

## IV.A.11 Effect of Gaseous Impurities on Long-Term Thermal Cycling and Aging Properties of Complex Hydrides for Hydrogen Storage

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### Objectives

- Determination of the effect of ppm level impurities in H<sub>2</sub> gas as well as the effect of impurities and defects in light-weight storage materials.
- Evaluate the long-term durability of light weight Li-N-based compounds.
- Understand the reaction pathways for both developing new materials with enhanced hydriding properties, modifying or controlling reaction pathways for improved performance.

### Technical Barriers

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

- (A) System Weight and Volume
- (D) Durability/Operability
- (E) Charging/Discharging Rates

- (K) System Life Cycle Assessments
- (P) Lack of Understanding of Hydrogen Physisorption and Chemisorption

### Technical Targets

This project is conducting fundamental studies of light-weight hydrogen storage materials, particularly of the Li-N-H system. Insights gained from these studies will be applied toward the design and synthesis of hydrogen storage materials that meet the following DOE 2010 hydrogen storage targets:

- System Gravimetric Capacity: 1.5 kWh/kg
- Storage System Cost: \$4/kWh net
- Cycle Life (1/4 tank to full): 1,000 cycles

### Accomplishments

- Determined causes for loss of cycle in Li<sub>3</sub>N-H<sub>2</sub> system, using calculation of phase diagrams (CALPHAD) thermodynamic modeling and experiments.
- It was determined that there was continuous loss of nitrogen from solid phases during the dehydrogenation cycle, leading to formation of rather stable LiH phase which traps the hydrogen at operating temperature of 528 K (255°C), consequently loss of cycle life.
- Significant cycle life improvements were achieved by using mixtures of H<sub>2</sub> and N<sub>2</sub> gases; using 80/20 molar mixtures of H<sub>2</sub>/N<sub>2</sub>; we obtained near starting Li<sub>3</sub>N-H<sub>2</sub> reversible capacity of ~10 wt% H at 255°C after 516 cycles.
- Critical phase reaction pathways determined using progressive incremental loading of hydrogen in Li<sub>3</sub>N, supported by CALPHAD developed Li-N-H phase diagrams.
- Determined the formation of the Li<sub>4</sub>NH phase at room temperature; this phase is stable up to 5 wt% H and is a peritectically decomposed phase observed at 25°C.
- Details of the amount of LiH phase formed under equilibrium loading of hydrogen determined.
- New higher capacity cubic phase of Li<sub>2-x</sub>NH<sub>1+x</sub> type was found.
- Improved kinetics observed at higher pressures in the Li-N-H system.



## Introduction

Trace amount (ppm levels) of gaseous impurities in ultra-high purity (UHP) hydrogen may loss in hydrogen capacity in solid state storage of hydrogen in complex hydrides. To evaluate these effects we performed extrinsic thermal cycling with impure hydrogen; these tests are rather long. We also performed thermal aging where necessary. These tests are important for long-term reliability of metal hydrides that is important for working towards achieving DOE technical targets for a hydrogen economy. In general, hydrogen is used in research laboratory for the development of metal hydrides. However, the commercial hydrogen will always have some very low level impurities (ppm levels); typically  $O_2$ ,  $H_2O$ ,  $CH_4$ ,  $N_2$ , hydrocarbons, and others in hydrogen. Typical industrial grade hydrogen, that we used showed: "Hydrogen min % (v/v) 99%, water ~32 ppm,  $O_2$  ~10 ppm,  $N_2$  ~400 ppm, total hydrocarbons: 10 ppm,  $CO_2$  ~10 ppm,  $CO$  ~10 ppm, argon may be present, reads as oxygen." We also used special 100 ppm  $O_2$  mixed with UHP hydrogen in this study. These low level impurities affect the performance of hydrogen storage materials in the hydride beds that are installed in automobiles. The purpose of the pressure cycling experiments (with fresh charges of impure hydrogen) is to simulate the long-term performance of metal hydrides if there are trace amounts of impurities in the hydrogen. Parallel contamination studies are essential for evaluation impurity effect, before design of any hydride bed for a vehicular application containing complex hydride materials. The degradation of the hydriding properties are generally associated with disproportionation or decomposition of hydrides surface or bulk properties of the material during normal periodic charges of impure hydrogen. These studies are expected to lead to the understanding of mechanisms that occur after "hydrogen filling" in tanks numerous times. In another situation, for example, a hydride bed is left unused for a prolonged period of time, i.e., hydrogen is fully loaded in a hydride bed from a gas station, this type of test is called thermal aging; performed at elevated pressures and temperatures.

