High Temperature-Pressure Processing of Mixed Alanate Compounds

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Abstract

Mixtures of light-weight elements and hydrides were investigated to increase the understanding of the chemical reactions that take place between various materials. This report details investigations we have made into mixtures that include $NaAlH_4$, $LiAlH_4$, MgH_2, Mg_2NiH_4, alkali(ne) hydrides, and early third row transition metals (V, Cr, Mn). Experimental parameters such as stoichiometry, heat from ball milling versus hand milling, and varying the temperature of high pressure molten state processing were studied to examine the effects of these parameters on the reactions of the complex metal hydrides.

Keywords: A. alloys, A. intermetallic compounds, B. chemical synthesis, C. X-ray diffraction.

1. Introduction

Solid state materials which store hydrogen with high gravimetric and volumetric densities are needed to develop a hydrogen economy for transportation applications. To this end, hydrogen containing solids of the light-weight elements are under intensive investigation.[1-6] Desirable materials properties include a high weight percent hydrogen (ideally $9 \geq wt\%$), rapid release and uptake of H_2 at pressures of 100 atm or less, and operation at fuel cell compatible temperatures ($T \leq 160\ °C$). This combination of properties is challenging to achieve in one material, and while there are a variety of materials that exhibit one or more of these properties, no material to date meets all of the criteria. For instance, magnesium is abundant, relatively inexpensive, and MgH_2 has a large weight percent hydrogen (7.9 %) but the temperature for hydrogen evolution is about 300 °C, and the kinetics of hydrogenation are too slow.[7] Titanium catalyzed $NaAlH_4$ is reversible and releases hydrogen at acceptable temperatures, but the kinetics are slow and the weight percent is mediocre (5.6 %).[8-10] One method of tailoring

properties is to combine two or more materials to create a new material with intermediate properties. One interesting example of this is Mg_2NiH_4, where the mixed metal hydride has a lower dehydrogenation temperature (~250 °C vs 300 °C for MgH_2) and better kinetics of hydrogenation. Researchers have investigated the effect of combining Mg_2NiH_4 with a variety of materials, e.g. transition metal oxides, Pd, and excess MgH_2, in order to improve properties.[11]

Even with the constraint of focusing on the light-weight elements, there is a wide variety of mixtures to be investigated with the goals of understanding the chemical reactions that take place between various materials, and possibly creating a new material with improved properties. This report details investigations we have made into mixtures that include $NaAlH_4$, $LiAlH_4$, MgH_2, Mg_2NiH_4, alkali(ne) hydrides, and early third row transition metals (V, Cr, Mn), where V is used in the hydrided form to compare the effect of transition metal hydride vs. bare transition metal.

2. Experimental

Mixtures were made by dry mixing in a Spex 8000 ball mill. The vial volume is 65 mL, and two 12.7 mm and four 6.4 mm balls was used for mixing. Mixture components and balls were loaded into ball mill vials under an inert atmosphere (Argon gas). Samples were typically milled for 60 minutes. Cold ball milling was performed by chilling the vial on dry ice for 5-10 minutes, then ball milling in 10 minute increments with chilling of the vial between each milling cycle. Hand milling was performed with an agate mortar and pestle under Argon atmosphere. X-ray diffraction (XRD) samples were pressed onto quartz plates, and sealed with a thin film of polyethylene under Argon atmosphere, then transferred to the diffractometer for analysis using Cu K_α radiation.

Molten state processing (MSP), an SRNL patented technique, was used to heat ball milled mixtures under high hydrogen gas pressure (usually 4500 psi) in order to synthesize possible new phases.[12, 13] The premise is that the high H_2 pressure will favor the formation of products with high hydrogen content, following Le Châtelier's principle. Vanadium forms hydrides rather easily, so vanadium powder was heated under vacuum/hydrogen cycles to create $VH_{0.81}$ for use in these experiments.[14]

3. Results and Discussion

Table 1 provides a comprehensive list of the various mixtures that were investigated in this study. The mixtures we have explored include $NaAlH_4$, $LiAlH_4$, MgH_2 and Mg_2NiH_4 as the primary materials, with addition of alkali(ne) hydrides, and/or early third row transition metals (V, Cr, Mn) and occasionally $TiCl_3$. The metals Cr and Mn were chosen because they do not easily form hydrides under the conditions of our experiment, so the effect of a transition metal in "metal" form could be examined. Vanadium, immediately to the left of Cr on the periodic table, does form a hydride so $VH_{0.81}$ was used to explore the difference in chemical reactions of a transition metal versus a transition metal hydride additive. Experimental parameters such as stoichiometry, temperature of ball milling, use of hand milling, and temperature of MSP were varied to study the effects of these parameters on the products. Starting materials are abbreviated SM for brevity in the table, and this indicates that all of the starting materials are seen in the XRD pattern of the indicated reaction step.

