IV.A.4j Development of Metal Hydrides at Sandia National Laboratories

Lennie Klebanoff (Primary Contact), Jay Keller
Sandia National Laboratories
7011 East Ave., MS 9401
Livermore, CA 94551
Phone: (925) 294-3471; Fax: (925) 294-3410
E-mail: lekleba@sandia.gov

DOE Technology Development Manager:
Carole Read
Phone: (202) 586-3152; Fax: (202) 586-9811
E-mail: Carole.Read@ee.doe.gov

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Introduction

There are currently no materials that meet the DOE hydrogen storage performance targets. In order to address this problem, we at Sandia National Laboratories (SNL) have undertaken the synthesis and testing of new high-hydrogen content hydrogen storage materials in FY 2006. The work is focused on the Complex Anionic Materials (Project B) and the Amide/Imide Materials (Project C). We have further modified these hydrides through catalyst doping and cation substitution in order to improve their reversibility and sorption properties. In addition to these activities, SNL has provided theoretical work in support of the materials discovery activity, fundamental studies to better understand underlying processes and engineering studies that have developed the analysis infrastructure necessary for developing practical storage systems for onboard vehicular storage of hydrogen.

Approach

The discovery process involves preparation methods in both the solid-state and liquid phase, followed by extensive characterization. For the solid state; mainly ball milling and the high-pressure sintering technique (P <140 MPa, T <773 K) have been applied. By utilizing different ball milling approaches in collaboration with our MHCoE partners, we are able to control the size of the particles which is crucial for creating diffusion paths for hydrogen. The high-pressure vessel that incorporates six sample holders has been proven to be an effective tool for discovering new hydrides in different ternary systems. For liquid phase synthesis, we have applied novel solution methods for preparation of new complex hydrides that are flexible in terms of dopants inclusion at all levels, thereby avoiding phase separation issues that can arise in alternative approaches. Structural characterization of the new materials is performed by x-ray diffraction (XRD), neutron diffraction, synchrotron x-ray, Raman, Fourier transform infrared (FTIR), and single crystal x-ray diffraction when possible. For fundamental studies, we have also applied ion scattering spectroscopy (ISS) to elucidate H-related surface diffusion at surfaces. Theoretical approaches include a new Monte Carlo (MC) based method for rapidly assessing compound stability, and more traditional density functional theory (DFT) calculations.

Results

Complex Anionic Materials (Project B)

Initially, our research focused on the development of new doping and substitution methods for the alanates without a loss in hydrogen sorption kinetics, however in FY 2006 our research efforts were expanded to include high-capacity metal borohydrides ($M(BH_4)_x$) since it was determined that these materials have the potential to meet the DOE goals. These borohydrides were synthesized by combining ball milling and Sandia's unique high-pressure station that enables hydrogen pressures of <140 MPa and temperatures <773 K or as an alternative route, solution methods were developed to increase our understanding of the H-properties of these novel materials. Again, computation modeling has been a critical component in the characterization of these high capacity $M(BH_4)_x$ materials.

A new bialkali alanate K_2LiAlH_6 was synthesized at 320-330°C and 100-700 bar. By powder x-ray diffraction, K_2LiAlH_6 was found to crystallize in the *R*-3*m* space group (no. 166) where $a = 5.62068(8)$ and $c = 27.3986(6)$ Å. The observed, calculated and difference plots from the Rietveld refinement are shown in Figure 1. First-principles total energy calculations

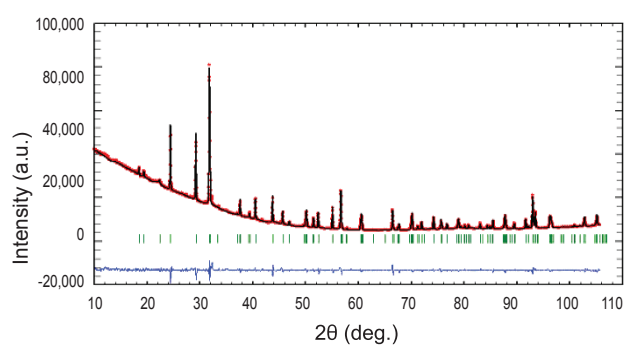

FIGURE 1. The observed and calculated x-ray diffraction pattern with the difference plot from Rietveld refinement of K_2LiAlH_6.

