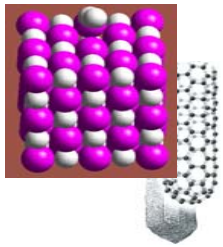
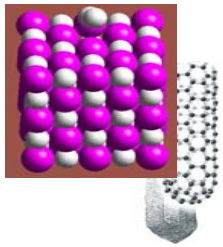


Standardized Testing Program for Emergent Chemical Hydride and Carbon Storage Technologies

2003 Hydrogen and Fuel Cells Merit Review Meeting

May 19-22, 2003

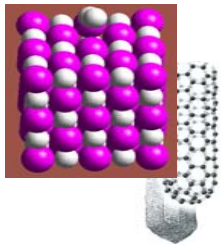


Relevance/Problem Definition

- DOE's Draft Hydrogen Storage Plan sets aggressive 2005, 2010 and 2015 targets for gravimetric and volumetric efficiency and cost for on-board hydrogen storage.

- Significant R&D activity is required since no current storage material or system meets even the 2005 targets.

- Variability in adsorption/desorption measurement equipment and protocols has made it difficult to assess the potential viability of many of the new solid-state storage materials, e.g., storage capacity of SWNTs can vary by 40 to 50% when measured by TPD vs. Seiverts in the same lab.



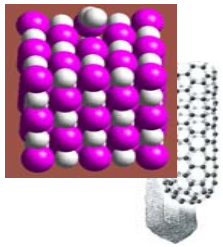
Relevance/Need and Objectives

DOE Program Need

An ability to accurately and independently assess the performance of the wide array of solid-state storage materials and focus efforts on those that show the most promise in meeting the 2005, 2010, and 2015 performance targets.

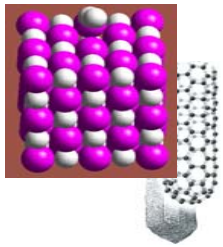
Project Objectives

- Develop and operate a standard testing and certification program specifically aimed at assessing the performance, safety and life cycle of emergent chemical hydride and carbon adsorption/desorption hydrogen storage materials and systems.
- Work with industry and the U.S. government to develop an accepted set of performance and safety evaluation standards.



Project Team

- Southwest Research Institute
- The National Hydrogen Association
- Teledyne Energy Systems
- Energy Conversion Devices, Inc.

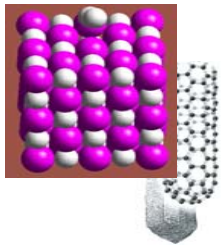


Technical Approach

Completed facility will contain two primary capabilities:

- Characterization of small quantities (milligram to gram) of candidate materials; and
- Characterization of the performance of full-size storage systems.

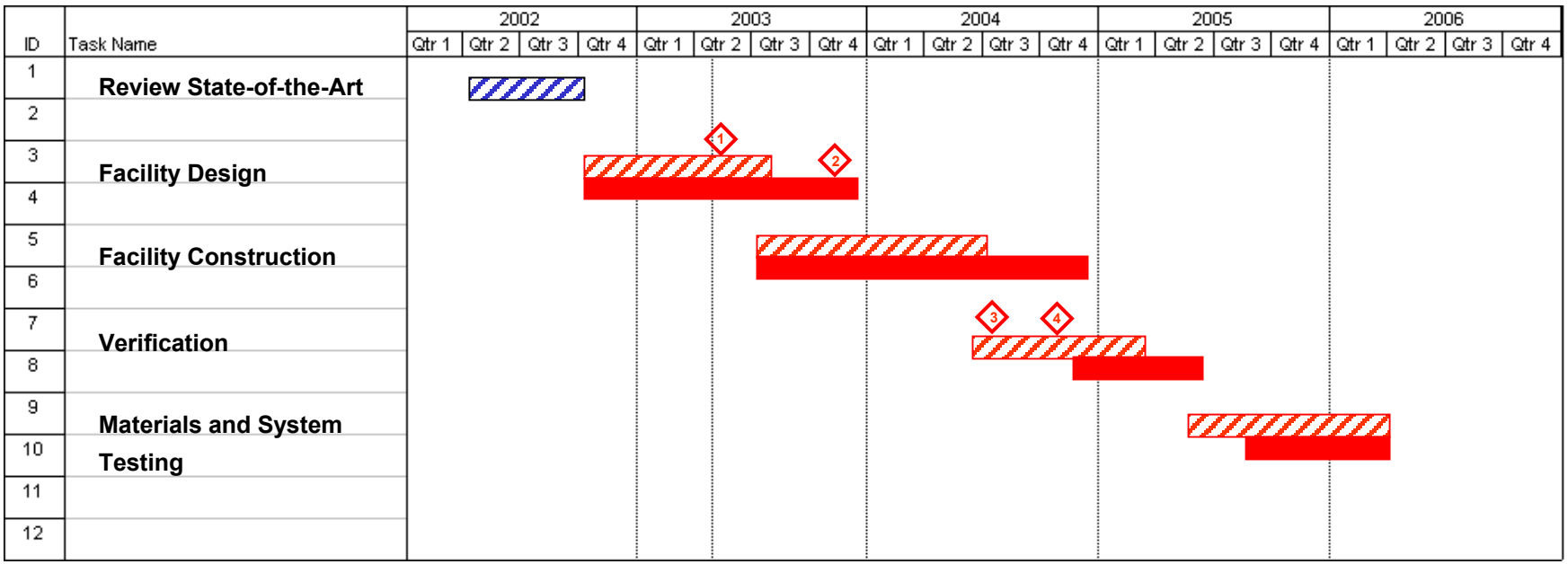
Completed facility will be available to all organizations involved in hydrogen storage.



