

HYDRIDE DEVELOPMENT FOR HYDROGEN STORAGE

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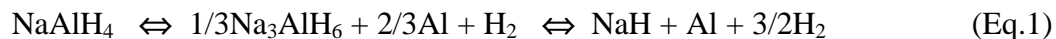
Abstract

The hydrogen storage properties of NaAlH_4 mechanically catalyzed with 2 mol % each of the liquid alkoxides $\text{Ti}(\text{O}i\text{Bu})_4 + \text{Zr}(\text{OPr})_4$ have been studied using a small (1.5 g sample) reactor and a 100 g “scaleup” reactor. The aim was to assess the potential for using this new hydride family for fuel-cell vehicle applications and to identify potential problems. Studies included low temperature equilibrium thermodynamic measurements, discharging and recharging kinetics and thermal effects, cyclic capacity, room temperature desorption kinetics, H_2 purity analysis and other practical properties. For the first time, low temperature equilibrium pressure measurements were made which showed that a hydrogen pressure of 1 atm can be obtained at 33°C by desorbing NaAlH_4 to Na_3AlH_6 . Also, recharging was possible at lower temperatures and pressures than generally reported. Cycling tests, however, indicated a much lower than expected capacity after the first desorption. The use of alkoxide-based Ti and Zr catalysts introduces oxygen and hydrocarbon impurities into the sample which were still present after several cycles and may contribute to the observed low reversible capacity. For this reason we have started to investigate inorganic catalysts as well as dry doping processes. Our latest results show reversible capacities close to the theoretical value of 5.6 wt.% (note added in proof).

Introduction

The need remains for safe, lightweight and compact means of hydrogen storage for vehicles. Liquid, compressed gas and reformer-derived hydrogen as well as metal hydride hydrogen storage systems all have well-known disadvantages. In the case of hydrides, metallic (interstitial) hydrides such as LaNi_5H_6 have excellent thermodynamic properties but suffer from low reversible gravimetric capacity (generally < 2 wt.% H). On the other hand, covalent hydrides such as Mg_2NiH_4 and other Mg-alloys offer good H-capacity (3-7 wt.%), but have unfavorable thermodynamics that require 250-300 C for positive desorption pressures. Overcoming this thermodynamic-capacity dilemma has been the hydride R&D objective of Sandia National Laboratories for the last few years.

Although alkali metal alanates have been known for many years as nonreversible “chemical hydrides” for the one-time hydrolysis generation of H_2 , recent demonstrations of catalyzed reversibility offer the distinct possibility of a new family of low-temperature hydrides. Bogdanovic et al (Bogdanovic' 1997) demonstrated that the well-known alkali metal alanate-complex hydride, NaAlH_4 , readily released and absorbed hydrogen when doped with a TiCl_3 or Ti-alkoxide catalysts according to the following two-stage reaction:



The net reaction represents a theoretical 5.6 wt.% reversible gravimetric H-storage, with the potential of liberating this hydrogen at less than 100 C. Such an achievement would be of significant practical value for on-board H_2 storage for a low-temperature fuel-cell vehicle.

There are two areas of R&D that are needed to develop Na-alanate into a practical vehicular system:

1. The optimization of the catalyst as to (a) type, (b) doping process and (c) mechanistic understanding.
2. Engineering development and determination of practical properties.

These are being addressed within the DOE Hydrogen Program by a cooperative effort between the University of Hawaii (with emphasis on area 1) and Sandia (with emphasis on area 2).

It is important to understand the history of catalyzed Na-alanate development and how it relates to the work to be presented here. The concept of catalyzed Na-alanate reversibility was first developed at the Max-Planck Institute für Kohlenforschung (Germany) using solution chemistry to add the catalyst and control the alanate particle size (Bogdanovic' 1997, 2000). The technique developed at Hawaii was quite different, with liquid Ti- and Zr-alkoxide catalysts added directly to the Na-alanate by mechanical ball-milling (Jensen 1999, Zidan 1999). Very recent work at Hawaii has also suggested that liquid TiCl_4 catalyst can also be added to NaAlH_4 by ball-milling (Jensen 2000). Ball-milling also offers the ability to control particle size and introduce solid catalysts or “activators”, such as carbon (Zaluska 2000). Sandia work with Ti- and Zr-alkoxide catalyzed NaAlH_4 (ball-mill-doped) began two years ago with detailed dynamic X-ray

diffraction studies to delineate the complex reaction sequence, along with some preliminary kinetic measurements (Gross 1999, Thomas 1999). During the last reporting year we continued with this same material with the aim of quantifying the engineering it's behavior, as well as identifying possible problems for the practical use of these materials.

There are a number of engineering problems that must be understood and/or solved before the catalyzed alanates can be applied to practical H-storage. For example, *low-temperature kinetics* may be slow because, unlike the interstitial hydrides, alanate H₂ absorption and desorption reactions take place by a series of disproportionation and repropotionation reactions (Eq.1) that must involve at least localized metal atom diffusion. Will low temperature kinetics be high enough for application in vehicles and other devices? Because of low kinetics, *low temperature equilibrium plateau pressures* have not been properly measured, causing us to rely on van't Hoff extrapolation from higher temperatures. A priority has been to gather experimental thermodynamic data. Most importantly, a *macroscopic* "engineering-scale" *catalyzed alanate bed* has never been made and tested. Are there any problems with large beds? What are the *reaction heat effects* (exothermic and endothermic)? Can the alanate bed be melted or sintered during exothermic charging and what are the resultant changes in gas impedance effects? What are the *volume changes* associated with hydriding and dehydriding? What effects on capacity and kinetics result from *cycling*? Do the liquid organometallic catalysts decompose in service, resulting in *contamination of the desorbed H₂*? These are the issues we address in this paper.