were performed for nine competing database structures of the stoichiometry A_2BCX_6, taken from fluoride and oxide X_6 compounds in the Inorganic Crystal Structure Database (ICSD). The relaxed structures were compared via their total energies and their agreement with experimental diffraction spectra and two database structures, K_2LiAlF_6 (*R*-3*m*) and Cs_2NaAlF_6 (*C*2/*m*), were found to have the lowest total energies. The Rietveld method indicated that the K_2LiAlF_6 structure type was the most favorable. Further, *ab-initio* total energy calculations support the validity of the structure determination. First principles calculations also indicate that cation mixing is energetically unfavorable and this was further proven by Rietveld refinements. Hydride properties such as plateau pressure are therefore more difficult to manipulate through alloying in this class of compounds. We also investigated the hydrogen storage properties and found that the material desorbs >200°C and absorbs >320°C and 100 bar H_2. Kinetics is slow (ca 2 weeks for recharging). The reversible hydrogen content is limited to 2.5 wt% because the reaction formula is:

$$K_2LiAlH_6 \leftrightarrow 2KH + LiH + Al + 3/2H_2$$

In FY 2006, we showed that it is feasible to prepare new hydride phases by solid state reactions yielding the novel Ca-B-H and Mg-B-H systems. We observed <1 wt% reversibility at 250-390°C for the Ca-B-H system. Alternatively, complex borohydrides $[M(BH_4)_x(solv)_y]$ were synthesized using solution metathesis preparatory routes wherein a metal halide is reacted with an alkali metal borohydride in selected solvents. The reaction formula can be written as:

$$MX_3 + ABH_4 \rightarrow M(BH_4)_3(solv)$$

where initial investigations have focused on $MX_3 = AlCl_3$, $AlBr_3$, $TiCl_4$, MgI_2, $ScBr_3$, $CaCl_2$, and $ABH_4 = NaBH_4$, and $LiBH_4$. In FY 2006 we prepared and characterized the following metal borohydrides: $Ca(BH_4)_2(THF)_2$, $Mg(BH_4)_2(THF)_2$, $Sc(BH_4)_3(THF)_2$, $Ti(BH_4)_3(DME)$. To better understand reaction mechanisms, structural characterization of the new materials is performed by XRD, neutron diffraction, synchrotron x-ray, Raman and FTIR. New compounds are investigated with respect to hydrogen desorption temperature, capacity and reversibility using thermally programmed desorption (TPD) and the pressure-composition-temperature (PCT)-station. The crystal structure of novel $Sc(BH_4)_3(THF)_2$ is shown in Figure 2. Additional investigations are underway to find new high-hydrogen content materials. Currently the occurrence of complex hydrides in the ternary Si-system is investigated in collaboration with Utah/HRL/JPL/NIST.

In order to calculate the cohesive energy, the first step in approximating the enthalpy of a reaction, first-principles codes require crystallographic information,

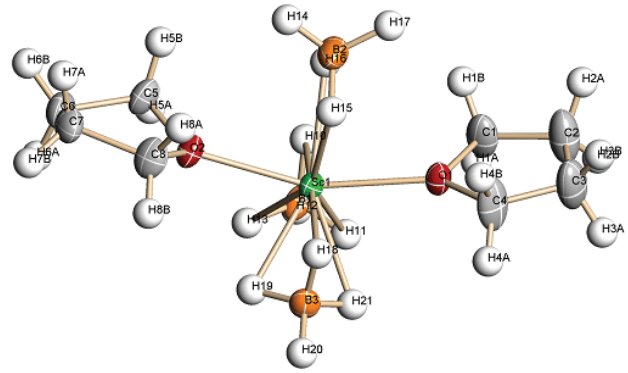

FIGURE 2. Single crystal x-ray diffraction crystal structure of novel $Sc(BH_4)_3(THF)_2$.

