:
  - Hydrogen embrittlement concerns and high capital costs. FRP pipelines?
  - H2 distribution lines in cities ? And at what pressure? At what cost? Odorants/Sensors?
  - High H2 content tube trailers could be cost effective for distribution
- Transition
  - Low volumes means higher delivery costs
  - Need a breakthrough: liquefaction, higher H2 content tube trailers, or a carrier approach
- System storage needs drive costs up
  - Need to better understand storage needs and demand cycles

Lessons Learned/Challenges

- 70 MPa Refueling
  - Higher compression and storage costs/greater challenge to meet targets
  - May require cooling at the refueling station
- Other potential needs for cooling at refueling stations
  - Metal hydride on-board storage
  - Cryo-gas on board storage
- High capacity tube trailers/storage vessels
  - Higher pressure? Cryo-gas approach? Composites tanks? Solid carriers ?
- H2 Quality Requirements
  - Will polishing purification be needed at the refueling site?
  - Geologic storage contamination issues?
- Can Carriers change the Delivery Paradigm?

Back-Up slides

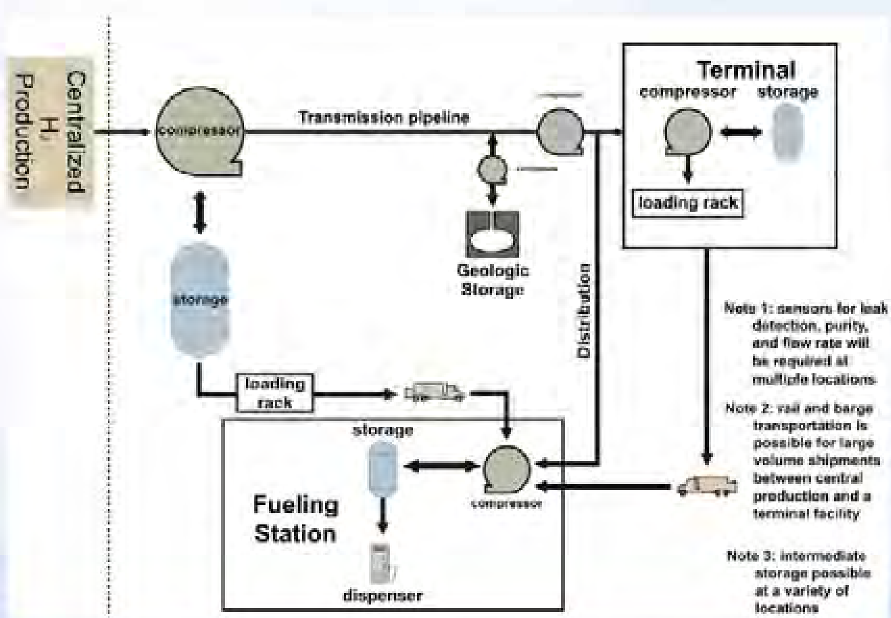
Delivery Projects

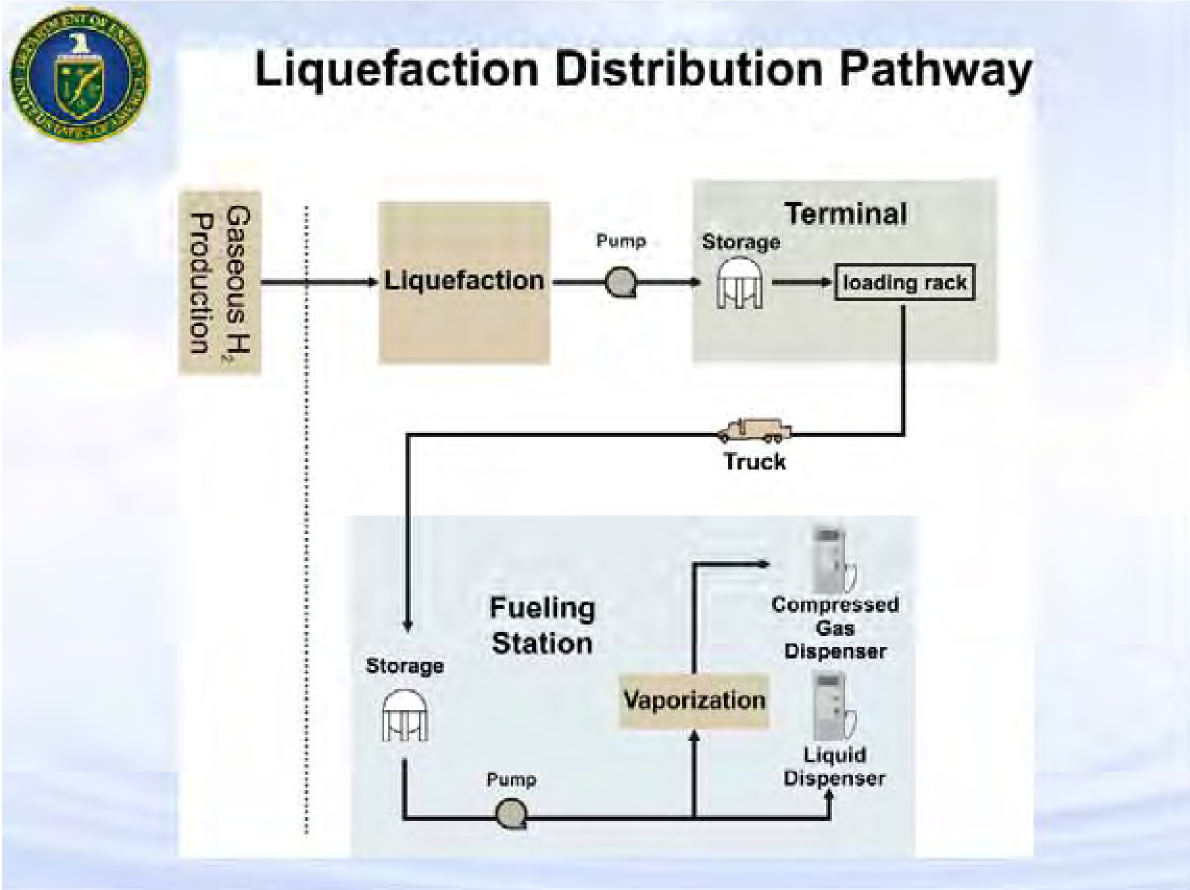
- Delivery Analysis
  - H2A Delivery (U.C. Davis, PNNL ANL, NREL)
  - Nexant collaborative project
- Compression
  - BOC/HERA: DG integrated Hydride Compression
  - MITI: Centrifugal compressor
  - Analytic-Power: Electrochemical compression
- Liquefaction
  - GEECO: Advanced turbo-compression/expansion

