IV.F.4 Best Practices for Characterizing Hydrogen Storage Properties of Materials

Karl J. Gross (Primary Contact)
and Russell Carrington
Hy-Energy LLC
8440 Central Ave., Suite 2B
Newark, CA 94560
Phone: (510) 793-3345; Fax: (510) 402-4705
E-mail: info@hy-energy.com

DOE Technology Development Manager:
Sunita Satyapal
Phone: (202) 586-2336; Fax: (202) 586-9811
E-mail: Sunita.Satyapal@ee.doe.gov

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Objectives

- To prepare a reference document detailing best practices and limitations in measuring hydrogen storage properties of materials.
- The document will be reviewed by experts in the field (International Energy Agency/International Partnership for the Hydrogen Economy [IEA/IPHE], etc.).
- The final document will be made available to researchers at all levels in the DOE Hydrogen Storage Program.

Technical Barriers

This project addresses the following technical barriers from the Hydrogen Storage section (3.3.4.2) of the Hydrogen, Fuel Cells and Infrastructure Technologies Program Multi-Year Research, Development and Demonstration Plan:

(A) System Weight and Volume
(C) Efficiency
(D) Durability/Operability
(E) Charging/Discharging Rates
(J) Thermal Management
(Q) Reproducibility of Performance

Technical Targets

The role of this project is to provide knowledge and experience in making critical performance measurements on materials to all projects within the Hydrogen Storage Program. The goal is to provide a resource to improve the accuracy and efficiency of performance measurements to aid the projects and ultimately the entire program to achieve or exceed the technical storage targets. In particular this project is focused on the following critical performance materials measurements and how they relate to the hydrogen system storage targets:

- **Kinetics** (Targets: System fill time for 5-kg hydrogen, minimum full-flow rate and start time to full-flow)
- **Capacity** (Targets: Gravimetric and volumetric capacity)
- **Thermodynamic Stability** (Targets: Maximum/minimum delivery pressure of H_2 from tank and impact on capacity and kinetic related targets)
- **Cycle-life Properties** (Targets: Cycle life and cycle life variation)

Accomplishments

- Compiled example measurements from the literature to illustrate key issues associated with the four tasks outlined above.
- Performed example measurements on classic hydride materials to illustrate key issues associated with kinetics and other measurements.
- Materials included in presentation for project H-27 "International standardized testing practices for hydrogen storage materials", IEA HIA Task 22 expert workshop for fundamental and applied hydrogen storage materials development, January 28-February 1, Monterey, CA.
- Within IEA, established collaboration with Dr. Nobuhiro Kuriyama and Dr. Tetsu Kiyobayashi of AIST (Advanced Industrial Science and Technology) Osaka, Japan.
- Contributions to this project from world experts have been received including written materials, examples, presentation or editorial review of draft documents.
- Delivered rough draft of kinetics task document to DOE for review on June 15, 2007.

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Introduction

The Hydrogen Storage Program goal is the development of hydrogen storage materials that meet or exceed the DOE's targets for the onboard hydrogen storage in a hydrogen-powered vehicle. The recent rapid expansion of research efforts in this field has brought the talents of a wide-range of researchers to bear in solving the grand challenge of hydrogen storage. There is a need to have common metrics and best practices for measuring the practical hydrogen storage properties of new materials that are being developed within the U.S. DOE Hydrogen Storage Program as well as at an international level. Hy-Energy is tasked with providing a clear and comprehensive resource that will provide detailed knowledge and guidelines to best practices in the measurements of these properties.

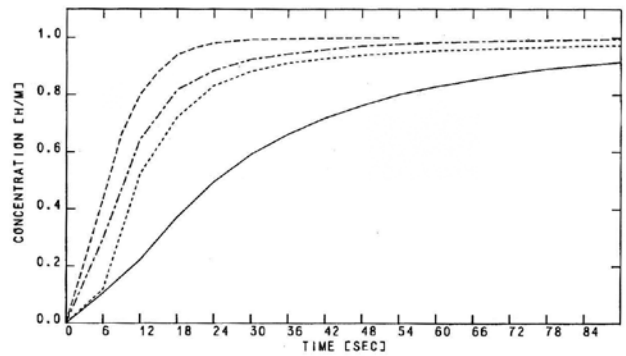
Approach

This project will be a combined approach of documenting the experience the principal investigator and other experts in the field have with these measurement, reviewing and incorporating examples from the literature, when necessary, performing experimental measurements to demonstrate important issues and finally, condensing key information into a concise reference guide. Participation from other experts in the field will be sought out for input, relevant examples and critical review at all levels.