Technical Approach (cont.)

- **Characterization of small quantities (milligram to gram) of candidate materials**
  - **Sorption/desorption behavior**
  - *Intrinsic thermodynamic characteristics*
  - *Elemental composition*
  - *Crystallographic properties*
- **Characterization of the performance of full-size storage systems**
  - **Sorption/desorption cyclic performance**
  - **Refueling time**
  - *Resistance to exogenous contaminates*
  - *Specific energy contained*
  - *Impact, vibration and fire resistance*

Project Timeline

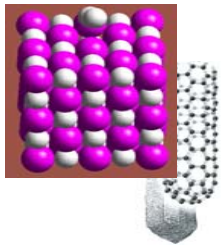


Milestones

1. Submit document describing test systems for measurements of storage materials.
2. Submit document describing test systems for measurement of full-scale storage systems.
3. Initiate internal verification with standard materials.
4. Initiate round-robin verification.

Storage Material Characterization

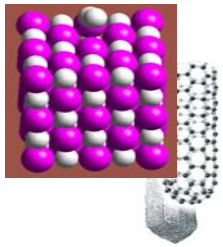
Storage System Characterization



Project Timeline (cont.)

- The testing facility will be completed and verified (2Q, 2005) in time to provide support for Task 3: Solid-State Materials R&D and Task 4: R&D of Adv. Solid-State Materials for 2010 Targets of DOE's Draft Hydrogen Storage Plan.

- Specifically, the facility will be available in time to provide input into the go/no-go decision point on carbon nanotubes (4Q, 2005) and the downselect of complex hydride materials (4Q, 2006).



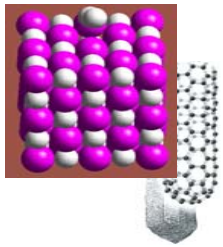
Project Timeline

Success Criteria

- Demonstrated ability to provide unbiased and accurate measurements of hydrogen storage capacities.
- Community acceptance and use of test facility.

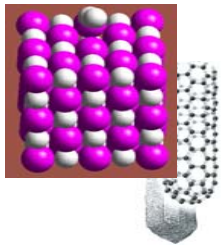
Business Plan

- Actively solicit representative storage materials and systems for use during facility verification and benchmarking, participate in Carbon Materials Working Group round robin testing program and initiate additional round robin program.



Current Status

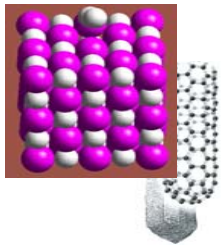
- Project activities initiated in April, 2002.
- Completed literature review of current measurement state-of-the-art.
- Developed preliminary facility and equipment design.
- Completed visits of leading laboratories involved in hydrogen adsorption/desorption measurements for critique of preliminary facility design.
  - GMR; 9/17/02 Air Products; 9/23/02
  - NREL; 9/30/02 Sandia; 11/18/02
- Submitted document specifying the equipment to be used for characterization of storage materials.



Current Status (cont.)

State-of-the-art Overview

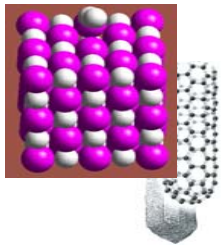
- Wide range of materials results in a wide range of measurement conditions.
  - mg to g sample quantities
  - vacuum to 100 atm
- Three prominent techniques being used.
  - Gravimetric (TGA)
  - Volumetric (Sieverts)
  - Thermally programmed desorption (TPD)
- Errors develop due to indirect nature of the techniques and operating near their sensitivity limits.
- SWNTs are currently the most challenging to measure due to combination of small mass and high pressure.
- Carbon materials are being measured using gravimetric (GM), volumetric (Air Products) and TPD (NREL).