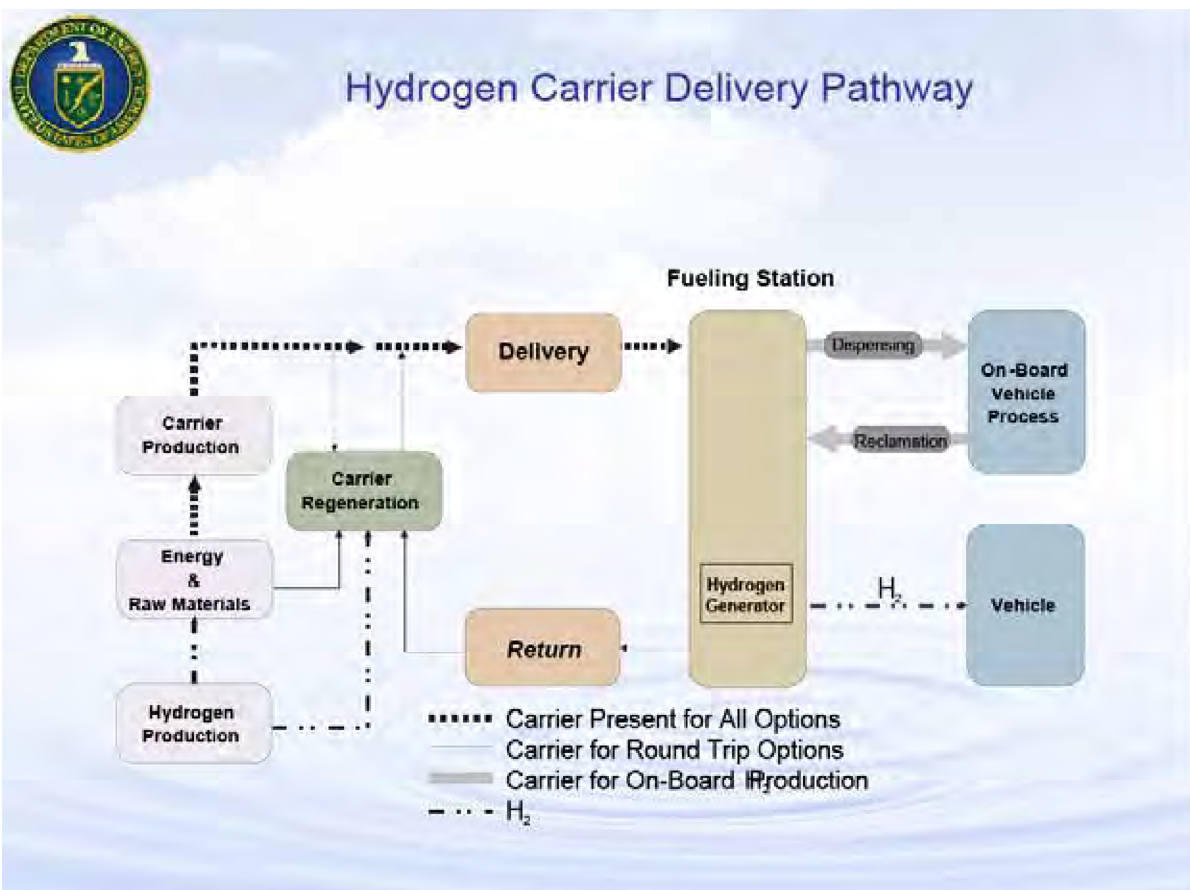
Delivery Projects

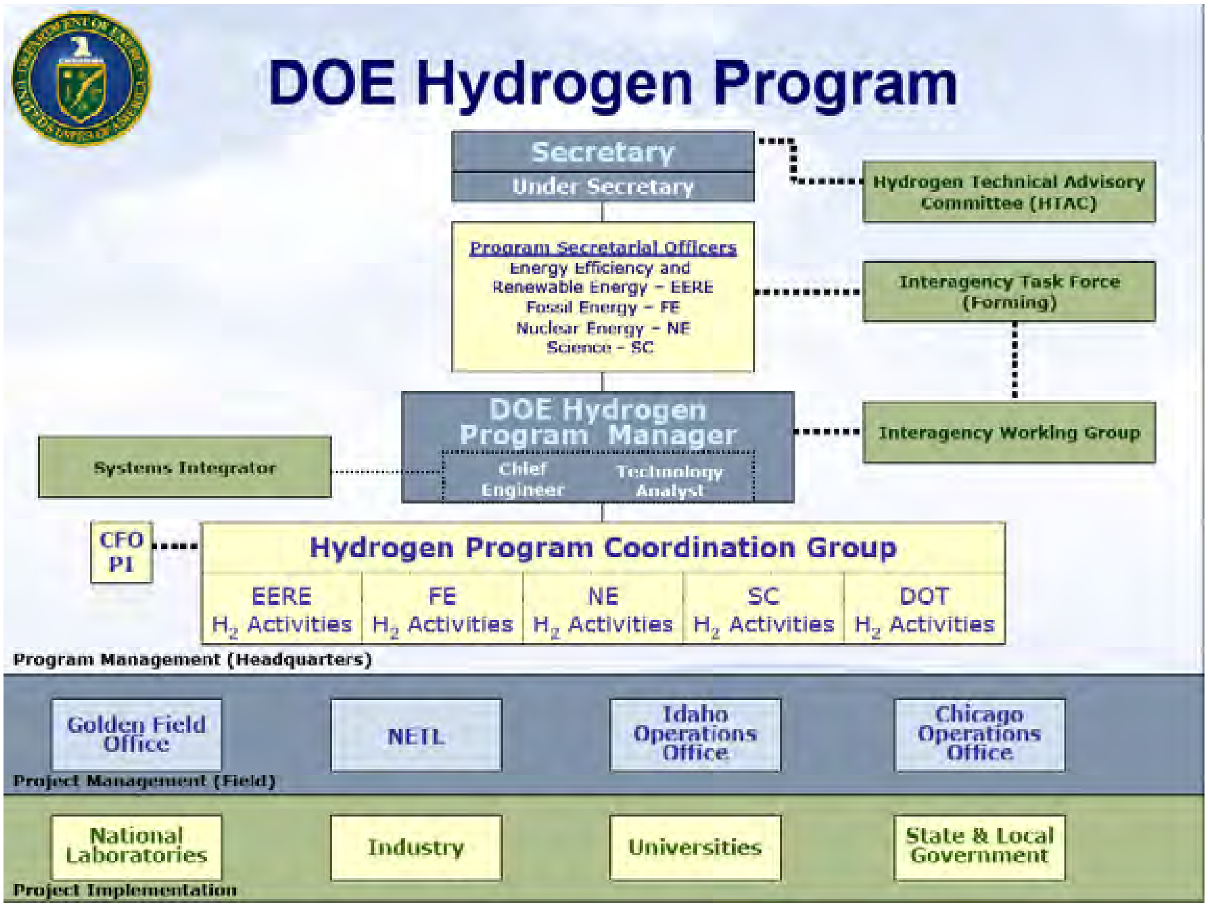
- Off-Board Storage
  - LLNL: Composites for high pressure storage and tube trailers
- Pipelines (Pipeline WG)
  - SECAT collaborative project: New steels and coatings
  - ORNL/SRNL: Composite pipelines
  - ORNL/SRNL: H2 permeability and embrittlement
  - U. of Illinois: Fundamentals of embrittlement
  - CTC: PA Infrastructure Project
  - (EC: Naturalhy Project: H2/NG mixtures in existing NG infrastructure)
- Carriers
  - APCi, UTRC, Penn State U: Liquid hydrocarbon

Gaseous Hydrogen Delivery Pathway



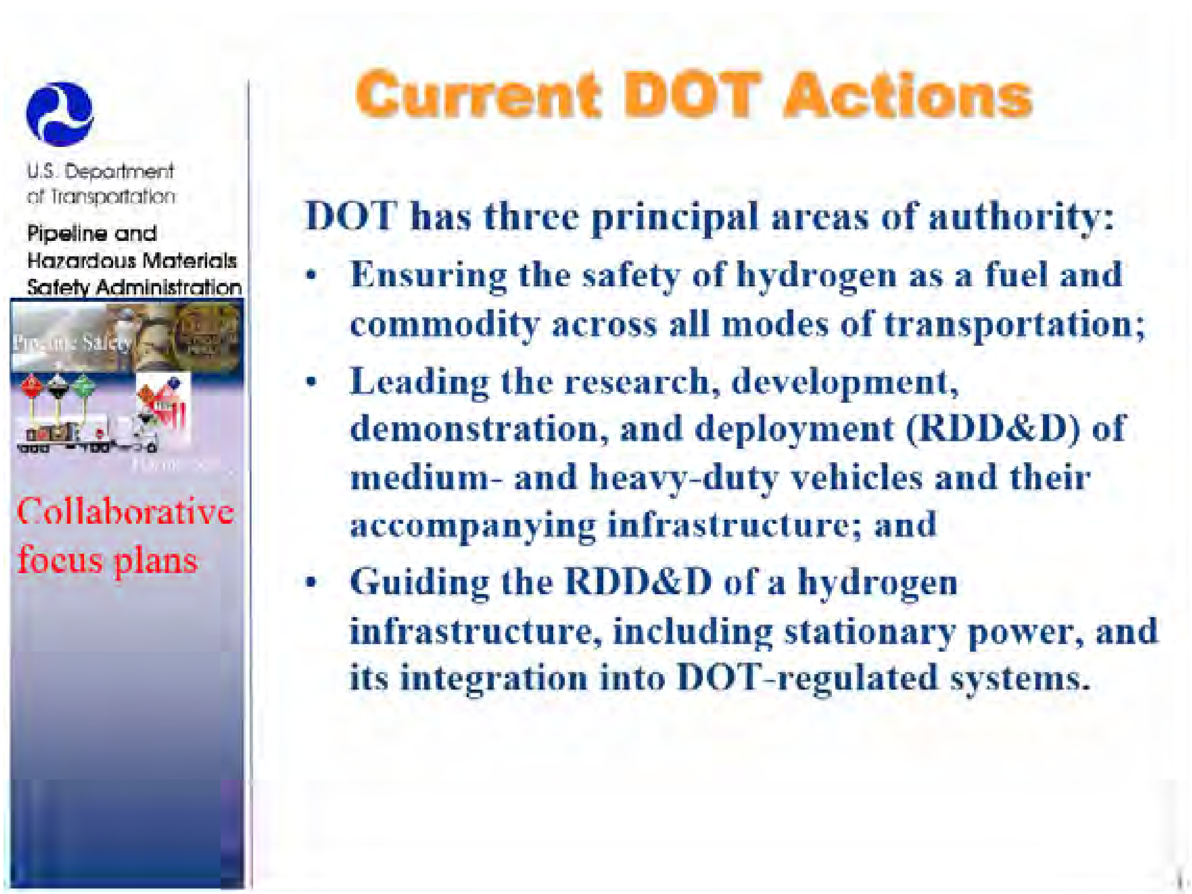

Liquefaction Distribution Pathway
Gaseous H2 Production
Liquefaction
Pump
Terminal
Storage
loading rack
Truck
Fueling Station
Storage
Vaporization
Compressed Gas Dispenser
Liquid Dispenser
Pump


Hydrogen Carrier Delivery Pathway
Fueling Station
Delivery
Dispensing
On-Board Vehicle Process
Reclamation
Carrier Production
Carrier Regeneration
Energy & Raw Materials
Hydrogen Generator
H2
Vehicle
Return
Hydrogen Production
Carrier Present for All Options
Carrier for Round Trip Options
Carrier for On-Board H2 Production
H2


DOE Hydrogen Program
Secretary
Under Secretary
Hydrogen Technical Advisory Committee (HTAC)
Program Secretarial Officers
Energy Efficiency and Renewable Energy – EERE
Fossil Energy – FE
Nuclear Energy – NE
Science – SC
Interagency Task Force (Forming)
DOE Hydrogen Program Manager
Chief Engineer
Technology Analyst
Systems Integrator
Interagency Working Group
CFO PI
Hydrogen Program Coordination Group
EERE H_2 Activities
FE H_2 Activities
NE H_2 Activities
SC H_2 Activities
DOT H_2 Activities
Program Management (Headquarters)
Golden Field Office
NETL
Idaho Operations Office
Chicago Operations Office
Project Management (Field)
National Laboratories
Industry
Universities
State & Local Government
Project Implementation


FreedomCAR and Fuel Partnership
Executive Steering Group
Fuel Operations Group
Energy Directors
DOE Program Managers
Joint Operations Group
FreedomCAR Operations Group
OEM Directors
DOE Program Managers
Fuel Tech Teams
•Hydrogen Production
•Hydrogen Delivery
•Fuel Pathway Integration
Joint Tech Teams
•On-board Hydrogen Storage
•Codes & Standards
Fuel Cell & Vehicle Tech Teams
•Fuel Cells
•Advanced Combustion Emission Control
•Electrochemical Energy Storage
•Vehicle Systems Analysis
•Materials
•Electrical & Electronics
FreedomCAR
Fuel Partnership


U. S. Department of Transportation
Pipeline and Hazardous Materials Safety Administration
DEPARTMENT OF TRANSPORTATION
UNITED STATES OF AMERICA
NIST Workshop on Materials Test
Procedures for Hydrogen Pipelines
August 21-22, 2007
www.phmsa.dot.gov


U.S. Department of Transportation
Pipeline and Hazardous Materials Safety Administration
Collaborative focus plans
Current DOT Actions
DOT has three principal areas of authority:
• Ensuring the safety of hydrogen as a fuel and commodity across all modes of transportation;
• Leading the research, development, demonstration, and deployment (RDD&D) of medium- and heavy-duty vehicles and their accompanying infrastructure; and
• Guiding the RDD&D of a hydrogen infrastructure, including stationary power, and its integration into DOT-regulated systems.

U.S. Department of Transportation

Pipeline and Hazardous Materials Safety Administration

Pipeline Safety Program Strategic Focus

- Improve safety of the Nation's pipelines
  - Reduce number of incidents
  - Reduce likelihood of major incidents
  - Mitigate consequences of incidents
- Provide basis for increased public confidence in:
  - pipeline safety
  - Security and reliability
  - environmental protection

U.S. Department of Transportation

Pipeline and Hazardous Materials Safety Administration

PHMSA Authority

DOT/PHMSA is Ready

- PHMSA is responsible for enforcing regulations to ensure public & environmental safety for over 2 million miles of interstate pipelines
- Includes: Hazardous liquids, natural gas, & other flammable, corrosive and toxic gases, including hydrogen.
- Regulations: Federal Pipeline Safety Laws codified in 49 U.S.C. 60101 *et seq*., implements regulations, 49 C.F.R. Parts 190–199.
- Specifically: 49 C.F.R. Part 192 regulates the transportation of natural gas and other gases in pipelines which are transported as a compressed, flammable gas. Part 192 includes in it's definition of "gas" "flammable gas, or gas which is toxic or corrosive."

2

U.S. Department of Transportation

Pipeline and Hazardous Materials Safety Administration

PHMSA Authority

Thus, current pipeline safety regulations apply to transporting hydrogen gas by pipeline.