Experimental Basics

Most of the work reported here was done with NaAlH₄ to which was added 2 mol % each of Ti(OBuⁿ)₄ and Zr(OPr)₄ in liquid form by mechanical mixing. The use of this formula was based on the University of Hawaii observation that combined Ti- and Zr-doping resulted in maximum desorption kinetics for both steps of Eq.1 (Zidan 1999).

Hydrogen absorption/desorption studies were done using two experimental reactors. The one shown in Figure 1 represents what we call the "100 g scaleup reactor" designed to simulate the heat transfer and gas impedance conditions of a larger engineering bed. This 316 SS reactor has a 4.6cm OD, 0.38cm wall thickness, about 11.4cm internal length and was pressure rated to 3000 psia (204 atm) at 350 C. It was loaded with about 100 g of bicatalyzed NaAlH₄ as described above (78 g of NaAlH₄ and 22 g of Ti(OBuⁿ)₄ + Zr(OPr)₄). The NaAlH₄ was synthesized at University of Hawaii and blended with the catalysts at SNL using a few minutes ball-milling. The reactor had internal thermocouples but no internal heat exchange structure. The reactor was heated with an air furnace. Absorption kinetics and capacity were measured volumetrically and desorption measured with flow meters backed up by a wet test meter. The data below is presented in terms of wt.% H₂ normalized to the alanate weight only (i.e., not including the 22 wt.% catalyst).



Figure 1 – Scaleup (100 g) reactor with end cap removed in Ar-glovebox to show catalyzed alanate

Low temperature kinetics and plateau pressures were obtained volumetrically with a smaller 316 SS reactor (1.3 cm OD and 0.12 cm wall thickness) containing about 1.5 g of catalyzed sample. This reactor also had a fine internal thermocouple located in the middle of the bed. This unit was heated with electrical heating tape, and of course had better heat exchange than the scaleup reactor described above. This reactor was also used for lower pressure data (below 100 atm H₂). Other experimental details will be given with the data presentations.

Scaleup (100 g) Bed Results

The 100 g bed was evaluated through five instrumented absorption/desorption cycles. (The synthesis of the starting NaAlH₄ is artificially considered the first absorption half-cycle). Absorption H₂ pressures generally ranged from 100-200 atm and nominal absorption temperatures ranged from 125-165 C. Desorption was generally performed at 150-165 C against a 1 psig (1.07 atma) backpressure. In addition to capacity and rate measurements, exothermic (charging) and endothermic (discharging) thermal effects could be recorded. Exit hydrogen was monitored semiquantitatively for possible gaseous impurities. Samples were examined by XRD and SEM/EDS surface analysis. The alanate volume change was measured for the last desorption half-cycle.

Cyclic Capacity

The measured absorption and desorption capacities of the 100 g scaleup bed are shown as a function of cycle number in Figure 2. Generally good agreement was found between absorbed gas quantities and the amounts subsequently desorbed. However, cycle 1D (first discharge) showed a capacity of 6.0 wt.% H₂, in significant excess of the 5.6 wt.% theoretical value from Eq.1. This is believed to be an indication of substantial quantities of non- H₂ impurities coming out of the bed during the first desorption (see impurities discussion below). In cycle 2 only a partial absorption was made. The charge pressure used was 14 atm. This is well below the NaAlH₄ plateau pressure at 125 C and, hence, only the Na₃AlH₆ phase was formed.

Cycles 3-5 demonstrates a serious capacity problem encountered not only with the scaleup bed but with all other samples we have tested with the liquid alkoxide catalysts. Cyclic capacity (both absorption and desorption) was only about 3 wt.%, well below the 5.6 wt.% expected for this system. X-ray diffraction measurements clearly showed that the low capacity is due to an inability to completely recharge to 100% NaAlH₄ (Eq.1, full left reaction). There was no problem discharging to NaH+Al (Eq.1, full right reaction). A mechanism for this incomplete recharging phenomenon is uncertain at the present time, although it may involve the uniformity of catalyst distribution or impurity effects to be discussed later. This reversible capacity problem must be solved if this material is to be a viable candidate for hydrogen storage. (See Note added in Proof at the end of this paper)

