Hydrogen Storage Materials Workshop

Proceedings

August 14th and 15th, 2002
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Argonne, IL

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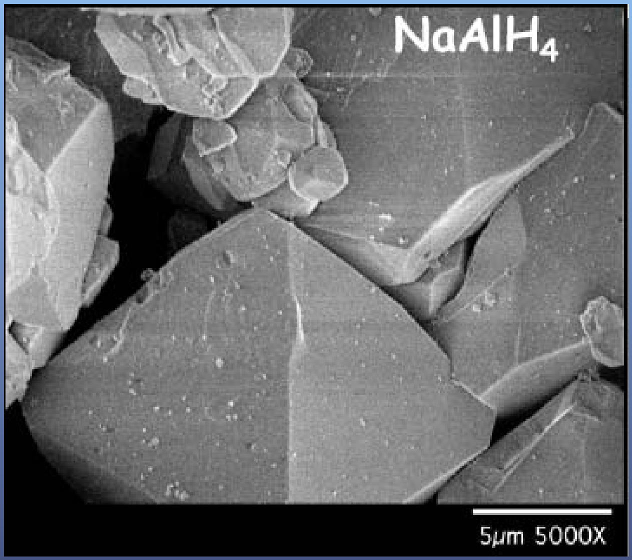
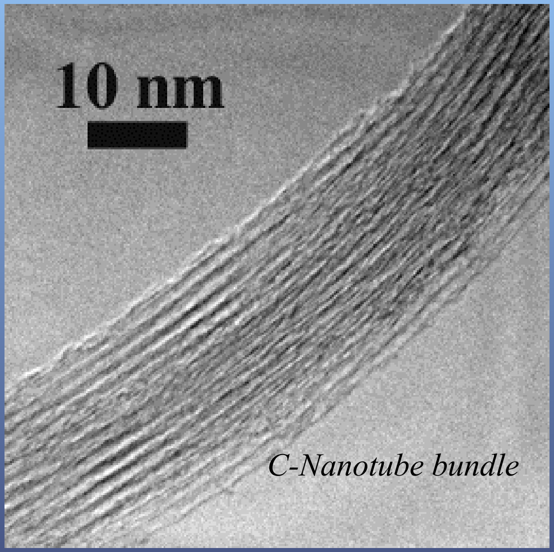
Office of Hydrogen, Fuel Cells and Infrastructure Technologies





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Acknowledgments

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Dr. JoAnn Milliken
Hydrogen Storage Team Leader
Office of Hydrogen, Fuel Cells and Infrastructure Technologies
U.S. Department of Energy

Executive Summary

President Bush launched the Hydrogen Fuel Initiative to ensure our nation's energy security and environmental viability. Pursuant to this directive and the President's National Energy Policy, the Hydrogen, Fuel Cells and Infrastructure Technologies Program of the U.S. Department of Energy (DOE) is developing a multi-year plan for research, development and demonstration of advanced technologies. This plan is being developed with input from researchers in industry, academia and the national laboratories.

BACKGROUND

Using hydrogen to fuel our economy can reduce U.S. dependence on imported petroleum, diversify energy sources and reduce pollution and greenhouse gas emissions. Fuel cells are an important enabling technology for a future hydrogen economy and have the potential to revolutionize the way we power our nation, offering cleaner, more efficient alternatives to today's technology. Safe, cost-effective and practical means of storing hydrogen is an important component for the advancement of hydrogen and fuel cell power technologies in transportation, stationary, and portable power applications. The Hydrogen Storage program element will focus primarily on the research and development of on-board vehicular storage systems that will allow for a driving range of 300 miles or more. In most situations, on-board hydrogen storage systems are more challenging than off-board due to space, weight and cost limitations.

Under the auspices of the DOE Program, a workshop was held at Argonne National Laboratory to identify on-board storage technical barriers and to explore promising research and development options to overcome them. Approximately one hundred technical experts from industry, academia and the national laboratories participated in the workshop. The specific objectives of the workshop were to:

- Review the current status of hydrogen storage technologies
- Identify the technical challenges that must be overcome to have safe, cost-effective and practical storage systems
- Identify promising technical approaches to overcome the challenges
- Prioritize the R&D needs for each of those promising approaches

The workshop included a plenary session in which the goals of the DOE Program and the perspective of an automotive original equipment manufacturer were presented. In addition, overview presentations of the status of hydrogen storage technology in each of the following topic areas were given:

- Advanced/Complex Hydrides

- Carbon-Based Materials
- Chemical Hydride Storage
- Advanced Concepts

The Advanced Concepts topic area included discussion of innovative materials and approaches for on-board storage that could be promising but have not yet received sufficient attention.

DOE and USCAR (U.S. Council for Automotive Research) representatives emphasized that the workshop was to consider hydrogen storage at a systems level, recognizing the fact that the weight and volume required to appropriately package a storage material will have an impact on fuel efficiency and on both the volumetric and gravimetric storage density. Thus, the hydrogen storage technical performance targets represent the total system, not the storage material alone.

Following the plenary sessions, the attendees participated in four working groups based on their area of expertise. Led by a facilitator, the working groups addressed each of the objectives of the workshop as they applied to their specific area. The facilitators then presented the findings of each of the working groups during the final summary session. The detailed findings of the working groups are presented in the last section of this report "Reports from Breakout Groups". The key recommendations that emerged from the four working groups are summarized below.

RECOMMENDATIONS

Advanced Complex Hydrides: Alanates have been the focus of extensive research and are considered to be the most promising of the complex hydrides studied to date for on-board hydrogen storage applications. While sodium alanate will not meet the 2010 targets, a thorough thermodynamic and kinetic understanding of the alanate system is needed to serve as the basis for systematically exploring other complex hydride systems. In a parallel effort, engineering studies must be initiated to understand the system level issues and to facilitate the design of optimized packaging and interface systems for on-board vehicular applications.

Carbon-Based Materials: Nanotubes have received considerable attention as possible hydrogen storage materials. There has been controversy regarding the storage capacity for hydrogen in these materials. Therefore, a coordinated experimental and theoretical effort is needed to characterize the materials, to understand the mechanism and extent of hydrogen absorption/adsorption and to improve the reproducibility of the measured performance. These efforts are required to obtain a realistic estimation of the potential to store and to release adequate amounts of hydrogen under practical operating conditions. It is recommended that research not be limited to single-walled nanotubes and that other carbon-based materials be examined regarding their ability to store and to release hydrogen.

Chemical Storage: Sodium borohydride is the most investigated of this class of materials. However, significant technical issues remain regarding the regeneration of the spent material. Other chemical storage systems have not been investigated to a similar extent. The workshop identified a variety of chemical storage materials that should be screened in the near-term. Detailed investigation of the process chemistry for preparing and regenerating the materials must then be investigated with a particular emphasis on the full fuel chain energy efficiency of the process. Life cycle cost analysis of the promising storage systems is also essential.

Advanced Concepts: Several classes of materials were identified that could be attractive for hydrogen storage. Very little experimental data are available to base an informed judgment regarding their potential for on-board storage applications. The workshop's recommendation is to begin a screening process to test their viability as hydrogen storage media.

Overview

The President's National Energy Policy, issued in May 2001, directs the Secretary of Energy "to develop next generation technology including hydrogen" and to "focus research and development efforts on integrating current programs regarding hydrogen technologies, fuel cells and distribution." In response, the U.S. Department of Energy (DOE) initiated a National Hydrogen Vision and Roadmap process through which it sought the guidance of stakeholders from industry, academia and the nonprofit sector on the vision and on the technical content of a robust R&D program in this crucial area. The National Hydrogen Roadmap clearly identified hydrogen storage as an issue critical to realizing a hydrogen economy. In particular, on-board hydrogen storage is recognized as a key enabling technology for automotive fuel cell systems. The National Hydrogen Roadmap can be downloaded from http://www.eere.energy.gov/hydrogenandfuelcells/. Figure 1 illustrates how policy documents and stakeholder input from the visioning and road mapping processes have been incorporated into the DOE planning activities. The Office of Hydrogen, Fuel Cells and Infrastructure Technologies Multi-Year Research, Development and Demonstration Plan is built upon these and other predecessor planning documents and is also integrated with other DOE hydrogen R&D plans.

FIGURE 1. THE DOE HYDROGEN, FUEL CELLS AND INFRASTRUCTURE TECHNOLOGIES PROGRAM PLAN BUILDS UPON SEVERAL PLANNING DOCUMENTS

The Hydrogen, Fuel Cells and Infrastructure Technologies Program plans to establish a National Hydrogen Storage Project. A workshop on hydrogen storage materials was a first step in identifying a path forward. Technical experts from industry, academia, and the national laboratories were assembled to identify the current status of hydrogen storage technologies and to solicit new ideas. Over one hundred technical experts convened at Argonne National Laboratory on August 14 and 15, 2002 for the Workshop on Hydrogen Storage Materials. The output of this workshop was also used to develop a multi-year R&D plan for the program. These proceedings describe the results of this workshop.

Workshop Organization

The workshop included a plenary session in which the goals of the DOE program and the perspective of an automotive original equipment manufacturer were presented. The plenary technical presentations addressed four key approaches for hydrogen storage: Advanced/Complex Hydrides, Carbon Materials, Chemical Hydrides and Advanced Concepts. The attendees then participated in working groups based on their area of technical expertise. The four working groups met in breakout sessions following the plenary session for more in-depth deliberation. The charge to each breakout group was to identify specific storage techniques/approaches including their performance and operating requirements and their technical, economic and environmental challenges. In addition, the groups were asked to determine a priority ranking for the storage approaches identified on the basis of the highest probability for success in the shortest time period. The workshop agenda and list of participants are provided in the appendices to this document.

Workshop Objectives

The overall goal of the workshop was to obtain technical input to shape a national activity and to be included as part of a multi-year R&D plan. Specifically, the workshop participants were asked to address the following issues:

- Review the current status of hydrogen storage materials R&D
- Identify the technical challenges that must be overcome to meet the goals of the FreedomCAR and the Hydrogen Fuel Initiative
- Identify promising technical approaches to overcome the challenges
- Prioritize the R&D needs for each of the promising technical approaches

Plenary Session - Introduction and Overview Presentations

Dr. Don Joyce, Deputy to the Director of Argonne National Laboratory, welcomed the participants and emphasized the importance that Argonne National Laboratory attaches to hydrogen and fuel cell R&D.

Following the welcoming remarks, the plenary session contained two overview presentations. The first, by Dr. JoAnn Milliken, provided an overview of the DOE Hydrogen, Fuel Cells and Infrastructure Technologies Program. The second presentation, by Mr. Brian Wicke of General Motors, contributed an automotive original equipment manufacturer's perspective on fuel cell vehicles and their hydrogen storage requirements. These two presentations were followed by several technical status reports summarizing the stage of development and performance of various approaches to hydrogen storage. The following subsection summarizes these technical presentations.

Overview Presentations

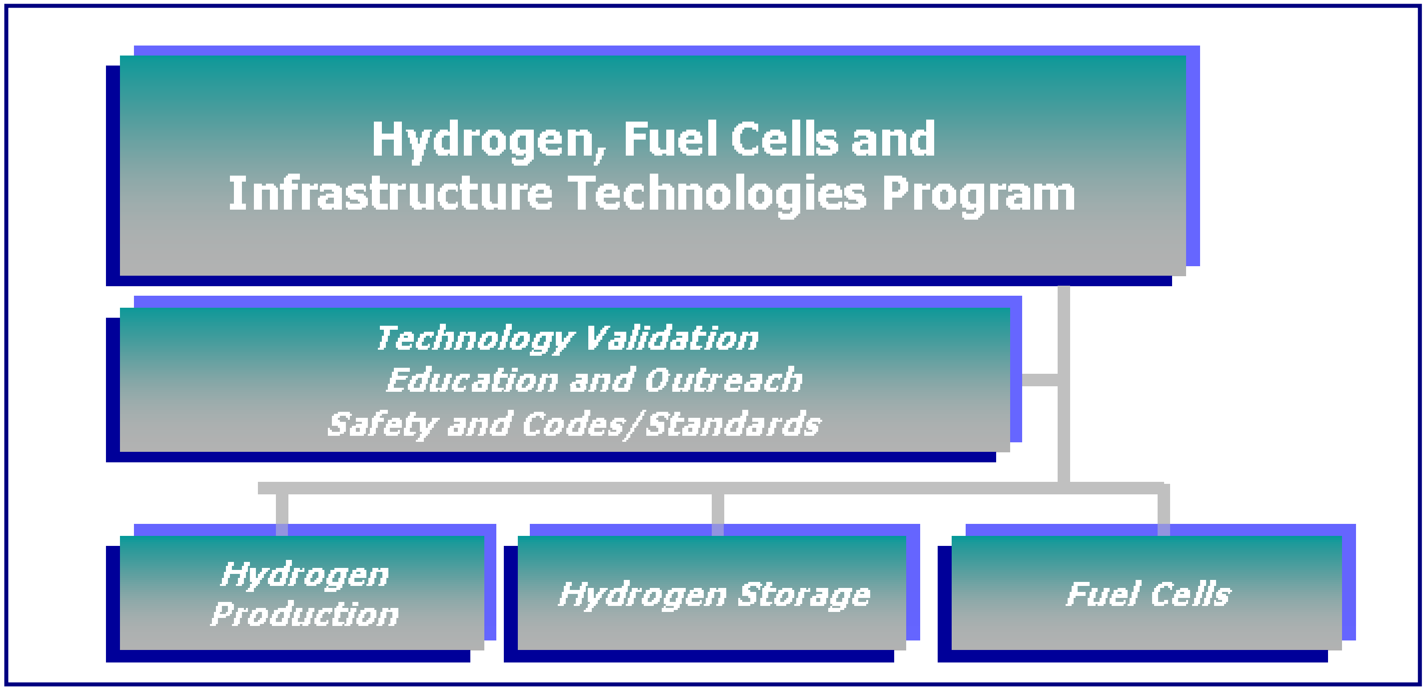
Overview of the DOE Program

Dr. Milliken provided an overview of the DOE Program activities in hydrogen and fuel cell technologies, covering five topics: (1) the Hydrogen, Fuel Cells and Infrastructure Technologies Program, (2) the Role of FreedomCAR and the Hydrogen Fuel Initiative, (3) DOE Fuel Cell & Hydrogen Activities, (4) DOE Targets/Status, and (5) Workshop Objectives. (Viewgraphs from her presentation can be found in Appendix B.) The key points made are summarized below.