Gas Integrity Management Rule (2002) requires inspection of high consequence area (HCA) pipelines

Proposed Distribution Integrity Management Rule will further drive need

U.S. Department of Transportation

Pipeline and Hazardous Materials Safety Administration

U.S. Pipeline Regulations

- 49 CFR, Parts 190 – 195
- http://www.gpoaccess.gov/cfr/

U.S. Department of Transportation

Pipeline and Hazardous Materials Safety Administration

Some Prescriptive Standards Used in Pipeline Regulations

Steel Pipe:	API 5L
Plastic Pipe:	ASTM D2513
Welding:	API 1104
Design and Operations:	ASME B31.4 (oil)
	ASME B31.8 (gas)
Corrosion:	NACE standards
Liquefied Natural Gas:	NFPA 59A

U.S. Department of Transportation

Pipeline and Hazardous Materials Safety Administration

DOT Hydrogen Working Group

DOT administrations have a broad yet common spectrum of needs across hydrogen & fuel cell-related RDD&D.

In 2003, the DOT Hydrogen Working Group formed to issue the DOT Hydrogen Road Map to coordinate hydrogen and fuel cell RD&D activities across DOT and other Federal agencies.

Four primary topics (Roads) are mapped:
Road 1: Safety, Codes, Standards, and Regulations
Road 2: Infrastructure Development and Deployment
Road 3: Safety Education, Outreach and Training
Road 4: Medium- and Heavy-duty Vehicle Development, Demonstration, & Deployment

U.S. Department of Transportation

Pipeline and Hazardous Materials Safety Administration

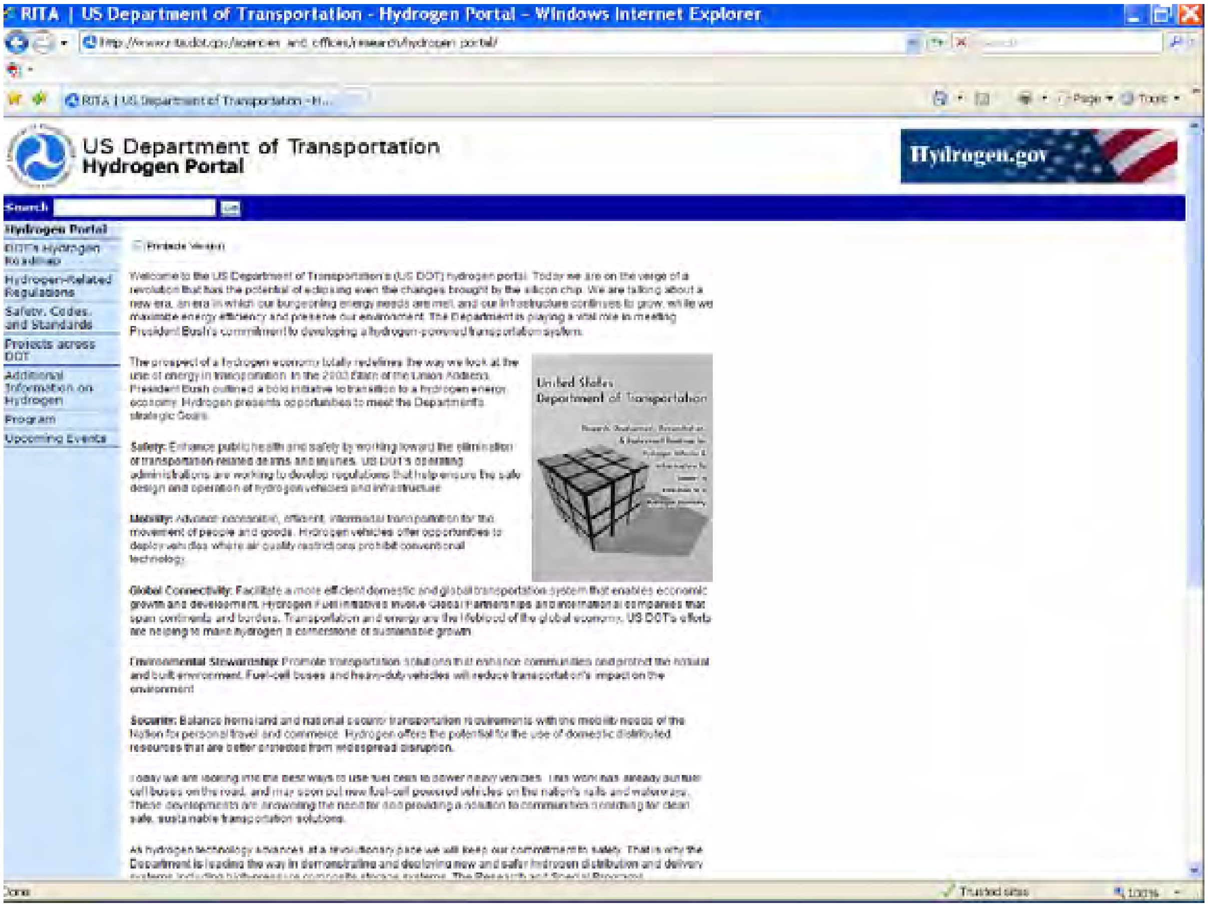
DOT Hydrogen Working Group

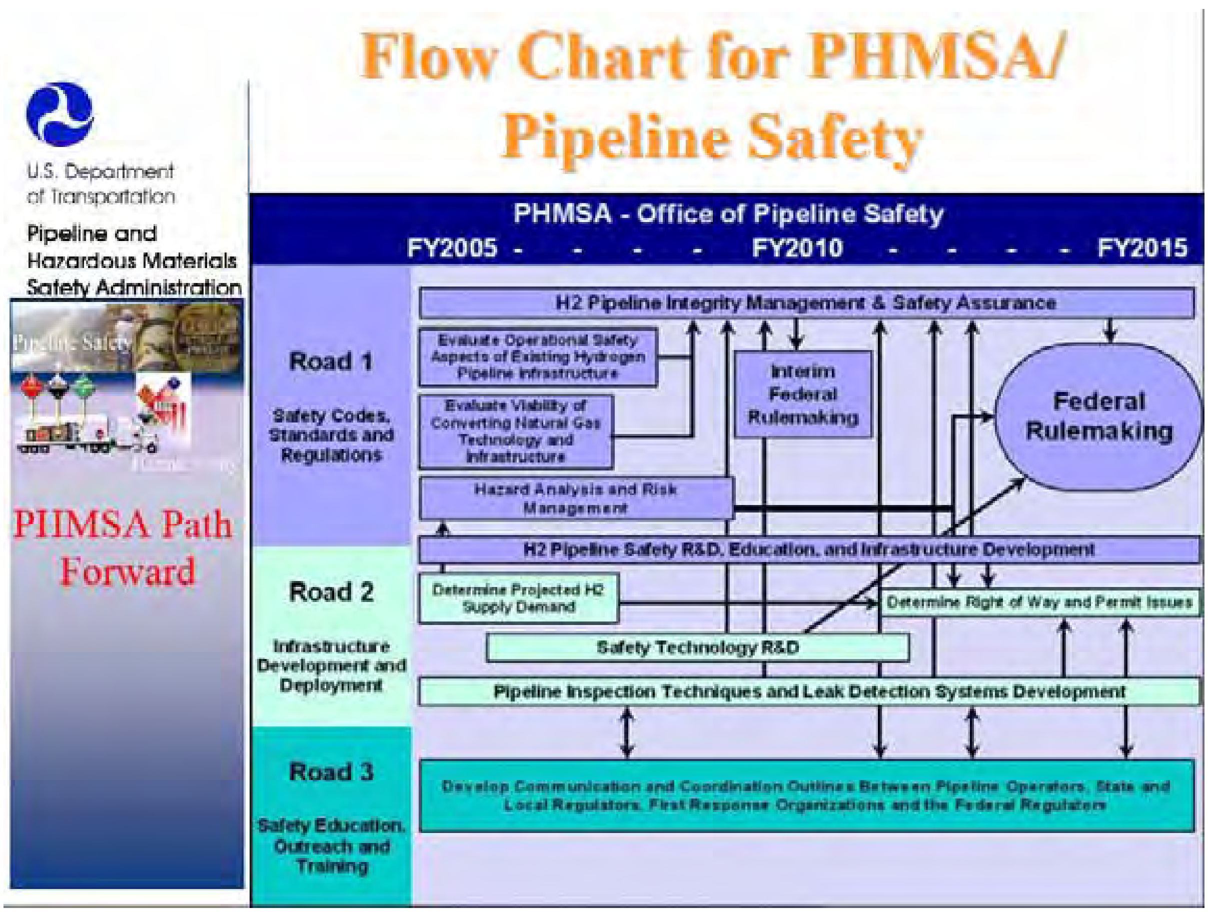
The Map for each Road considers four areas for planning purposes:

- Anticipated long-term outcomes (11 to 20 years)
- Challenges and requirements
- Pathways, projects and products
- Timelines

The Map also illustrates pathways for convergence, program goals and joint activities with DOE, DOD, EPA, DOC, and others

The DOT Hydrogen Road Map & other information Is available at: **http://hydrogen.dot.gov**


RITA | US Department of Transportation - Hydrogen Portal - Windows Internet Explorer
US Department of Transportation
Hydrogen Portal
Hydrogen.gov
Search
Hydrogen Portal
DOT's Hydrogen Roadmap
Hydrogen-Related Regulations
Safety, Codes, and Standards
Projects across DOT
Additional Information on Hydrogen
Program
Upcoming Events
United States Department of Transportation
Safety:
Mobility:
Global Connectivity:
Environmental Stewardship:
Security:


Flow Chart for PHMSA/ Pipeline Safety
U.S. Department of Transportation
Pipeline and Hazardous Materials Safety Administration
PHMSA Path Forward
PHMSA - Office of Pipeline Safety
FY2005 - - - - FY2010 - - - - FY2015
Road 1
Safety Codes, Standards and Regulations
H2 Pipeline Integrity Management & Safety Assurance
Evaluate Operational Safety Aspects of Existing Hydrogen Pipeline Infrastructure
Evaluate Viability of Converting Natural Gas Technology and Infrastructure
Hazard Analysis and Risk Management
Interim Federal Rulemaking
Federal Rulemaking
Road 2
Infrastructure Development and Deployment
H2 Pipeline Safety R&D, Education, and Infrastructure Development
Determine Projected H2 Supply Demand
Determine Right of Way and Permit Issues
Safety Technology R&D
Pipeline Inspection Techniques and Leak Detection Systems Development
Road 3
Safety Education, Outreach and Training
Develop Communication and Coordination Outlines Between Pipeline Operators, State and Local Regulators, First Response Organizations and the Federal Regulators

U.S. Department of Transportation

Pipeline and Hazardous Materials Safety Administration

Current R&D

In-Situ Hydrogen Analysis in Weldments

Main Objective

The Colorado School of Mines and the NIST- Boulder will collaborate in the development of non-destructive technology for weld inspection, assessment, and repair in high strength pipeline steels and their weldments.