Current Status (cont.)

State-of-the-art Overview – Conclusions

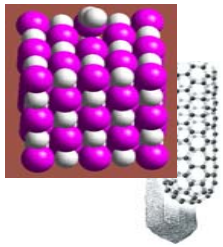
- No single measurement technique will be appropriate for all of the materials that are currently being considered for hydrogen storage.
- A comprehensive testing facility will need to have multiple techniques to cover the full range of materials.
- The presence of multiple techniques will provide higher confidence in results by enabling cross checks of storage capacity using more than one technique.
- Whenever possible, gas speciation should be included in the measurement of storage capacity.



Interactions/Collaborations

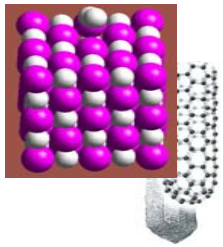
- Visits have been completed with hydrogen storage research teams at GMR, NREL, Air Products and Sandia. Engaged in ongoing discussions with these teams regarding the best approaches for accurate storage measurements.

- Participating in the Carbon Materials Working Group which is initiating a round robin testing program.



Future Work and Milestones

- A testing facility equipped to perform gravimetric, volumetric and TPD measurements on small quantities of solid-state hydrogen storage materials has been defined 2Q, 2003).
- The design for the facility to test full-scale storage systems will be finalized (3Q, 2003).
- Construction of approximately 50% of the testing facility should be completed (2Q, 2004).



Future Plans (cont.)

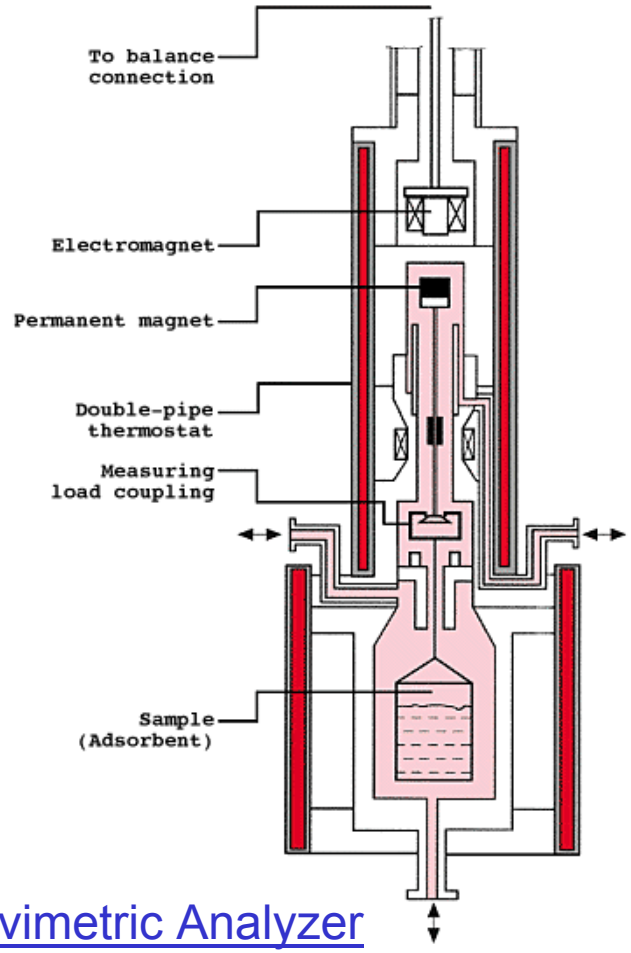
Testing Facility

- 1,000 sq ft of dedicated laboratory space
- Titan HM-200 Hydrogen Generator
  - 424 scfh
  - 99.9998% hydrogen
- Two stage hydrogen compressor for service up to 2,500psig
- Pressurized hydrogen storage vessels for 15kg hydrogen at 2,000 psi
- Source-gas manifolds with control valves and regulators
  - H_2
  - He
  - Lecture bottle source-gas manifold (CO, CO_2, H_2S)
- Electropolished supply lines and point of use purifiers
- H_2 safety sensors

Future Plans (cont.)