The Hydrogen, Fuel Cells and Infrastructure Technologies Program

The Hydrogen, Fuel Cells and Infrastructure Technologies Program at DOE was created to implement the directive in the National Energy Policy to "focus research and development efforts on integrating current programs regarding hydrogen, fuel cells, and distribution…." Three technical teams implement R&D based on the objectives of the program; these are the Fuel Cell, Hydrogen Storage and Hydrogen Production teams. In addition, cross-cutting activities include efforts in the areas of safety/codes and standards, technology validation, and education. An organization chart of the program is shown below.

FIGURE 2. HYDROGEN, FUEL CELLS AND INFRASTRUCTURE TECHNOLOGIES PROGRAM ORGANIZATION



Role of FreedomCAR and President's Hydrogen Fuel Initiative

FreedomCAR's long-term goal is to develop advanced automotive technologies, which will require no foreign oil and emit no harmful pollutants or greenhouse gases. Specifically, the FreedomCAR partnership is focusing on developing technologies to enable mass production of affordable hydrogen-powered fuel cell vehicles. The Hydrogen Fuel Initiative is designed to accelerate development of advanced technologies for producing, delivering, storing and using hydrogen.

DOE Fuel Cell & Hydrogen Activities

The Hydrogen Vision and Roadmap Workshops were held in November 2001 and April 2002, respectively, and included the participation of a wide spectrum of stakeholders. The results of these workshops were incorporated into the National Hydrogen Roadmap, which can be downloaded from http://www.eere.energy.gov/hydrogenandfuelcells/.

The DOE program focuses its R&D activities on removing high-risk technical barriers. In Fiscal-Year 2002, fuel cell activities were funded at $47 million, while the hydrogen activities were funded at $29 million. Funding appropriations for Fiscal-Year 2003 represent a 19 percent increase in fuel cell technology R&D and a 38 percent increase for hydrogen technology. Request for Fiscal-Year 2004 funding will be $88.0 million for hydrogen technology R&D and $77.5 million for fuel cell technology R&D.

DOE Performance Targets/Status

Through the FreedomCAR partnership, DOE established R&D targets for hydrogen storage; see Table 1. This table lists the targets as of June 2003. Minor changes may be made as the targets are analyzed and updated. Updated targets will be included in the hydrogen storage solicitation to be released in July 2003. The primary focus of the program is on-board transportation applications since these are technically more challenging compared to off-board storage of hydrogen.

TABLE 1. DOE TECHNICAL TARGETS, ON-BOARD HYDROGEN STORAGE SYSTEMS

TECHNICAL TARGETS, ON-BOARD HYDROGEN STORAGE SYSTEMS [A, B, C]				
STORAGE PARAMETER	**UNITS**	**2005**	**2010**	**2015**
Usable, specific-energy from H_2 (net useful energy/max system mass)[d]	kWh/kg (kg H_2/kg)	1.5 (0.045)	2 (0.06)	3 (0.09)
Usable energy density from H_2 (net useful energy/max system volume)	kWh/L (kg H_2/L)	1.2 (0.036)	1.5 (0.045)	2.7 (0.081)
Storage system cost [e]	$/kW$_e$hr net ($/kg H_2)	6 (200)	4 (133)	2 (67)
Fuel cost [f]	$ per gallon gasoline equivalent at pump	3	1.5	1.5
Operating ambient temperature[g]	°C	-20/50 (sun)	-30/50 (sun)	-40/60 (sun)
Cycle life (1/4 tank to full)[h]	Cycles	500	1000	1500
Cycle life variation[i]	% of mean (min) @ % confidence	N/A	90/90	99/90
Minimum and Maximum delivery temperature of H2 from tank	°C	-20/100	-30/100	-40/100

TECHNICAL TARGETS, ON-BOARD HYDROGEN STORAGE SYSTEMS [A, B, C]

STORAGE PARAMETER	UNITS	2005	2010	2015
Minimum full flow	(g/sec)/kW	0.02	0.02 FC 0.027 ICE	0.02 FC 0.033 ICE
Minimum delivery pressure of H2 from tank FC=fuel cell, I=ICE	Atm (abs)	2.5 FC 10 ICE	2.5 FC 35 ICE	2 FC 35 ICE
Transient response 10%-90% and 90%-0% [j]	Sec	0.5	0.5	0.5
Start time to full flow at 20°C	Sec	4	0.5	0.5
Start time to full flow at minimum ambient	Sec	8	4	2
Refueling rate[k]	kg H_2/min	0.5	1.5	2
Loss of useable hydrogen[l]	(g/hr)/kg H_2 stored	1	0.1	0.05
Permeation and leakage[m]	SCCM/hr	Federal enclosed-area safety-standard		
Toxicity		Meets or exceeds applicable standards		
Safety		Meets or exceeds applicable standards		
Purity[n]		98%		

a. Based on the lower heating value of hydrogen and a minimum of 300-mile vehicle range; targets are for the complete system, including tank, material, valves, regulators, piping, mounting brackets, insulation, added cooling capacity and/or other balance-of-plant components.
b. Unless otherwise indicated, all targets are for both internal combustion engine and for fuel cell use, based on the low likelihood of power-plant specific fuel being commercially viable.
c. Systems must be energy efficient - for reversible systems, greater than 90% energy efficient; for systems generated off-board, greater than 70% life-cycle efficiency. Useful constants: 0.2778 kWh/MJ, 33.3 kWh/gal gasoline equivalent.
d. Generally the ‘full’ mass (including hydrogen) is used, for systems that gain weight, the highest mass during discharge is used.
e. 2003 U.S. $; total cost includes any component replacement if needed over 15 years or 150,000 mile life.
f. 2001 U.S. $; includes off-board costs such as liquefaction, compression, regeneration, etc; 2015 target based on H_2 production cost of $1.50/gasoline gallon equivalent untaxed.
g. Stated ambient temperature plus full solar load
h. Equivalent to 100,000; 200,000; and 300,000 miles respectively (current gasoline tank spec).
i. All targets must be achieved at end of life
j. At operating temperature.
k. 2015 target is equivalent to 3-5 minutes refueling time.
l. Total hydrogen lost from the storage system, including leaked or vented hydrogen; relates to loss of range.
m. Total hydrogen lost into the environment as H_2; relates to hydrogen accumulation in enclosed spaces. Storage system must comply with CSA/NGV2 standards for vehicular

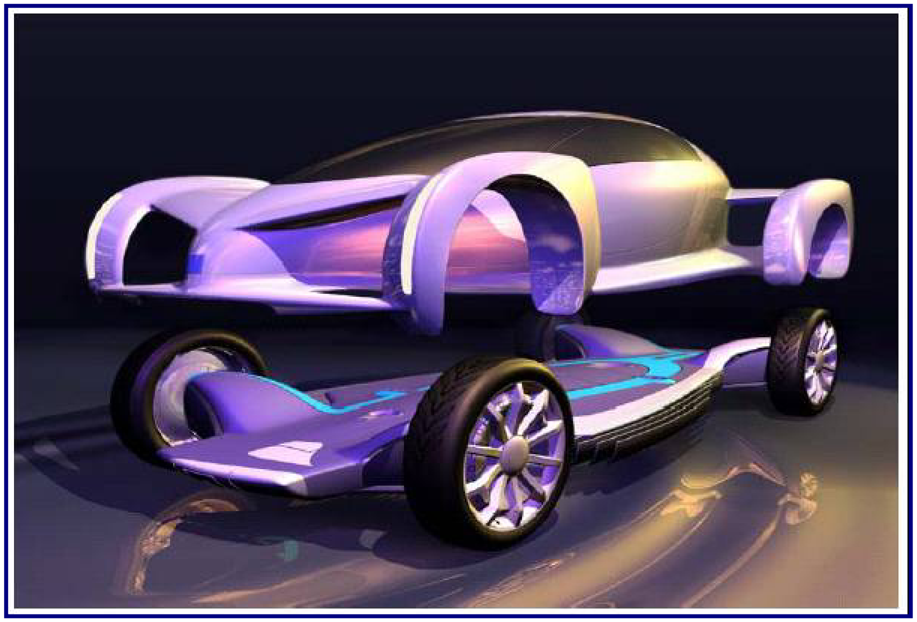
Technical Targets, On-Board Hydrogen Storage Systems [A, B, C]				
Storage Parameter	**Units**	**2005**	**2010**	**2015**
tanks. This includes any coating or enclosure that incorporates the envelope of the storage system. n. For fuel cell systems: less than 10 ppb sulfur, 1 ppm carbon monoxide, 1 ppm carbon dioxide, 1ppm ammonia, 100 ppm hydrocarbons; and oxygen, nitrogen and argon can not exceed 2% on a dry basis.				

Perspective from an Automotive Original Equipment Manufacturer (OEM) – Automotive Fuel Cell Systems

Presented by Mr. Brian Wicke, General Motors R&D and Planning

Mr. Wicke of General Motors (GM) presented an overview of automotive fuel cell systems and hydrogen storage requirements from the perspective of an automotive OEM. Mr. Wicke emphasized the need to consider the challenges facing the automobile industry and was frank about the industry's primary goals – satisfying customers and making a profit.

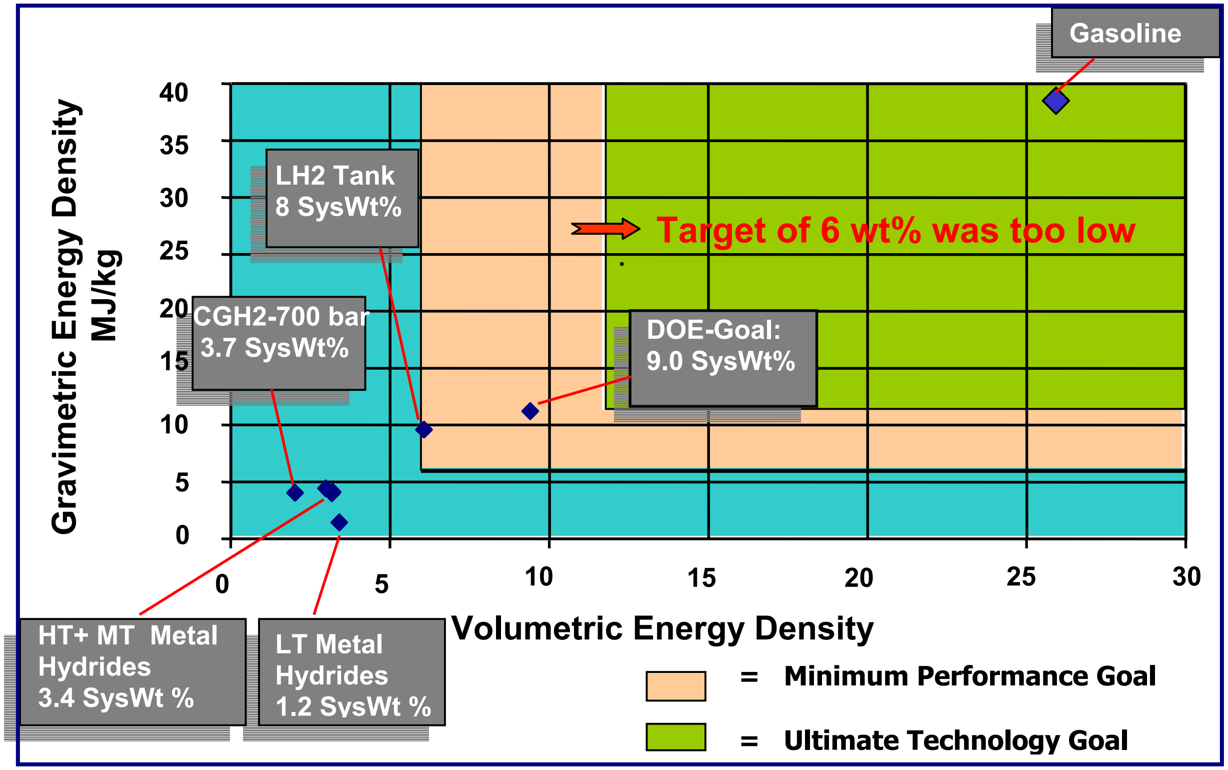
GM's fuel cell vehicle goal is to be the first company to profitably sell one million fuel cell vehicles. However, to succeed at GM, fuel cell vehicles must be affordable, suitable for a broad range of customer usage and vehicle styles and have safety performance comparable to today's vehicles. In addition, they must exhibit uncompromised performance and reliability. These fuel cell vehicles will be developed with evolutionary and revolutionary vehicle designs.