such as atom positions, and unit cell dimensions. These are not always available. In many cases, we wish to consider compounds which differ in chemistry from known compounds. For example, it might be of interest to investigate $NaLi(AlH_4)_2$, since this would increase the weight percent of hydrogen substantially in comparison to the standard $NaAlH_4$. In most cases, mixed compounds in the alanate (AlH_n), and borohydride (BH_n) systems have not been synthesized, and the crystal structures remain unknown. We have developed a Monte Carlo-based method which allows crystal structures to be generated by minimizing the electrostatic energy of a combination of alkali or alkaline earth metals with negatively charged anions. The procedure is rapid, and yields results equivalent to, or better than, database structure searching. Figure 3 shows the calculated stability of idealized candidate hydride materials $Mg(AlH_4)_2$, $Mg(BH_4)_2$, and $Ca(BH_4)_2$. Energy changes are with respect to the presumed reaction equations in the figure. Both ICSD structure searching, and MC methods produce, within error, the same results.

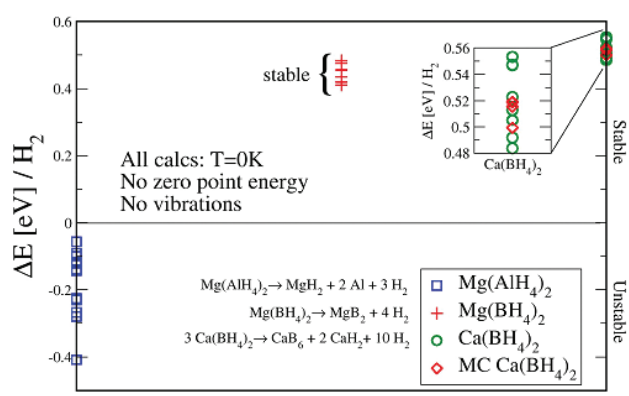

FIGURE 3. Calculated stability of candidate hydride materials $Mg(AlH_4)_2$, $Mg(BH_4)_2$, and $Ca(BH_4)_2$. Energy changes are with respect to the presumed reaction equations in the figure. Both ICSD structure searching, and Monte Carlo methods produce the same results.

Promising materials identified with MC calculations can be more intensively investigated with DFT calculations. Figure 4 shows DFT calculations of the equilibrium H_2 pressure for the decomposition of $Mg(BH_4)_2$ and $Ca(BH_4)_2$. The presumed reaction equations are shown above the graphs. Different lines in the graphs correspond to different crystal structures taken from the ICSD. The results indicate that the equilibrium pressure is promising, and synthesis attempts are worthwhile. Estimates of the formation enthalpy of Ca and Mg borohydride were promising, and we have performed full calculations including the full phonon DOS to obtain the free energy as a function of temperature. Synthetic efforts for both materials are currently underway.

Amide/Imide Storage Materials (Project C)

In FY 2006 we elucidated the reaction pathway for the hydrogen release and uptake from ($2LiNH_2$ + MgH_2). The determined reaction path is:

