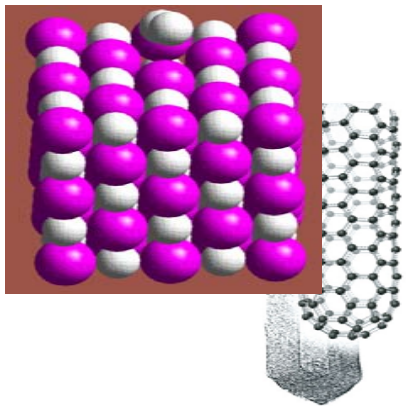
National Testing Laboratory for Solid-State Hydrogen Storage Technologies



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DOE Annual Merit Review
Washington DC
May 18, 2006

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

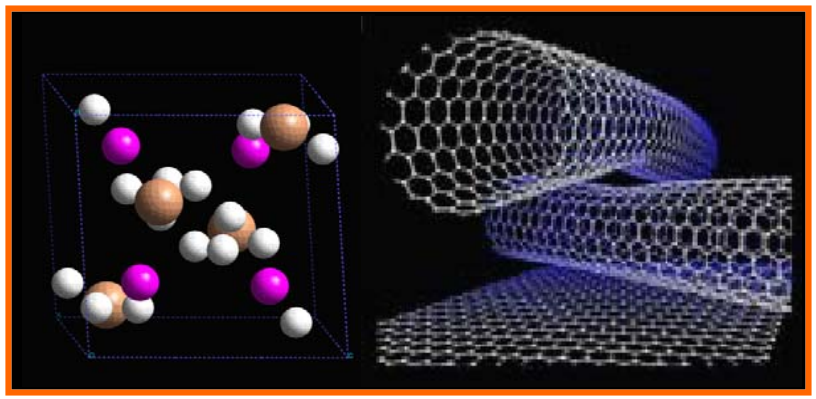
Partners / Collaborations

- 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



- **Capacity?**
- **Kinetics?**
- **Thermodynamics?**
- **Cycle life?**

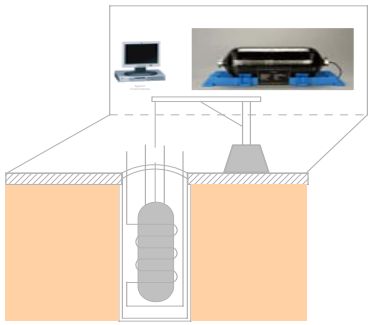
Current

- 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

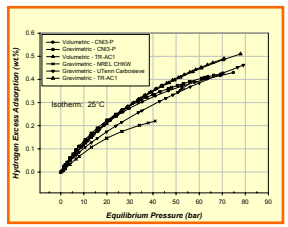
05/01/2005

Qualification of Small-Scale Lab:
- Internal controls
- Round-Robin Testing
  - Carbon
  - Metal hydride

05/06/2005

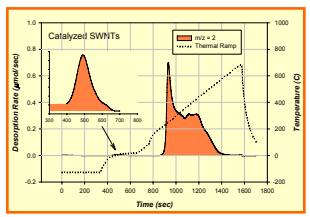
Completed Analysis of R-R Carbon Samples & Submitted Results

08/31/2005



Completed Independent Validation of NREL Measurements:
1. Two SWNT Samples
2. Three Catalyzed SWNT Samples

09/05/2005



Refine Analytical Methods
1. Low-temperature measurements
2. Thermal gradient correction
3. Small sample mass
4. Sample vessel optimization