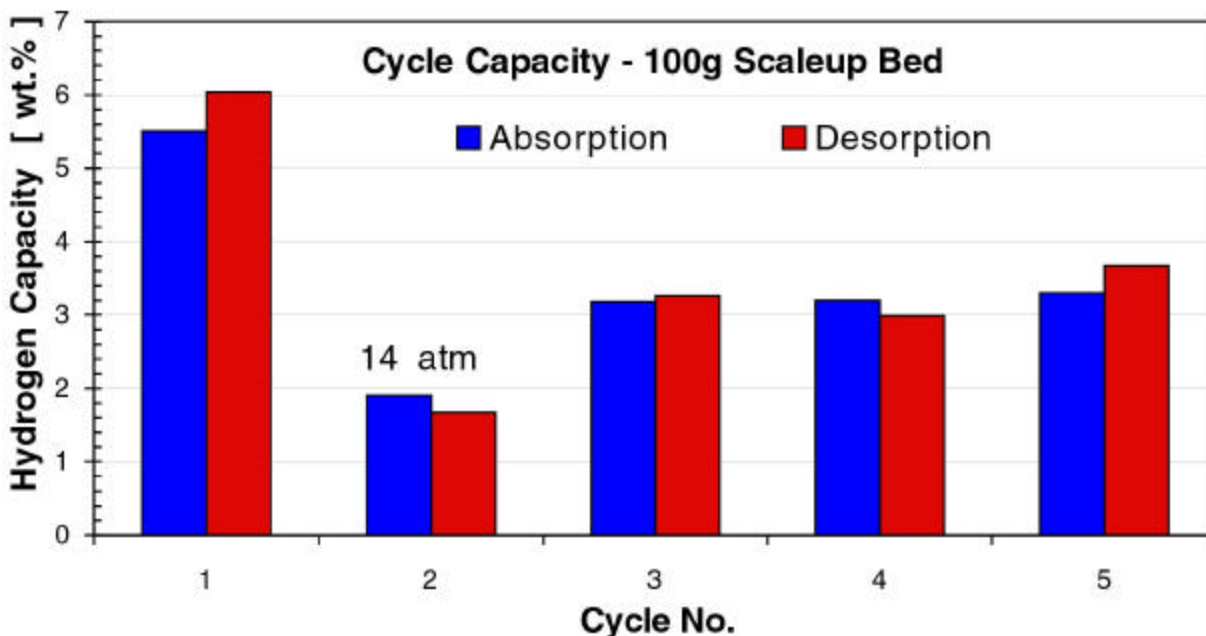


Figure 2 – Cyclic capacities of the 100 g scaleup bed.
Charging was done at 100-200 atm and 125-165 C,
except for cycle 2 which charged only through the low P phase.

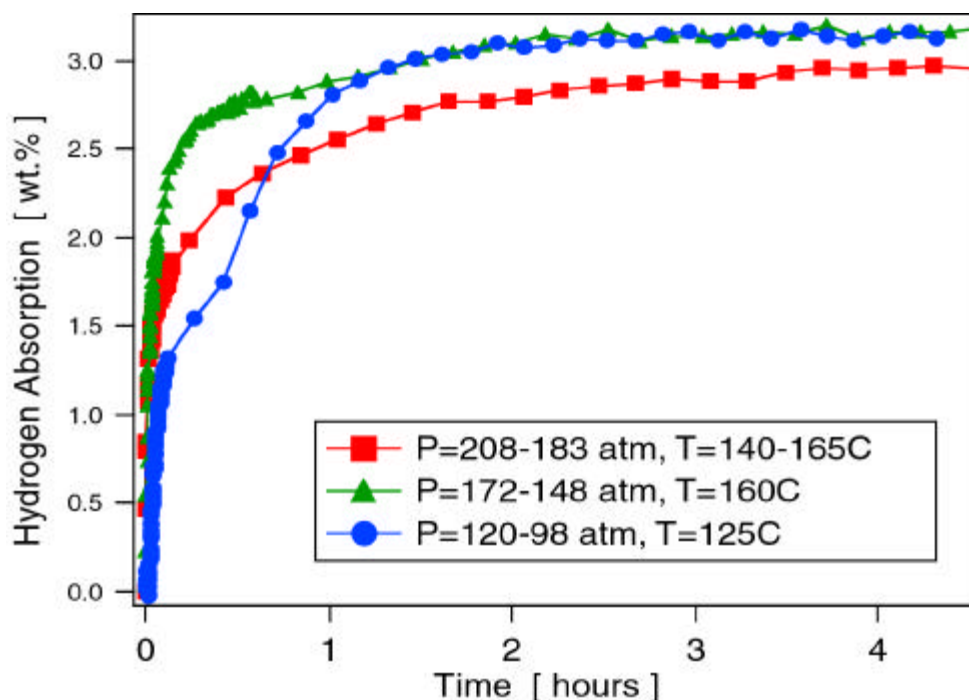


Figure 3 – Charging curves for the 100 g scaleup bed at various applied H₂ pressures and initial temperatures

Charging/Discharging Rates

Charging curves for half-cycles 3A-5A are shown in Figure 3. Although a range of applied pressures and starting temperatures were tried, in all cases charging was more rapid than we had expected from the literature and from University of Hawaii results, albeit with the lower than expected capacity discussed above. Most of the H₂ uptake was accomplished in the first hour or two, even for the lowest pressure and starting temperature. Although not shown, discharge was nearly as fast at 150-165 C; most of the charge was desorbed in about 3 hours. Using the small test bed (with better heat transfer), we have been able to achieve desorption times of 4-5 hours at 125 C. We need to achieve such rates at 100 C or less by improvements in particle size, catalyst and/or engineering design.

Thermal Effects

The higher than expected initial charging kinetics, combined with limited heat transfer, results in exothermic temperature excursions, as is common with all “fast” hydrides. An example is shown in Figure 4 for half-cycle 4A. At time zero, 172 atm H₂ was quickly applied to the fully dehydrided 100 g bed which had been equilibrated at 155 C. Within one minute, the exothermic hydriding reaction resulted in an internal temperature of 234 C. This is essentially the van’t Hoff temperature for NaAlH₄ at this applied pressure (see Figure 9, later). It is

important to recognize that the melting point of NaAlH_4 is only 182 C (Dymova 1974); thus, any NaAlH_4 formed during the first 0.5 hr of Fig.4 would do so directly into the liquid phase. As one can see that during the absorption (Fig.4), a 182 C thermal arrest occurs due to solidification during cooling (about $t = 0.6-0.8$ hr). This is precisely as expected and shows that liquid NaAlH_4 was formed during the exothermic temperature excursion associated with the rapid initial charge.