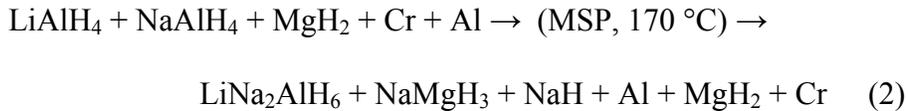### 3a. Mixtures of alanates and transition metals:

Mixtures with Cr metal:

The quaternary mixture, which has $NaAlH_4$, $LiAlH_4$, and MgH_2 mixed with Cr was first investigated with a simple 1:1:1:1 mixture. This treatment produced starting materials and aluminum. Based on the fact that the binary mixture of $LiAlH_4$ and various transition metals yields aluminum and starting materials, we conclude that some of the $LiAlH_4$ has decomposed with ball milling, which is known to occur.[15] The decomposition reaction is expected to take place as shown below in equation 1.
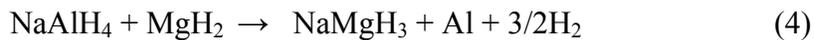
$$3LiAlH_4 \rightarrow Li_3AlH_6 + 2Al + 3H_2 \tag{1}$$

Note that only the Al product is seen in the XRD pattern, any Li_3AlH_6 produced does not show up in the XRD pattern, which is not uncommon as many of the expected Li containing species are not seen in XRD patterns of the products in this study. There are several possible reasons for the lack of XRD signal, including x-ray absorption by heavier elements, formation of nanoparticles, or formation of an amorphous phase.

When the mixture is further reacted with MSP conditions at 170 °C, the reaction yields mixed metal products as follows (XRD scans are qualitative, not quantitative, so this is not a balanced reaction equation, rather it is a list of products formed):

$$LiAlH_4 + NaAlH_4 + MgH_2 + Cr + Al \rightarrow (MSP, 170\ °C) \rightarrow$$

$$LiNa_2AlH_6 + NaMgH_3 + NaH + Al + MgH_2 + Cr \tag{2}$$

One can make assumptions about the reactions that took place to form this mix of products.

$$LiAlH_4 + 2NaAlH_4 \rightarrow Na_2LiAlH_6 + 2Al + 3H_2 \tag{3}$$

$$NaAlH_4 + MgH_2 \rightarrow NaMgH_3 + Al + 3/2H_2 \tag{4}$$

The product mixture contains NaH as well, which likely comes from the partial decomposition of Na_2LiAlH_6, as shown in reaction 5 below.

$$Na_2LiAlH_6 \rightarrow 2NaH + LiH + Al + 3/2H_2 \qquad (5)$$

No evidence of LiH is seen in the XRD pattern, though it is likely present unless the NaH is formed from the decomposition of a possible Na_3AlH_6 reaction intermediate. The NaH product is seen with quaternary mixtures containing Cr or Mn, which indicates that transition metals, when employed as metals, assist the partial decomposition of the alanates. When $VH_{0.81}$ is used as the quaternary element, the NaH is not seen. Additionally, with the $VH_{0.81}$ reactant, $LiAlH_4$ remains in the product mixture, while it does not for either Cr or Mn. This suggests that the $VH_{0.81}$ is not catalyzing the decomposition of the alanates relative to the Cr and Mn reactants, which are in the elemental state. The XRD patterns of the MSP product for mixtures containing Cr and $VH_{0.81}$ are shown in Figures 1 and 2. The XRD patterns do not show evidence of LiH formation, but because the Li containing products, especially in small quantities, are apt not to show up in XRD, the possibility that LiH is also present is not ruled out as the scattering from LiH is relatively weak compared to the other components of the sample.