The GM Autonomy is an example of revolutionary vehicle design. The vehicle disassembles and its tops or skins may be interchanged (chassis adaptation), e.g., from a sports car to a family sedan. It currently caters to a niche market, but keeps the public excited about revolutionary vehicle concepts.

GM's focus is on PEM fuel cell technology. GM takes a systems approach to its analysis of fuel cells, considering all unique requirements for storage, including the fuel delivery system support hardware. With respect to hydrogen storage requirements, it is the energy density that is critical. **Error! Reference source not found.**, which has been modified slightly from that presented by GM, illustrates the on-board hydrogen storage requirements, based on GM's perspective, and relative to the previous DOE target of 6 weight percent.

FIGURE 3. HYDROGEN STORAGE REQUIREMENTS AND STATUS FROM AN OEM PERSPECTIVE



The minimum acceptable performance for one or two light-duty vehicle platforms is 6 MJ/L and 6 MJ/L. To meet the needs of a broad range of vehicle platforms, the goal is about 12 MJ/L and 12 MJ/L. (These targets were subsequently modified by the FreedomCAR Hydrogen Storage Technical Team, which includes representatives from DaimlerChrysler, Ford, GM, and DOE.) Other requirements noted by GM at the workshop and addressed in the FreedomCAR targets include the following:

- Refueling time less than 5 minutes
- Durability (total miles maintaining 80% capacity) of 150,000 miles
- Hydrogen release rate of 0.025 g/s * kW_{max} (e.g. 1.25 g/s @ 50 kW)
- Hydrogen release temperature less than 80ºC
- Energy penalty for hydrogen release less than 5 percent
- On-board heat dissipation during refueling equivalent to 0 kW

Technical Presentations

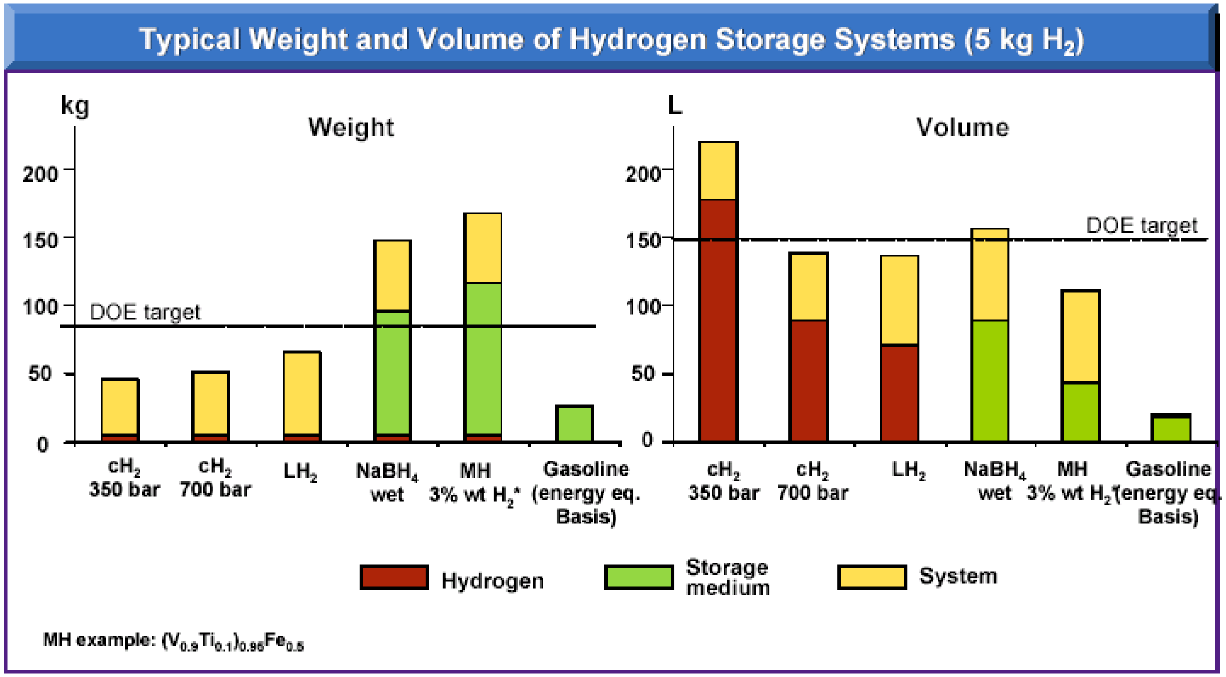
Advanced Hydrogen Storage: A System's Perspective and Some Thoughts on Fundamentals

Presented by Dr. W. Peter Teagan and Dr. Mehmet Rona, TIAX LLC

TIAX stressed the importance of systems-level analysis and fundamental materials improvement for hydrogen storage, stating that, "Fundamental material properties and their impact on system design are both critical." TIAX is involved in performing technical due diligence activities for advanced hydrogen storage systems, investigating on-board storage options and modeling of advanced materials. TIAX contributed the following insights.

- Advanced hydrogen storage requires a complex thermal and flow management system that has an impact on the system weight, volume and cost. For example, with current metal hydride technology, the material itself comprises less than half of the system volume, while the remaining volume consists of packaging, insulation, etc.
- Due to system-level limitations, current hydrogen storage systems meet some of the requirements but none meet all of the requirements. Figure 4 is taken from the TIAX presentation; it illustrates how various current approaches for hydrogen storage compare to the 2010 DOE targets of 6 wt% (2 kWh/kg) and 1.1 kWh/L. As shown in the figure, current storage materials do not appear to offer clear advantages over compressed or liquid hydrogen storage. (It must be noted that the 2015 DOE performance targets are even more aggressive than that shown in Figure 4.)

FIGURE 4. TIAX CONTRIBUTED EXAMPLE OF CURRENT STATUS OF HYDROGEN STORAGE APPROACHES



The following bullet points summarize the rest of the TIAX presentation:

- Current high-density storage material systems also appear to require higher energy either to store or to liberate the hydrogen. This is an important trade-off that must be considered. Thermodynamically stabilized systems have higher capacity with a deeper storage well and require work input to release hydrogen in the free gas form. Examples include metal hydrides, carbon and liquefied hydrogen. Kinetically stabilized systems have higher capacity with higher stored chemical potential; examples include compressed hydrogen and chemical hydrides.
- Improving storage capacity will require improvements in material performance that will also enable a better system design. Better storage materials will have lower weight, smaller volume, lower cost and better stability. In addition, better materials will require lower energy use for hydrogen liberation, easy and energy-efficient “recharging” or recycling and low temperature and low pressure operation.
- The fundamental characteristics of storage materials can be estimated using a combination of first principles-based models. As a preliminary step, TIAX modeled a hydrogen molecule between two graphitic planes and found that, although there is some

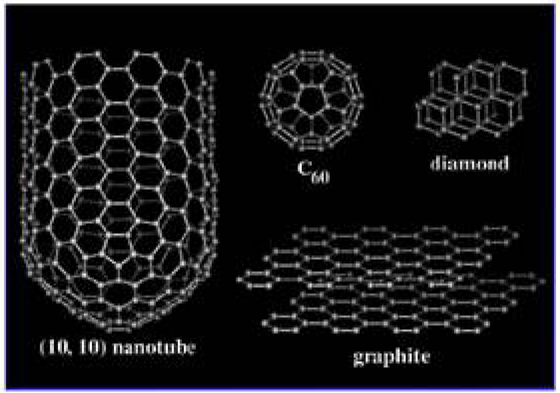
electron charge transfer from the graphite to the H_2 molecule, there is no localization potential pinning the hydrogen molecule. They saw that any charge transfer in or out of the graphitic planes results in a decrease of the in-plane lattice vector's magnitude. From their previous work on doped graphite, they expected the (inter-plane) c-spacing to diminish with electron charge transfer from graphite to the hydrogen molecule. This preliminary step suggests that it may be more fruitful to look at interactions of hydrogen with irregular clusters of doped-carbon.

Carbon Materials—Hydrogen Storage in Carbon Nanotubes

Presented by Professor John E. Fischer, University of Pennsylvania

Hydrogen storage in carbon nanotubes is an active and controversial area of research. The theory to explain hydrogen absorption/adsorption in carbon nanotubes has been evolving. Much of the controversy arises from the fact that materials are often poorly characterized. In addition, critical experiments to determine the nature of hydrogen bonding to the nanotubes have not yet been accomplished under well-defined, reproducible conditions.

Dr. Fischer provided a broad overview of the current status of research on hydrogen absorption/desorption on carbon nanotubes. As shown in the figure on the right, nanotubes are one example of the many allotropes of carbon. Nanotubes can be single-walled or be formed of multiple concentric tubes of carbon or multi-walled. Single-walled nanotubes of carbon are currently favored because it has been difficult to demonstrate storage of hydrogen in between the concentric layers of carbon in the tube. Thus these extra layers of carbon are rendered inefficient in terms of their storage ability and contribute to extra weight to the system. Single walled nanotubes can be isolated as individual strands or be present in bundles; a picture of a bundle is shown below to the right.



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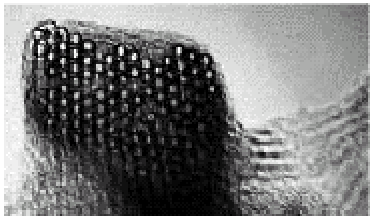
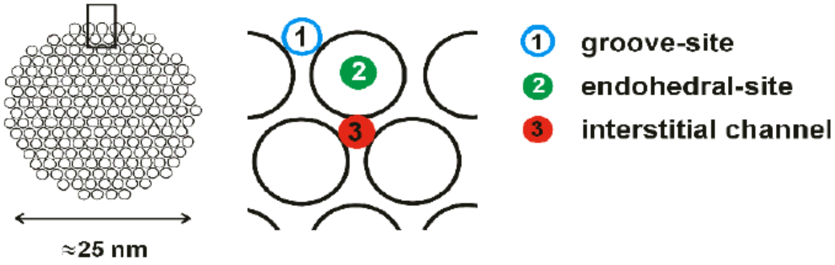
"rope"	1.3



TEM of a single walled nanotube, typical tube diameter is 1.4 nm, taken from http://cnst.rice.edu

Dr. Fischer outlined critical experiments that need to be conducted to identify the nature of the bonding of hydrogen and carbon in the nanotubes. Once that is established, it would be possible to estimate the maximum amount of hydrogen that can be stored in these types of materials. Some of the issues raised in Dr. Fischer's presentation were:

- Theoretical calculations indicate that physisorption of hydrogen on the exterior surfaces of carbon nanotubes is insufficient to achieve the storage density required at practical operating temperatures and pressures. The interactions between hydrogen and the carbon surface needs to be stronger than that of van der Waals forces.
- Practical storage densities can only be achieved if hydrogen is able to occupy the interior and exterior volumes of the nanotubes. In addition, the hydrogen/surface bond strength must be sufficient in order to fill and to release hydrogen at operating temperatures and pressures compatible with PEM fuel cell systems.
- Experimental efforts must therefore be focused on understanding the nature of sites of hydrogen bonding on carbon nanotube surfaces.
    - There are three commonly accepted bonding sites on doped nanotubes: grooved, endohedral and interstitial channel.
    - There are three methods for filling nanotubes: capillarity (with metals and oxides in large diameter tubes), immersion in molten salts (use in multi-walled carbon nanotubes shown) and gas phase (shown with fullerene peapods).