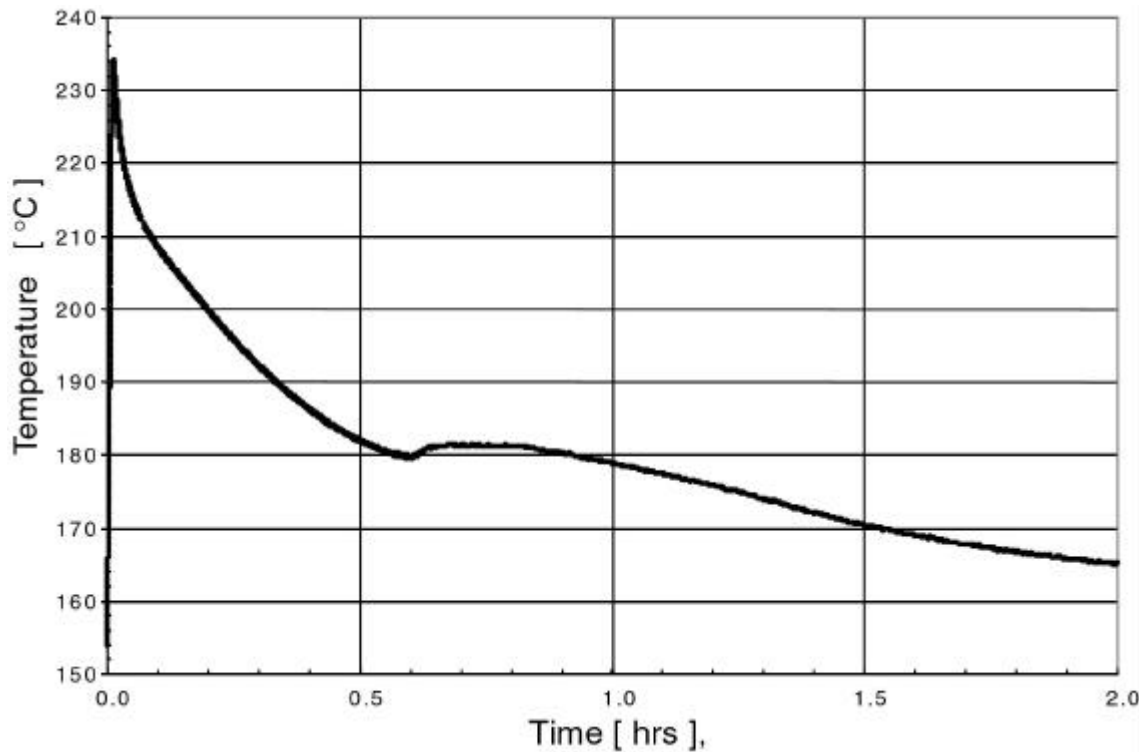


Figure 4 – Exothermic temperature excursion during scaleup bed charge half-cycle 4A ($P_i = 172$ atm, $T_i = 155$ C)

It is natural to ask if such melting could be detrimental to the subsequent performance of the alanate bed. The answer seems to be “no” or at least “not much”. We have not observed any negative effects of partially melting the bed at least three times. It is interesting to note that there was a furnace malfunction late in the half-cycle 3A that led to an increase in the temperature to 234 C with the material essentially in the fully charged state. This seemed to have had no significant negative effect on kinetics or capacity for subsequent half-cycle 3D, as well as cycles 4 and 5. In fact, there may be a benefit of partial melting. We opened the reactor between cycles 4 and 5 (the time at which the photograph shown in Fig.1 was taken) and found the bed to be sintered into a porous, solid mass. Such a structure may have distinct advantages for actual applications. In particular, such a sintered structure should reduce particulate migration, increase

packing densities, allow expansion, and provide a constant internal gas impedance and enhanced safety.

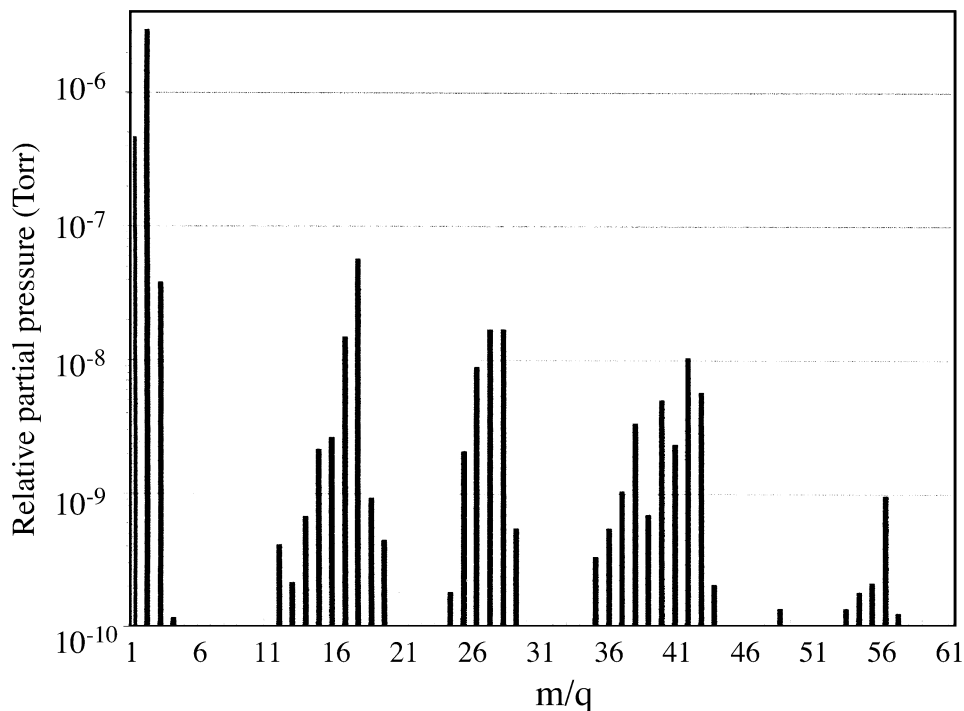


Figure 5 – Typical RGA pattern of H₂ desorbed from the Ti-Zr-alkoxide catalyzed NaAlH₄ 100 g scaleup bed