The research would be further advanced by the characterization of hydrogen in pipeline steel weldments.

Public Abstract

The assessment of hydrogen content in pipeline steel weldments is an essential requirement to monitor loss of weld integrity with time and to prevent failures. With use of pipeline steels of increasing strength, the threshold of hydrogen concentration for hydrogen cracking is significantly being reduced.

For additional information go to:
http://primis.phmsa.dot.gov/matrix/PrjHome.rdm?prj=224

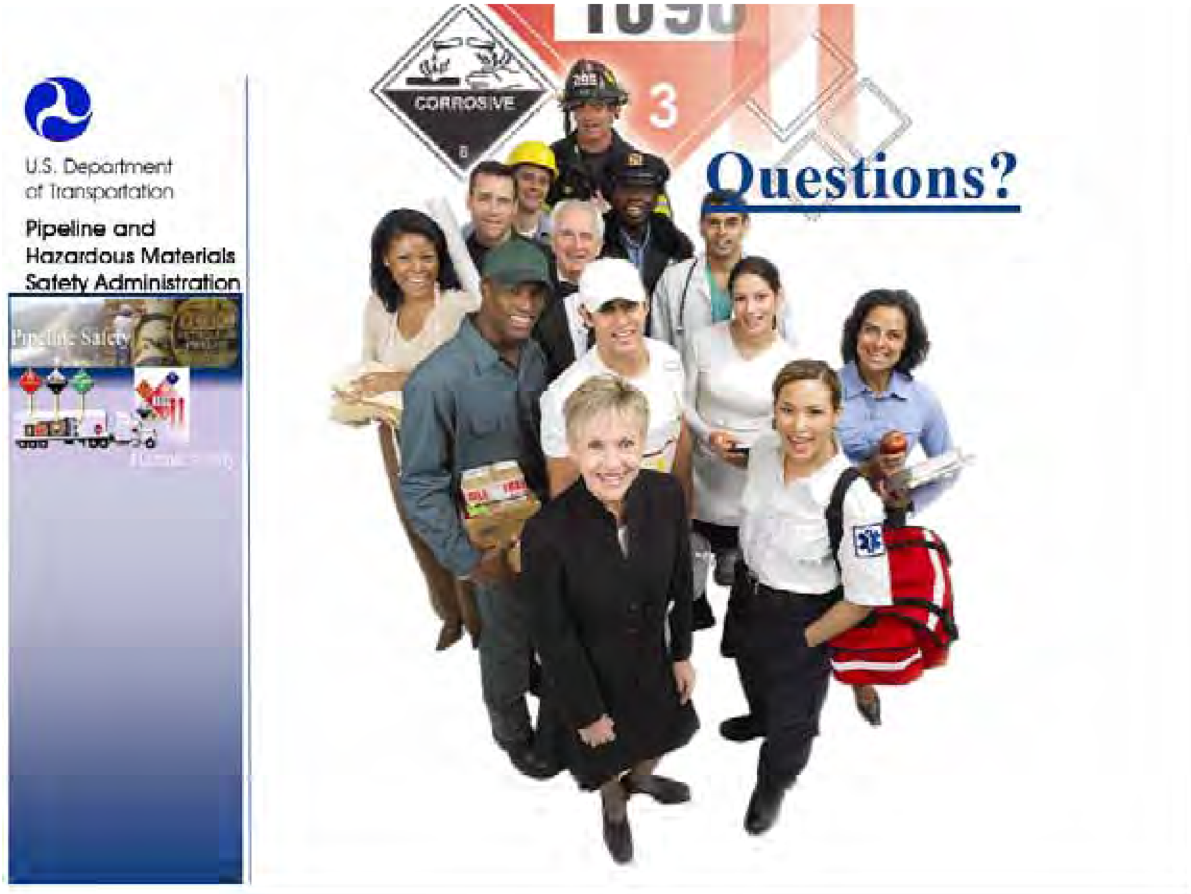
U.S. Department of Transportation

Pipeline and Hazardous Materials Safety Administration

PHMSA RD&T Contacts

James Merritt
P(303) 638-4758
F(303) 346-9192
Email
james.merritt@dot.gov

Robert Smith
P(202) 366-3814
F(202) 366-4566
Email
robert.w.smith@dot.gov


U.S. Department of Transportation
Pipeline and Hazardous Materials Safety Administration
Pipeline Safety
CORROSIVE
3
Questions?

ASME

SETTING THE STANDARD

1

ASME

SETTING THE STANDARD

ASME Hydrogen Infrastructure Codes

NIST Workshop on Materials Test Procedures for Hydrogen Pipelines

August 21 – 22

Boulder, CO

Louis E. Hayden, PE
Chair ASME
Hydrogen Piping and Pipeline Code B31.12
Vice Chair ASME BPTCS

2

Topics

- ASME Overview
- H_2 Standards Development Activities
- Code Rules for Hydrogen Pipelines
- Research Needs

3

ASME Codes and Standards Applicable to H_2 Infrastructure

- Piping and Pipelines:
  - B31.12 – Hydrogen Piping and Pipelines (Draft)
  - B31.1 – Power Piping
  - B31.3 - Process piping
  - B31.4 - Liquid pipelines
  - B31.8 - Gas pipelines
  - B31.8S - Managing gas pipeline integrity
- B16 Standards for Valves, Flanges, and Fittings
- Section VIII, Div.3 Article KD-10

4

Status of Standards Actions

- Two years ago BPTCS authorized the development of consensus standards for hydrogen infrastructure applications including piping, pipelines and Pressure Vessels
  - Draft new B31.12 Code. ***Draft 80+% complete***
  - Anticipated publication date: ***Winter* 2007**
  - Section VIII, Div.3 Article KD-10 is complete.

5

H_2 Piping and Pipelines

- B31.12 to develop a new code for H_2 pipelines
  - Include requirements specific to H_2 service for transportation and distribution applications
  - Balance reference and incorporation of applicable sections of B31.8 and B31.8S along with applicable sections from other recognized standards (ASME, API, CGA, etc.)
  - Have separate parts for general requirements and pipelines
  - Include new requirements for construction, Conversion, operation, maintenance and integrity management

6

ASME B31.12 Structure and Basis

- B31.12 is divided into four Parts
  - Part G : General Reference Material
  - Part A: Industrial Piping
  - Part B: Pipelines and Distribution Piping
  - Part C: Residential and Commercial piping

7

Part B: H_2 Pipeline and Distribution Piping

- Model document for Part B is ASME B31.8 and B31.8S
- Anticipated operating ranges:
  - Pressure: full vacuum to 20Mpa/3,000psig
  - Temperature: - 40°C/- 40°F to 150°C/300°F

8

H_2 Pipeline and Distribution Piping

- The B31.12 code offers two design approaches. Option A which is prescriptive and utilizes material performance factors to take into account the loss of mechanical performance of carbon and low allow steels and Option B which is performance based and uses a fracture mechanics approach derived from the pressure vessel design rules in Section VIII, Div. 3 Article KD-10.

9

Hydrogen Pressure Vessels

- A "Project Team on Hydrogen Vessels" was formed in 2004. The charter of this project team is to develop Code rules for all metal and composite pressure vessels to be used in transport and stationary application for 15,000 psi (103 MPa) hydrogen gas at ambient temperature. A series of rules are under development for these vessels.

10

Hydrogen Pressure Vessels

- The first set of rules were developed and approved for fatigue and fracture analysis of Section VIII, Division 3 vessels in 2006. These rules have been incorporated into new Article KD-10 in Division 3.
- The new rules require determining the fatigue crack growth rate and fracture resistance properties of the materials to be used in the construction of pressure vessels in high pressure hydrogen gas. Test methods have been specified to measure these properties, which are required to be used in establishing the vessel fatigue life.

11

Carbon and low alloy steels in H_2 Service

- **Carbon and low alloy steel materials are affected by dry hydrogen gas service. They show reduction in ductility, fatigue strength, burst strength and could be subject to sustained load cracking. It is noted that there are many carbon and low alloy steel pressure vessels and pipeline systems operating in hydrogen service with no history of failure that can be attributed to any of these factors. Carbon and low alloy steel materials with successful long term use in hydrogen service are generally low strength alloys. Typical materials are SA-106 Gr.B, API 5LX42 and API 5LX52 and SA 372. Most of the vessels are of non-welded construction.**

12

Carbon and low alloy steels in H_2 Service

- In reviewing system design data and discussions with engineers from industrial gas companies, the industry trend is to operate carbon steel hydrogen pipeline systems at low stress levels, sometimes at 30% to 50% SMYS. This trend probably accounts for operation without any major reported failures. Research has shown that increasing stress levels in a gaseous hydrogen environment does decrease the resistance of carbon steel to hydrogen related failures. (Current pressures are ≤ 2000psig)
- Low alloy seamless vessels also have a good service record. This can probably be attributed to the absence of welds and the steel and vessel manufacturing processes. (Current pressures are about 3000psig)
- With the lack of comprehensive material test data for carbon and low alloy steel in a high pressure hydrogen environment, additional design conservatism should be utilized to account for these diminished mechanical properties until such time as comprehensive test data is available and has been reviewed by piping and vessel engineers.

13

Carbon steels in H_2 Service

- Design factors for carbon steels used in pipeline systems expressed as a function of specified minimum yield strength, class location and the square root of pressure are shown in the following Table. This table is for use with prescriptive Design Option A.
- This table provides design factors for pressures from zero to 3000 psi and SMYS from 52ksi to 80 ksi.

14

Prescriptive Pipeline Material Performance Factor

Performance Factors For Pipeline Carbon Steel, Location Class 3

		Pressure, PSI						
SMUTS KSI	SMYS KSI	≤1000	2000	2200	2400	2600	2800	3000
		Square Root. Pressure						
		≤31.62	44.72	46.90	48.99	50.99	52.92	54.77
≤66	≤52	0.5	0.5	0.477	0.455	0.44	0.42	0.39
≤75	≤60	0.437	0.437	0.417	0.398	0.385	0.367	0.341
≤82	≤70	0.388	0.388	0.371	0.353	0.342	0.326	0.303
≤90	≤80	0.347	0.347	0.331	0.316	0.305	0.292	0.271

15

Pipeline Material Performance Factor

- Material performance factor table is for use in designing pipeline systems that will operate or have a design temperature within the embrittlement range of recommended lowest service temperature up to 150ºC (300ºF). If the system temperature is out of this range, use standard design allowables.
- Example: For a carbon steel pipeline material used in a location class 3 system, having a SMYS of 60ksi whose design temperature is 100°C and design pressure is 2200psi, the % of SMYS used for the system design would be would be 41.7 % or 25.02ksi.