- Binding energies and relative abundances of the three types of binding sites have shown that a single, isolated single-walled nanotube can achieve over 5.5 weight percent excess gravimetric storage of hydrogen at 133 K and 10 MPa. The challenge is to demonstrate that bulk materials operating at practical temperatures and pressures can achieve acceptable storage densities.
- Another challenge for preparation and isolation of these materials is cutting the domed ends of the tubes so that the interior volumes of the tubes can be accessed for storage. Techniques that prove that the ends have been cut and storage is occurring inside the tubes need to be proven.
- Experiments have shown that lithium ion (Li^{+1}) could fill the interior of single-walled nanotubes and has fast diffusion properties at 300K. It has not been proven that diatomic hydrogen (which has a larger kinetic diameter than Li^{+1}) could fill the interior of the tubes.

- In the literature[1], a MD simulation at 300K has shown a weak covalent bond between diatomic hydrogen and carbon inside distorted single-walled carbon tubes. However, at high loadings, most hydrogen molecules will not "feel" or interact with the tube walls.
- There are a number of experimental hydrogen specific probes that should be used to complement theoretical approaches to studying and characterizing these materials:
  - Temperature programmed desorption (TPD) with mass spectrometry
  - In situ proton NMR
  - In situ spectroscopy such as Raman and IR (characterizes diatomic hydrogen molecular vibrations)
  - Elastic neutron scattering with diatomic deuterium substitution (locates molecules in the structure)
  - Incoherent inelastic neutron scattering (identifies binding sites and molecular dynamics and diffusion)
  - Electrochemical charge and discharge

Advanced/Complex Hydrides

Presented by Dr. George Thomas, Sandia National Laboratories – Livermore

The challenge for complex hydrides is to identify those materials that not only meet the stringent storage density requirements but also demonstrate reversibility so that the hydrogen can be stored (uptake) and released under practical operating temperatures and pressures. A large number of metal hydrides have been investigated as hydrogen storage media. The website Hydpark.@.sandia.gov is a database that lists over 2,000 elements, alloys and compounds that form hydrides. They generally fall into two categories: transition metals and light metals. Transition metals (Group IIIB, IVB, and VB) form metallic bond hydrides with moderate temperature/pressure properties. The equilibrium properties can be adjusted over a wide range by alloying. These materials have good kinetics for interstitial hydrogen but low capacity (H/M ratio). Light metals (Group IA, IIA) form ionic or covalent bond hydrides. They have high energy bonds (high temperature, low pressure) and have high capacity.

Dr. Thomas provided an overview of the current status of research on reversible hydride materials. The issues raised in his presentation include:

- Complex hydrides consist of a hydrogen-metal complex with an additional bonding element. Hydrogen complexes include alanates [$(AlH_4)^-$] in which the hydrogen is covalently bonded to aluminum and borohydrides [$(BH_4)^-$] in which the hydrogen is bonded to boron. These anionic entities are complexed with Group VIII elements. A wide variety of complex hydrides are available but only the alanates have been studied in detail to date. Figure 5 is a table taken from Dr. Thomas' presentation showing example hydrogen content in example alanates.

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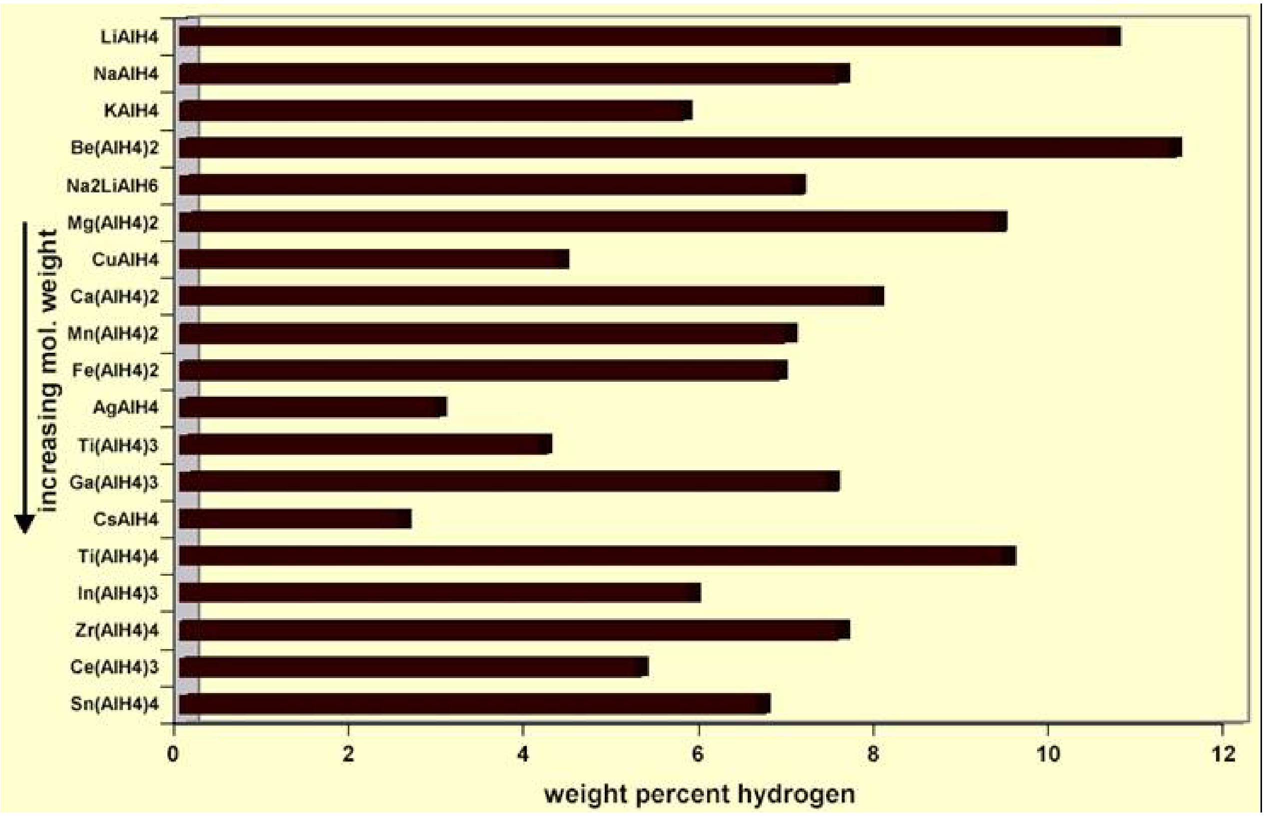
[1] JACS **2001**, *123*, 5845

FIGURE 5. TOTAL HYDROGEN CONTENT OF EXAMPLE ALANATES



- Complex hydrides must exhibit the following characteristics: reversibility (usually requiring a catalyst or dopant), favorable thermodynamics (at acceptable pressures and temperatures), fast kinetics, cyclic stability, viable synthesis methods, compatibility and safety in use.
- Understanding sodium alanate [$NaAlH_4$] "charge/discharge" mechanisms will help in developing higher capacity hydrides. There is renewed interest in sodium alanate since reversibility has been demonstrated with a titanium catalyst. Major R&D efforts are underway in the United States, Germany, Canada and Japan.
- Three factors affect the performance of catalysts/dopants: the specific catalyst/dopant, the method of introduction (e.g. wet or dry preparation and precursor) and the amount of catalyst or dopant. There is also a trade-off to consider because higher levels of catalyst promote faster kinetics but also reduce hydrogen storage capacity.
- Another complex hydride receiving significant attention is based on borohydrides [$(BH_4)^-$]. Millennium Cell is developing a

proprietary system that demonstrates 4 to 10 weight percent capacity. Reversibility is an issue; the impact of possible off-board regeneration of the spent material needs to be understood.

- The capacity of most hydrides to date appears limited to 5 weight percent. Accordingly, modifications or new complexes, as well as improvements from system engineering, are needed.

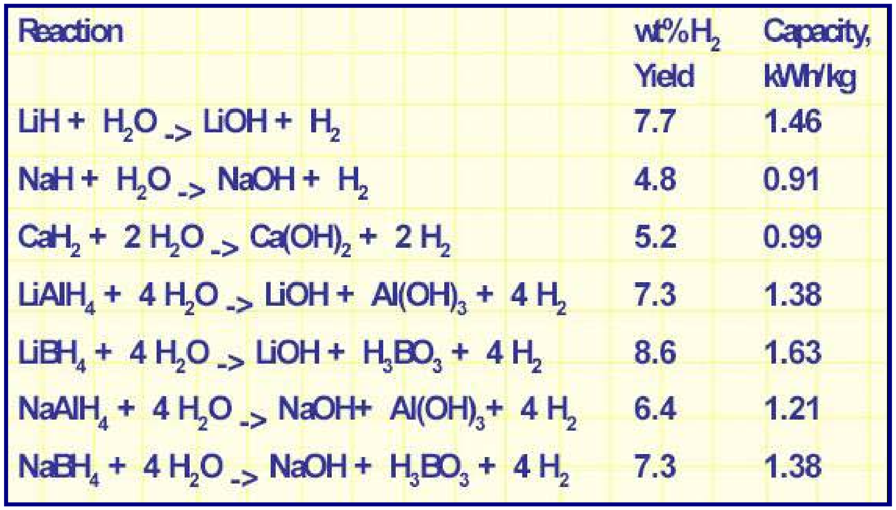
Chemical Hydride Storage

Presented by Dr. Ali T-Raissi, Florida Solar Energy Center

Dr. T-Raissi presented an overview of chemical hydride storage systems. Chemical hydrides provide secondary storage methods, in which the storage medium is expended upon use (and regenerated either on or off the vehicle). An example system is a chemical reaction of a reactant containing hydrogen in the "minus-1" oxidation state (e.g. a hydride) with a co-reactant containing hydrogen in the "plus-1" oxidation state (e.g. H_2O, NH_3 and H_2S). To be feasible, a chemical storage system must be thermodynamically spontaneous (ΔG less than zero) and kinetically tractable. It should use readily available reactants and produce hydrogen of purity compatible with PEM fuel cells. Finally, the system should be able to operate in a load following mode (applicable to all hydrogen storage methods), i.e., provide hydrogen to the fuel cell as needed.

Hydrogen generation by hydrolysis is impressive, in terms of capacity, compared to batteries. The reactions are spontaneous, highly exothermic and irreversible. Figure 6 contains examples of hydrolysis reactions of hydrides yielding hydrogen.

FIGURE 6. EXAMPLES OF HYDROGEN GENERATION BY HYDROLYSIS



Reaction	wt%H_2 Yield	Capacity, kWh/kg
$LiH + H_2O \rightarrow LiOH + H_2$	7.7	1.46
$NaH + H_2O \rightarrow NaOH + H_2$	4.8	0.91
$CaH_2 + 2 H_2O \rightarrow Ca(OH)_2 + 2 H_2$	5.2	0.99
$LiAlH_4 + 4 H_2O \rightarrow LiOH + Al(OH)_3 + 4 H_2$	7.3	1.38
$LiBH_4 + 4 H_2O \rightarrow LiOH + H_3BO_3 + 4 H_2$	8.6	1.63
$NaAlH_4 + 4 H_2O \rightarrow NaOH + Al(OH)_3 + 4 H_2$	6.4	1.21
$NaBH_4 + 4 H_2O \rightarrow NaOH + H_3BO_3 + 4 H_2$	7.3	1.38

The by-products require recycling/regeneration or disposal to complete the cycle. The issues are the control of the rate of reaction and the choice of the lightest possible system. Generally, the rate of hydrolysis is inhibited by high pH and the insolubility of the products.

Another method of hydrogen generation from hydrides is by pyrolysis or thermal decomposition. One application combines primary hydrides with NH_4Cl or a similar halide salt:

$$NH_4F + LiBH_4 = LiF + BN + 4\,H_2$$

This particular system yields a material hydrogen storage density of 13.6 weight percent. $[NH_4X + MH]$ formulations render the compound storable and insensitive to air and moisture. However, these pyrolytic reactions are highly exothermic and can not be stopped once initiated. In general, it is important to find a system that is stable at room temperature range conditions and could be initiated using waste heat from a PEM fuel cell system.

The inorganic hydrides (e.g., lithium alanate, $LiAlH_4$) and common reactants (e.g., water or ammonia) may have the best chance to meet cost goals. Successful implementation of chemical hydrides for fuel cell vehicle applications requires a substantial reduction in production costs, development of new synthesis routes for their preparation and development of feasible regeneration/recycling methods for the spent material.

Advanced Concepts for Hydrogen Storage

Presented by Dr. John Petrovic, Los Alamos National Laboratory

Dr. Petrovic's literature review revealed seven new material areas that have potential for improved hydrogen storage. These materials/concepts are described below. Further details are provided in the summary of the Advanced Concepts breakout group. It should be noted that disagreement exists concerning the potential for some of the proposed approaches to meet the DOE targets.