The *objective* of this research is to investigate the effect of trace impurities in complex hydrides which can be potentially used for fuel cell and other applications and aid in the development of materials solutions to potential hydrogen sorption degradation problems. The *relevance* to the Metal Hydride Center of Excellence (MHCoe) is that, trace impurities in hydrogen gas will have an impact on the hydrogen loading capacities during periodic recharging with fresh hydrogen in practical applications. These tests are being performed by accelerated laboratory testing which are extremely useful to understand the reliable use of hydrogen fuel.

The central problem is that  $Li_2NH$  will not reverse easily to either  $Li_4NH$  or  $Li_3N$ , at 255°C, leading to

approximately 5.8 wt% H reversible capacity. In addition, a LiH phase forms during hydriding of  $Li_3N$  which is difficult to reduce back to  $Li_3N$  at 255°C. However, Huq et al. [1] showed that at ~320°C decomposition of  $LiH \rightarrow Li_2NH$  was possible. To elucidate the various phase transition mechanisms we developed ternary phase diagrams of Li-N-H at various pressures and temperatures.

We also found that during repeated cycling there was a loss of nitrogen in the system [2]. The data indicated that during the desorption cycle (under dynamic vacuum) we were desorbing nitrogen and hydrogen or possibly LiN gas. Thus, we decided to use mixtures of  $N_2$ - $H_2$  to compensate for the lost nitrogen. Nitrogen compensation cycling results that show up to 10 wt% H reversibility are discussed.

## Approach

In this research, we are conducting experiments on cycling and aging tests of materials (as described in the above section) developed by the MHCoe partners. Pressure cycling apparatus was constructed at UNR for hydrogen loading and unloading. The overall approach to perform cycling on currently reversible complex hydrides such as imide/amide system (using  $Li_3N$  as precursor), Li amide-alanate that has theoretical hydrogen capacity ranging from ~7 to ~10.5 wt% hydrogen. The specific tasks were to determine the changes in the pressure-composition isotherm after cycling with impure hydrogen. A reference temperature of 255°C and a hydrogen pressure range from vacuum to 2 bar was chosen, based on pioneering research of Prof. Ping Chen's *Nature* paper [3].

We have studied the Li-N-H system in which an unusual improvement of hydrogen capacity is observed during pressure cycling at 528 K using mixed nitrogen/hydrogen gases. Without mixing in nitrogen, we observe a loss in hydrogen capacity, from ~10.2 to 3.1 wt% H after 501 cycles, during cycling due to the formation of a small amount of liquid lithium metal which forms a stable LiH phase during each cycle; this leads to a loss of nitrogen that significantly degrades the reversible hydrogen capacity of the  $Li_3N$ -H system. In this paper we report results from data obtained by prolonged pressure cycling of  $Li_3N$  with  $H_2$ -100 ppm  $N_2$  and 80/20  $H_2/N_2$  mixed gases. We observe a significantly enhanced reversible capacity of up to ~10 wt% H after 516 cycles with 80/20  $H_2/N_2$  mixtures, an improvement of ~7.1 wt% H as compared to using hydrogen with nitrogen additives. We attribute this enhancement to the reaction of nitrogen with liquid lithium during cycling as the Gibbs energies of formation of  $Li_3N$  ( $\Delta G^\circ = -100.16$  kJ/mol) are more negative than that of LiH phase ( $\Delta G^\circ = -50.74$  kJ/mol). We propose that there is continuous accumulation of the  $Li_2NH$  phase and minimizations of LiH formation as these materials

are subjected to many cycles. The cyclic behavior mechanism of  $\text{Li}_3\text{N}$  with  $\text{H}_2/\text{N}_2$  gas mixtures is quite a different phenomenon as compared to mechanisms occurring in classical hydrides.