High-Pressure TGA

- Rubotherm magnetic suspension balance
- Mass spectroscopic desorption speciation
- Milligram – gram sample size
- 30 atm operating pressure
- Located within a glove box for air sensitive samples
- External material activation facilities
- Estimated accuracy of 3×10^{-4} wt % for 300 mg sample at 1 atm

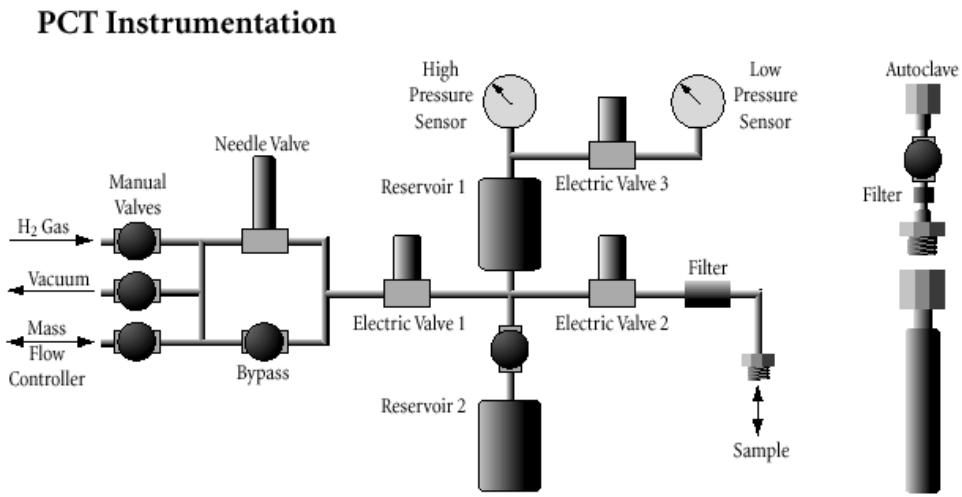


Gravimetric Analyzer

Future Plans (cont.)

Sieverts Apparatus

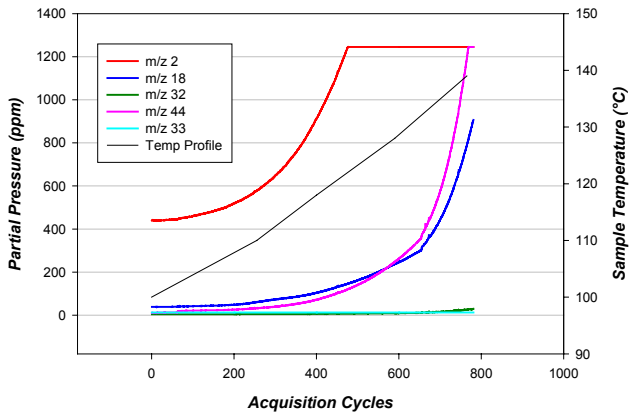
- Pressures up to 200 atm
- Sample temperatures to 400°C
- Controlled gas temperatures throughout
- Pneumatically controlled valves
- Variable sample volumes
- Estimated accuracy of 0.2 wt. % with 300 mg sample at 100 atm

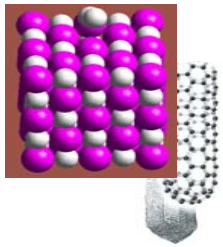


Future Plans (cont.)

Thermally Programmed Desorption

- Thermal Desorption & Recoiling Mass Spectrometry (TDARMS) system developed at SwRI
  - Real- time gas-phase speciation
  - High-pressure MS interface
  - Automated leak pressurization
  - Ultra-clean components
- Real-time vibrational spectroscopy of sample (fiber optically coupled)
- TDARMS being converted to TPD
- Accuracy to be determined

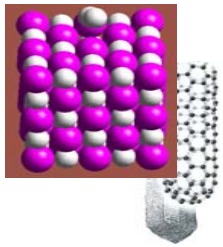




Future Plans (cont.)

Volumetric Sieverts apparatus for complete system testing

- >35 atm operating pressures
- > kg sample sizes
- 5 kg delivery in < 5 min
- Calibrated reservoirs
- Mass flow controller
- Heat exchanger (point-of-service chiller)
- Control and data acquisition computer
- System containment (Hydride-bed device)
- Gravimetric backup measurement



Contact Information

Point-of-Contact/Principal Investigators:

Richard A. Page, Institute Scientist

Michael A. Miller, Manager-R&D

Southwest Research Institute

6220 Culebra Rd., San Antonio, TX 78238

Phone (210)522-3252, FAX (210)522-6220

Email rpage@swri.org

Email mmiller@swri.org