- Advanced hydride materials are the most promising in terms of volumetric and gravimetric hydrogen storage densities, e.g., $LiBH_4$ and $[Al(BH_4)_3]$. Lithium tetrahydroboride ($LiBH_4$) is a salt-like, hydroscopic crystalline material. The change of free energy of reaction becomes negative at 450°C. Below these temperatures, the reaction is endothermic with a total release potential of 13.8 weight percent hydrogen. However a low temperature H_2 release has been observed (2.3 wt. % hydrogen released at 118°C). One issue to resolve is whether this lower temperature release could be made reversible.
- Hydride "alcoholysis," (chemical reaction of hydrides with alcohols) provides for controlled production of hydrogen at or below room temperature. The highest capacity (in terms of weight percent including the alcohol methanol) is with $LiBH_4$.
- Boron nitride nanotubes. These materials can be synthesized through a chemical vapor deposition process by pyrolyzing a B-N-

O precursor at 1730°C in a N_2/NH_3 atmosphere. Hydrogen uptake measurements yielded 1.8 weight percent hydrogen at 10MPa and 20°C.

- Zeolites/Alumina silicates: Zeolite ZSM-5 is commercially available and absorbs 0.7 weight percent hydrogen by physisorption at 77K and 1 bar pressure. Modeling suggests that Zeolite A can store at least 2 weight percent hydrogen if all cage sites are filled (at 10 MPa at T* of 8.0). Hydrogen storage in large pore zeolites such as UTD-1 has not been examined.
- Ordered carbon molecular sieves obtained through template synthesis techniques should be explored. An example is mesoporous silica molecular sieve MCM-48 impregnated with sucrose and then pyrolyzed.
- Silica dioxide aerogels and xerogels have not been explored as hydrogen storage materials.
- Other mesoporous materials include: metal-organic MOF-5 and organosilica material (e.g. benzene-silica hybrid material)
- Nano-scale metal and ceramic powders are commercially available at ten to one-hundred nanometer diameters. Nano-scale metal powders include: Au, Ag, Ni, Ti, Mo, Pt and W; while nano-scale ceramic powders include Al_2O_3, ZrO_2, CeO_2, CuO, MgO, SiO_2 and TiO_2. The literature reports synthesis of single benzene-1,4-dithiolate molecule between atomically-sharp gold electrodes. This approach could be explored for hydrogen storage with nano-scale metal powders.
- Hydrogen may also be produced from iron hydrolysis and by the optimized milling of iron powders, with hydrogen storage in modified iron oxides. For this process, 3.3 weight percent hydrogen has been shown, including both Fe and H_2O from the reaction, $3Fe + 4H_2O = Fe_3O_4 + 4H_2$.

Reports from Breakout Groups

A facilitator directed the discussions of each of the four breakout groups listed below, and a scribe assisted by keeping notes of the group's deliberations. The full list of participants is provided in Appendix C to this report.

Advanced/Complex Hydrides Breakout Group
Facilitator: Gary Sandrock (SunaTech)
Scribe: Carolyn Elam (NREL)

Carbon Materials Breakout Group
Facilitator: Jim Ohi (NREL)
Scribe: Matthew Rowles (SENTECH)

Chemical Storage Breakout Group
Facilitator: Ken Stroh (LANL)

Scribe: Cathy Gregoire Padró (NREL)

Advanced Concepts Breakout Group
Facilitator: John Petrovic (LANL)
Scribe: Sherry Marin (SENTECH)

The four breakout groups addressed research and development issues regarding potential hydrogen storage systems that are in various stages of development. For example, some systems using chemical hydrides are in a prototype stage of development, while other systems such as those discussed in the Advanced Concepts Group are in the exploratory phase. Thus, the levels of discussions in the four breakout groups were significantly different. Although an effort has been made to present the findings in a standardized format, considerable differences do exist in what was reported from the four breakout groups.

Advanced/Complex Hydrides

Objective

The objective of this session was to review the current state of complex metal hydrides for hydrogen storage and to identify opportunities for reversible hydrides. In particular, the goal was a material hydrogen storage capacity of at least 8 weight percent with acceptable charging and discharging kinetics. It was important that the material could utilize waste heat from a PEM fuel cell to discharge the hydrogen. The material must demonstrate 8-weight percent storage capacity if the 2010 system level goal of 6-weight percent is to be achieved. The following performance guidelines were suggested. Please note that these guidelines were suggested by the breakout group and do not necessarily agree with the DOE performance targets (see Table 1).

- At least 8 percent reversible hydrogen storage capacity for the material is needed with an interim goal of 6 percent hydrogen storage capacity by 2008.
- The materials need to have sufficient charging/discharging kinetics: less than 5-minute tank fill and 0.025 g/sec per kW discharge.
- The hydrogen should discharge at nominally 1 atm and at less than 80°C (current standard); at less than 110°C for higher fuel cell operating temperature.
- Retention of 80 percent capacity over 500 charging/discharging cycles is required.
- The safety of the hydride tank needs to be comparable to that of a passenger car gasoline tank.

Approach

To date, the storage performance of sodium alanate is closest to meeting the targets. However, an 8-weight percent reversible storage capacity would not be possible with sodium alanate. Therefore, other complex hydrides, such as lithium complexes, will need to be addressed to meet the necessary targets. The group agreed that a rigorous fundamental effort using model materials is required in order to develop a robust understanding of how the materials can be manipulated and fine-tuned. Sodium alanate was suggested as a model system. The impact of poisons and impurities and the system safety issues related to their use on-board vehicles must also be understood. The knowledge of the model systems could then be used to design other complex hydride systems. The group recommended that this fundamental effort be fifty percent of the focus for metal hydride work over the next 2 to 3 years.

In parallel, screening of other materials for their potential to meet the targets should continue. The reversibility of lithium borohydride and other borohydrides should be studied. Other transition metal complexes should also be screened. Aluminum hydride was identified as a possibility for being reversible at low temperature and pressure typical of a PEM-based system. The group recommended that a standing research group be available to screen and to test materials and high-risk ideas.

Once a fundamental understanding is established, ninety percent of the metal hydride effort should be focused on a few materials that have the highest probability of achieving greater than 6-weight percent hydrogen storage. However, screening of other materials should also continue.

By 2008, a material should be available that can provide 6 weight percent reversible hydrogen storage under the charging/discharging parameters discussed above. The effort should then focus on engineering 1 to 2 materials that have a potential for 8 weight percent or greater hydrogen storage capacity.

In addition to materials development, the group pointed out that reactor or bed engineering is needed. The storage system needs to be, at least, 90 percent efficient overall. Lower efficiencies will require greater storage capacity for the material development effort. Uniform heat and mass transfer is a concern. Safety testing must also be a priority.

Finally, the group recommended that a mechanism be in place to quickly pursue novel ideas and opportunities, e.g., small, one-year projects. Funding mechanisms that require long time lags for proposals and awards and cost-share, etc., are barriers to pursuing many of the ideas that could lead to a breakthrough.

In addition to research and development directly related to hydride materials, other research areas suggested included:

- Non-thermal discharge mechanisms – Although heat has been the primary mechanism for discharging hydrogen, other discharging means should also be investigated, including mechanical, chemical and electrical means. These could enable the use of the higher temperature hydrides with higher storage capacities.
- Scalability – Cost-sensitivity analyses need to be performed to understand the major cost drivers for materials synthesis and for scaling up hydride production. The materials may need to be doped; it is unclear how this will be done cost effectively or at large scale.
- First principle calculations should be employed for evaluating and designing materials.
- Understanding the decomposition or hydrogen discharge pathways for the materials and how this impacts safety is important.
- Nanocomposites are showing promise in a number of areas and should be addressed.
- Hybridized Systems – It is possible that no single material will be able to meet the targets. Hybridized systems should be investigated as a means of overcoming the barriers in order to achieve the needed properties.
- A combinatorial screening approach to material synthesis should be used.

R&D Priorities

Material Development:

- Fundamental studies on $NaAlH_4$. The mechanisms by which these materials are made reversible and optimized for storage capacity and how to apply this understanding to other compounds should be better understood.
- Achieve 8 weight percent (by 2010) for the storage material. A material with 8-weight percent (or greater) hydrogen storage capacity that also meets the charging/discharging targets should be developed. The material should be suitable for on-board use. An intermediate target should be materials that achieve 6 weight percent and are suitable for on-board use by 2008.

Storage System:

- Engineering studies on reactor engineering and system integration should be conducted on a laboratory-scale system with a capacity of 40-gram hydrogen and over time progress to a full-size vehicle prototype with a 5-kg hydrogen capacity.
- Preliminary system cost analysis should be completed using the 5-kg hydrogen bed as the basis. Large-scale material production should be modeled and the economics assessed.
- Full-size vehicle reactor bed meeting the 6 weight percent system criteria needs to be certified.
- An independent safety consultant and/or laboratory should be identified during the early prototype work. The consultant/laboratory should provide an independent evaluation of the safety issues and support certification of the reactor/bed for vehicle use.

Summary

The group recommended fundamental studies on sodium alanate be, at a minimum, doubled within the next year. They also recommended a similar doubling of effort on the screening of new materials. A sustained facility and/or research group needs to be established to facilitate the rapid testing and screening of materials. The facility/group needs to be independent of the material developers. Testing protocols and evaluation tools will need to be established and agreed upon by the technical community. Intellectual property issues will need to be addressed. Industry involvement in the field needs to be strengthened. Industry should also be directly involved in the cost evaluations for scale-up, system optimization, reactor engineering and certification.

Carbon Materials

Objectives

The carbon materials breakout group discussed the current state of the technology with the objective of:

- Identifying approaches to develop these materials
- Identifying the barriers to their development and eventual commercialization
- Developing priorities for a R&D program on carbon-based materials

Approach

The breakout group suggested two key areas of focus for the DOE program:

- Strengthen the understanding of scientific principles governing carbon materials and their hydrogen storage capacity
- Conduct the search for new materials guided by a fundamental understanding of hydrogen storage charge/discharge mechanisms in carbon-based materials

The consensus was that DOE should not focus exclusively on single-walled nanotubes; but rather, investigate a broad spectrum of carbon-based materials. The panel suggested that a major effort be made to integrate theory, experiment and engineering. There is a continuous need for groups working in all three areas to be aware of developments among their peers.

The basic interactions of hydrogen with carbon materials, such as the effect of curvature, should be understood. A fundamental understanding of these types of interactions ranging from physisorption to chemisorption would provide guidance for designing new materials and in modifying existing materials. The DOE program should consider other carbon-based materials such as:

- Chemically modified nanotubes
- Chemically modified nanoporous carbons
- Strained mix-hybridized carbons
- C-60 fullerenes as a model of a highly curved system
- Random amorphous carbons
- Polymer precursors
- Carbon metal hybrids
- Multi-walled tubes or carbon fibers

R&D Priorities

The group recommended that R&D focus on the following areas:

Theory

- Provide "directional" guidance for experiments (and vice-versa). It was felt that theory could guide experimentation and engineering, by modeling situations that could be favorable for increased storage. This can also work in the reverse, with engineering or experimentation highlighting certain systems that should be modeled.
- Provide baseline theory to elucidate parameters affecting the type and number of binding sites and the heat of adsorption of $H2$ for a broad range of modified carbon materials. One of the most significant issues facing researchers in the area of carbon materials and their hydrogen uptake is the lack of a baseline example for comparison. Thus, the panel suggested that a baseline be created so that results could be better characterized and potential new structures and modifications could be identified.
- Understand the effect of modifying shape and the degree of curvature on hydrogen adsorption.
- Predict heat and entropy of hydrogen adsorption to rank order candidate materials.
- Understand the chemical and electronic effects of additives and defects; optimize capacity by structural design.

Experimentation

- Conduct definitive experiments to show where and how hydrogen is stored in single-walled nanotubes and in other forms of carbon materials. The group felt that it was important to characterize single-walled nanotubes. However, there was a strong opinion that other carbon materials also be considered so that new possibilities are not overlooked. Several specific studies were suggested:

  - Develop 2 to 3 pure single-walled nanotube standards for synthesis, purification, activation and hydrogen adsorption/desorption measurements. This will help to determine the validity of measurement techniques and to standardize results.
  - Conduct round robin testing. This could be accomplished by providing samples to labs for measurement and by soliciting feedback for a database. The roles of research institutes, national labs, universities and industry need to be defined to address issues such as ownership of intellectual property.
  - Measure isosteric heat at low temperature and low pressure.
  - Develop adsorption isotherms at high pressure.
  - Characterize samples using in-situ Raman and IR spectroscopy and neutron diffraction.

- Conduct definitive experiments to show where and how hydrogen is stored
  - Measure IR stretch.
  - Measure rate, path and mechanism of hydrogen diffusion.
  - Vary material properties systematically for evaluation, particularly diameter of nanotubes and chirality.

- Develop reproducible syntheses and process routes.
- Develop standard reproducible measurement techniques.
- Develop a measurement on perturbation of hydrogen-hydrogen and carbon-carbon bonds with degree of interaction.
- Synthesize and evaluate new compositions, especially highly curved carbons.