## Results

In this study we present the results of hydriding of  $\text{Li}_3\text{N}$ , with no catalyst, under different conditions. In our previous work (Chien et al. Ref. [2]) the results from  $\text{Li}_3\text{N}$  and industrial hydrogen with ppm levels of impurities were discussed. It was found that during pressure cycling of  $\text{Li}_3\text{N}$  with  $\text{H}_2$  (under similar conditions as this study) there was a loss of nitrogen during cycling, and  $\text{LiH}$  formation increased with the number of cycles.

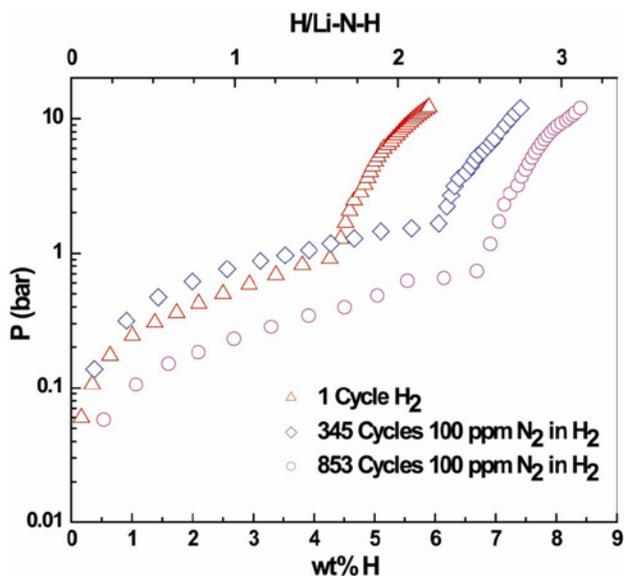
### Pressure Cycling With Industrial Hydrogen

Pressure cycling under hydrogen without nitrogen additions showed a steady decline of the hydrogen capacity from  $\sim 10.1$  to  $3.2$  wt% H after 501 cycles [2]. The mechanism suggested for this is the formation of  $\text{LiH}$  due to the loss of nitrogen, possibly in the form of  $\text{LiN}$  or  $\text{NH}_3$ , during the (dynamic vacuum) desorption cycle.

To mitigate the effect of lost nitrogen, we used mixtures of hydrogen and nitrogen to observe if the loss of hydrogen capacity was affected. Initially, we used 100 ppm levels of nitrogen in hydrogen. Then we increased the amount to 80/20  $\text{H}_2/\text{N}_2$  molar ratio, and finally to an excess of nitrogen 25/75  $\text{H}_2/\text{N}_2$ .

### Nitrogen Compensated Cycling Results

Initial experiments were performed by hydriding  $\text{Li}_3\text{N}$  with (2 bar) hydrogen mixed with 100 ppm nitrogen at 528 K followed by dynamic vacuum desorption for half hour each, in the cycling apparatus. The sample was then transferred to a Sieverts apparatus to obtain isotherms using UHP hydrogen gas after 345, and 853 cycles. Isotherms taken at 528 K and loading up to  $\sim 10$  bar hydrogen showed that the cycling with 100 ppm nitrogen improved the hydrogen capacity as illustrated in Figure 1. We include data from our previous work designated as “1 cycle  $\text{H}_2$ ,” which was taken after the “zeroth cycle.” When the hydrogen is added to  $\text{Li}_3\text{N}$  for the very first time, we refer to this as “zeroth cycle.” We obtained  $\sim 10.5$  wt% H capacity, similar to reported by Chen et al. [2]. Then during the very next cycle, referred to as “1 cycle  $\text{H}_2$ ,” the majority of the hydrogen could not be desorbed from the solids; only  $\sim 5.8$  wt% H is reversibly absorbed. This is due to fact that the rest of hydrogen is trapped in  $\text{LiH}$  and  $\text{Li}_2\text{NH}$  phase that did not revert back to the  $\text{Li}_3\text{N}$  phase at 528 K. This “1 cycle  $\text{H}_2$ ,” is used as a bench mark isotherm for comparison purposes. We