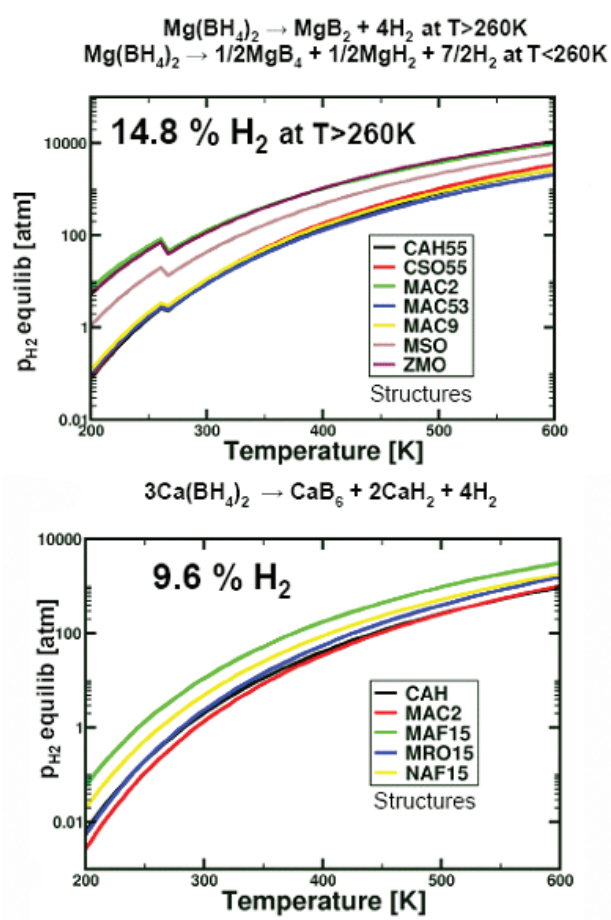

FIGURE 4. Calculations of the equilibrium H_2 pressure for the decomposition of $Mg(BH_4)_2$ and $Ca(BH_4)_2$. The presumed reaction equations are shown above the graphs. Different lines in the graphs correspond to different crystal structures taken from the ICSD. The results indicate that the equilibrium pressure is promising.

$$2LiNH_2 + MgH_2 \rightarrow Mg(NH_2)_2 + 2LiH \leftrightarrow MgLi_2(NH)_2 + 2H_2$$

The first step is in irreversible transformation of the starting material into magnesium amide ($Mg(NH_2)_2$) and lithium hydride. After this initial irreversible transformation, by heating one can release hydrogen producing $MgLi_2(NH)_2$, the imide. The imide can be hydrogenated to produce magnesium amide and lithium hydride back again. As a result, the system is reversible, as indicated in the equation above. However, the H contained in the imide seems not to be accessible, limiting the realizeable hydrogen weight percent to ~5.5%. Experiments were also conducted to examine the effect of Ti, ZnO, Pd, Pt, Ni and Cr_2O_5 catalysts on the kinetics of hydrogen release and uptake. Only very slight (<10%) effects were observed on hydrogen uptake and release times.

A difficulty in using amide materials for hydrogen storage is that these materials are at risk for ammonia release via thermal decomposition. The ammonia contamination in the desorbed hydrogen was measured as a function of desorption temperature. The result of these experiments is shown in Figure 5. Summarizing, from 180-240°C, the ammonia concentration varies from ~190 ppm to ~700 ppm.

Potential hydrogen storage materials must be extensively tested for their cycling lifetime. Such measurements on ($2LiNH_2$ + MgH_2) were conducted in FY 2006. The overall results indicate that the kinetics of the material are slow, and that as a result, the capacity is affected by charging time. After 264 cycles, approximately 23% of the original storage capacity is lost. Some of this capacity lost is due to the volatilization of the material by ammonia release.

In collaboration with Professor Ping Chen at the National University of Singapore, the reactive chemistry and structural properties of ($2LiNH_2$ + $LiAlH_4$) were

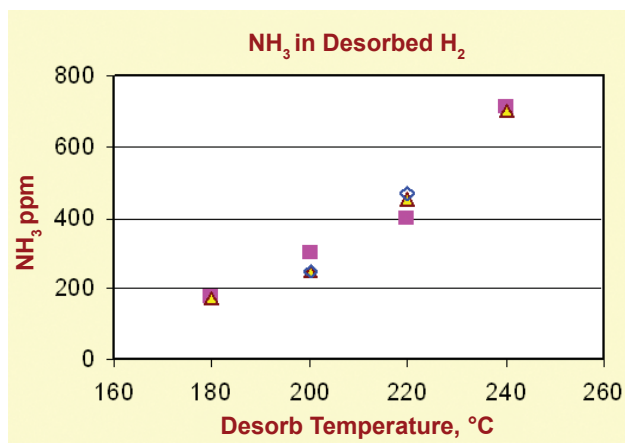

FIGURE 5. Concentration of ammonia in desorbed hydrogen as a function of temperature for the material $2LiNH_2$ + MgH_2. The estimated uncertainty in ordinate values is ± 60 ppm.

