National Testing Laboratory for Solid-State Hydrogen Storage Technologies

Michael A. Miller
Staff Scientist
Richard Page
Institute Scientist

Southwest Research Institute®
San Antonio, TX

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Project ID #: STP 36
## Overview

### Timeline

**Phase I**
- **Program Start**: March 2002
- **Program End**: September 2006
- **100% Complete**

**Phase II**
- **Program Start**: October 2006
- **Program End**: September 2010
- **20% Complete**

### Barriers

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

### Budget

**Phase I**
- **DOE Share**: $2.475M
- **SwRI Share**: $0.62M

**Phase II**
- **DOE Share**: $2.0M
- Funding Received in FY07: $405K

### Partners / Collaborations

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

Overall
• Support DOE’s Hydrogen Storage Program by operating an independent national-level reference laboratory aimed at assessing and validating the performance of novel and emerging solid-state hydrogen storage materials and full-scale systems
• Conduct measurements using established protocols to derive performance metrics: capacity, kinetics, thermodynamics, and cycle life
• Support parallel efforts underway within the international community, in Europe and Japan, to assess and validate the performance of related solid-state materials for hydrogen storage

Current
• Provide an in-depth assessment and validation of hydrogen physisorption in MOF-177
• Assess hydrogen adsorption and spillover phenomena in catalytically-doped IRMOF-8 compounds
• Develop method based on laser desorption mass spectrometry to evaluate hydrogen binding interactions in spillover compounds
Approach

MOF-177 (210 mg)

Precondition 45°C (1°C/min), 10⁻⁶ Torr, 18 hr

Volumetric Analysis, 298 K
Volumetric Analysis, 77 K

IRMOF-8, AX-21 Pt/Bridged-Spillover Compound (10 mg)

Precondition 180°C (1°C/min), 10⁻⁶ Torr, 24 hr

Volumetric Analysis, 298 K
Gravimetric Analysis, 298 K
Laser Thermal Desorption Mass Spectrometry (LTDMS)

UCLA
U. Michigan INER, Taiwan
Experimental Approach

(a) High-pressure volumetric analyzer and dewar system used to measure hydrogen adsorption in framework compounds (MOF-177) at low-temperatures.

(b) High pressure gravimetric analyzer contained in glove box used to measure the hydrogen adsorption in catalytically-doped framework compounds (IRMOF-8 Pt / bridged spillover).
Experimental Approach

Complete LTDMS spectrometer system attached to an ultra-high purity gas manifold and electronic controls (a); vacuum chamber, QMS analyzer, and laser driver (b); optical bench for steering beam of laser through a variable density filter (VDF), acousto-optic tunable filter (AOTF), and collimating lenses before entering the sample chamber (c).
Technical Accomplishments

Hydrogen Physisorption in MOF-177

MOF-177†

Low-Temperature Isotherm Validated
by SwRI: 7.5 wt.% at 60 bar and 77 K


Mechanism for hydrogen spillover in catalytically doped nanostructures, involving the adsorption of gaseous hydrogen onto a catalytic site, followed by dissociation and migration of atomic hydrogen into the nanostructured substrate.

High-pressure gravimetric sorption isotherm measured for IRMOF-8 Pt/bridged-spillover compound (from INER) at room temperature.
Laser-induced thermal desorption profile measured for IRMOF-8 Pt / bridged-spillover compound (from U. Mich.), indicating multiple occurrences of stable binding sites between -10 and 25°C.
Future Work

• Continue to examine and validate spillover compounds (AX-21 and IRMOF-8 Pt / bridged-spillover) at room temperature
• Aerogel materials at 77 K
• Round-Robin testing of metal hydride materials
• International Round-Robin testing of carbon and metal hydride materials in partnership with EU’s NESSHY program
Summary

Relevance: Provide DOE with facilities and analytical methods to independently assess and validate the sorption properties of promising new materials for hydrogen storage.

Approach: Develop analytical methodologies to accurately measure hydrogen sorption in challenging forms of material chemistries and structures.

Technical Accomplishments:
Validated hydrogen saturation uptake in MOF-177 at 77 K (7.5 wt.% at 60 bar); validated hydrogen spillover uptake in catalytically-doped IRMOF-8 (Pt / bridged-spillover compound) at room temperature (2.5 wt.% at 74 bar); measured stable binding sites of hydrogen in such compounds using LTDMS.

Collaborations:
Active collaborations with UCLA, U. Mich., NREL, LBNL, Ovonic Hydrogen Systems, NESSHY (EU), and INER (Taiwan).

Future Research:
Further evaluate reproducibility of hydrogen uptake in spillover framework compounds; evaluate aerogel materials at low temperature; continue Round-Robin testing for metal hydrides; commence Round-Robin testing of carbon and metal-hydride materials via international collaboration (NESSHY).