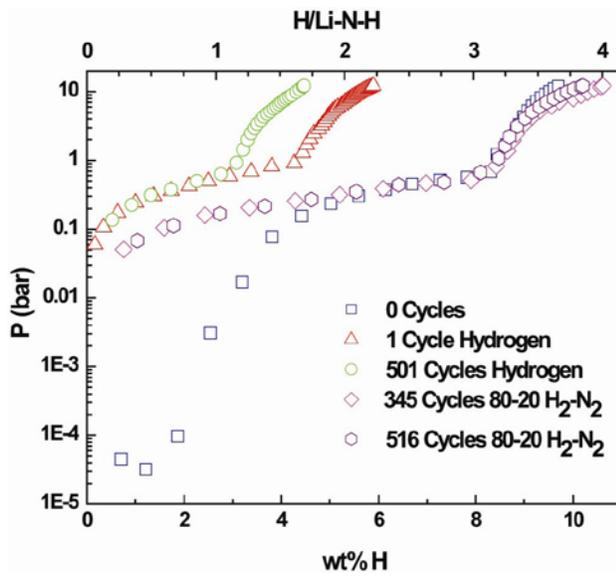


**FIGURE 1.** Pressure-composition isotherms obtained after 345, and 853 cycles using  $\text{H}_2$ -100 ppm  $\text{N}_2$  gas. For comparison 1 cycle  $\text{H}_2$  isotherm is included.

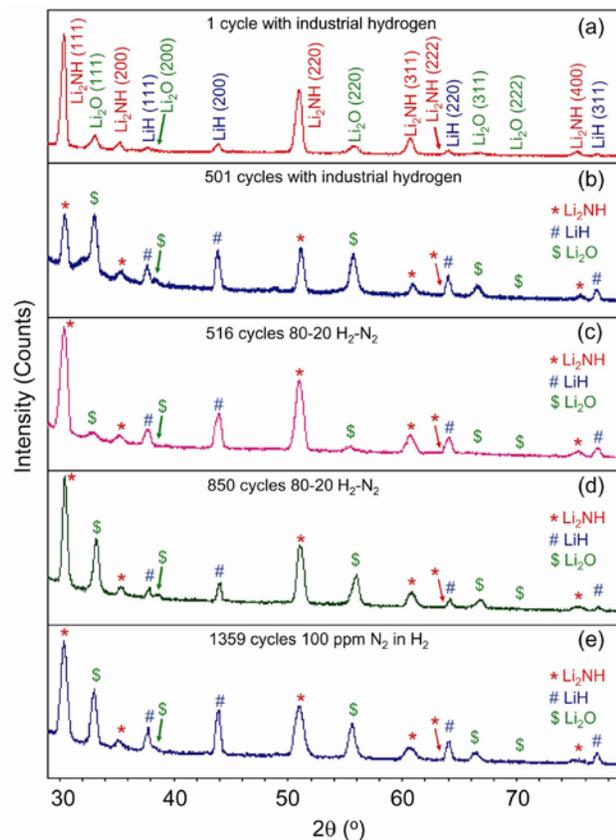
now compare results obtained by cycling  $\text{Li}_3\text{N}$  with  $\text{H}_2$ -100 ppm  $\text{N}_2$  mixed gases, up to 345 cycles, and noted an increase in capacity from 5.8 to  $\sim 7$  wt% H. A further increase in the number of cycles to 853 led to a further enhancement from  $\sim 7$  to  $\sim 8.5$  wt% H (Figure 1). This trend in the increase of the capacity as a function of cycles-is quite opposite to what we observed using hydrogen gas with no nitrogen.