also investigated. The expectation for this reaction was as follows:

$$2LiNH_2 + LiAlH_4 \rightarrow Li_3AlN_2 + 4H_2$$

The storage capacity of the reaction above is 9.5 wt% theoretical. This reaction was investigated in the forward direction by ball milling (at room temperature) a mixture of ($2LiNH_2 + LiAlH_4$). Structural characterization (Al NMR, XRD) of the dehydrogenated product indicated that an amorphous structure had formed, but with evidence for significant Al-N bonding, and a four-coordinated aluminum, suggestive of a [LiAlH4-xNx] complex. The reaction was also investigated in the backward direction by purposefully hydrogenating Li_3AlN_2. The studies indicated that for complete hydrogenation of Li_32AlN_2 one needs a high hydrogen pressure (120 bar), high temperature (330°C) and long times (three days to 5.17 wt%).

Although primarily solid-state synthetic approaches have been used to make amide/imide materials, an additional thrust on this project was to develop a high-yield route to state-of-the-art hydrogen storage materials that meet the DOE goals. Novel solution-based synthesis methods of complex metal amides ($M\text{-}NR_2$) were developed in FY 2006 and the surface area controlled through shear mixing during synthesis to yield metal hydrides with high surface areas to improve the thermodynamics and kinetic properties of the hydrogen storage materials.

Simple and complex amides were devised using commercially available metal alkyls and NH_3(g) in a solvent. Bubbling ammonia (NH_3 (g)) through this reaction immediately transformed into precipitate which was identified by micro-powder x-ray diffraction as a variety of products shown in the respective equation (eq 1–7).

$$n\text{-BuLi} + (xs)\ NH_3(g) \rightarrow LiNH_2 + H\text{-}Bu^n \quad \text{(amide) (1)}$$

$$t\text{-BuLi} + (xs)\ NH_3(g) \rightarrow Li_2NH + H\text{-}Bu^t \quad \text{(imide) (2)}$$

$$n\text{-}Mg(Bu)_2 + (xs)\ NH_3(g) \rightarrow Mg(NH_2)_2 + 2\ H\text{-}Bu^t \quad \text{(amorphous) (3)}$$

$$Mg(Bu)_2 + x\ BuLi + (xs)\ NH_3(g) \rightarrow MgLi_x(NH_2)_{3+x} + 3{+}x\ H\text{-}Bu^n$$
(imide) (4-7)

n-Bu, t-Bu were mixed for an array of 4 sets of compounds.

Scanning electron microscopy (SEM) studies of these powders were found to be large (< 0.5 µm) and irregular in shape. Therefore, we undertook shear mixing during the reaction process which led to nanometer materials. Figure 6 shows the mixed metal ceramic nanoparticles.

Testing of the simple material for H-cyclability indicated that solvent was present which prevented the normal observed values noted for these materials. Therefore, after several attempts, a methodology that involved heating under vacuum for extended times successfully removed the trace amounts of solvent. The resulting materials were found to be comparable with the ball milled materials. Analysis of the 1;1 LiMg materials were not highly cycleable as expected; 2:1 Li:Mg compounds are expected to give high cycleability and testing is underway.

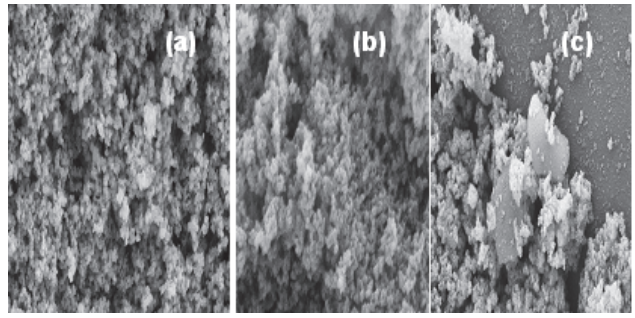

FIGURE 6. SEM images of powders generated through shear mixing following (a) eq. 6, (b) eq. 7, and (c) eq. 8 in the text.

Engineering Properties (Project E)

Hydride materials experience expansion during hydrogen absorption. For some hydride materials the volumetric expansion is significant, and large pressures are generated on the containment boundary. Therefore pressure containment vessel design engineering calculations need to include both internal gas pressure and hydride expansion pressure. We used the $LiNH_2$–MgH_2 material as a vehicle to construct the experimental methodology and capability, which we will use to determine the expansion and other engineering properties (transport) of promising materials in the future.