Continuous

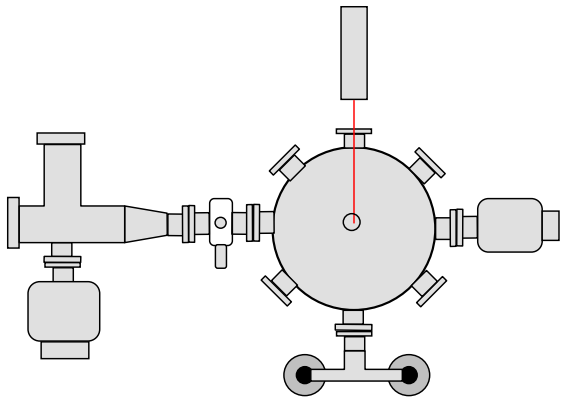
Revise SOPs (Small-Scale)

Completion of Full-Scale Lab

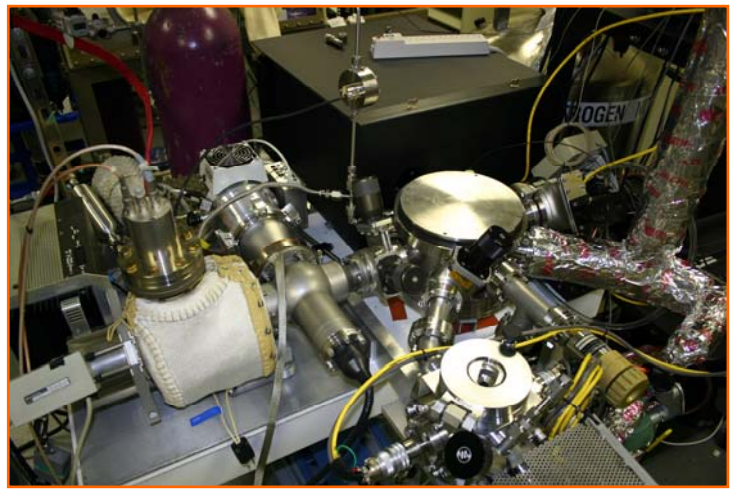
03/16/2006

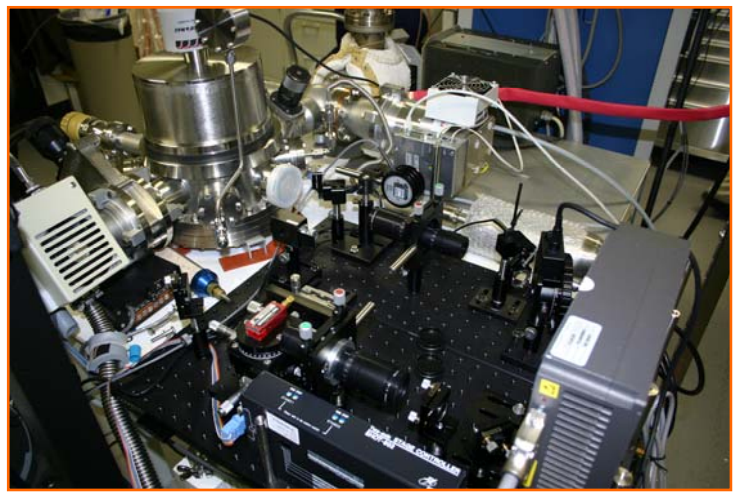
Technical Accomplishments

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

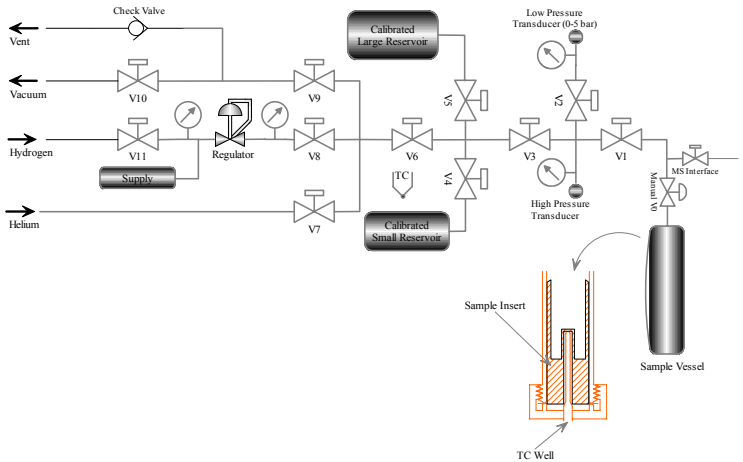




Ytterbium Laser & Optical Bench

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

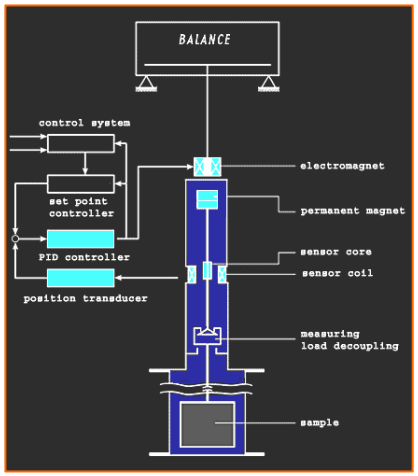