16

What We Need to Know About Materials Used for Pressure Boundary Construction

- The context of the pressure boundary construction is for hydrogen delivery systems after production, which is expected to include hydrogen pressures from full vacuum to 1000 bar (15,000 psi) and temperatures from liquid to 150ºC (300ºF).
- The most common materials being used for current and planned construction are carbon steel (for pipelines) and Type 316 stainless steel (for piping). Hence, the high priority testing should be done on carbon steel and Type 316 stainless steel. Testing of other metals such as aluminum alloy 6061-T6 and Type 304 stainless steel should be done as a second priority.

17

Carbon Steels with Yield Strengths up to 360 MPa (52,000) psi

- Testing of C-Mn, C-Mn-Microalloy (HSLA), and C-Mn-Alloy steels used in pipeline and piping systems for the transportation of high purity hydrogen gas needs to proceed.
- Samples representing various levels of chemistry (C, Mn, microalloy, solute alloy, etc.) typical of what is used in the current pipeline and piping systems needs to be evaluated. More importantly these samples should have a range of microstructural differences in ferrite, pearlite, acicular ferrite, bainite, martensite, etc. By choosing various C-Mn and microalloy/solute alloy samples the testing should yield a variety of microstructures with various volume fractions of any one microstructural constituent. Grades that can be considered are:
- A53, A106, A134 and API grades X42-52 from current production as well as pipe material that has been in hydrogen or natural gas service for over 20 years (metallurgy has been evolving over the years and therefore not all API grades are created equal in regard to their ability to perform in hydrogen service).

18

Tests and Data Requirements C-Mn and Microalloy Steels

	Need to Know	Current Knowledge?
Base Metal	Reduction in ultimate strength	Reductions are reported
	Reduction in yield strength	Reductions are reported
	Reduction in ductility	Significant reductions have been measured
	Fracture resistance (K_{IH} values)	Mostly unknown
	Fatigue resistance (da/dn values)	Mostly unknown
	What changes when the material is cold formed	Unknown
	How does a corroded surface affect the performance?	Unknown
	Diffusion coefficients for various microstructures and the amount of hydrogen that gets trapped in the matrix	Unknown?
Weld Metal	Reduction in ultimate strength	Unknown
	Reduction in yield strength	Unknown
	Reduction in ductility	Unknown
	Fracture resistance (K_{IH} values)	Unknown
	Fatigue resistance (da/dn values)	Unknown
	Effect of post weld heat treatment	Unknown
Heat Affected Zone	Reduction in ultimate strength	Unknown
	Reduction in yield strength	Unknown
	Reduction in ductility	Unknown
	Fracture resistance (K_{IH} values)	Unknown
	Fatigue resistance (da/dn values)	Unknown
	Effect of post weld heat treatment	Unknown

19

Tests and Data Requirements

Testing Environment

- The immediate information need is over the range of pressures up to 200 bar (3,000 psi) and temperatures from ambient to 150°C (300°F). As a second priority, information about increasing pressures up to 1000 bar (15,000 psi) is needed to determine the practical upper limit for use of carbon steel. The samples should be tested under different environmental conditions. This includes various pressures and exposure times, unless these can be shown to be unimportant variables.

Information to be Captured Includes

- Complete chemistry characterization.
- Complete microstructural characterization including volume fractions of each microstructural constituent, degrees of banding, cleanliness (inclusion size, frequency and shape), voids, cracks, grain size, dislocation/residual stress analysis, etc.
- The product form from which the specimens were taken.
- The product form production process, including either hot or cold forming.
- Any thermal treatments of the product or Mechanical forming (bending).
- The preparation of the test specimens, including operations such as cutting, grinding, machining, flattening, bending, weld procedure used for sample fabrication, thermal treating, hydrogen charging and the lab environment.

20

Tests and Data Requirements Stainless Steels

- Testing of stainless steels in high purity hydrogen needs to proceed to support the current assumption that stable grades of austenitic stainless steels behave well in hydrogen environments. The assumption is based on a long history of using stainless steel for hydrogen service, but almost all of the experience is for piping and equipment operating a pressures much lower than 1000 bar (15,000 psi).

Samples to be tested

- Most Type 316 stainless steels are dual certified; i.e. they meet the specification requirements for both the traditional and low carbon grades. Samples to be tested should be dual certified or a combination of traditional and dual certified grades.

21

Tests and Data Requirements Stainless Steels

	Need to Know	Current Knowledge?
Base Metal	Reduction in ultimate strength	Modest reductions are reported
	Reduction in yield strength	Modest reductions are reported
	Reduction in ductility	Modest reductions are reported
	Fracture resistance (K_{IH} values)	Unknown
	Fatigue resistance (da/dn values)	Unknown
	What changes when the material is cold formed	Unknown
	Diffusion coefficients for various microstructures and the amount of hydrogen that gets trapped in the matrix	Unknown?
	Effect of alloy shaving	Unknown?
Weld Metal	Reduction in ultimate strength	Unknown
	Reduction in yield strength	Unknown
	Reduction in ductility	Unknown
	Fracture resistance (K_{IH} values)	Unknown
	Fatigue resistance (da/dn values)	Unknown
Heat Affected Zone	Reduction in ultimate strength	Unknown
	Reduction in yield strength	Unknown
	Reduction in ductility	Unknown
	Fracture resistance (K_{IH} values)	Unknown
	Fatigue resistance (da/dn values)	Unknown

22

Tests and Data Requirements Stainless Steels

- The practice of "alloy shaving" we needs to be investigated. The affect of alloy content (austenite formers) must be determined to verify that current chemistry ranges are adequate for hydrogen service at high hydrogen pressures.
- Additionally we need to verify the affects of strain (cold work) on the same alloys. The martensite transformation needs to be evaluated to determine if large strains, over 10%, have a detrimental effect on austenitic stainless steel resistance to hydrogen embrittlement at high hydrogen pressures.
- Welding of stainless must also be investigated and delta ferrite content correlated against weld performance at high hydrogen pressures.

23

Tests and Data Requirements

Testing Environment

- The information is needed over the range of pressures up to 1000 bar (15,000 psi) and temperatures from liquid to 150°C (300°F). The samples should be tested under different environmental conditions. This includes various pressures and exposure times, unless these can be shown to be unimportant variables.

Information to be Captured Includes

- Complete chemistry characterization.
- Complete microstructural characterization including volume fractions of each microstructural constituent, degrees of banding, cleanliness (inclusion size, frequency and shape), voids, cracks, grain size, dislocation/residual stress analysis, and percent ferrite.
- The product form from which the specimens were taken.
- The product form production process, including either hot or cold forming.
- Any thermal treatments of the product.
- The preparation of the test specimens, including operations such as cutting, grinding, machining, flattening, bending, weld procedure used for sample fabrication, thermal treating, hydrogen charging and the lab environment.

24

Fatigue in A Hydrogen Environment

- Hydrogen gas enhances the fatigue crack growth rate of carbon steels. The fatigue crack growth rates in hydrogen become increasingly greater relative to crack growth rates in air or inert gas as ΔK increases. In the higher range of ΔK, fatigue crack growth rates are at least ten-fold greater than crack growth rates in air or inert gas. While the da/dN vs ΔK relationships in air and inert gas are remarkably similar, the da/dN vs ΔK relationships in hydrogen are noticeably more varied.

In the higher range of ΔK, crack growth rates in hydrogen can vary by more than a factor of 10.

The da/dN *vs* ΔK relationships in hydrogen gas can be affected by numerous variables, including gas pressure, load ratio, load cycle frequency, and gas composition.

Ref. Sandia Report:

Technical Reference on Hydrogen Compatibility of Materials
Carbon Steels: C-Mn Alloys (code 1100)
Prepared by: B.P. Somerday, Sandia National Laboratories
Editors:C. San Marchi, B.P. Somerday; Sandia National Laboratories

25

More test data needed

Effect of gas pressure

- Fatigue crack growth rates generally increase as hydrogen gas pressure increases

Effect of load cycle frequency

- Fatigue crack growth rates in hydrogen gas generally increase as the load cycle frequency decreases.

Effect of gas composition

- Additives to hydrogen gas can reduce fatigue crack growth rates, however this phenomenon has not been explored at low load cycle frequencies.

26

Testing

- The preceding has shown what is being done, what information is lacking and what results we need to support our code writing activities.
- What testing activities and how the tests are run and documented needs to be a joint agreement with engineers involved in the codes and testing labs.
- Standardization of testing and documentation must be accomplished. Data from all test labs must be able to be correlated with one-another.

27

ASTM Test Standards

- ASTM E 1820, "Standard test method for measurement of fracture toughness", ASTM, West Conshohocken, PA, 19428
- ASTM E 338,"Standard test method of sharp – notch tension testing of high- strength sheet materials", ASTM, West Conshohocken, PA, 19428
- ASTM E 602 "Standard test method for sharp- notch tension testing with cylindrical specimens", ASTM, West Conshohocken, PA, 19428
- ASTM E 1681, "Standard test method for determining threshold stress intensity factor for environment – assisted cracking of metallic materials", ASTM, West Conshohocken, PA, 19428
- ASTM E 647, "Standard Test Method for measurement of fatigue crack growth rates", ASTM, West Conshohocken, PA, 19428
- API RP- 579, "First Edition 2000 "Recommended Practice for Fitness–for–Service", American Petroleum Institute, Washington D.C., 20005
- ASTM E399, "Standard test method for linear – elastic plane – strain fracture toughness KIC of metallic materials", ASTM, West Conshohocken, PA, 19428

28


Contact Information
Louis Hayden
Louis.hayden1@verizon.net
610-694-0868
Fax: 610-694-8023 USA
29

NIST Workshop on Materials Test Procedures for Hydrogen Pipelines

August 21-22, 2007

NIST

Materials Science and Engineering Laboratory

Materials Reliability Division

Boulder, CO

Why NIST?

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

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

A few observations on hydrogen…

- Market is growing rapidly, so we should support efforts of others (especially DOE) by performing complementary research to fill in gaps and use our existing expertise and equipment.
- Market is moving in multiple directions
- We should make sure that the research base is ready to support whatever delivery and storage technologies are eventually adopted

Observations (con't)…..

- More researchers are becoming active in this area
- We need to maintain a coordinated and efficient roadmap or plan. There are not enough resources to afford duplication of effort or large overlaps.
- Some overlap is necessary to confirm research results between laboratories

Boulder Hydrogen Test Facility

Construction Plans finalized May 2007

Construction starts Oct 2007

Estimated completion Feb 2008

Operational status June 2008

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
    - 5000 psi max pressure (vacuum???)
    - Temperature range 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)

Expectations from this 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:**
  - **Test Methods**
  - **Materials**
  - **Codes and Standards**
- **Preliminary results at end of today open for discussion for entire group**
- **Final breakout sessions tomorrow AM with summary of results at 10:45**

Expectations from this workshop….

Develop a 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.

Expectations from this workshop….

Materials:

Research materials depository (documentation, storage, handling and distribution), Old pipeline steels in use as well as new pipeline materials proposed for use, pipeline welds, composites (FRP) and poly pipelines and pipes, others???

Expectations from this workshop….