Engineering

- Conduct system analysis to determine trade-offs among hydrogen storage capacity and fundamental material properties.
- Address parameters for system engineering development
- Address cyclability and durability of carbon materials.
- Address system poisoning.

Coordination

- Create a secure website to post information and to enhance the efficiency of information exchange.
- Establish a Carbon Materials Working Group.

Chemical Hydride Storage

Objectives

The chemical storage breakout group discussed the current state of technology with the objective of:

- Identifying recycling/recovery options by building on existing chemical hydride storage technologies.
- Identifying additional chemical hydrides that have the potential to meet storage goals.
- Identifying the barriers to development and to eventual commercialization.
- Drafting an R&D plan.

Approach

The group identified several challenges that are discussed below.

Critical Research and Engineering Issues. System integration, e.g. water self-sufficiency and heat rejection, is an important engineering issue. For chemical hydride systems, regeneration of the spent material is another key issue. It was agreed that small companies might not have resources to explore the fundamental chemistry of a large number of chemical hydride systems. Chemistry is the first priority, followed by industrial process development for the recycling of the spent chemical hydride.

Addressing the Challenges. The regeneration process for various chemical hydride cycles needs to be identified and developed. Alternatively, a disposable "waste" such as nitrogen from ammonia decomposition could be considered. The group felt that the quantification of externalities is an important issue for all hydrogen production and storage systems and should play a role in any selection criteria. A full life cycle analysis for materials needs to be performed for each chemical hydride with respect to energy requirements, process integration, efficiency and cost.

Approaches. Only a few complexes have been thoroughly studied – additional R&D on a large number of compounds and cycles is needed to develop synthesis/regeneration processes and to engineer packaged systems. As a first cut, the thermodynamic limit of efficiency and of energy density for each cycle can be determined, since this represents the theoretical maximum.

Potential near-term complexes include (but are not limited to):

- NaH
- $LiAlH_4$
- $NaBH_4$
- Al
- $NaAlH_4$
- Cyclohexane (e.g. cyclohexane $\rightarrow$ benzene + $3H_2$)
- Na
- NH_3BH_3
- $Al (BH_4)_3$
- CH_3OH
- NH_3
- LiH

R&D Priorities

The Chemical Hydride group's recommended R&D plan includes four phases with a go/no-go decision point between phases 1 and 2:

Phase 1: Screen a large number of chemical complexes for:
- Hydrogen storage density potential (without system considerations)
- Thermodynamic energy requirements including regeneration
- Availability of basic components
- Safety
- Down-select to a "manageable" number of systems (approximately 10)

Phase 2: For the subset of systems identified above, investigate improved/new process chemistry
- Identify routes and energy requirements
- Develop processes, including catalysts and operating window conditions (temperature and pressure)

Phase 3a: Develop process designs for "best" complexes and evaluate economic potential
- Reactor engineering, including safety issues
- Energy-efficient new processes
- Overall emissions for entire cycle

- Cost of delivered fuel

Phase 3b: Develop detailed life cycle analyses of top complexes
- Primary energy use
- Cost, emissions and resource depletion

Phase 4: Demonstrate (industry-government partnership)
- Scale-up process for production and recovery
- Optimize system integration for energy efficiency and cost.
- Conduct fleet demonstration to assess consumer interface/acceptability.

Advanced Concepts

Objective

The advanced concepts breakout group discussed thirteen proposed areas for research with the objective of prioritizing the approaches. It should be noted that disagreement exists concerning the potential for some of the proposed approaches to meet the DOE targets.

R&D Priorities

It should be noted that the group felt that a more useful priority ranking of the Advanced Concepts would require far more in-depth consideration. This is a preliminary ranking; several overarching R&D needs were identified which apply to any/all of the advanced concepts. These are:

- Maximum storage capacity – theoretical predictions
- Energy balance / life cycle analysis
- Hydrogen absorption / desorption kinetics
- Preliminary cost analysis – potential for low-cost, high-volume manufacturing
- Safety

Crystalline Nanoporous Materials

Dr. David Sholl of Carnegie Mellon University took the lead on presenting crystalline nanoporous materials for hydrogen storage. He described them and their current status as:

- Advanced zeolites
- Maximum H_2 capacity measured to date: 2.5 wt.%, 5.1 kg/m^3
- H_2 capacity at practical conditions (e.g., T=77K, P=1atm): 1.3 wt%, 2.6 kg/m^3 for NaA, 0.7 wt.%, 13.4 kg/m^3 for ZSM-5
- H_2 adsorbs much more weakly in zeolites compared to other common gases.
- Evidence points to physisorption alone

The advantages are: they are readily available at low cost; chemically and thermally robust; good structural reproducibility; modifiable; and environmentally benign and safe. Six R&D needs were identified:

- Maximum weight percent of H_2 that can be absorbed by physisorption
- Chemical modifications of zeolite surfaces for hydrogen chemisorption
- Best structures for maximum absorption – e.g. small versus large pore
- Characterization of internal surface structures
- Advanced material characterization
- Zeolite chemistry (e.g., Si/Al ratio)

Polymer Microspheres

Robert Dye of LANL took the lead on presenting polymer microspheres for hydrogen storage. He described them and their current status as:

- Hollow spheres from glassy polymers
- Hydrogen membranes, e.g. high temperature polymer meniscus membrane
- Segmented polymers hold H_2 at room temperature
- Goal: Hold liquid H_2 at room temperature
- Operate greater than 300 atm
- PTMSP has very high gas permeability
- For each polymer, there is an optimum permeability at a set operating condition
- Permeation curve as a function of temperature – starting at zero permeation at room temperature
- Metal, boron spheres: 4 wt%

- Material costs inexpensive

The advantages are: microspheres are flowable (which is consistent with conventional automobile designs); portable; safe (due to microencapsulation); light; inexpensive and rechargeable/recyclable. Five R&D needs were identified:

- Pressure inside the sphere
- Means and rate for H_2 transport into and out of the microspheres
- Identifying correct polymer
- Initiation and cessation of hydrogen flow
- Room temperature leak rate

Self-Assembled Nanocomposites

Robert Botto of ANL took the lead on presenting self-assembled nanocomposites for hydrogen storage. He described them and their current status as:

- Aerogels are the scaffold; template with organic functional groups; physisorption, acid-base reaction
- Usable capacity ratio (wt% absorbed in material compared to cylinder): 1.4
- Roughly a few wt% hydrogen storage
- Adsorption/desorption exchange rate: 0.8 seconds; 100% desorption

The advantages are: they are lightweight; self assembly takes place in a one-step process; flexibility to control properties (including the chemistry of surface groups, pore structure, and incorporation of dispersed metallic clusters); the materials are stable; environmentally benign and inexpensive. Four R&D needs were identified for self-assembled nanocomposites, as follows:

- Studying silica aerogels
- Modifying aerogels
- Theoretical modeling - various chemical structures / materials
- Functionalization strategies

Advanced Hydride Materials

The nature and status of advanced hydride materials were summarized as follows:

- $LiBH_4 = LiH + B + 1.5H_2(g)$: endothermic reaction; 13.8 wt% H_2 released

- A low temperature H_2 release has been observed: 2.3 wt% H_2 released at 118°C
- $LiBH_4$ combined with organics – to reduce the severity and heat of the hydrolysis reaction: 2.5 wt% H_2 produced from one compound

The advantages of these materials include their high weight percent hydrogen storage potential, light weight and reversibility potential (to be explored). Three R&D needs were identified for advanced hydride materials, as follows:

- Hydrogen generation from $LiBH_4$.
- Understanding of hydrogen uptake and release.
- Incorporation of $LiBH_4$ into nanoporous materials to see effects on the chemical reaction (for lowering reaction temperature).

Metal Organics

Christopher Marshall of ANL took the lead on presenting metal organics for hydrogen storage. He described them and their current status as:

- Zeolite materials using carbon as the backbone, polymeric synthesis, using carbon and metals; cross between carbon and zeolite materials; organic microporous
- Form large cage-like structures, approximately the size of a methane molecule
- MOF-5 optimized for methane: 3% methane
- Want strong van der Waals interactions.

The advantages of metal organics include: flexibility in material composition/structure; larger pore structures with tailored properties; and potential to incorporate functional groups and capillary effect. Two R&D needs were identified for metal organics, as follows:

- Initial studies of weight percent hydrogen absorption
- Chemical modification – functional groups

Boron Nitride Nanotubes

The nature and status of boron nitride nanotubes were summarized as follows:

- Nanotubes based on boron nitride instead of carbon.
- Roughly equivalent to carbon nanotubes in terms of advantages, but less pyrophoric.

Four R&D needs were identified for boron nitride nanotubes, as follows:

- Verify weight density of hydrogen storage
- Understand adsorption mechanisms
- Estimate costs
- Understand desorption behavior

Bulk Amorphous Materials

C.T. Liu of ORNL took the lead on presenting bulk amorphous materials for hydrogen storage. He described them and their current status as:

- A new approach – new class of metallic materials based on multi-component alloy systems; loosely packed with porous defects (interstitial holes for H_2 storage) in super cooled liquid phase.
- Ti-Al-Fe based - light weight/low cost; can meet 6% target if H/M=3.
- Thermal treatment may be used to control size and distribution of porous defects.
- Lack of systematic study.
- Theoretically, stability will increase with addition of hydrogen.
- Various materials/alloys; zirconium base.

The advantages of bulk amorphous materials include: fast adsorption/desorption kinetics; resistance to embrittlement and disintegration; multiple types of interstitial sites for hydrogen absorption and/or chemisorption; and low cost/volume production. Five R&D needs were identified:

- Verify weight percent for Ti-Al-Fe material
- Low density / low cost materials
- Demonstrate H_2 release
- Calculate / optimize environment and bonding strengths
- Detailed experimental information on bond lengths and ordering

Hydrogenated Amorphous Carbon

George Fenske of ANL took the lead on presenting Hydrogenated Amorphous Carbon for hydrogen storage. He described them and their current status as:

- Carbon skeleton made up in part of stressed graphitic “cages” (nanotube sponge)
- Plasma-assisted chemical deposition process
- Tests indicate rapid H_2 release between 200-300ºC

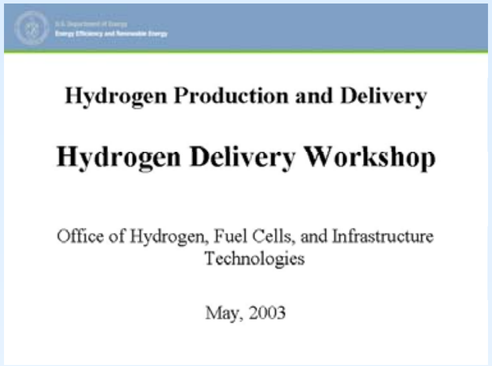
The advantages of this approach are: 6-7 wt% hydrogen; stable up to 300ºC; and potential for high hydrogen content. Four R&D needs were identified:

- Reversibility
- Kinetics – uptake/release rates
- Structure / Modeling - to determine whether paths are stable, diffuse back and forth, interconnected.
- Fabrication of powders

Appendix A. Hydrogen Storage Materials Workshop -- AGENDA

DAY ONE – August 14, 2002	
8:30 am	Overview of the Workshop ▪ Argonne Welcome, Don Joyce, Deputy to the Director, Argonne National Laboratory ▪ Meeting Arrangements/Logistics, Walt Podolski, Argonne National Laboratory
9:00 am	Plenary Presentations: DOE Hydrogen Program and the Status of Hydrogen Storage Systems ▪ DOE Storage Program & Targets/Workshop Expectations, JoAnn Milliken, DOE ▪ An Auto Company's Perspective, Brian Wicke, General Motors ▪ Analysis of Hydrogen Storage Materials and Systems, P. Teagan and M. Rona, TIAX LLC
10:40 am	Hydrogen Storage Material Technology Status Reports ▪ Advanced Hydrides, George Thomas, Sandia National Laboratory ▪ Carbon Materials, Jack Fischer, University of Pennsylvania ▪ Chemical Storage, Ali T-Raissi, Florida Solar Energy Center ▪ Advanced Concepts, John Petrovic, Los Alamos National Laboratory
12:30 pm	LUNCH
1:30 pm	Four Facilitated Breakout Sessions – organized by technical topic areas: ▪ Advanced/Complex Hydrides (Facilitator: G. Sandrock, Scribe: C. Elam) ▪ Carbon Materials (Facilitator: J. Ohi, Scribe: Cathy Grégoire Padró) ▪ Chemical Storage (Facilitator: K. Stroh, Scribe: M. Rowles) ▪ Advanced Concepts (Facilitator: J. Petrovic, Scribe: S. Marin)
4:30 pm	Breakout Group Initial Reports
5:30 pm	ADJOURN
DAY 2 – August 15, 2002	
8:30 am	Breakout groups meet
10:30 am	Breakout groups report results to the group
12:00 pm	ADJOURN