Gaseous Impurities

The direct addition of the catalysts in the form of liquid organometallic alkoxides (the University of Hawaii method) seems to result in serious problems relative to the purity of the desorbed H₂ and may be a contributing factor to the low cyclic capacity we see (cf. Figures 2 and 3). During the first desorption (1D) of the 100 g scaleup bed the measured quantity of gas indicated a capacity above the stoichiometric 5.6 wt.% H₂. Hence, during subsequent desorption half-cycles, semiquantitative analyses of the desorbed gas composition were done by RGA (Residual Gas Analysis [Mass Spectroscopy]) and occasionally by GC (Gas Chromatography).

A typical RGA pattern of the desorbed H₂ is shown in Figure 5. For pure H₂, one would expect only the mass 2 peak (with satellites m 1 and m 3), along with the ubiquitous background RGA peaks for H₂O (m 18,17,16), CO (m 28) and CO₂ (m 44). In fact, many more impurity peaks are present. A GC analysis taken of cycle 2D desorbed gas showed that butane (Bu) and propane (Pr) were present in the hydrogen. Both of those species can be deduced from the Fig.5 RGA pattern. Thus the Ti(OBuⁿ)₄ and Zr(OPr)₄ wet catalysts seem to be decomposing into O-free Bu and Pr. As shown in Figure 6, EDS (X-ray Energy Dispersive Spectroscopy) surface analysis clearly shows that most of the oxygen introduced by the Ti(OBuⁿ)₄ + Zr(OPr)₄ catalysts remain on the hydride surface after both stages of the desorption reaction (Eq.1). It may be that surface

damage associated with this oxygen is a contributing factor to the lower than stoichiometric cyclic capacity.

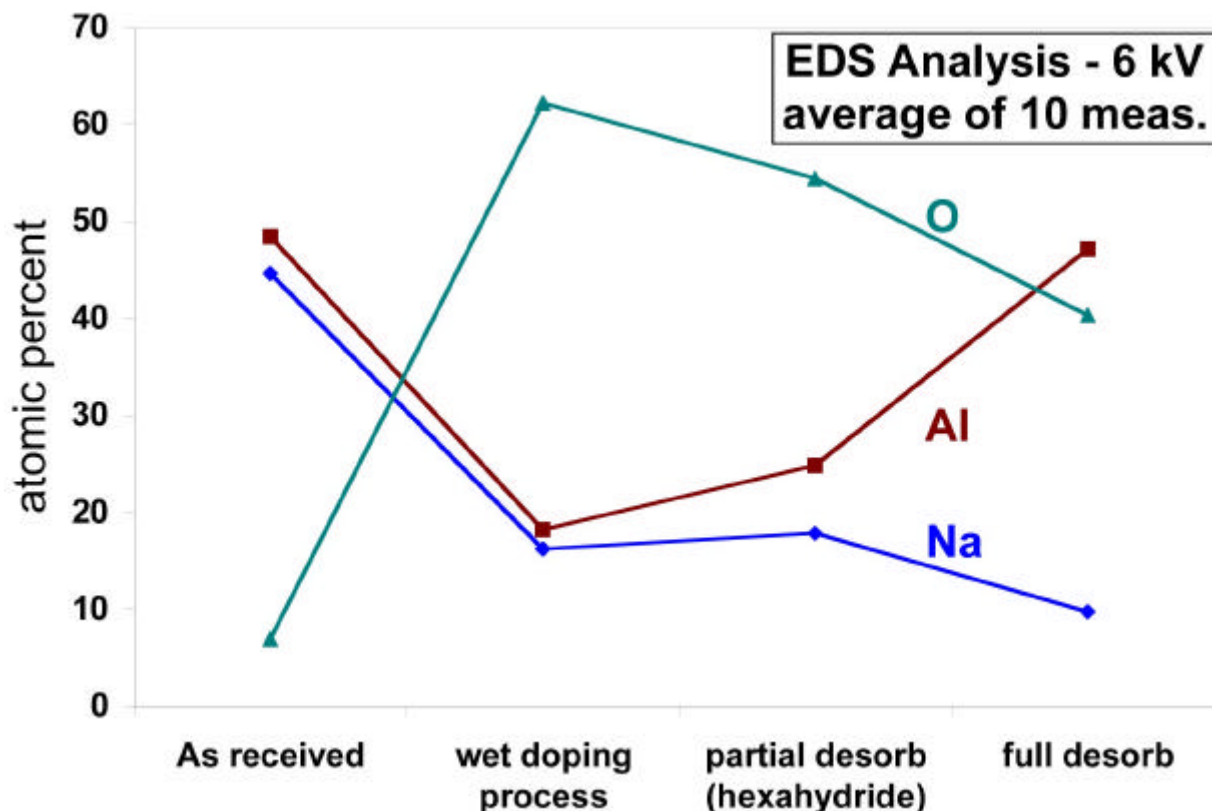


Figure 6– EDS analysis of NaAlH_4 sample before and after wet doping with Ti and Zr alkoxide catalysts, after desorbing to Na_3AlH_6 , and fully desorbing to NaH .

