IV.E.3 Best Practices for Characterizing Hydrogen Storage Properties of Materials

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Project End Date: Project continuation and direction determined annually by DOE

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 [IEA]/ International Partnership for the Hydrogen Economy, 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 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 is to provide knowledge and experience in making critical performance measurements on materials to all projects within the Hydrogen Storage sub-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.
- Contributions to this project from world experts have been received including written materials, examples, presentation or editorial review of draft documents.
- Final reviewed documents for introduction and kinetics sections delivered to DOE May 2008.
- Document posted to DOE Web site May 2008: http://www1.eere.energy.gov/hydrogenandfuelcells/hydrogen_publications.html#h2_storage
- Presentation: project H-27 "International standardized testing practices for hydrogen storage materials" IEA Hydrogen Implementing Agreement Task 22 Expert Workshop for fundamental and applied hydrogen storage materials development, 2 – 5 March 2008, Sacacomie, Québec, Canada.
- Established new collaborations with Pacific Northwest National Laboratory, the University of

California, Berkeley, and California Institute of Technology.

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Introduction

The Hydrogen Storage sub-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 Hydrogen Storage sub-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 of 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 as well as a general introduction and a preface section (Tasks 1 and 2) of the overall "Best Practices" document. A highly productive collaboration was established with Professor Sam Mao and Russell Carrington of the University of California, Berkeley. Dr. Gary Sandrock and Dr. George Thomas, consultants to the U.S. Department of Energy, Dr. Michael Miller of Southwest Research Institute in San Antonio, TX, Dr. Anne Dailly, Dr. Eric Poirier, and Dr. Frederick Pinkerton of General Motors Research and Development Center, Dr. Ole Martin Løvvik of the Institute for Energy Technology in Kjeller, Norway, Professor Channing Ahn of the California Institute of Technology in Pasadena, CA, Professor Sam Mao of the University of California, Berkeley in Berkeley, CA, and Dr. Nobuhiro Kuriyama and Dr. Tetsu Kiyobayashi of the National Institute of Advanced Industrial Science and Technology in Osaka, Japan all provided valuable review and editing assistance, as well as, 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). Contributor's edits and comments were integrated into the final rough draft of the preface, introduction and kinetics documents and forwarded to NREL and the DOE for comment in early 2008. The preface section includes a foreword and terminology used throughout the Best Practices document. The introduction section includes an overview of hydrogen storage materials and the common techniques used to characterize the storage properties of such materials. The kinetics section 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.

Examples are included in the following of the different ranges and purposes of kinetics measurements. Figure 1 shows hydrogen desorption rates of $NaAlH_4$ with the addition of different additives all measured under the same conditions to aid research at a materials development level [1]. Figure 2 shows wide exothermic temperature excursion during hydrogen absorption in the formation of $NaAlH_4$ in a kinetics study with the aim of understanding materials performance under scaled-up application-level conditions. These examples serve to illustrate that the experimental setup and procedures will vary greatly depending on the overall purpose of the measurements [2].

Conclusions and Future Directions

In Fiscal Year 2008 we were able to establish important collaborations and technical assistance from experts in the field. We were able to complete the final

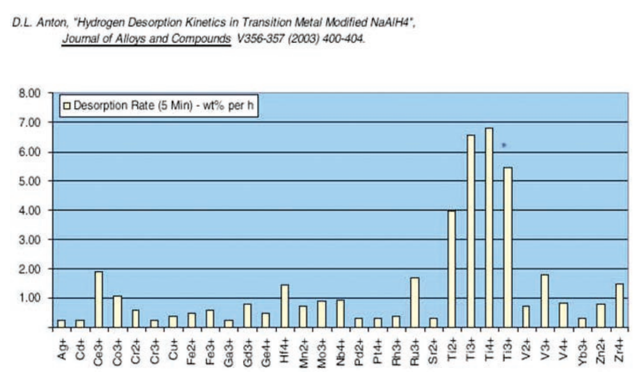

FIGURE 1. Example of a comparative analysis of the improvement in kinetics of hydrogen release from $NaAlH_4$ through the addition of different additives [1].

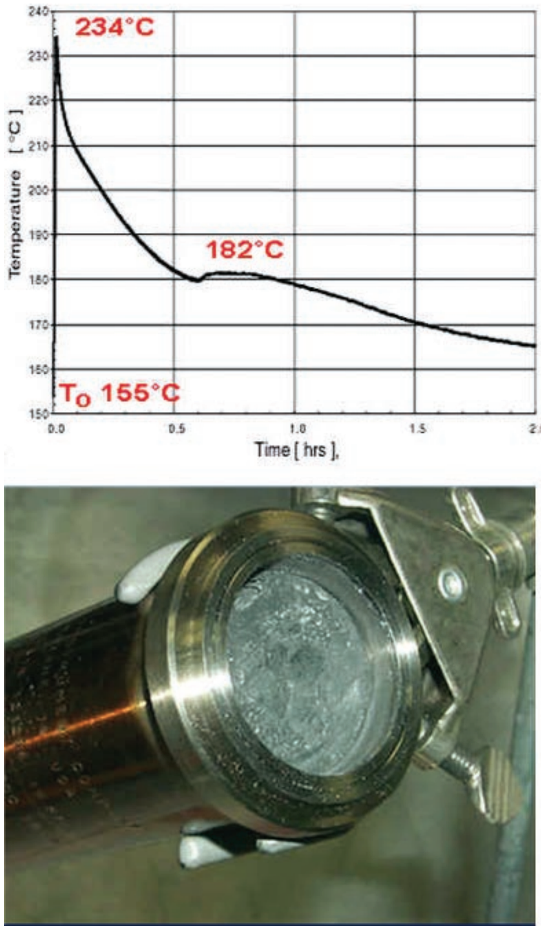

FIGURE 2. Example of a scale-up measurement of hydrogen absorption in a 100 gram sample of $NaAlH_4$ doped with Ti/Zr-alcoxide additives [2]. a) rapid exothermic temperature excursion on charging with hydrogen at ca 150 bar pressure as measured by thin thermocouple positioned in the center of the bed, b) image of material in bed after hydrogen absorption/desorption cycles showing evidence of meting and re-solidification of the $NaAlH_4$ phase (melting point 182°C).

rough draft document for the first and second tasks which included the preface, introduction and kinetics sections in a timely manner. This set of sections was submitted to NREL and the DOE for review and was posted for public comment on the DOE Web site in early May 2008. The following task will be the focus of our work in the future:

- Capacity
  - Hydrogen storage capacity is 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 and evaluate new measurement techniques for the rapid determination of thermodynamic stabilities.
- Cycle-Life Properties
  - This task will focus on 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.

FY 2008 Publications/Presentations

1. Presentation: 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, 2 – 5 March 2008, Sacacomie, Québec, Canada.

References

1. D.L. Anton et al, "Hydrogen Desorption Kinetics in Transition Metal Modified NaAlH4", Journal of Alloys and Compounds V356-357 (2003) 400-404.

2. Gary Sandrock, Presentation MH2000 October 1-6, 2000 - Noosa, Australia.