Appendix B. Plenary Presentations



U.S. Department of Energy
Energy Efficiency and Renewable Energy

Hydrogen Production and Delivery

Hydrogen Delivery Workshop

Office of Hydrogen, Fuel Cells, and Infrastructure Technologies

May, 2003

AIR LIQUIDE

Hydrogen Delivery Technologies and Systems

Pipeline Transmission of Hydrogen

Strategic Initiatives for Hydrogen Delivery Workshop ■ May 7- 8, 2003
U.S. Department of Energy ■ Hydrogen, Fuel Cells, and Infrastructure Technologies Program

PRAXAIR

Hydrogen Delivery
Liquefaction & Compression

Raymond Drnevich

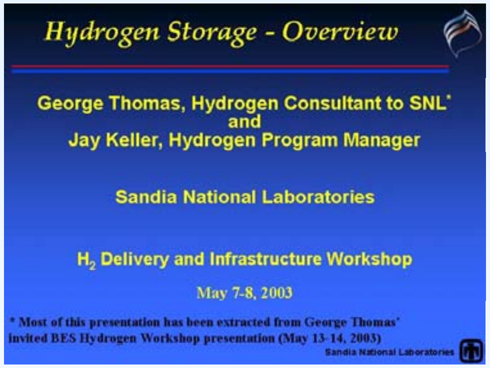
Praxair - Tonawanda, NY

Strategic Initiatives for Hydrogen Delivery Workshop -
May 7, 2003

AIR PRODUCTS

Toward new solid and liquid phase systems for the containment, transport and delivery of hydrogen

By Guido P. Pez



Hydrogen Storage - Overview

George Thomas, Hydrogen Consultant to SNL*
and
Jay Keller, Hydrogen Program Manager

Sandia National Laboratories

H_2 Delivery and Infrastructure Workshop

May 7-8, 2003

* Most of this presentation has been extracted from George Thomas' invited BES Hydrogen Workshop presentation (May 13-14, 2003)

Sandia National Laboratories

Appendix C. Hydrogen Storage Materials Workshop List of Participants

Channing C. Ahn
Division of Engineering & Applied Science
California Institute of Technology
MS 138-78
1200 East California Boulevard
Pasadena, CA 91125
Phone: 626.395.2174
Fax: 626.795.6132
E-mail: cca@caltech.edu

Khalil Amine
Chemical Technology Division
Argonne National Laboratory
Building 205
9700 South Cass Avenue
Argonne, IL 60439
Phone: 630.252.3838
Fax: 630.252.4176
E-mail: amine@cmt.anl.gov

Arlene F. Anderson
Office of Hydrogen, Fuel Cells & Infrastructure Technologies
U.S. Department of Energy
Forrestal Building, EE-2H
1000 Independence Avenue, SW
Washington, DC 20505
Phone: 202.586.3818
Fax:
E-mail: arlene.anderson@ee.doe.gov

Ming Au
Westinghouse Savannah River Company
999W-407
Aiken, SC 29808
Phone: 803.819.8442
Fax: 803.819.8416
E-mail: ming.au@srs.gov

Joseph F. Birdwell
Nuclear Science & Technology Division
Oak Ridge National Laboratory
P.O. Box 2008
Oak Ridge, TN 37831-6305
Phone: 865.607.9474
Fax: 865.574.4624
E-mail: birdwelljfjr@ornl.gov

Larry S. Blair
Office of Hydrogen, Fuel Cells and Infrastructure Technologies
U.S. Department of Energy
1550 Bridger Road
Rio Rancho, NM 87144
Phone: 505.259.5009 (Cellular)
Fax: 505.896.6686
E-mail: larry.blair@ee.doe.gov

Bradley C. Bockrath
Department of Fuels & Process Chemistry
U.S. DOE/National Energy Technology Laboratory
P.O. Box 10940
Pittsburgh, PA 15236
Phone: 412.386.6081
Fax: 412.386.5936
E-mail: bockrath@netl.doe.gov

Robert E. Botto
Chemistry Division
Argonne National Laboratory
Building 200
9700 South Cass Avenue
Argonne, IL 60439
Phone: 630.252.3524
Fax: 630.252.9288
E-mail: rbotto@anl.gov

Keith Bradley
Nanomix, Inc.
1295A 67th Street
Emeryville, CA 94608
Phone: 510.428.5300
Fax: 510.658.0425
E-mail: info@nano.com

Darryl P. Butt
Department of Materials Science & Engineering
University of Florida
206 Rhines Hall
P.O. Box 116400
Gainesville, FL 32611-6400
Phone: 352.846.3333
Fax: 352.392.6359
E-mail: dbutt@mse.ufl.edu

Lucito B. Cataquiz
Office of Hydrogen, Fuel Cells & Infrastructure Technologies
U.S. Department of Energy
1000 Independence Avenue, SW
Washington, DC 20585
Phone: 202.586.0729
Fax: 202.586.9811
E-mail: lucito.cataquiz@ee.doe.gov

Bharat S. Chahar
Department of Carbon Businesses
Conoco, Inc.
1000 South Pine
Ponca City, OK 74601
Phone: 580.767.4354
Fax: 580.767.3995
E-mail: bharat.s.chahar@conoco.com

Richard Chahine
Hydrogen Research Institute
Université du Québec a Trois-Rivières
P.O. Box 500
Trois-Rivières, Québec G9A 5H7
CANADA
Phone: 819.376.5011 ext. 3588
Fax: 819.376.5154
E-mail: chahine@uqtr.ca

Dhanesh Chandra
Department of Metallurgical & Materials Engineering
University of Nevada-Reno
Mackay School of Mines
MS 388
Reno, NV 89557
Phone: 775.784.4960
Fax: 775.784.4316
E-mail: dchandra@unr.edu

Dennis R. Cheney
Dept. of Technology Transfer and Commercialization
INEEL
2525 Freemont Street
Idaho Falls, ID 83401
Phone: 208.526.7950
Fax: 208.526.0876
E-mail: dcy@inel.gov

Terry M. Copeland
Millennium Cell, Inc.
Building E, Ste. L
1 Industrial Way West
Eatontown, NJ 07724
Phone: 732.544.5724
Fax: 732.542.4010
E-mail: copeland@millenniumcell.com

David H. DaCosta
Ergenics, Inc.
373 Margaret King Avenue
Ringwood, NJ 07456
Phone: 973.728.8815
Fax: 973.728.8864
E-mail: dacosta@warwick.net

Sara Anne Dillich
Industrial Technologies Program, EE-20
U.S. Department of Energy-EERE
1000 Independence Avenue, SW
Washington, DC 20585-0121
Phone: 202.586.7925
Fax: 202.586.7114
E-mail: sara.dillich@ee.doe.gov

Anne C. Dillon
Division of Basic Sciences
NREL
1617 Cole Boulevard
Golden, CO 80401
Phone: 303.384.6607
Fax: 303.374.6655
E-mail: adillon@nrel.gov

Robert C. Dye
Materials Department
Los Alamos National Laboratory
MS E549
Los Alamos, NM 87545
Phone: 505.667.3404
Fax: 505.667.8109
E-mail: rcdye@lanl.gov

Carolyn C. Elam
Hydrogen Program
NREL
1617 Cole Boulevard
Golden, CO 80401
Phone: 303.275.2925
Fax: 303.275.2905
E-mail: carolyn_elam@nrel.gov

George R. Fenske
Energy Technology Division
Argonne National Laboratory
Building 212
9700 South Cass Avenue
Argonne, IL 60439
Phone: 630.252.5190
Fax: 630.252.4798
E-mail: gfenske@anl.gov

Karl F. Fiegenschuh
FreedomCAR/THINK Technologies
Ford Motor Company
Scientific Research Laboratory
2101 Village Road, MD 2247
Dearborn, MI 48124
Phone: 313.337.3125
Fax: 313.594.7303
E-mail: kfiegens@ford.com

John E. Fischer
Department of Materials Science & Engineering
University of Pennsylvania
3231 Walnut Street
Philadelphia, PA 19104-6272
Phone: 215.898.6924
Fax: 215.573.2128
E-mail: fischer@lrsm.upenn.edu

Matthew Fisher
Powerball International, Incorporated
2095 West 2200 South
West Valley City, UT 84119-1323
Phone: 801.974.9120
Fax: 801.972.9269
E-mail: matt@powerball.net

Henry C. Foley
Department of Chemical Engineering
The Pennsylvania State University
160 Fenske Laboratory
University Park, PA 16802
Phone: 814.865.2574
Fax: 814.865.7846
E-mail: hcf2@psu.edu

William W. Freise
Powerball International, Incorporated
2095 West 2200 South
West Valley City, UT 84119-1323
Phone: 801.974.9120
Fax: 801.972.9269
E-mail: bill@powerball.net

Nidia C. Gallego
Metals & Ceramics Division-Carbon Materials Group
Oak Ridge National Laboratory
MS 6087
1 Bethel Valley Road
Oak Ridge, TN 37716
Phone: 865.241.9459
Fax: 865.576.8424
E-mail: gallegonc@ornl.gov

Catherine E. Gregoire Padro
Hydrogen Program
NREL
MS 1613
1617 Cole Boulevard
Golden, CO 80401
Phone: 303.275.2919
Fax: 303.275.2905
E-mail: cathy_pedro@nrel.gov

Leonid Grigorian
FRL Department
Honda R&D Americas, Inc.
1381 Kinnear Road, Ste. 116
Columbus, OH 43212
Phone: 614.340.6095
Fax: 614.340.6082
E-mail: lgrigorian@oh.hra.com

Karl J. Gross
Department of Analytical Materials Science
Sandia National Laboratories
P.O. Box 969, MS 9403
Livermore, CA 94551-0969
Phone: 925.294.4639
Fax: 925.294.8410
E-mail: kjgross@sandia.gov

Dieter Gruen
Materials Science Division
Argonne National Laboratory
Building 200
9700 South Cass Avenue
Argonne, IL 60439
Phone: 630.252.3513
Fax: 630.252.9555
E-mail: dmgruen@anl.gov

Michael J. Heben
Department of Basic Sciences
NREL
1617 Cole Boulevard
Golden, CO 80401
Phone: 303.384.6641
Fax:
E-mail: mikeh@nrel.gov

Hugh W. Hillhouse
Department of Chemical Engineering
Purdue University
1283 Chemical Engineering Building
West Lafayette, IN 47907
Phone: 765.496.6056
Fax: 765.494.0805
E-mail: hugh@purdue.edu

Michael Hirscher
Schutz
Max-Planck-Institut for Metallforschung
Heisenbergstrasse 1
D-70569Stuttgart
GERMANY
Phone: 49.711.689.1808
Fax: 49.711.689.1952
E-mail: hirscher@mf.mpg.de

Craig M. Jensen
Department of Chemistry
University of Hawaii
Honolulu, HI 96822
Phone: 808.956.2769
Fax: 808.956.5908
E-mail: jensen@gold.chem.hawaii.edu

D. Ray Johnson
Metals & Ceramics Division
Oak Ridge National Laboratory
P.O. Box 2008
Oak Ridge, TN 37831-6066
Phone: 865.576.6832
Fax: 865.574.6098
E-mail: johnsondr@ornl.gov

Karl Johnson
Department of Chemical & Petroleum Engineering
University of Pittsburgh
1249 Benedum Hall
Pittsburgh, PA 15261
Phone: 412.642.5644
Fax: 412.624.9639
E-mail: karlj@pitt.edu

Russell H. Jones
Materials Science Division
Pacific Northwest National Laboratory
P.O. Box 999
Richland, WA 99352
Phone: 509.376.4276
Fax: 509.376.0418
E-mail: rh.jones@pnl.gov

George Kervitsky
SENTECH, Inc.
4733 Bethesda Avenue
Bethesda, MD 20814
Phone: 301.961.4922
Fax: 301.654.7832
E-mail: gkervits@sentech.org

David L. King
ESTD-Materials Department
Battelle-PNNL
P.O. Box 999
Richland, WA 99352
Phone: 509.375.3708
Fax: 509.375.2167
E-mail: david.king@pnl.gov

Kerry M. Klingler
Department of Energy Efficiency & Natural Resources
INEEL
MS Z110
2095 North Boulevard
Idaho Falls, ID 83415
Phone: 208.526.4516
Fax: 208.526.9822
E-mail: klinkm@inel.gov

Arthur J. Kropf
Chemical Technology Division
Argonne National Laboratory
Building 205
9700 South Cass Avenue
Argonne, IL 60439
Phone: 630.252.9398
Fax: 603.252.9373
E-mail: kropf@cmt.anl.gov

David A. Lesch
Department of Corporate Research
UOP
50 East Algonquin Road
Des Plaines, IL 60017
Phone: 847.311.3899
Fax: 847.375.7985
E-mail: dalesch@uop.com