We believe the high level of impurities in the desorbed H_2 (not to mention possible surface damage from the catalysts) constitutes a fatal flaw in the University of Hawaii process of wet mechanical catalyst-alanate homogenization. One likely application for an onboard storage bed will be the PEM fuelcell which is not very tolerant of impurities in the H_2 fuel, especially carbonaceous species that might include or lead to CO. It might be argued that the wet-alkoxide-catalyzed beds can be cleaned up by vacuum baking and repeated cycling, but we feel this will be expensive and not easy. We observed significant impurity levels even after 5 cycles and temperature excursions above 200 C. Furthermore, if the capacity loss is really due to damage

by the catalysts, that problem will be particularly hard to surmount. Thus, we plan to direct our future work toward inorganic catalyst species.

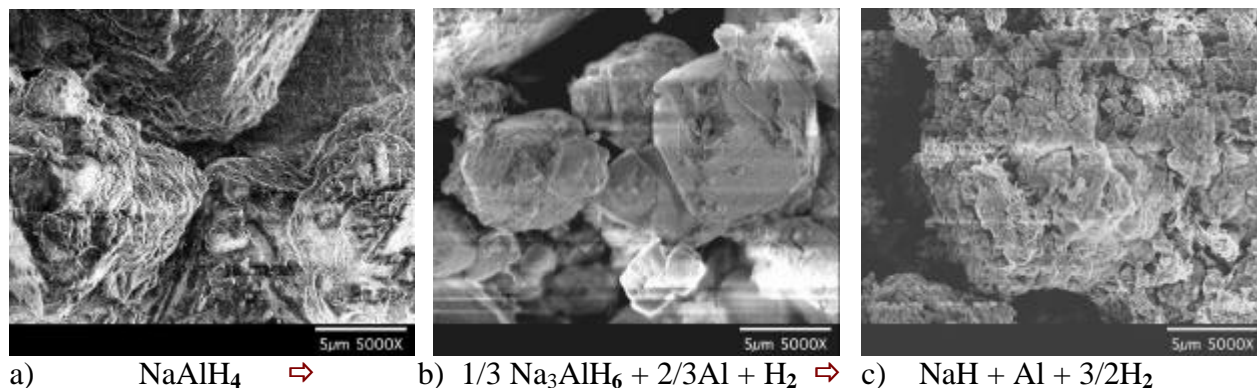


Figure 7 – SEM images showing the morphology changes during the two-step dehydriding reaction

Packing Densities and Hydriding Volume Changes

For the traditional metallic hydrides typically experience volume expansions on the order of 25% during hydriding. Because such large volume changes pose engineering problems (e.g., bed packing, expansion, deformation, etc.), it is important to know how the complex alanates behave. As shown by the SEM (Scanning Electron Microscopy) images in Figure 7, Na-alanate exhibits rather pronounced changes in morphology during the two stages of decomposition (Eq.1). From published X-ray densities the first stage of decomposition ($\text{NaAlH}_4 \rightarrow \frac{1}{3}\text{Na}_3\text{AlH}_6 + \frac{2}{3}\text{Al}$) would be expected to give a contraction of -30.3 % and the second stage ($\frac{1}{3}\text{Na}_3\text{AlH}_6 + \frac{2}{3}\text{Al} \rightarrow \text{NaH} + \text{Al}$) an expansion of 13.8 %. The net ΔV for the complete dehydriding reaction of Eq.1, including the excess Al produced in both stages, would then be expected to be -16.5 %. The actual volume change measured from the beginning to the end of the dehydriding cycle 5D was -14.7%. This large measured volume change is presumably due to the incomplete reaction to the final phases as shown by the low capacity (3.3 wt.%) of the bed. Unlike the traditional metallic hydrides, the alanates have the distinct advantage of starting with the material in it's fully hydrided state. This means that fully packed beds can be constructed which makes engineering the container easier and allows a higher volumetric density to be achieved compared to traditional hydrides. The aforementioned ability to liquid-phase-sinter the alanates into porous structures is also an engineering plus.

Results of Small Reactor Studies

We performed a number of parallel experimental studies on alkoxide catalyzed NaAlH_4 using the small 1.5 g reactor. In general, the kinetics and capacity data were very similar to that reported above for the 100 g scaleup bed and so it will not be presented here. We also did synthesis studies and testing of the Norway-Hawaii catalyst Ti_3Al (Maeland 1999) which is said to increase the rehydriding kinetics when added as a third component to the Ti-Zr-alkoxide bicatalyst system. The results of that work is included in the University of Hawaii contribution

to these Proceedings (Jensen 2000). The unique results obtained from the small reactor studies we report here are low temperature NaAlH_4 kinetics (especially room temperature) and low temperature NaAlH_4 van't Hoff (plateau pressure) data that were obtained experimentally for the first time.

Room Temperature Desorption Measurements

It was our original intent to perform detailed absorption and desorption kinetic measurements, starting at 100 C and down to the lowest measurable temperature. This effort was discontinued after our capacity and impurity observations suggested the liquid-alkoxide-catalyzed alanate system was probably not practical. Before that, however, we made the rather surprising observation that desorption kinetics could be measured even down to room temperature. Examples of room temperature desorption data are shown in Figure 8. As shown in Fig.8b, the initial kinetics are strongly dependent on particle size, as would be expected. The sample designated “large particles” represents the bicatalyzed sample used in the 100 g scaleup bed where the NaAlH_4 and catalysts were simply mixed with relatively brief ball-milling. The “fine particle” data were taken with an identical blend that was ball-milled for 3 hours (Spex-mill). There was nearly an order of magnitude difference in initial desorption kinetics between these two samples.