Tests to determine $LiNH_2$-MgH_2 material's expansion pressure behavior during hydrogen absorption were performed. These experiments are performed by loading a mass of desorbed material into a volume to achieve the required bulk density. An aluminum beam is outfitted with high temperature strain gages and is positioned in contact with the hydride containment vessel. Hydride expansion pressure causes small deflections/strains in the beam that are recorded as voltage outputs from the strain gage bridge circuit. The absorption conditions for $LiNH_2$-MgH_2 are approximately 200°C and 100 bar. Results from tests performed with $LiNH_2$-MgH_2 material indicate expansion pressures are below 200 psig, approximately four times less than that for sodium alanate under similar conditions.

This year we also initiated development of a system analysis tool for engineering simulations heat and mass transport of the hydrogen solid storage materials. The model provides information on the influence of the

transport properties of a material on the performance of a simplified hydride system. This is critically important in order to optimize the trade-off between thermodynamics and kinetic behavior of a material with the transport properties needed to effect heat management and to evolve and re-absorb hydrogen in a hydride storage system. The goal for this effort is to provide information on the transport behavior of potential materials and not to provide design guidance to construct a storage system. The model considers a unit-cell within the hydride storage bed. The unit-cell model considers a one-dimensional, axisymmetric geometry in the bed surrounding a coolant tube. Partial differential equations for the transient absorption/desorption reaction kinetics and heat transfer are integrated to determine the temperature and hydrogen concentration in the bed. While the reaction chemistry can be generalized, the initial simulations have been performed with a two-step reaction scheme for sodium alanate. The model is linked to the Simulink software to allow dynamic simulations to be coupled with other aspects of the system in the future.

Supporting Fundamental Studies

Catalysts can play an essential role in improving the performance of hydrogen storage materials. We have initiated a study of surface catalysts, and in particular, how hydrogen affects the stability of Ti on a model Al(001) surface. The thermal stability of Ti atoms evaporated onto an Al(100) surface was studied using the surface sensitive technique of ion scattering spectroscopy (ISS). Measurements were made in vacuum and in the presence of an atomic deuterium beam directed at the sample. When heated in vacuum, Ti begins to disappear from the surface at substrate temperatures below 100°C and is completely absent (<0.001 monolayer) at temperatures above 250°C. When a similar measurement is made while the sample is being exposed to a flux of about 10^{14} D/cm^2-sec, the Ti persists on the surface to noticeably higher temperatures. This is illustrated in Figure 7. When more than a monolayer of Ti is present on the surface, the effect may in large part result from the formation of a thin titanium hydride layer. The hydride evidently immobilizes Ti. At a lower Ti coverage, the effect of hydrogen on Ti thermal stability is subtle, but is still detectable. To investigate this in detail, diffusion coefficients for monolayer quantities of Ti on Al were obtained from ISS data in both cases at selected substrate temperatures in the range 50–150°C. The activation energy for diffusion of Ti from the Al(100) surface appears to be altered in the presence of D. The experimental results are being interpreted using density functional theory, which indicates that H-Ti interactions can stabilize surface Ti.

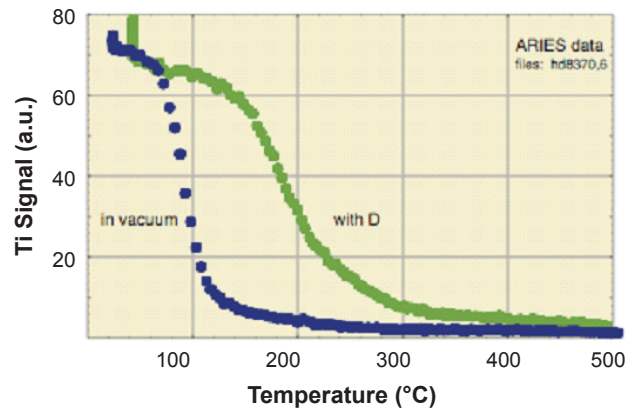

FIGURE 7. ISS data showing the surface concentration of Ti, which had been evaporated as a thin layer onto an Al surface, as a function of temperature. When the sample is exposed to a flux of D atoms, the Ti persists at the surface to higher temperatures, compared to heating in vacuum.