C. T. Liu
Metals & Ceramics Division
Oak Ridge National Laboratory
1 Bethel Valley Road
Oak Ridge, TN 37831
Phone: 865.574.4459
Fax: 865.574.7659
E-mail: liuct@ornl.gov

Di-Jia Liu
Chemical Technology Division
Argonne National Laboratory
Building 205
9700 South Cass Avenue
Argonne, IL 60439
Phone: 630.252.4511
Fax: 630.252.4176
E-mail: liud@cmt.anl.gov

John J. Low
UOP Research Center
50 East Algonquin Road, Box 5016
Des Plaines, IL 60017-5016
Phone: 847.391.3046
Phone: 847.391.3727
E-mail: jjlow@uop.com

Sherry L. Marin
SENTECH, Inc.
4733 Bethesda Avenue
Bethesda, MD 20814
Phone: 301.961.4918
Fax: 301.654.7832
E-mail: smarin@sentech.org

Christopher L. Marshall
Chemical Technology Division
Argonne National Laboratory
Building 205
9700 South Cass Avenue
Argonne, IL 60439
Phone: 630.252.4310
Fax: 630.972.4408
E-mail: marshall@anl.gov

Vincent Meunier
Computer Science & Mathematics Division
Oak Ridge National Laboratory
MS 6232, Building 4500N
P.O. Box 2008
Oak Ridge, TN 37831
Phone: 865.574.7632
Fax: 865.574.4839
E-mail: meunierv@ornl.gov

James F. Miller
Chemical Technology Division
Argonne National Laboratory
Building 205
9700 South Cass Avenue
Argonne, IL 60439
Phone: 630.252.4537
Fax: 630.252.9505
E-mail: millerj@cmt.anl.gov

JoAnn Milliken
Office of Hydrogen, Fuel Cells & Infrastructure Technologies
U.S. Department of Energy
Forrestal Building
1000 Independence Avenue
Washington, DC 20003
Phone: 202.586.2480
Fax: 202.586.9811
E-mail: joann.milliken@ee.doe.gov

Eric C. Moloy
Thermochemistry Facility, CHMS
University of California-Davis
4440 Chem Annex
One Shields Avenue
Davis, CA 95616-8779
Phone: 530.752.9289
Fax: 530.752.9307
E-mail: ecmoloy@ucdavis.edu

Daniel A. Mosher
Department of Structural Integrity & Reliability
United Technologies Research Center
MS 129-73
411 Silver Lane
East Hartford, CT 06108
Phone: 860.610.7011
Fax: 860.610.7526
E-mail: mosherda@utrc.utc.com

Theodore Motyka
Hydrogen Technology Laboratory
Savannah River Technology Center
Building 773-41A, 144
Aiken, SC 29808
Phone: 803.725.0772
Fax: 803.725.4553
E-mail: ted.motyka@srs.gov

Renée M. Nault
Information and Publishing Division
Argonne National Laboratory
Building 362
9700 South Cass Avenue
Argonne, IL 60439
Phone: 630.252.6064
Fax: 630.252.1393
E-mail: rmnault@anl.gov

William J. Nellis
Department of Physics and Applied Technology

Lawrence Livermore National Laboratory
Livermore, CA 94550
Phone: 925.422.7200
Fax: 925.422.2851
E-mail: nellis1@llnl.gov

Nobuyuki Nishimiya
School of Materials Science
Toyohashi University of Technology
441-8580 Tempaku-cho, Toyohashi JAPAN
Phone: 81.532.44.6816
Fax: 81.532.48.5833
E-mail: nisimiya@tutms.tut.ac.up

James M. Ohi
National Renewable Energy Laboratory
1617 Cole Boulevard
Golden, CO 80401-3393
Phone: 303.275.3706
Fax: 303.275.4695
E-mail: jim_ohi@nrel.gov

Kevin C. Ott
Chemistry Division
Los Alamos National Laboratory
MS J514
Los Alamos, NM 87545
Phone: 505.667.4600
Fax: 505.667.3314
E-mail: kcott@lanl.gov

Richard A. Page
Southwest Research Institute
6220 Culebra Road
San Antonio, TX 78238
Phone: 210.522.3252
Fax: 210.522.6220
E-mail: rpage@swri.org

Philip A. Parilla
NREL
1617 Cole Boulevard
Golden, CO 80401
Phone: 303.384.6506
Fax: 303.384.6430
E-mail: philip-parilla@nrel.gov

George D. Parks
Phillips Petroleum Company
356 PL PRC
Bartlesville, OK 74004
Phone: 918.661.7780
Fax: 918.662.1097
E-mail: gdp@ppco.com

Vitalij K. Pecharsky
Department of Materials Science and Engineering
Ames Laboratory
Iowa State University
242 Spedding
Ames, IA 50011-3020
Phone: 515.294.8220
Fax: 515.294.9579
E-mail: vitkp@ameslab.gov

John J. Petrovic
Los Alamos National Laboratory
Group MST-8, MS G770
P.O. Box 1663
Los Alamos, NM 87545
Phone: 505.667.0125
Fax: 505.667.5268
E-mail: petrovic@lanl.gov

Guido P. Pez
Corporate Science & Technology Center
Air Products & Chemicals, Inc.
7201 Hamilton Boulevard
Allentown, PA 18195-1501
Phone: 610.481.4271
Fax: 610.481.7719
E-mail: pezgp@apci.com

Walter F. Podolski
Chemical Technology Division
Argonne National Laboratory
Building 205
9700 South Cass Avenue
Argonne, IL 60439
Phone: 630.252.7558
Fax: 630.252.4176
E-mail: podolski@cmt.anl.gov

A. C. Raptis
Energy Technology Division
Argonne National Laboratory
Building 308
9700 South Cass Avenue
Argonne, IL 60439
Phone: 630.252.5930
Fax: 630.252.3250
E-mail: raptis@anl.gov

James J. Reilly
Materials Science Department
Brookhaven National Laboratory
Building 815
Upton, NY 11973-5000
Phone: 631.344.4502
Fax: 631.344.2887
E-mail: jreillys@bnl.gov

Stephen J. Riley
Chemistry Division
Argonne National Laboratory
Building 200
9700 South Cass Avenue
Argonne, IL 60439
Phone: 630.252.6793
Fax: 630.252.4954
E-mail: sjriley@anl.gov

Terry Ring
Department of Chemical & Fuels Engineering
Powerball International, Incorporated/Univ. of Utah
Merrill Engineering Building 3290
Salt Lake City, UT 84112
Phone: 801.585.5705
Fax: 801.585.9291
E-mail: t.ring@m.cc.utah.edu

James A. Ritter
Department of Chemical Engineering
University of South Carolina
Swearingen Engineering Center
Columbia, SC 29208
Phone: 803.777.3590
Fax: 803.777.8265
E-mail: ritter@engr.sc.edu

Jerry D. Rogers
R & D Department
General Motors
30500 Mound Road
Warren, MI 48090-9055
Phone: 586.986.1607
Fax: 586.986.1910
E-mail: jerry.rogers@gm.com

Mehmet Rona
TIAX LLC
20 Acorn Park
Cambridge, MA 02140
Phone: 617.498.5070
Fax: 617.498.7119
E-mail: rona.m@tiax.biz

Jules Routbort
Energy Technology Division
Argonne National Laboratory
Building 212
9700 South Cass Avenue
Argonne, IL 60439
Phone: 630.252.5065
Fax:
E-mail: routbort@anl.gov

Matthew P. Rowles
SENTECH, Inc.
4733 Bethesda Avenue
Bethesda, MD 20814
Phone: 301.941.2541
Fax: 301.654.7832
E-mail: mrowles@ssentech.org

Tecle S. Rufael
Department of Energy Research & Technology
Chevron Texaco
4800 Fournace Place
Bellaire, TX 77401
Phone: 713.432.2589
Fax: 713.432.6908
E-mail: tecle.rufael@chevrontexaco.com

Gary D. Sandrock
SunaTech, Inc.
113 Kraft Place
Ringwood, NJ 07456
Phone: 973.962.1158
Fax: 973.962.1158
E-mail: sandrock@warwick.net

Jeff A. Schmidt
Ball Aerospace & Technologies Corp.
1600 Commerce Street
Boulder, CO 80301
Phone: 303.939.5703
Fax: 303.939.6307
E-mail: jschmidt@ball.com

Joachim H. Schneibel
Metals & Ceramics Division
Oak Ridge National Laboratory
P.O. Box 2008
Oak Ridge,TN 37831-6115
Phone: 865.576.4644
Fax: 865.574.7659
E-mail: schneibeljh@ornl.gov

Rajat K. Sen
Sentech, Inc.
4733 Bethesda Avenue, Ste. 608
Bethesda, MD 20814
Phone: 301.961.4924
Fax: 301.654.7832
E-mail: rsen@sentech.org

David S. Sholl
Department of Chemical Engineering
Carnegie Mellon University
Pittsburgh, PA 15213
Phone: 412.268.4207
Fax: 412.268.7139
E-mail: sholl@andrew.cmu.edu

Darlene K. Slattery
Hydrogen Research & Applications Center
Florida Solar Energy Center
1679 Clearlake Road
Cocoa, FL 32922-5703
Phone: 321.638.1449
Fax: 321.638.1010
E-mail: dkslatt@fsec.ucf.edu

Randall Q. Snurr
Department of Chemical Engineering
Northwestern University
2145 Sheridan Road, E136
Evanston, IL 60208
Phone: 847.467.2977
Fax: 847.467.1018
E-mail: snurr@northwestern.edu

Kenneth R. Stroh
Energy & Sustainable Systems Programs Office
Los Alamos National Laboratory
MS C 331
Los Alamos, NM 87544
Phone: 505.567.7933
Fax: 505.667.4098
E-mail: stroh@lanl.gov

William A. Summers
Hydrogen Technology Laboratory
Savannah River Technology Center
Building 773-42A, Savannah River Site
Aiken, SC 29808
Phone: 803.725.7766
Fax: 803.725.8829
william.summers@srs.gov E-mail:

Ali T-Raissi
Hydrogen Research & Applications Center
Florida Solar Energy Center
1679 Clearlake Road
Cocoa, FL 32922--5703
Phone: 321.638.1446
Fax: 321.638.1010
E-mail: ali@fsec.ucf.edu

Morse N. Taxon
Department of Liberty & Technical Affairs
DaimlerChrysler Corporation
2730 Research Drive
Rochester Hills, MI 48309
Phone: 248.838.5227
Fax: 248.838.5300
E-mail: mnt@daimlerchrysler.com

William P. Teagan
TIAX LLC
20 Acorn Park
Cambridge, MA 02140
Phone: 617.498.6054
Fax: 617.498.7206
E-mail: teagan.w@tiax.biz

George J. Thomas
Sandia National Laboratories
537 Spirit Ridge Court
Reno, NV 89511
Phone: 775.851.2702
Fax: 775.851.2780
E-mail: george.thomas@srcglobal.net

Susan H. Townsend
Hydrogen Energy Adv. Technology Program
GE Global Research
1 Research Circle
Niskayuna, NY 12309
Phone: 518.387.5554
Fax:
E-mail: townsends@research.ge.com

John J. Vajo
Materials Engineering Department
HRL Laboratories
3011 Malibu Canyon Road, RI 62
Malibu, CA 90265
Phone: 310.317.5745
Fax: 310.317.5840
E-mail: vajo@hrl.com

Mike J. Veenstra
Department of Energy Systems
Ford Motor Company
15050 Commerce Drive, North
Dearborn, MI 48120
Phone: 313.322.3148
Fax: 313.248.4077
E-mail: mveenstr@ford.com

Brian G. Wicke
Planning Department
General Motors R&D and Planning
MC 480-106-336M
30500 Mound Road
Warren, MI 48090
Phone: 586.986.1308
Fax: 586.986.1647
E-mail: brian.g.wicke@gm.com

Christopher M. Wolverton
Department of Physical & Environmental Sciences
Ford Motor Company
MD 3083/SRL, P.O. Box 2053
Dearborn, MI 48121-2053
Phone: 313.390.9552
Fax: 313.322.7044
E-mail: cwolvert@ford.com

Alicja Zaluska
HERA, Hydrogen Storage Systems, Inc.
577 Le Breton
Longview, PQ J4G 1R9
CANADA
Phone: 450.651.1202 ext. 226
Fax: 450.651.3801
E-mail: az@herahydrogen.com

Ragaiy Zidan
Hydrogen Technology Laboratory
Savannah River Technology Center-Savannah River Site
773-41A/247 Savannah River Site
Aiken, SC 29808
Phone: 803.725.1726
Fax: 803.725.4129
E-mail: ragaly.zidan@srs.gov

William T. Zollinger
Department of Energy Efficiency & Natural Resources
INEEL
MS 2220
2095 North Boulevard
Idaho Falls, ID 83415
Phone: 208.526.3484
Fax: 208.526.7688
E-mail: zollwt@inel.gov