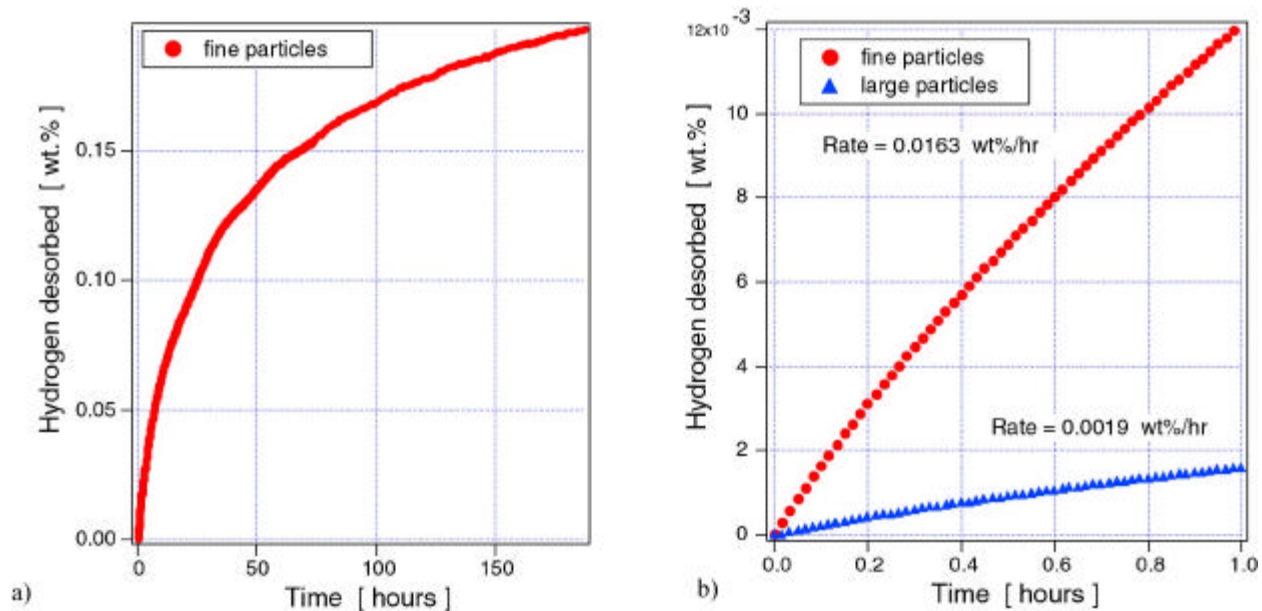


Figure 8 – Room temperature H_2 desorption from Ti- + Zr-alkoxide catalyzed NaAlH_4

We believe that, this is the first time that NaAlH_4 has been reported to decompose at room temperature, and that quantitative low temperature H_2 desorption kinetics have been measured. This has practical significance for applications. For example, high-capacity catalyzed- NaAlH_4 could be used for long-term, low-demand devices that use H_2 . Examples might be low-power remote fuel-cells or portable gas analyzers that use H_2 .

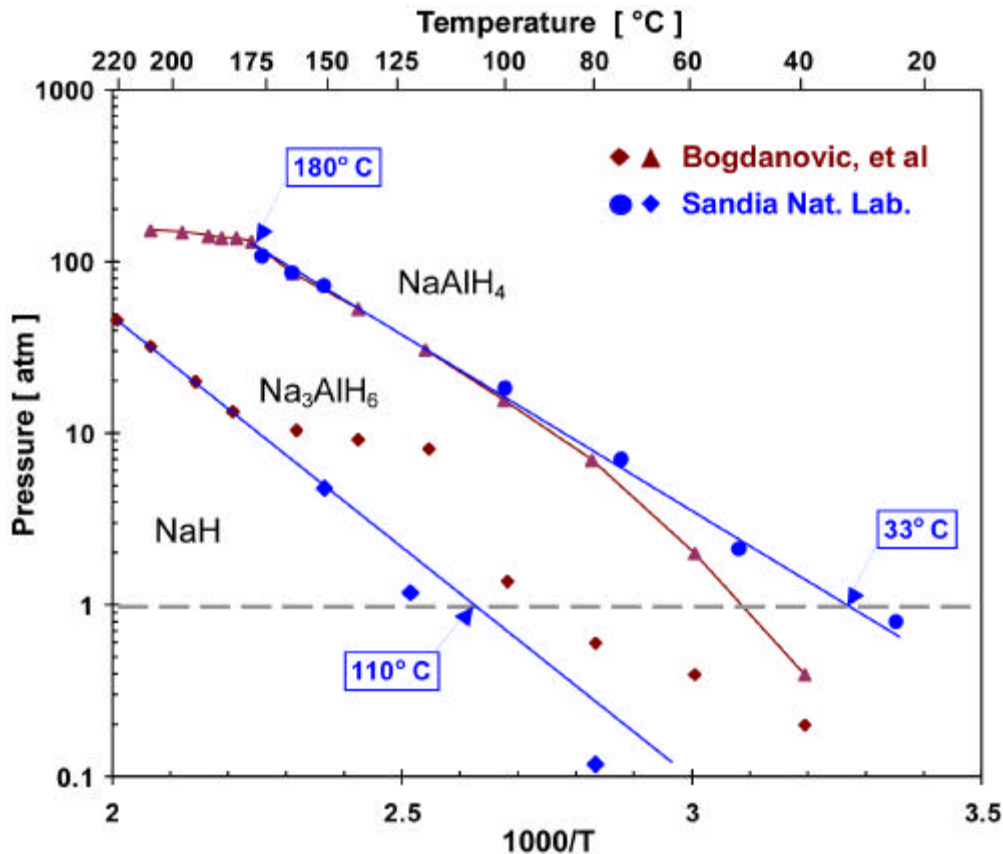


Figure 9 – Van't Hoff Diagram showing equilibrium pressures as a function of temperature for both hydride phases

