National Testing Laboratory for Solid-State Hydrogen Storage Technologies

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This presentation does not contain any proprietary or confidential information

Project ID #: STP 34
Overview

Timeline

- Program Start: March 2002
- Program End: September 2006
- 99% Complete

Barriers

- Standardization of Methods
- “Gold Standard” Measurements
- Verification of Material Performance
  - Understanding of Physisorption & Chemisorption Processes
  - Reproducibility of Performance
- Verification of System Performance
  - Reproducibility of Performance
  - System Life-Cycle Assessment
- Codes & Standards

Budget

- Total Program Funding:
  - DOE Share: $2.4M
  - SwRI Share: $0.62M
- Funding Received in FY05: $415k
- Funding Received in FY06: $50K

Partners / Collaborations

- National Hydrogen Association (Standards)
- Ovonic Hydrogen Systems (Full-scale storage systems)
- NESSHY (EC-JRC)
- NREL
- INER (Taiwan)
Objectives

**Overall**
- Construct and operate a national-level research and core reference laboratory aimed at assessing and validating the performance of emergent solid-state hydrogen storage materials and full-scale systems
- Establish “gold standard” measurement techniques for hydrogen sorption and related performance metrics

**Current**
- Qualify laboratory based on outcome of double-blind Round-Robin testing
- Assist NREL in independent analysis of SWNT materials
- Improve and refine measurement techniques to accommodate most any structure of matter, thermal condition, and sample quantity
- Complete testing laboratory for full-scale hydrogen storage systems
**Approach**

- **Lab Facility Design & Instrumentation Selection**
  - 01/16/2004

- **Completion of Small-Scale Lab**
  - System Shakedown
  - Draft SOPs
  - 05/01/2005

- **Refine Analytical Methods**
  - Low-temperature measurements
  - Thermal gradient correction
  - Small sample mass
  - Sample vessel optimization

- **Continuous**

- **Qualification of Small-Scale Lab:**
  - Internal controls
  - Round-Robin Testing
    - Carbon
    - Metal hydride
  - 05/06/2005

- **Completed Analysis of R-R Carbon Samples & Submitted Results**
  - 08/31/2005

- **Completed Independent Validation of NREL Measurements:**
  - Two SWNT Samples
  - Three Catalyzed SWNT Samples
  - 09/05/2005

- **Completion of Full-Scale Lab**
  - 03/16/2006

- **Revise SOPs (Small-Scale)**
Technical Accomplishments

Completion & Validation of Laser Thermal Desorption Mass Spectrometer

- Quantitative and highly sensitive
- Suitable for desorption of small sample quantities (< 50 mg)
- Resolves relative energies of different physisorbed or chemisorbed hydrogen binding sites

Ytterbium Laser & Optical Bench
Technical Accomplishments

Completion & Validation of High-Pressure Volumetric Analyzer

- Full-pressure PCT and sorption isotherms
- Extended vacuum capability using logic control
- Real-time mass spectrometry
- Sample vessel optimized for better sample compactness and consistent thermal diffusivity
Technical Accomplishments

Completion & Validation of High-Pressure Gravimetric Analyzer

- Full-pressure PCT and sorption isotherms
- Sample vessel optimized for more accurate buoyancy correction
- Real-time mass spectrometry
- Cryogenic cooling attachment under development
Technical Accomplishments

Understanding Critical Point Effects and Free Volumes in H₂ Sorption

- Refined technique and apparatus for low-temperature volumetric measurements by examining the relationship between critical point effects and free volume
- Sample compaction crucial in minimizing susceptibility of system to density fluctuations and measurement inaccuracies
- Determined an optimum vessel design
- Improved thermal diffusivity and stability
Technical Accomplishments

Round Robin Results: Volumetric & Gravimetric Measurements

- Room temperature results consistent with those of outside participant labs (n=6)
- Gained detailed understanding of important factors affecting the accuracy of low-temperature physisorption measurements
- Low temperature isotherm results also consistent with those of outside participant labs after refining measurement technique and apparatus
Technical Accomplishments