Results

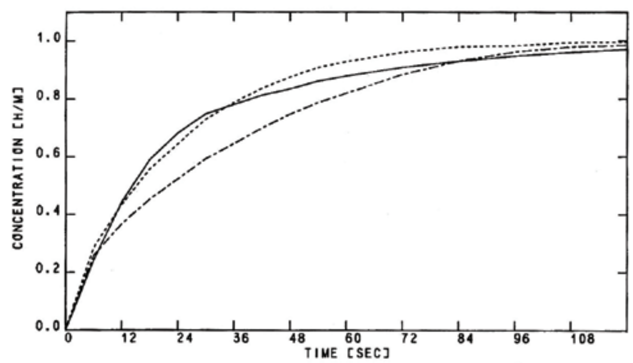
The first task in this project focused on creating a best practices document for kinetics measurements. A collaboration was established with Professor Sam Mao and Russell Carrington of the University of California, Berkeley. Dr. Gary Sandrock, Dr. Channing Ahn, and Professor Evan Gray provided valuable assistance and content for this initial document. In addition, the work has been coordinated and has received important scientific input through our contract monitor, Dr. Phil Parilla, at the National Renewable Energy Laboratory (NREL). The first rough draft of the document has been completed and forwarded to NREL and the DOE for comment. This kinetics document covers such topics as the overall purpose of kinetics measurements, some basic theory, experimental consideration depending on the purpose of the measurements, methods of measurement, and many details on both material properties and experimental factors that may strongly influence the final results and conclusions.

An example is included as one of the many important considerations to be taken into account when making kinetics measurements. Figures 1 and 2 are hydrogen absorption kinetics measurements on a $LaNi_{4.7}Al_{0.3}$ alloy made by Supper et al [1]. These show the significant difference in sorption rates obtained for the same material depending on the thickness of the powder sample that was used. This indicates the importance of the experimental setup and the effects of heat transfer. It demonstrates that one should be cautious in ascribing measured absorption rates to the intrinsic hydrogen sorption kinetic properties of any hydrogen storage material.



Absorption reaction rates for $LaNi_{4.7}Al_{0.3}$ (bed thickness, 1 mm; distance from equilibrium, 2 bar): ———, 293 K; ---------, 303 K; — • —, 313 K; – – – – –, 323 K.

FIGURE 1. Measurements taken on $LaNi_{4.7}Al_{.3}$ with a specially designed cell to investigate the effects of temperature and sample thickness on kinetics. Sample bed thickness of 1 mm [1].



Absorption reaction rates for $LaNi_{4.7}Al_{0.3}$ (bed thickness, 2 mm; distance from equilibrium, 2 bar): ———, 293 K; ---------, 313 K; — • —, 333 K.

FIGURE 2. Measurements taken on $LaNi_{4.7}Al_{.3}$ with a specially designed cell to investigate the effects of temperature and sample thickness on kinetics. Sample bed thickness of 6 mm [1].

Conclusions and Future Directions

In FY 2007 we were able to establish important collaborations and technical assistance from experts in the field. We were able to complete the rough draft document for the first task on kinetics measurements in a timely manner. This document will be reworked with comments received and will also be reviewed through our expert collaborators before final submission to the DOE. The following tasks will be the focus of our work in the future:

- Capacity
  - Hydrogen storage capacity is a key metric for practical hydrogen storage.

  - Effect of activation, kinetics and poisoning on capacity to be considered.
- Thermodynamics
  - The objective of this task is to establish methodologies for determining equilibrium thermodynamics of hydrogen storage materials.
  - We will define measurement protocols to separate true equilibrium conditions from kinetic effects.
  - We will examine details of new measurement techniques for the rapid determination of thermodynamic stabilities.
- Cycle-Life Properties
  - This task will focus on developing better definitions of how such tests should be performed.
  - We will detail what parameters may impact results and what properties are the most critical in performance evaluation (e.g. capacity fade or degradation in kinetics).

FY 2007 Publications/Presentations

1. Presentation "International standardized testing practices for hydrogen storage materials" IEA HIA Task 22 Expert Workshop for fundamental and applied hydrogen storage materials development, January 28 – February 1, Monterey, California, USA.

References

1. Supper, W., Groll, M., Mayer, U. "Reaction Kinetics in Metal Hydrides Reaction Beds with Improved Heat and Mass Transfer." *Journal of the Less-Common Metals*, 104 (1984): 279-286.