Test Methods:

Standardize test procedures through interlaboratory RR testing, specimen design (geometry), in-chamber instrumentation (extensometers, LVDT's, etc.), gas purity measurement (before, during and/or after testing?), chamber purging procedure and assurance of "pure (desired)" environment, effects of inhibitors and/or odorants (good/bad, problems with removal at end of line), pressure and temperature.

Expectations from this workshop….

Codes and Standards:

Identify critical test parameters for suitable test methods, define fields that contribute to a database (Sandia?) for hydrogen pipeline designers and operators, Definition of a comprehensive test plan for NIST, utilizing standardized test methods and following a prioritized list of materials.

General thoughts for all sessions…

- **Collaboration with other labs in developing safe operational procedures in the handling of hydrogen and hydrogen testing systems.**
- **Coordination of R&D with other labs to minimize duplication of efforts and maximize research efforts per dollar.**
- **What are the major hurdles we face in this R&D effort? What can NIST do to overcome these hurdles?**


Breakout Sessions: Rooms 1103, 1105 and 1107
Codes and Standards:
Session Chair: Lou Hayden
Test Methods:
Session Chair: Thad Adams or Andrew Duncan
Materials:
Session Chair: Brian Somerday

3.1 Breakout Session: Materials

Session Chair: Brian Somerday (SNL/CA)
August 22, 2007

Attendees:

Tim Armstrong (ORNL)
Dorian Balch (SNL/CA)
Elizabeth Drexler (NIST)
Chris McCowan (NIST)
Jim Merritt (DOT)
Govindarajan Muralidharan (ORNL)
Dave Olson (Colorado School of Mines)
Martin Prager (Materials Properties Council)
Richard Ricker (NIST)
Joe Slusser (Air Products)
Petros Sofronis (Univ. Illinois)
Samuel Vasquez (El Paso Corp)
Kevin Widenmaier (TransCanada)

The objective of the materials breakout session was to identify a set of goals related to testing of structural materials for hydrogen transportation. A list of priorities was then created for each goal. The end result was an outline consisting of three overall goals and a detailed list of priorities under each goal.

Goal 1: Test relevant materials in hydrogen transportation infrastructure

Linepipe steels
- Current best practice, industry standard steels with low strength (less than X70)
- Current best practice, industry standard steels with high strength (greater than X70)
- Steels currently in the ground

Materials used in components associated with pipeline (valves, compressors, fittings)
Linepipe composites
Storage vessel materials
Pressure manifold component materials (e.g., stainless steels)

Goal 2: Consider important variations in materials

Welds (fusion zone, heat-affected zone)
- Field (girth) welds
  - Current industry practice (single pass, multiple pass)
  - Repair procedures for welds
  - Future practices (e.g., friction stir welds, hybrid laser gas metal arc welds)
- Manufacturing (seam) welds
  - Current industry practice (single pass, multiple pass)
  - Repair procedures for welds
  - Future practices (e.g., friction stir welds, hybrid laser gas metal arc welds)

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 stress

Goal 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

3.2 Breakout Session: Test Techniques and Methods

Session Chair: Andrew Duncan, Savannah River National Lab

Attendees:

Dorian Balch (Sandia-Livermore)
Robert Burgess (NREL)
Ian Cannon (Pratt & Whitney Rocketdyne)
Jenny Collins (Colorado School of Mines)
Phillippe Darcis (NIST)
Andrew Duncan (SRNL)
Zhili Feng (ORNL)
Walter Gerstle (Univ. New Mexico)
Kevin Klug (CTC)
Zvi Livne (NIST)
David McColskey (NIST)
Aryeh Meisels (Pratt & Whitney Rocketdyne)
Kevin Nibur (Sandia-Livermore)
Steve Pawel (ORNL)
David Pitchure (NIST)
Avi Shtechman (NIST)
Paul Tibbals (Pacific Gas & Electric)

Summary:

Initially, the panel began by discussing the six questions that were introduced as primary questions. The discussion was couched within the scope of the presentations from the previous morning. Specifically, the data needs for consensus codes and standards which were: tensile properties, fracture toughness, Kth, fatigue crack growth rate in base metal and welds for piping alloys with specified minimum yield below 70 ksi. In addition, properties for alloys intended for use in consumer distribution/refueling systems would also be desirable (SA 372, 316L). An understanding of the role of microstructure, purity and test environment on properties was emphasized to the panel.

Based on the panel discussions, three critical needs in the area of test methods were identified for the community to address in order to further the potential for a hydrogen based infrastructure. They are listed in order of importance.

3 Critical needs/Areas of required development/Deficiencies

1. **Testing capabilities for the new test facility at NIST**

   Scale
   - Materials from pipe sizes: 4” to 48” (up to 1” wall thickness)
   - Base Metal & Welds
   - Archive and New

   Environment
   - High purity hydrogen *(up to 6-9’s)*
   - Evacuation and purge capability
   - Air or inert gas, as well

   Loading Rate

Down to slow strain rate (i.e., 10^{-7}/s)

Pressure

Overlapping testing capabilities between labs would be good,
Don't want to limit to just pipeline materials (i.e., major data need is 316 stainless steel)
up to 20ksi in small (1 liter) chamber
NIST large chamber (15" dia. X 30") unique in the country

Temperature

-40F to 300F
What happens if there is thermal cycling?
-80 C for oil and gas in artic- lots of issues just with carbon steel,
-(Consensus: −40C would be the highest minimum temperature)
-40F to 300F in small chamber
-only room temperature to 300 F in large chamber
-threshold tests without feed-throughs are easier for temperature control

Major data needs

Tensile-
Threshold – results usually independent of method, eliminates many problems such as strain rate, easier, but how do you set initial load? – use multiple specimens, need additional separate test chamber?
Fracture-
LEFM, Elastic-plastic FM?
Fatigue-

Number of Repeats How many specimens? Depends on how reliable you want to be (undecided)

Test capabilities should support the validation and further development of consensus codes and standards (e.g., ASME B31.12)

2. Inter-laboratory cooperation/ test program

Cross-compare test results/ test methods
-purging, sample machining, hydrogen purity
Understand test methods/results
Compare laboratory abilities
For example: choose same sample, and compare test methods
See what test procedures need to be identical, what can be varied from lab to lab w/o changing results
Is there really a benefit if manufacturers will be self-certifying materials?
Testing hydrogen concentration in material is possible
Testing hydrogen gas purity

3. Component Testing

Composites-piping, FRP
Running cracks might not be an issue
Develop validation approach for tests
Standardized tests for component testing?
Generate properties for welds, joints

Synopsis of Breakout Session for Test Techniques and Methods:

Meeting Notes from August 21:

Initial questions that were posed to the panel for discussion were:

Which attributes have adequate techniques?
Which attributes do not have adequate techniques?
In-Chamber instrumentation for properties and load measurements?
Effects of impurities in H2? How to measure gas concentration levels? To what accuracy?
Purging (purity) techniques?
Testing environment?
Sample Geometry?

Comments were gathered from around the room on why it is difficult to compare results from different studies on the same materials.

Measuring hydrogen-the panel felt that a major reason for this is that inconsistent experimental techniques result in different hydrogen contents in the sample during testing. For example, some studies charge their samples in high pressure gaseous hydrogen for various lengths of time, while other charge their samples electrochemically. A need that was put forth was for the ability to sample the hydrogen concentration in the alloy. This is sometimes done by plating the sample with a metal resistant to H diffusion (e.g., Cu, Sn), charging the sample and then sending it out for chemical analysis. Other studies assume an equilibrium concentration in the metal for charging conditions. **In any case,** a method to quantify H in metal BEFORE testing would be highly desirable. One such method was suggested by a panel member:

1. Angelique Lasseigne at NIST is working on non-destructive, contact and non-contact (ideal) methods. She's saturating samples and measuring how much is in samples by non-contact induced current impedance measurements. The technique could be adapted to perform H content measurements on samples, *in-situ*.
   - Ref. her PhD thesis on thermo-electric power to determine hydrogen content (Colorado school of mines) and a paper given at QNDE 2006.
   - Limitations and topics still to be worked out include: need to demonstrate the in-situ measurement, effect of pressure on equipment, need to make standards first to calibrate.
   - Benefits: simple and cheap ($5,000 for entire unit)

Hydrogen Transport Phenomena: It was mentioned that the equilibrium hydrogen in the lattice amounts for only a small amount of hydrogen in the sample. A need to understand the role of traps/sinks, interfaces and strain on hydrogen content is important.

1. Trapping, diffusivity, and permeability measurements?

2. Concentration throughout lattice can vary at traps and crack tips, saturation? (Thus "Equilibrium" is nebulous)
3. Time constants (scale) sec, min, hours
4. Surface effects can also play a large role.
   - What happens to H in a "used" pipeline? Welds? Moisture? Corrosion? Etc? (Paul Tibble)
5. Concentration gradients my also be important.
   - It would be interesting to try to simulate H gradient in wall thickness of a regular pipeline (Zvi Livne)

Baseline Test Parameters: Their does not appear to be a standard procedure for previous H testing (exc. slow strain rate test as per ASTM G129). Baseline ASTM Standards for mechanical testing can be adapted for hydrogen testing (ref. Lou Hayden presentation from morning session), however certain parameters should be tightly controlled. How can we provide this?

- **Q:** What are the important parameters to keep constant?
  A: Environment of baseline tests (helium, N_2, air?), charge time? (sec, min, hours), Environmental Purity? (4-9's, 5-9's, 6-9's), surface cleanliness, surface roughness, sample geometry, strain rate (10^{-4}-10^{-7}/sec)
- **Q:** What parameters are important to determine the impact on varying on properties.
  A: Pressure Range (0-3000 psi), Temperature (-40 to 300F, but emphasis on ambient temperature), surface cleanliness, contaminants, surface roughness, sample geometry.
- **Q:** What do the codes and standards people need?
  A: The C&S need certain properties for validation of the design approach in Option A for given materials in hydrogen and benign environments (minimum specified YS, UTS & ductility to failure). For validation of Option B (i.e., KD-10) approach they need fatigue crack growth rates, K_{th} values and fracture properties for given materials in hydrogen and benign environments.

Test Methods: certain methods for hydrogen testing need to be standardized. Concerns over the effect of hydrogen environment/ temperature on testing techniques were raised

- Cathodic charging and Gaseous charging
  1. Cathodic charging is an established technique though frequently it is not applied correctly to control hydrogen content (R. Ricker)
  2. Gaseous charging is sensitive to gas purity and contaminants on surface of sample.
- Pre-test characterization (hydrogen measurement)
- Instrumentation measurements during test

  hydrogen may cause drift in sensor readings

  temperature may cause the same
  1. strain gage (open foil vs. closed foil)
  2. clip gage extensometer (MTS makes clip gage for hydrogen environments Model # 632.03)

3. piezo-electric load cell internal vs. external
4. bolt-on LVDT for crack opening displacement
5. heating can be performed by induction coil (thereby minimizing the thermal load to which the instruments/vessel are subjected: A. Meisels).