In the next set of experiments, we increased the amount of nitrogen by mixing hydrogen with 20 mol% nitrogen (80/20  $\text{H}_2/\text{N}_2$  molar mixtures) for the reaction with  $\text{Li}_3\text{N}$ , and obtained an isotherm after 345 cycles as shown in Figure 2. The same sample was used to cycle up to 516 cycles whose results are also shown in Figure 2. In this case we include the data from “zeroth cycle,” from reference [2]. Results show that our 516 times cycled data with 80/20  $\text{H}_2/\text{N}_2$  is comparable with “zeroth cycle” data, showing  $\sim 10.5$  wt% H reversible absorption capacity.

Later, the samples from both the  $\text{H}_2$ -100 ppm  $\text{N}_2$  and 80/20  $\text{H}_2/\text{N}_2$  samples were subjected to 1,359 and 850 cycles respectively. We used the samples that were used earlier for these mixtures. However, the results showed a decrease in reversible capacity as compared to the “1 cycle” with industrial hydrogen (not shown, available from the principal investigator on request). Although reversible capacity is 7.2 wt% H after 1,359 cycles, these are better than “1 cycle” ( $\sim 5.8$  wt% H) with no nitrogen additions to hydrogen gas. The decrease at a high number of cycles is thought to be caused by oxygen contamination in the nitrogen gas.



**FIGURE 2.** Pressure-composition isotherms obtained after 345, and 516 cycles using 80/20 H<sub>2</sub>/N<sub>2</sub> mixed gas. For comparison zeroth, 1, and 501 cycle using hydrogen gas are included.

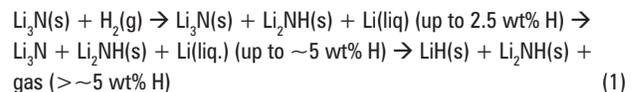


**FIGURE 3.** XRD patterns obtained using Cu-K $\alpha$  from the desorbed samples (a) 1 cycle industrial hydrogen (b) 501 cycles industrial hydrogen (c) 516 cycles with 80/20 H<sub>2</sub>/N<sub>2</sub> gas (d) 850 cycles with 80/20 (e) 1,359 cycles with H<sub>2</sub>-100 ppm N<sub>2</sub> gas.