Vacuum Logic Control

Capillary MS Interface

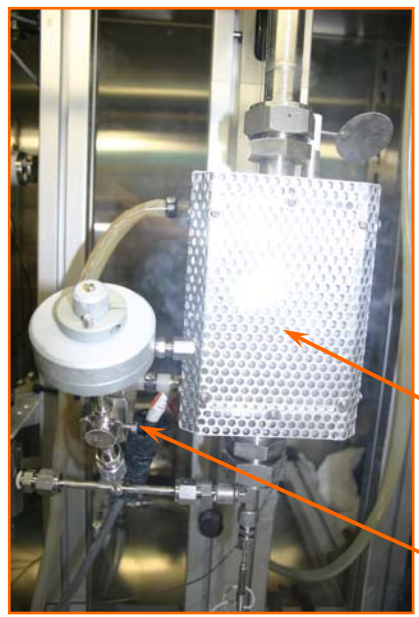
Backside

Technical Accomplishments

Completion & Validation of High-Pressure Gravimetric Analyzer

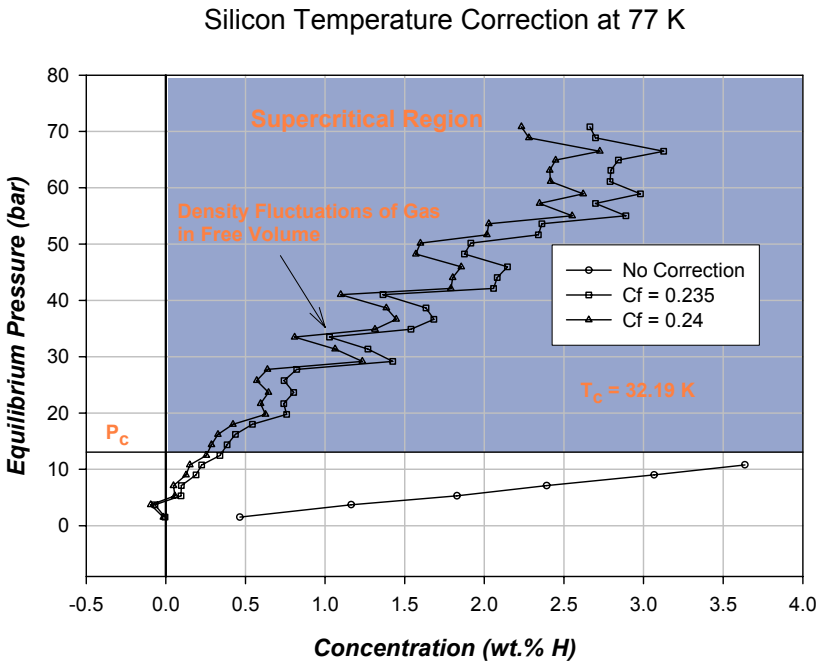


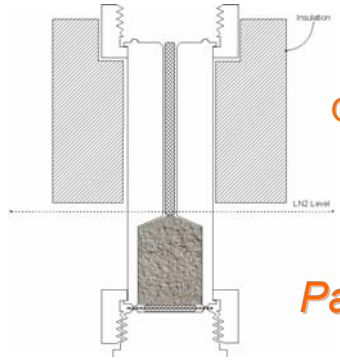
- Full-pressure PCT and sorption isotherms
- Sample vessel optimized for more accurate buoyancy correction
- Real-time mass spectrometry
- Cryogenic cooling attachment under development