- Post-test analysis and interpretation

Consensus: technology has been developed that would allow accurate measurement prior to, during and after testing. The laboratories/ experimentalists must consistently implement this technology to generate data that can be effectively compared/contrasted between facilities. It would be a good idea to create a matrix of capabilities of each laboratory. A correlation between established charging techniques (gas pressure vs. cathodic charging) for classes of materials would be important. It was agreed that collaboration between laboratories is important.

Exchange samples between laboratories (compare data with at least some amount of overlap)
See if data trends can be duplicated
Are all load frames or type of load frames the same?
Actual component testing (Valuable Capability for NIST)
- pressure/depressurization of new and an old "real world" pipeline
- allow for validation and usefulness of laboratory testing
- find trouble spots that may not have otherwise been observed

Tensile Tests- ASTM E 8 and E 338

Strain rate an important parameter to control (10e-3/sec is conventional test)
10e-4 to 10e-6/sec would be more applicable but slower is better (within reason).

Fatigue Testing- ASTM E 647

High frequency testing is NOT representative since low frequency (0.5-1Hz) fatigue promotes hydrogen embrittlement. Need to identify maximum frequency allowable.
Are there accelerated tests that can be done?
Consensus: Before we can address frequency, we first need a better understanding of H diffusion rates (which includes trapping, diffusion along grain boundaries, diffusion through the grains), permeability, etc. (hopefully Sofronis' work will provide insight). Until then, slower is better (within reason) or the frequency that shows worse case.
Some discussion about needing S-N curves rather than just crack growth rate occurred but no clear consensus was achieved.

Fracture Testing - ASTM E 1820 and E 399

Standards appear to be comprehensive enough
How to test fracture toughness on weldments?

C-shape specimen (ASTM E399), cut specimen out of pipe and grow a crack, can take sections out of a girth weld

WOL Testing for K_{th} – ASTM E 1681

Standards appear to be comprehensive enough

Short Rod test ASTM E 1304 may be useful for this data

- used in short, transverse tests, there is a standard for it.

C-shape specimen (ASTM E399) may be useful, as well.

Environmental Parameters during testing

Hydrogen Charging: Electrochemical vs. Gas

Ricker believes that a gas system is necessary to accurately emulate service conditions especially the effects of gas purity and surface reactions. However, since properly controlled electrochemical charging can be used to reproducibly charge samples with homogenous distributions of hydrogen, he believes that laboratories studying microstructural effects need not use gas phase charging. He will be working with NIST-Boulder to compare. Once hydrogen is in sample, it doesn't matter where is came from

-A. Lasseigne: cathodic charging results in saturation sometime as much as 3X lower than with gas charging

-R. Ricker: surface contamination issues may play a role in cathodic charging, as in gas phase charging

Is it only the hydrogen that is in the material is contributing to embrittlement? Stress-intensity issues?

A good idea to make a list of scenarios of pipeline failures due to hydrogen and simulate those environments in test environment.

Hydrogen concentration charging from one side of the sample would be interesting to compare to hydrostatic charging

We should re-visit some of these old H diffusion testing (work on permeation is ongoing at ORNL and SRNL)

What is an adequate number of purging cycles to maintain bottle purity in the vessel? (2, 5, 10?)

- Sandia-Livermore recommends 3 He followed by 3 H_2 purge cycles w/full evacuation in between using 6-9's purity hydrogen.
- SRNL currently does 2 Ar and 2 H_2 using 5-9's purity hydrogen and felt that 6-9's was overkill for carbon steels.
- Others are somewhere in between.

The point was made by several that since we do not have a mature understanding of the role of impurities on charging kinetics that we should all strive for highest purity gas possible (at least initially).

Sampling before and after the test would demonstrate the equivalence or differences of each charge method.

Closing thought: Component testing may be an important need in order to characterize the performance behavior of FRP (composite piping) and none metallic components.

What kind of testing on fiber reinforced polymer is out there?

What kind of permeation barriers (metallic liner, polymer liner, etc?)

Meeting Notes for Aug 22nd

Discussion on testing capabilities for the new test facility at NIST

Maybe a good idea to develop a needs matrix based on what customer/stakeholder needs are:

Where do we stop in terms of focus on testing –transmission lines, piping all the way to household? (McColskey-NIST)

Testing needs:

All agree that composite testing leads more to full component test

What sizes are we talking about?- 4" – 48, 52" diameter w/ 1" wall

Problems associated with **higher strength steels**

Thicker wall and lower strength are often chosen by designers to guard against secondary damage rather than use high strength steels with smaller wall thicknesses
If the ASME standard degrades higher strength steels with the prescriptive design approach, why use higher strength steels

need to get away from prescriptive method with fracture mechanics tests

Pipeline industry is also tending toward strain based design

Focus **should first be on the lower strength steels**

Develop test methods/procedures to obtain necessary data for standard design methods

Test charged samples and establish test method

Do we test in Hydrogen or Air? (*Consensus was Hydrogen*)

Fracture-failure assessment diagram (FAD) often uses SENT test data since it is more conservative

Fatigue- S-N curve, or fatigue crack growth curve? (SN takes longer, but FCG has more scatter)

Codes and standards need to tell us what they need, and we can tell them how we can get it (Need to find out KD-10, option B31-12, code approach test needs, i.e., K_{th}, to develop test program)

Lower constraint of test- get results similar to actual pipe, but you get much more scatter.

Also need to consider what type of cracking is occurring to know what should be tested for (axial crack growth, through thickness crack growth??)

NIST plans 2 chambers for facility

1-3 liter capacity

Larger one with 15” diameter OD x 30” tall to be able to run full wall thickness tests

Larger chamber is beneficial to be able to meet a lot of standard specimen geometries, and contribute to other labs that don’t have the larger chamber capability, even if the larger chamber is at lower pressures

Can we compare tests from natural gas pipelines to hydrogen pipelines and then see what additional tests need to be performed?

If you test in hydrogen you know crack will grow faster than in air- what does that say about constraint?

May be more highly constrained because of hydrogen going to crack tip

To reduce the amount of volume inside the chamber, fill chamber with filler blocks, is this on the right track? (*Consensus was yes*)

The smaller the volume of hydrogen the fewer the problems

It is often more applicable to do threshold tests than crack propagation; they more represent the pipeline in service threshold test are often easier to perform.

NIST-Gaithersburg has a slow strain test rig that is capable of gas phase testing up to 1,000 psi and cathodic charging and may be able to support this capability. (Ricker-NIST)

Discussion on Inter-laboratory cooperation/ test program

Is it important to perform Round Robin testing if the KD-10 approach relies on the vendor to test and certify the components? (*Consensus is yes*)

Duplication of capabilities seems to be more beneficial; it is not encroaching on others capabilities.

Will enable Round Robin test/validation

Samples need to be prepared by one source

Need to standardize hydrogen gas purity & charging techniques

Need to perform test in hydrogen and compare to helium data to see the reduction in properties

This program would enable pooling of data and assembly of a database on materials properties

We are talking about developing a national capability

NIST-Gaithersburg has a slow strain test rig and may be able to participate in RR testing that is capable of 1,000 psi gas phase and cathodic charging. (Ricker-NIST)

Other potential organizations include ORNL, Sandia-Livermore, SRNL and NIST-Boulder (once they're up and running).

Reemphasize- hydrogen charging content through pipe wall is probably irrelevant to the concentration around the crack tip.

Is the pressure relevant at the crack tip or is it more based on the hydrogen content? (Livne-NIST)

Though to not depend on it highly, think there may be an upper limit where higher pressures will not affect it any. (McColskey –NIST)

Discussion on Component Testing program

The consensus was that the need for component testing might be a niche that NIST could fill.

- FRP certification lends itself more to component testing
- Component testing would enable the evaluation of joining techniques
  - Welds
  - Joints
- Need to develop a validation approach

3.3 Breakout Session: Codes and Standards and Safety

Session Chair: Lou Hayden

Attendees:
Juana Williams
Thomas Gross
Steve Pawel
Paul Tibbals
Angelique Lasseigne

Objectives:

Determine how to process and evaluate data and techniques
Develop design allowables
Safety considerations for test facilities and personnel

Directives:

Identify critical test parameters for suitable test methods
Define fields that contribute to a database for hydrogen pipeline designers/operators
Define a comprehensive test plan for NIST, utilizing:

- Standardized methods
- A prioritized list of materials

Establishment of goals:

1. Testing commonly used (API 5LX52, SA106B) linepipe steel base metal and weldments:
    a. For loss of ductility, loss of toughness, fatigue, low cycle and high cycle, at varying K, da/dN, and R values.
    b. Test materials over a range of temperatures to determine the scope of the embrittlement range.
    c. Support the prescriptive design method currently planned for B31.12.
    d. Document and archive test results in a database.

	Need to Know*	**Current Knowledge?**
Base Metal	Reduction in ultimate strength	Reductions are reported
	Reduction in yield strength	Reductions are reported
	Reduction in ductility	Significant reductions have been measured
	Fracture resistance (K_{IH} values)	Mostly unknown
	Fatigue resistance (da/dn values)	Mostly unknown
	What changes when the material is cold formed	Unknown
	How does a corroded surface affect the performance?	Unknown
	Diffusion coefficients for various microstructures and the amount of hydrogen that gets trapped in the matrix	Unknown?
Weld Metal	Reduction in ultimate strength	Unknown
	Reduction in yield strength	Unknown
	Reduction in ductility	Unknown
	Fracture resistance (K_{IH} values)	Unknown
	Fatigue resistance (da/dn values)	Unknown
	Effect of post weld heat treatment	Unknown
Heat Affected Zone	Reduction in ultimate strength	Unknown
	Reduction in yield strength	Unknown
	Reduction in ductility	Unknown
	Fracture resistance (K_{IH} values)	Unknown
	Fatigue resistance (da/dn values)	Unknown
	Effect of post weld heat treatment	Unknown

2. a. Verify the effect of pressure on embrittlement of commonly used (API 5LX52, SA106B) linepipe steel base metal and weldments.
   b. Test up to 3000 psi
   c. Test up to 15000 psi to determine the maximum pressure limit (if any) for carbon steels.

4. a. Evaluation of microstructure of materials commonly used (API 5LX52, SA106B) linepipe steel base metal and weldments for performance in H_2.
   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.

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?