Conclusions and Future Directions

For SNL's work in Project B, a new bialkali alanate, K_2LiAlH_6, was synthesized and characterized in the space group *R*-3*m* (no. 166) being isostructural with the high-temperature form of K_2LiAlF_6. *Ab-initio* total energy calculations were performed, including relaxations of the Rietveld refined structure for comparison to database and further support the validity of the structure determination. The calculations also indicate that the energy for the entropy of mixing of the cations is much smaller in magnitude than the increase in electronic energy, and therefore cation mixing is energetically unfavorable in this bialkali alanate. The effort on discovering new alanates will be abandoned since this group of materials will not meet the FreedomCAR Program targets.

We will continue to focus on discovering new high-capacity complex metal hydrides by using the high-pressure hot-sintering technique. The work on synthesizing metal borohydrides by both solid state and solution based routes will continue. Both the surface and bulk material properties will be thoroughly investigated in order to elucidate methods that improve the kinetics and thermodynamic behavior of the novel materials. In FY 2007, we will also continue our discovery process to find complex metal hydrides in new ternary systems. In addition, in FY 2006 we observed the formation of new phases in the Na-Si-H system depending on the reaction conditions and thus we will in FY 2007 continue exploring Si-containing systems and their potential as new reversible hydrogen storage materials.

The MC method has been validated using $NaAlH_4$, K_2LiAlH_6, and $Ca(BH_4)_2$ as test cases. For the remainder of this fiscal year, and into the next, we will use this

novel method to identify potential candidate bi-alkali borohydride materials for H-storage. If a candidate hydride is found to have an enthalpy which suggests proper stability, the synthesis of these compounds will be undertaken along with additional theoretical studies (DFT) that will include lattice vibrations contributions to the free energy.

For SNL's work in Project C, the reaction pathway by which the ($2LiNH_2 + MgH_2$) system absorbs and releases hydrogen was elucidated. The extent of ammonia contamination in the released hydrogen was quantitatively characterized; however, experimental work on this ($2LiNH_2 + MgH_2$) system will be suspended, pending a clear theoretically based path forward on how to enable a greater hydrogen storage capacity in the system, beyond the ~5 wt% limit. We will continue to support our MHCoE partners in exploring and assessing other (non-Mg) modifications to lithium amide (for example adding Li_3AlH_6), which support the FreedomCAR goals for a high hydrogen storage capacity.

For SNL's work in Project E, the expansion pressure character of ($2LiNH_2 + MgH_2$) was measured and found to be approximately 4x less than the expansion pressure produced by sodium alanate. In addition, in FY 2006 we also initiated development of a system analysis tool for engineering simulations of the hydrogen storage materials. These engineering activities will be continued, albeit at a modest level, to improve our analysis infrastructure so that when a truly viable storage material is found, the analytical infrastructure is in place so that tank design can proceed quickly.

For Supporting Fundamental Studies, we have initiated a study of surface catalysts, and in particular, how hydrogen affects their stability. A model system consisting of Ti on a clean Al surface has been selected for this study. The surface stability of the Ti in the presence of hydrogen and elevated heating has been examined. In FY 2007 we plan to extend our fundamental studies to understand more clearly the role of contamination in the synthesis and characterization of metal hydrides storage materials. In particular, the extent of oxygen contamination will be examined, and will provide feedback to the synthetic methodologies to help improve the purity with which these materials are made.

FY 2006 Publications/Presentations

1. Structural Investigation and Hydrogen Storage Properties of a New Li,K Bialkali Alanate, E. Rönnebro, E. Majzoub, S. Sickafoose, Poster presented at Hydrogen-Metal Systems, Gordon Research Conference, Waterwille, ME, July 10-15, 2005.

2. On the road to make new metal hydrides, E. Rönnebro, E. Majzoub, Oral presentation at 2006 MRS Spring Meeting, Session EE; Hydrogen Storage Materials, San Francisco, CA, April 17-21, 2006.