Technical Accomplishments

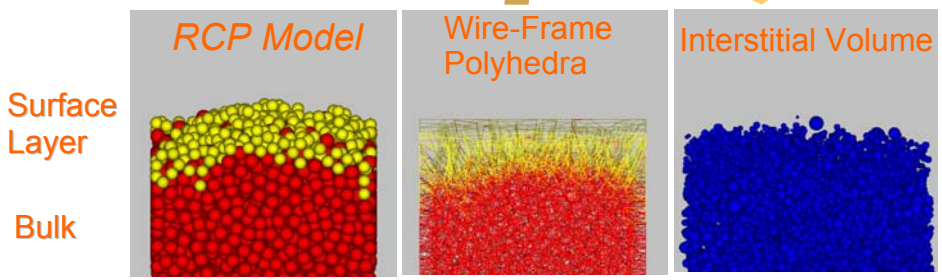
Understanding Critical Point Effects and Free Volumes in H_2 Sorption





Optimized Sample Vessel

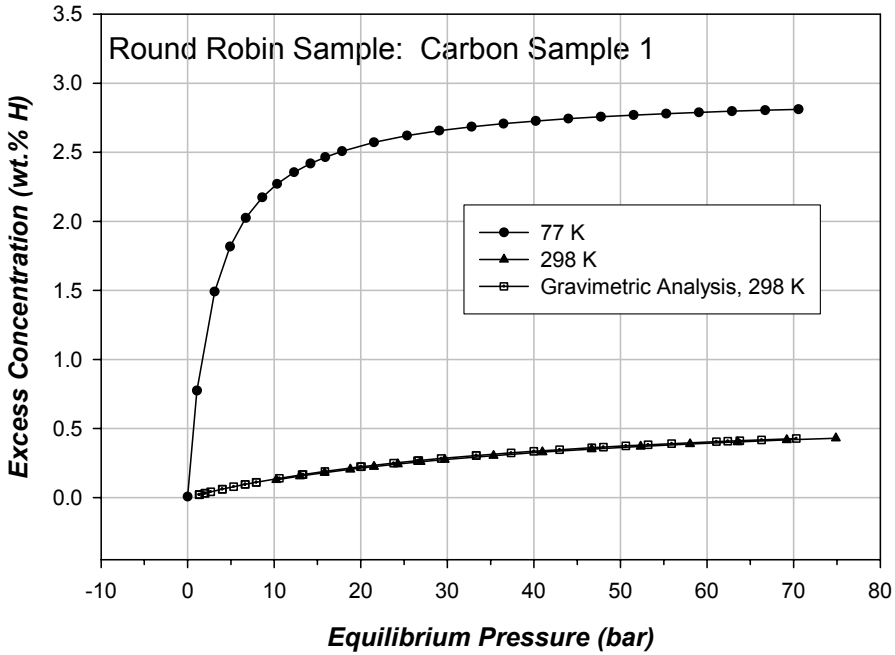
Packing Density Considerations

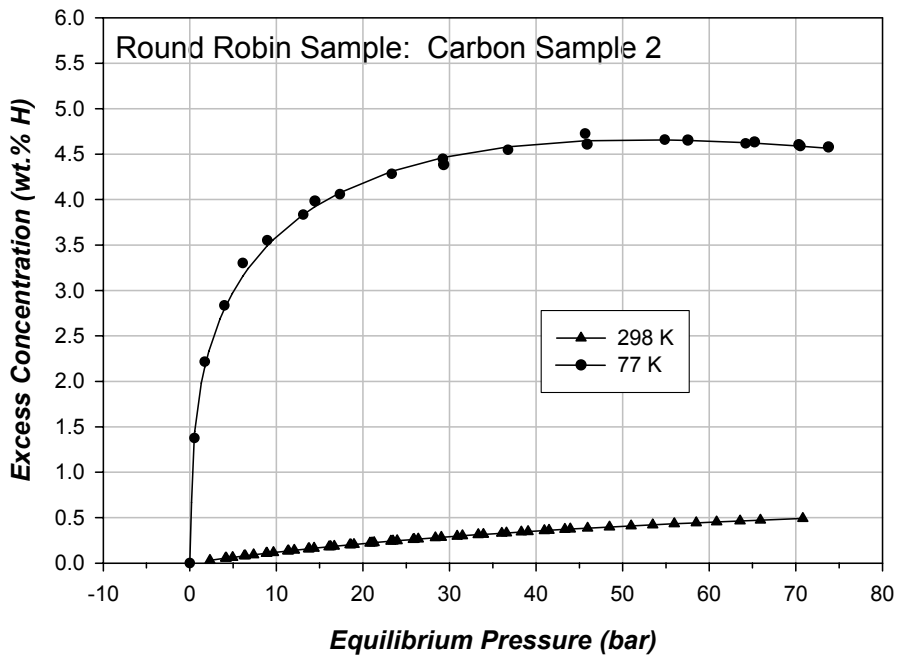


- Refined technique and apparatus for low-temperature volumetric measurements by examining the relationship between critical point effects and free volume
- Sample compaction crucial in minimizing susceptibility of system to density fluctuations and measurement inaccuracies
- Determined an optimum vessel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