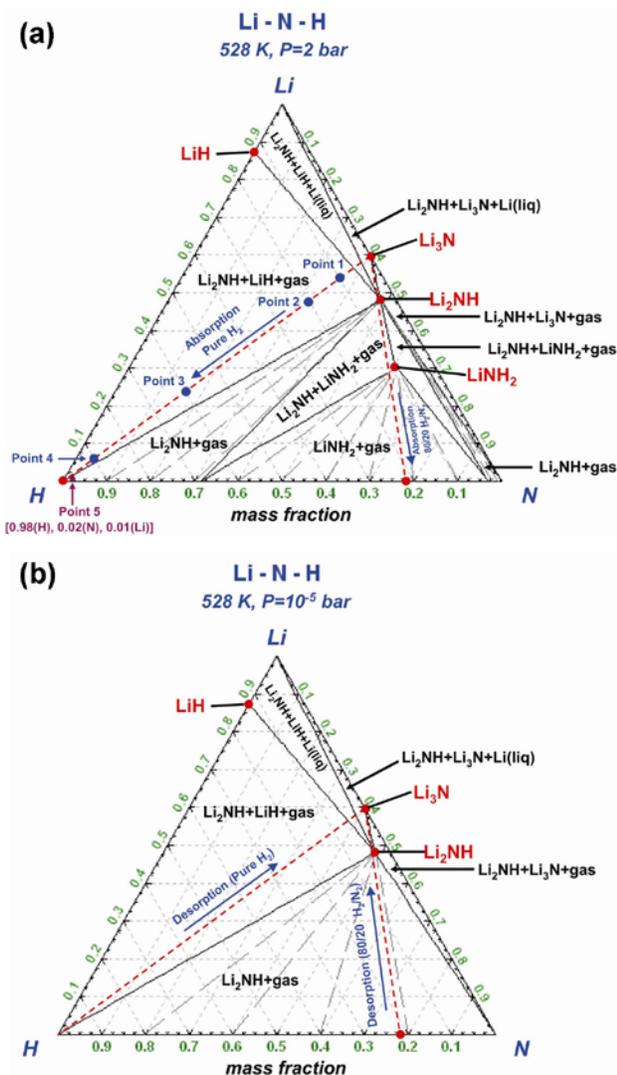
X-ray diffraction (XRD) patterns of the products from desorbed samples obtained from the Sieverts experiments, after cycling, are presented in Figure 3. In the XRD pattern after “1 cycle” with industrial hydrogen, we detected Li<sub>2</sub>NH as the major phase along with LiH and Li<sub>2</sub>O as minor phases. After 501 cycles with industrial hydrogen, we found a significant increase in the LiH and Li<sub>2</sub>O phases, and a decrease in the amount Li<sub>2</sub>NH phase (noting that the reversible hydrogen capacity dropped to ~ 3.8 wt% H) [2]. Next, we compare the 516 cycle product from the 80/20 data with the 501 cycle data of industrial hydrogen. It can be seen in Figure 4c that the amount of Li<sub>2</sub>NH is significantly higher than the 501cycled hydrogen and also the Li<sub>2</sub>O phase is lower in amount, as indicated by the increased intensities of the Bragg peaks. Figure 4 (d) show the XRD patterns from 850 cycled 80/20 H<sub>2</sub>/N<sub>2</sub> gases, and Figure 4(e) form the 1,359 cycle H<sub>2</sub>-100 ppmN<sub>2</sub>, taken after the desorption isotherm, yielding similar powder patterns with much higher amount of Li<sub>2</sub>O phase. It is possible that the increase in Li<sub>2</sub>O phase was responsible for the decrease in H-capacities after prolonged cycling. A plausible reason for this is accidental trapping of air in the volume between the valve and the sample holder of the Sieverts apparatus during switching from the cycling to Sieverts apparatus. But it is surprising and coincidental that both the high cycled materials show similar XRD patterns.

To understand this phenomenon of increased reversibility we constructed ternary Li-N-H phase diagrams using the CALPHAD approach. Results from the FactSage software are shown in Figure 4. The Li-N-H phase diagram at 528 K and 2 bar hydrogen is shown in Figure 4(a). During absorption of hydrogen along the “Absorption pure H<sub>2</sub>” line with the arrow pointing towards H, the phases formed in regions where the line intersects in the phase diagram progress as follows in Equation 1:

Absorption at 528 K, 2 bar H<sub>2</sub> (Figure 4(a)):



During desorption, along the H-Li<sub>3</sub>N in Figure 4(b) under dynamic vacuum we ideally can go back in a reverse fashion. From the experiments, we note that the reversibility is not complete, in other words there persists always LiH phase as Li<sub>2</sub>NH in the residual products. It can be noted that the maximum capacity reported so far is ~11 wt% H [2-6] in the Li<sub>3</sub>N-H system. The LiH phase contains ~12.4 wt% H. Theoretically, Equation 1 shows ~8.58 wt% H but the trapping of hydrogen in the LiH phase increases the amount of hydrogen to about 11 wt% H as reported by Chen et al. [3,7,8]. The XRD patterns in Figure 4 are all taken after cooling the