3. Hydrogen Storage in Sodium Alanates, E.H. Majzoub, American Crystallographic Association Annual Meeting, Orlando, Florida, June 2005.

4. Global Optimization of Complex Hydride Crystal Structures, E.H. Majzoub, V. Ozolins, Materials Research Society, Spring 2006 Symposium on Materials for Hydrogen Storage, April 17-21, San Francisco, CA. Oral presentation.

5. Prediction of the Formation Enthalpy and Free Energy of AB_2H_8 Hydrides (A= Mg, Ca and B= B, Al), R. Stumpf, Materials Research Society, Spring 2006 Symposium on Materials for Hydrogen Storage, April 17-21, San Francisco, CA. Oral presentation.

6. In-situ Raman Spectra of NaAlH4 : Evidence of Highly Stable AlH-1 Anions, E.H. Majzoub, K.F. McCarty, V. Ozolins, Materials Research Society 2004 Symposium on Materials for Hydrogen Storage, November 29-December 3, Boston, MA. Oral presentation.

7. X-ray Diffraction and Raman Spectroscopy Investigation of Titanium Substitution in Sodium Aluminum Hydride, E.H. Majzoub, K.F. McCarty, V. Ozolins, 2004 TMS Annual Meeting & Exhibition, March 14-18, 2004, Charlotte, North Carolina. Oral Presentation.

8. Crystal structure, Raman Spectroscopy and ab-initio calculations of a new bialkali alanate K2LiAlH6, E. Rönnebro, E. Majzoub, paper submitted.

9. Catalytically modified hydrogen properties of novel complex hydrides, Annual Report IEA, Task 17, Project H-10, K. Gross, E. Rönnebro, 2006.

10. Al and 1 H MAS NMR and 27 Al Multiple Quantum Studies of Ti-doped NaAlH4 , J.L. Herberg, R.S. Maxwell, E.H. Majzoub, accepted J. Al. Comp., 2005.

11. XRD and NMR investigation of Ti-compound formation in solution-doping of sodium aluminum hydrides: Solubility of Ti in NaAlH4 crystals grown in THF, E.H. Majzoub, J.L. Herberg, R. Stumpf, S. Spangler, R.S. Maxwell., J. Al. Comp., 394, 265-270, 2005.

12. Li-Mg-N-H: A viable hydrogen storage system for transportation application, W. Luo, K. Gross, S. Sickafoose, P. Crooker, J. Wang, invited presentation, ICMAT-MRS meeting, Singapore, 3-8 July, 2005.

13. Metal Hydride for Reversible Hydrogen Storage, W. Luo, Tutorial lecture, MRS Fall meeting, Boston, MA, Nov. 27-Dec. 02, 2005.

14. Recent Development on Li-Mg-N-H Storage System, W. Luo, J. Wang, K. Gross, T. Boyle, P. Crooker, S. Sickafoose, K. Stewart, MRS spring meeting, San Francisco, CA, Apr. 17-20, 2006.

15. Thermodynamic and Structural Characterization of the Mg-Li-N-H Hydrogen Storage System, W. Luo and S, Sickafoose, J. Alloys and Compounds, 407 (2006) 274-281.

16. Thermodynamic and kinetic investigation of the hydrogen storage in the Li-Mg-N-H system, Z. Xiong, J. Hu, G. Wu, P. Chen, W. Luo, K. Gross and J. Wang, J. Alloys and Compounds, 398 (2005) 235-239.

17. Towards a viable hydrogen storage system for transportation application, W. Luo and E. Rönnebro, J. Alloys and Compounds, 404-406 (2005) 392-395.

18. New Hydrogen Storage Material: Metal-N-H system, W. Luo, J. Wang, K. Gross, S. Sickafoose, P. Crooker, K. Stewart, New Hydrogen Storage Material: Metal-N-H system", Sandia Report, in press.

19. Metal Hydride Center of Excellence, L. Klebanoff, E. Ronnebro and E. Majzoub, oral presentation to the DOE Hydrogen Program Review, May 17, 2006, Arlington, VA.