Technical Accomplishments

Round-Robin Results: Volumetric & Gravimetric Measurements

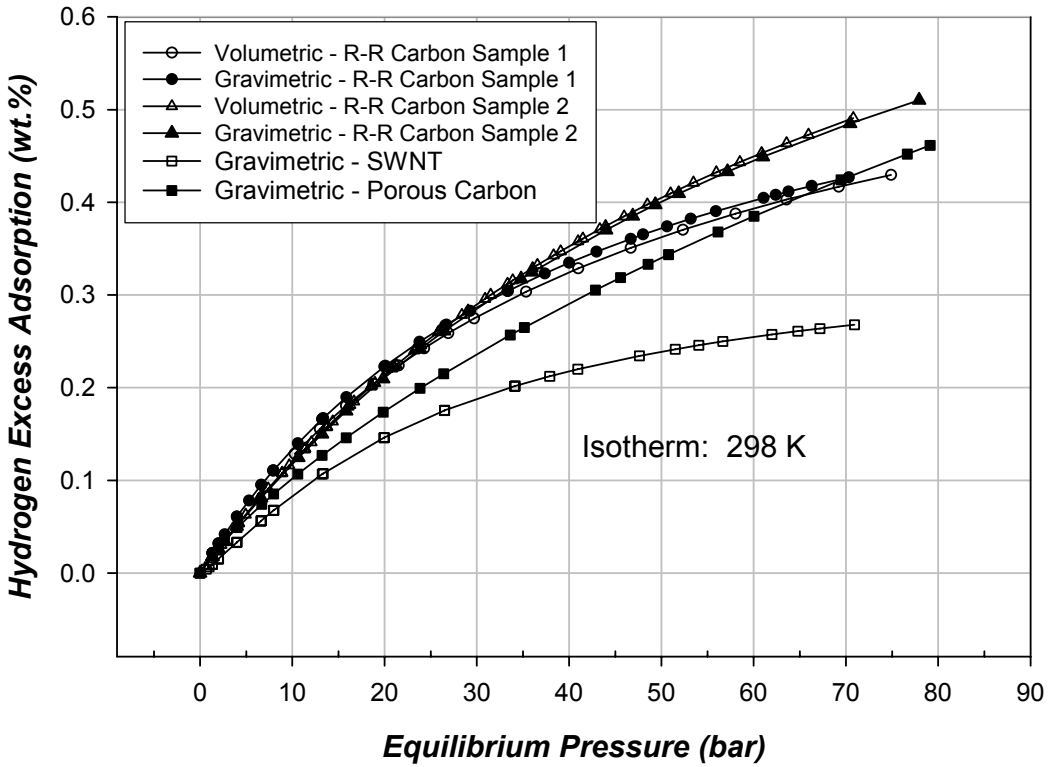




- Room temperature results consistent with those of outside participant labs (n=6)
- Gained detailed understanding of important factors affecting the accuracy of low-temperature physisorption measurements
- Low temperature isotherm results also consistent with those of outside participant labs after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