**FIGURE 4.** (a) Li-N-H ternary phase diagram showing various phase fields at 2 bar hydrogen absorption pressure used for cycling purposes with all the experiments performed in this set. It is expected that the reactions proceed along the line (a) starting from the point  $\text{Li}_3\text{N}$  to H (without nitrogen additions), (b) starting from  $\text{Li}_3\text{N}$  to point 0.14 wt. fraction nitrogen along the axes H-N ( $\text{H}_2$ -100 ppm  $\text{N}_2$ ), and (c) starting from point  $\text{Li}_3\text{N}$  to 0.78 nitrogen fraction along the axes H-N (80/20  $\text{H}_2/\text{N}_2$ ). (b) Li-N-H ternary phase diagram showing various phase fields during dynamic vacuum desorption cycle at approximate pressure 10-5 bar hydrogen. The desorption paths are reversed in this case as compared to the one in Figure 4(a).

sample to room temperature. At 528 K, when desorbed, the path followed is shown in the Figure 4(b).

## Conclusions and Future Directions

- Cycling lithium nitride with hydrogen containing 100 ppm nitrogen gas showed improved hydrogen storage properties over that cycled without the nitrogen addition. Specifically, the reversible

capacity approached ~10 wt% H after 516 cycles, and showed improved cycling stability. We propose that the ability of the nitrogen to have an effect in such small quantities is due to formation of  $\text{Li}_3\text{N}$  which transforms to  $\text{Li}_2\text{NH}$ . As the number of cycles increases, more  $\text{Li}_2\text{NH}$  is formed that leads to an increased hydrogen capacity.

- Cycling with hydrogen containing 20 mol% nitrogen was able to improve the cycling stability over a large number of cycles and allowed the lithium nitride to maintain a capacity close to its starting capacity. A reversible capacity of ~10 wt% at 255°C was observed up to 516 cycles. However, the capacity decreased to ~5.8 wt% H at ~10 bar pressure after 850 cycles; we attribute this to oxidation of the particles due possible entrapment of air during sample transfer.
- CALPHAD modeling showed formation of pure lithium, which reacts with nitrogen to form  $\text{Li}_3\text{N}$  in preference to  $\text{LiH}$ . The modeling also enabled us to determine the path of reaction at different temperature and pressures.
- In a practical sense, when we load the hydrogen mixed with nitrogen, in batches, in a hydride bed (as opposed to continuous supply) there is always partial pressure of nitrogen gas, but the majority of hydrogen gas is absorbed in the solid complex hydride phases.
- Isothermal data collected using a 25/75  $\text{H}_2/\text{N}_2$  mixture showed that these high nitrogen concentrations tend to stop hydriding processes prematurely, which is thus not desirable.

## FY 2010 Publications/Presentations

- J.-C. Crivello, M. Gupta, R. Černý, M. Latroche, and D. Chandra, "Density functional study of  $\text{Li}_4\text{NH}$  and  $\text{Li}_1.5\text{NH}_{1.5}$  as intermediary compounds during hydrogenation of  $\text{Li}_3\text{N}$ ," *PHYSICAL REVIEW B* 81, 104113, 2010.
- Dhanesh Chandra, Joshua Lamb, Wen-Ming Chien, "Effect of Gaseous Impurities on Long-Term Thermal Cycling/Aging Properties of Complex Hydrides for Hydrogen Storage" DOE - Sandia National Laboratory Program 2009 Review Meeting, December 15, 2009 at University of Utah, Salt Lake City, Utah.
- D. Chandra, "Extrinsic Effects of Impurities on Long-Term Behavior of Complex Hydrides and Phase Equilibria Studies" International Energy Agency Task 22 – Workshop IEA Task-22 Death Valley California April 11–15, 2010 Organizers: B. Hauback (Norway), Carol Read (US DOE), Channing Ahn (Caltech) Tom Autrey (PNL) USA.
- D. Chandra, J. Lamb, W-M Chien, N. Pal, J. Olsen, K. Yvon, R. Cerny, D. Phanon, and N. Penin, J-C Crivello, M. Latroche, M. Gupta, "Effect of Trace Elements on Long-Term Cycling/Aging Properties and Thermodynamic Studies of Complex Hydrides for Hydrogen Storage," 2010

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