Measurements of Various Carbon Materials at Room Temperature

- Demonstrated excellent complementarity between volumetric and gravimetric measurements
- Hydrogen physisorption on various carbon materials (non-catalyzed) is limited to < 0.5 wt.% at room temperature
Technical Accomplishments

Evaluation of Catalyzed SWNTs from NREL: Validation of H$_2$ Capacity by Thermal Desorption Mass Spectrometry

- Demonstrated capability of measuring very small sample quantities (< 10 mg)
- SwRI measured hydrogen capacity consistent with previously published data†

Technical Accomplishments

Completion of Full-Scale Laboratory Facility

Characterization of the performance of full-size storage systems to include:

- Sorption/desorption performance
- Refueling time
- Resistance to exogenous contaminates
- Specific energy contained
- Impact, vibration and fire resistance
Future Work

Program / Sample Backlog
1. US DOE
2. US Commercial
3. NESSHY (EC)
4. INER (Taiwan)

Future Work

Small-Scale Lab Facility → Complete Round-Robin Testing of Metal Hydride Samples → Facility Benchmarking

07/01/2006

Full-Scale Lab Facility → Facility Benchmarking

07/01/2006

Ovonic Hydrogen Systems

<table>
<thead>
<tr>
<th></th>
<th>Fiber-Wrapped Vessel</th>
<th>Aluminum Tubular Vessel</th>
</tr>
</thead>
<tbody>
<tr>
<td>H₂ Capacity</td>
<td>1.7 kg</td>
<td>0.7 kg</td>
</tr>
<tr>
<td>Diameter</td>
<td>11 in.</td>
<td>6 in.</td>
</tr>
<tr>
<td>Length</td>
<td>31.3 in.</td>
<td>60 in.</td>
</tr>
<tr>
<td>Weight</td>
<td>127 kg</td>
<td>81 kg</td>
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</table>
Summary

Relevance: Provide DOE with facilities to independently assess the performance of solid state storage materials.

Approach: Construct and operate a national-level research and core reference laboratory aimed at assessing and validating the performance of emergent solid-state hydrogen storage materials and full-scale systems.

Technical Accomplishments: Completed laboratory containing gravimetric, volumetric and TPD instrumentation; room temperature and low temperature results on round robin carbon samples consistent with those of outside participant labs.

Collaborations: Active collaborations with NESSHY (EC-JRC), NREL and INER (Taiwan).

Future Research: Improve and refine measurement techniques to accommodate most any structure of matter, thermal condition, and sample quantity; complete testing laboratory for full-scale hydrogen storage systems.
Response to Reviewer’s Comments

• Sample shipping restrictions may be a significant impediment
  • Began to address shipping issues with presentation at workshop
  • Shipping and receipt protocols have been developed

• Need more effort on characterizing smaller samples
  • Current TGA and TPD systems are available for 10-100mg samples
  • Future plans to modify volumetric system for 10-100mg samples

• Burst chamber for tanks too small for automotive scaled tanks
  • Chambers are adequate for current tanks up to at least 2 kg capacity
  • Modifications are possible for larger or unusual shaped tanks


Critical Assumptions & Issues

Assumption: Sufficient quantities of research grade samples will require independent verification
• Round-robin testing program designed to optimize acceptance of facility
• Rate of development of promising materials that require independent verification is uncertain

Assumption: Progress in the development of full-scale storage systems will be sufficient to adequately utilize the testing facility
• Independent verification of promising results on new materials should speed system development
• Rate of system development is uncertain

Assumption: Facility will have the instrumentation and protocols necessary to evaluate as yet undeveloped materials
• Facility team needs to keep abreast of current developments within community
• Facility team and DOE need to remain flexible in defining the instruments and protocols in the facility