Technical Accomplishments

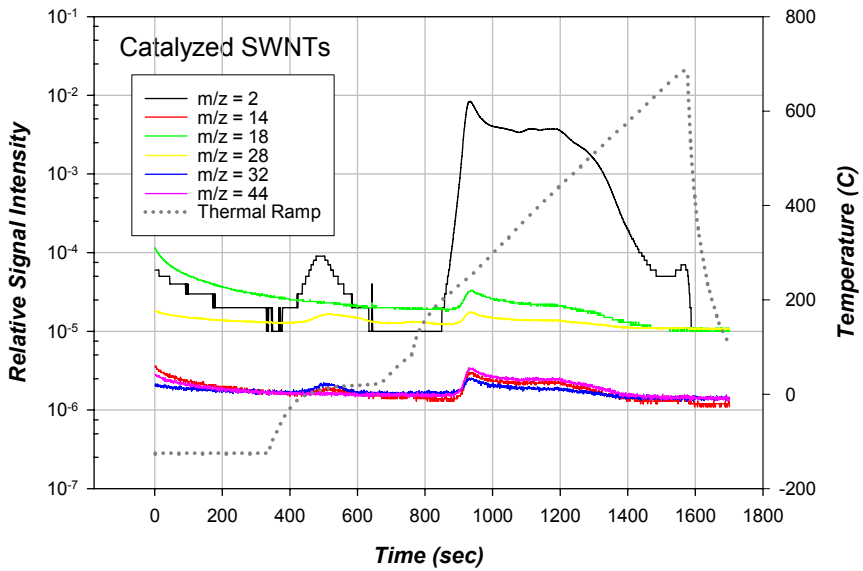
Measurements of Various Carbon Materials at Room Temperature



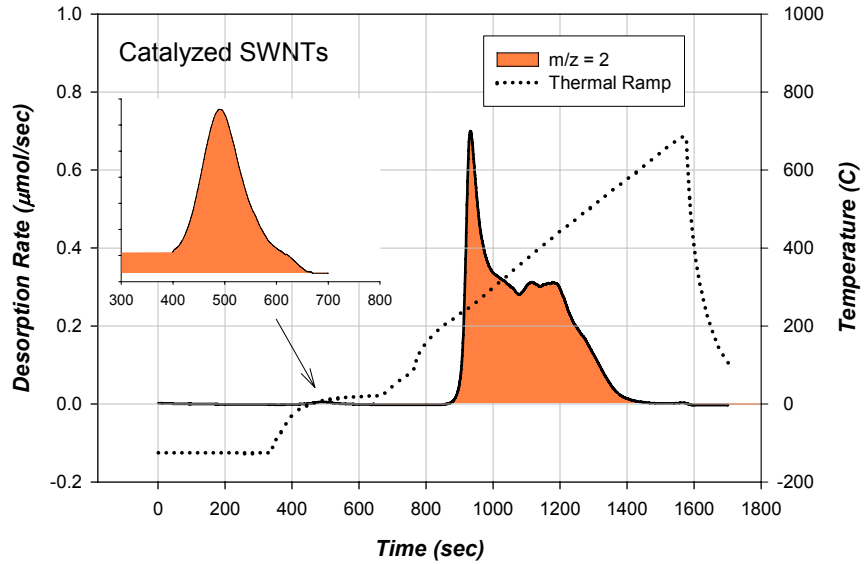
- Demonstrated excellent complementarity between volumetric and gravimetric measurements
- Hydrogen physisorption on various carbon materials (non-catalyzed) is limited to < 0.5 wt.% at room temperature