Low Temperature van't Hoff Data

The reasonably high kinetics at low temperatures allowed the reliable determination of equilibrium desorption plateau pressures down to room temperature for the NaAlH₄ phase. These data are plotted in Figure 9 in the form of a van't Hoff plot (lnP vs 1/T) and compared to the data previously published by the Bogdanovic' and co-workers (Bogdanovic'2000). The low temperature SNL data looks more reasonable because it falls along the extrapolation of the high temperature data. It has now been accurately shown that the temperature for 1 atmosphere absolute desorption pressure is 33 C. This is a very convenient temperature for fuel-cell applications.

The Na₃AlH₆ plateau pressure is also shown in Figure 9. Included are the MPI data (Bogdanovic'2000) and the previously published SNL data (Thomas 1999). Because of the relatively low pressures and kinetics involved, the low-temperature Na₃AlH₆ van't Hoff data are not as reliable as the NaAlH₄ data. Be that as it may, the 1 atma van't Hoff temperature for Na₃AlH₆ is estimated to be about 110 C. This temperature is somewhat higher than desired for a PEM fuel-cell and future work will have to aim at lowering that temperature well below 100 C. This can be accomplished only by modification of the alanate thermodynamics.

Summary and Conclusions

Studies of a 1.5 g experimental and a 100 g scaleup bed of NaAlH₄ wet catalyzed with 2 mol % each of the alkoxides Ti(OBuⁿ)₄ + Zr(OPr)₄ have indicated some promising engineering properties:

1. H₂ absorption/desorption kinetics for the scaleup bed were surprisingly fast, at least at 125 C, and certainly good enough for some practical applications.
2. Rehydriding could be accomplished at lower pressures and temperatures than originally thought.
3. We were not been able to achieve full capacity after the first cycle. Instead of the 5.6 wt.% theoretical reversible H₂ capacity, we consistently achieved only about 3 wt.%.
4. The reason for the low H-capacity is associated with difficulty rehydriding all the Na₃AlH₆ to NaAlH₄.
5. NaAlH₄ has measurable desorption kinetics down to room temperature, and thus, may be useful in its present state for practical applications where H₂ rate demands are low.
6. Because of 5, we have been able to measure plateau pressures of NaAlH₄ down to room temperature and, therefore, experimentally complete the van't Hoff plot. The temperature required for 1.0 atma desorption plateau pressure is 33 C.
7. The initial absorption kinetics were so high that reaction self-heating was significant, easily exceeding the 182 C melting temperature of NaAlH₄. This means that, like all other known hydrides, adequate heat transfer will be important for achieving the best hydrogen absorption and desorption rates.
8. Partial melting of the alanate bed did not appear to hinder subsequent A/D kinetics. On the contrary, partial melting of the alanate results in a moderately sintered porous structure that effectively fixes the particulates, thus simplifying large bed design.
9. The A/D volume change of alanate beds is nearly zero which is much lower than conventional metallic hydrides. This also simplifies large bed design and allows far more material to be used in storage beds.
10. The current Ti+Zr alkoxide catalyst doping procedure has significant drawbacks:
 - A. The alkoxide based catalysts are too heavy. Standard loadings of 2 mol % each results in a 22 wt.% burden on the bed.
 - B. The liquid organometallic catalysts contribute substantially to hydrocarbon contamination of the exit H₂, in particular with butane and propane.
 - C. A large amount of oxygen from the alkoxide catalysts remains in the bed, perhaps damaging the bed and causing the less than ideal cyclic capacity.

Future Work

Our principal plan for the coming year is to continue work on the catalyzed complex hydrides, with an emphasis on moving these materials toward practical storage applications and devices. Our focus on advanced studies of the engineering properties of these materials will also be complimented by efforts to develop improved methods of materials preparation, as well as some mechanistic studies on the reaction processes. More specifically, we will focus on the following list of objectives:

1. Understand the factors that lead to the much lower than ideal capacity in the alanate beds.
2. Work with the University of Hawaii to optimize chloride-catalyzed beds.
3. Investigate other inorganic and non-chloride catalysts.
4. Complete engineering data (especially low-temperature kinetics) on an optimized inorganic catalyst-alanate system.
5. Develop substituted alanates and characterize their hydriding properties.
6. Begin safety studies and economic analyses.

Acknowledgements

This is an extension of work originally started at Sandia by S. Guthrie. We gratefully acknowledge all of his contributions and are saddened by his untimely death. We also acknowledge the valuable collaboration of C. Jensen and S. Takara of the University of Hawaii. We wish to thank D. Meeker of SNL for his expert technical help in all aspects of the experimental measurements.

Note added in proof:

Recent experiments using an inorganic catalyst have achieved reversible hydrogen capacities nearly at the theoretical limit with no degradation in kinetic properties or gas impurities. Reversible capacities in this material have been measured for a small number of cycles at this time. In addition, analysis of the material has identified the source of the small loss in capacity and work is in progress leading to the attainment of essentially full hydrogen capacity.

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