   Strength in bending
   Pressure retaining capacity
   - Burst strength (strain) at Temperature and Hydrogen Pressure

   Time-related hydrogen degradation of composite material

  - Chemical Reaction
  - Delamination
  - Internal Damage due to Hydrogen Accumulation
- Environmental degradation (other than hydrogen)
  - Ultraviolet
  - Soil Chemistry
  - Moisture Absorption
  - Temperature (High and Low)
  - Corrosion of Metallic Components (Joints)
- Fatigue performance
  - Specifications to be determined

	Need to Know*	**Current Knowledge?**
Base Material	Reduction in burst strength	Unknown
	Reduction in flexural strength	Unknown
	Reduction in ductility	Unknown
	Fracture resistance	Unknown
	Fatigue resistance (da/dn values)	Unknown
	What changes when the material is mechanically distorted? i.e. reeled or kinked	Unknown
	How does a degraded/altered surface affect the performance?	Unknown
	Diffusion coefficients for different classes of materials and the amount of hydrogen that gets trapped in the matrix	Unknown
Joint	Reduction in burst strength	Unknown
	Reduction in flexural strength	Unknown
	Reduction in ductility	Unknown
	Fracture resistance	Unknown
	Fatigue resistance (da/dn values)	Unknown

Discussion Notes:

Establish breakout discussion guidelines of materials, divided into three categories:

Existing metallic

Novel metallic

Nonmetallic (ASME B31.12 code doesn't mention non-metallics at this time due to lack of engineering data)

Based on DOE cost analysis, over 70% of pipeline of cost is the materials and labor. It is doubtful that we are going to make hydrogen pipelines cost effective that incorporate new materials that require alloy development and other associated costs. (This might be true if the right of way was already in existence.)

FRP, fiber reinforced plastic, said to be the solution for low-cost/low-risk pipelines to transport renewable energy. Capital in a material derived from nonrenewable sources and FRP is not proven in this environment where most pipeline incidents come from third-party damage.

The highest cost in placing a pipeline is obtaining the right of way. How would you establish a pipeline from Boston to Washington, D.C.?

What prevents the establishment of a hydrogen powered car as a primary means of transport is the underlying lack of infrastructure. The initial economically justified city-centered development would prevent a cross-country driving trip.

How do you select a representative sample of old pipeline?

Regarding linepipe pressure levels, we started out 5 years ago at 2,000 psi and increased the pressure to 3,000 psi after second meeting of the hydrogen code development task group.

NIST Workshop on Materials Test Procedures for Hydrogen Pipelines

Dates: August 21-22, 2007
National Institute of Standards and Technology
Materials Reliability Division
325 Broadway Boulder, CO
Building 1 Room 1107

Purpose: Develop roadmap for materials, test procedures, mechanical properties data and standards for future hydrogen pipelines. NIST/Materials Reliability Division can use this data as input into the research plan for the new hydrogen test facility being constructed in Boulder.

Agenda:
August 21

8:30-8:45 Welcome: David McColskey (NIST)
8:45-9:00 Overview of NIST and its mission (Stephanie Hooker, NIST)
9:00-9:30 NIST Hydrogen Program (Richard Ricker, NIST)
9:30-10:00 Government Hydrogen Research Activities (Tim Armstrong, DOE)
10:00-10:15 Break
10:15-10:45 Government Hydrogen Research Activities (Jim Merritt, DOT/PHMSA)
10:45-11:15 Codes and Standards (Lou Hayden)
11:15-11:45 Workshop goals and Breakout Sessions (Tom Siewert, NIST)
11:45-1:00 Lunch

1:00-4:00 Breakout Sessions:
1) Materials (Leader: Brian Somerday, Sandia)
Which have adequate data?
What recent materials have been overlooked?

New materials in development?
Metals
Composites and plastics

2) Test Techniques and Methods (Leader: Andrew Duncan, Savannah River National Lab)
Which attributes have adequate techniques?
Which attributes do not have adequate techniques?
In-chamber instrumentation for properties and load measurement?
Effects of impurities in H2? How to measure gas concentration levels? To what accuracy?
Purging (purity) techniques?

3) Codes and Standards and Safety (Leader: Lou Hayden)
How to process and evaluate data (to develop design allowables) and techniques.
Safety considerations for test facilities and personnel

4:00-5:00 Preliminary results from breakout sessions.

August 22

8:30-10:30 Breakout sessions (continued)
10:30-10:45 Break
10:45-12:30 Results of breakout sessions

Contact: Tom Siewert (siewert@boulder.nist.gov) (303) 497-3523
David McColskey (mccolske@boulder.nist.gov) (303) 497-5544

Pre-registration (mandatory) at:
http://www.nist.gov/public_affairs/confpage/blconf.htm

Updates on agenda at:
http://www.boulder.nist.gov/div853/Pipeline_Workshop/index.htm

Report: STATUS OF ATTENDEES   Criteria: ALL   Sort by: NAME

Last Name	First Name	Company Name	e-Mail Address / Phone	Amt Due	Amt Paid	Registration Type	Cancelled/ Noshow
Adams	Thad	Savannah River National Laboratory	thad.adams@srnl.doe.gov (803)725-5510	$0		No Fee Attendee	No Show
Armstrong	Timothy	Oak Ridge National Laboratory	armstrongt@ornl.gov (865)574-7996	$0		No Fee Attendee	
Bakke	Paul	DOE - Golden Field	paul.bakke@go.doe.gov (303)275-4916	$0		No Fee Attendee	
Balch	Dorian	Sandia National Laboratories	dkbalch@sandia.gov (925)294-2368	$0		No Fee Attendee	
Burgess	Robert	National Renewable Energy Laboratory	robert_burgess@nrel.gov (303)275-2823	$0		No Fee Attendee	
Cannon	Ian	Pratt & Whitney Rockedtyne	ian.cannon@pwr.utc.com (818)586-9673	$0		No Fee Attendee	
Collins	Jenny	Colorado School Of Mines	jcollins@mines.edu (303)931-2561	$0		No Fee Attendee	
Darcis	Philippe	NIST - Materials Reliability Division	darcis@boulder.nist.gov (303)497-4866	$0		No Fee Attendee	
Drexler	Liz	NIST - Materials Reliability Division	drexler@boulder.nist.gov (303)497-5350	$0		No Fee Attendee	
Duncan	Andrew	Savannah River National Laboratory	andrew.duncan@srnl.doe.gov (803)725-4996	$0		No Fee Attendee	
Feng	Zhili	Oak Ridge National Laboratory	fengz@ornl.gov (865)576-3797	$0		No Fee Attendee	
Gerstle	Walter	NIST - Materials Reliability Division	gerstle@boulder.nist.gov (303)497-7624	$0		No Fee Attendee	
Gross	Thomas	IF,LLC/LMI	tgenergy@cox.net (703)273-0631	$0		No Fee Attendee	
Hayden	Louis	Asme	louis.hayden1@verizon.net (610)694-0868	$0		No Fee Attendee	
Jackson	Joshua	Colorado School Of Mines	joshjack@mines.edu (303)895-7146	$0		No Fee Attendee	No Show
Klug	Kevin	Concurrent Technologies Corporation	klugk@ctc.com (910)437-9904	$0		No Fee Attendee	
Koenig	Kamalu	Colorado School Of Mines	kkoenig@mines.edu (720)935-8401	$0		No Fee Attendee	
Lasseigne	Angelique	NIST-Materials Reliability Division	lasseign@boulder.nist.gov (303)497-3032	$0		No Fee Attendee	
McColskey	Joseph	NIST	mccolske@boulder.nist.gov (303)497-5544	$0		No Fee Attendee	
McCowan	Chris	NIST	mccowan@boulder.nist.gov (303)497-3699	$0		No Fee Attendee	
Mcqueen	Shawna	Energetics	smcqueen@energetics.com (410)290-0370	$0		No Fee Attendee	
Meisels	Aryeh	Pratt And Whitney Rocketdyne	aryeh.meisels@pwr.utc.com (818)586-7827	$0		No Fee Attendee	
Merritt	James	DOT/PHMSA	james.merritt@dot.gov (303)683-3117	$0		No Fee Attendee	
Moyer	David	Concurrent Technologies	moyerd@ctc.com (814)269-2578	$0		No Fee Attendee	
Muralidharan	Govindarajan	Oak Ridge National Laboratory	muralidhargn@ornl.gov (865)574-4281	$0		No Fee Attendee	
Nibur	Kevin	Sandia National Laboratory	kanibur@sandia.gov (925)294-3270	$0		No Fee Attendee	
Olson	David	Colorado School Of Mines	dolson@mines.edu (303)273-3955	$0		No Fee Attendee	
Pawel	Steve	Oak Ridge National Laboratory	pawelsj@ornl.gov (865)574-5138	$0		No Fee Attendee	
Pitchure	David	Nist	pitchure@nist.gov (301)975-3814	$0		No Fee Attendee	

Criteria: ALL Sort by: NAME

Last Name	First Name	Company Name	e-Mail Address / Phone	Amt Due	Amt Paid	Registration Type	Cancelled/ Noshow
Prager	Martin	Materials Properties Council Inc.	mprager@forengineers.org (212)534-4275	$0		No Fee Attendee	
Rana	Mahendra	Praxair, Inc.	mahendra_rana@praxair.com (716)879-2408	$0		No Fee Attendee	No Show
Ricker	Richard	NIST	richard.ricker@nist.gov (301)975-6023	$0		No Fee Attendee	
Robertson	Ian	Materials Science and Engineering, UIUC	lmelchi@uiuc.edu (217)333-1440	$0		No Fee Attendee	No Show
Rocha	Adriana	Inmetro	acrocha@inmetro.gov.br 55 21 26799050	$0		No Fee Attendee	
Roubidoux	John	Colorado School Of Mines	jroubido@mines.edu (720)227-2939	$0		No Fee Attendee	
Shtechman	Avigdor	NIST - Materials Reliability Division	avigdor.shtechman@nist.gov (303)497-5050	$0		Comp Attendee ($0)	
Siewert	Tom	NIST	siewert@boulder.nist.gov (303)497-3523	$0		No Fee Attendee	
Simoni De Cannon	Fiorella	Lmi	fsimoni@lmi.org (571)633-7685	$0		No Fee Attendee	No Show
Slusser	Joseph	Air Products and Chemicals, Inc	slussejw@airproducts.com (610)481-5887	$0		No Fee Attendee	
Sofronis	Petros	University of Illinois at Urbana-Champaign	sofronis@uiuc.edu (217)333-2636	$0		No Fee Attendee	
Somerday	Brian	Sandia National Lab	bpsomer@sandia.gov (925)294-3141	$0		No Fee Attendee	
Tibbals	Paul	Pacific Gas & Electric Co.	pht1@pge.com (925)866-5356	$0		No Fee Attendee	
Treinen	John	NIST - Materials Reliability Division	jtreinen@boulder.nist.gov (303)497-7065	$0		No Fee Attendee	
Vasquez	Samuel	El Paso Corp	samuel.vasquez@elpaso.com (915)587-3757	$0		No Fee Attendee	
Widenmaier	Kevin	TransCanada	kevin_widenmaier@transcanada.com (403) 920-5646	$0		No Fee Attendee	
Williams	Juana	Nist Weights And Measures Division	juana.williams@nist.gov (301)975-3989	$0		No Fee Attendee	

Total Collected: $0

Number Paid and Unpaid Registered To Date:	0
Number of Comp Attendees:	1
Number of Badge Only Attendees:	0
Number of Press Attendees:	0
Number of Comp Speakers:	0
Number of No Fee Attendees:	45
Total Count:	46