Technical Accomplishments

Evaluation of Catalyzed SWNTs from NREL: Validation of H_2 Capacity by Thermal Desorption Mass Spectrometry



Multi-Ion Monitoring of Desorbed Species



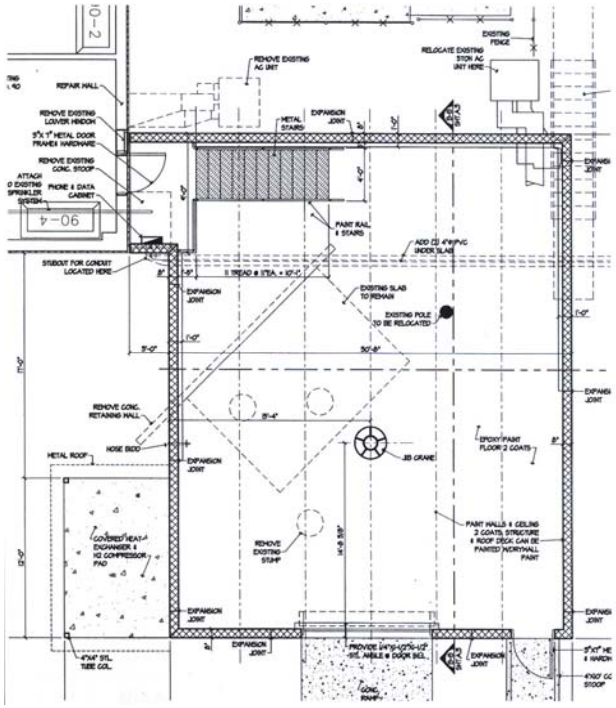
Measured Desorption: 2.78 wt.%

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

† A.C. Dillon, K.M. Jones, T.A.Bekkedahl, C.H. Kiang, D.S. Bethune, & M.J. Heben, Nature (386) 377, 1997.

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

Small-Scale Lab Facility → Complete Round-Robin Testing of Metal Hydride Samples → Program / Sample Backlog
1. US DOE
2. US Commercial
3. NESSHY (EC)
4. INER (Taiwan)

07/01/2006

Full-Scale Lab Facility → Facility Benchmarking

Commence 07/01/2006

Ovonic Hydrogen Systems

	Fiber-Wrapped Vessel	Aluminum Tubular Vessel
H_2 Capacity	1.7 kg	0.7 kg
Diameter	11 in.	6 in.
Length	31.3 in.	60 in.
Weight	127 kg	81 kg

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

Publications & Presentations

1. M.A. Miller and R.A. Page, National Testing Laboratory for Solid-State Hydrogen Storage Technologies, NHA National Hydrogen Conference, Washington, DC, March 29 – April 1, 2005
2. M.A. Miller and N. Sridhar, Measurement and Prediction of Material Performance Subject to Hydrogen Exposure, Hydrogen Gas Embrittlement Workshop, ASTM T.G. 01.06.08, ASTM Meeting, Reno, NV, May 17, 2005
3. M.A. Miller, Development and Application of Standard Methods for Quantifying Hydrogen Sorption in Nanostructured Materials, 2005 Taiwan Symposium on Hydrogen Storage in CNMs, Institute of Nuclear Energy Research (INER), Taipai, Taiwan, Oct. 18-19, 2005

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**