These Cr mixtures were explored further by varying the ratio of starting materials away from the simple 1:1:1:1 mixtures that had been explored for the other transition metals. The Cr and the MgH_2 mol ratios were varied to see if this would change the reaction. When the Cr content was doubled, regardless of the MgH_2 content, ball milling formed starting materials and Al. When the mol ratio of Cr is increased to 4, ball milling alone causes the $LiAlH_4$ to fully decompose to Li_3AlH_6. This is a confirmation that the presence of excess Cr catalyzes the decomposition of $LiAlH_4$, which is expected from a transition metal.

The temperature of ball milling is another experimental parameter that was varied. A typical 60 minute ball milling reaction creates enough heat to warm the vials to approximately 60 °C. In order to separate the effect of mechanical mixing from the effect of heating the mixture during mixing, the 1:1:1:1 Cr mixture was ball milled "cold" by cooling the vial every 10 minutes for a total of 60 minutes of milling; the vials did not exceed room temperature with this method. The cold ball milling prevented the decomposition of $LiAlH_4$ and only the starting materials were seen in the XRD pattern, where regular ball milling shows an XRD pattern with starting material and aluminum. The cold ball milled mixture, and the regular ball milled mixture, were further reacted under MSP conditions at a low temperature of 60 °C (usually done at 170 °C or higher). The low temperature MSP yields SM, Al, and Li_3AlH_6 regardless of ball milling conditions. Note that in this case the Li_3AlH_6 is present in the XRD pattern. The Al peak is much more intense after the MSP step versus after the ball milling step, so presumably the 3 hour heating provides energy for crystallization of the products allowing Li_3AlH_6 to be seen, and/or the quantity of products has increased with heat, to a level of Li_3AlH_6 detectable by XRD.

Mixtures with Mn metal:

The quaternary mixture of both alanates, MgH_2 and Mn is interesting in that it yields $MnH_{0.07}$ with just ball milling, in addition to the non-Mn starting materials and Al. The H is assumed to originate with $LiAlH_4$ that decomposes with ball milling. When this mixture is heated for MSP, the $MnH_{0.07}$ decomposes leaving Mn, in addition to MgH_2, Al, Na_2LiAlH_6, $NaMgH_3$, and NaH.

Mixtures with $VH_{0.81}$:

The quaternary mixture of $NaAlH_4$:$LiAlH_4$:MgH_2:$VH_{0.81}$ 1:1:1:1 forms starting materials and Al with ball milling. The MSP reaction of this mixture at 170 °C results in the formation of starting materials $LiAlH_4$, MgH_2 and $VH_{0.81}$ in addition to Al, Na_2LiAlH_6 and $NaMgH_3$.

There is clearly a difference in behavior between the Mn and Cr metal samples, and the $VH_{0.81}$ hydride samples. We see that the transition metals assist in the partial decomposition of the alanates.

3b. Mixtures of alanates and Mg_2NiH_4:

The mixed metal hydride Mg_2NiH_4 contains the transition metal Ni, which assists in the decomposition of this material relative to MgH_2, much as the transition metals assist in the decomposition of the alanates. The Mg_2NiH_4 was ball milled with 1)$LiAlH_4$, 2)$NaAlH_4$, and 3)$NaAlH_4$ with 4 mol% $TiCl_3$ to explore the affect of the Ni component on the alanate materials. The mixture with $LiAlH_4$ results in the starting Mg compound completely decomposing with ball milling to form 4 products: MgH_2, Mg_2NiH, and $Mg_2NiH_{0.3}$ and $Al_{1.1}Ni_{0.9}$. Apparently the Mg_2NiH_4 has partially disproportionated to MgH_2 and Ni, and presumably the Ni has catalyzed the decomposition of $LiAlH_4$ resulting in LiH and Al formation, where the Ni and Al go on to form the $Al_{1.1}Ni_{0.9}$ alloy.

The ball milled mixture with $NaAlH_4$ has only one product in common with the $LiAlH_4$ mixture, and that is $Mg_2NiH_{0.3}$. There are also starting materials present after ball milling, as well as the previously known mixed metal $NaMgH_3$. When 4 mol% $TiCl_3$ is added in addition to $NaAlH_4$, $Mg_2NiH_{0.26}$ is formed along with $NaMgH_3$ and Al, and starting materials remain. A likely reaction path is that Ti catalyzes the dehydrogenation of $NaAlH_4$ in the ball mill vial, and the NaH formed reacts with MgH_2 to form the

NaMgH$_3$. If the mixture with TiCl$_3$ is hand ground in a mortar and pestle (much lower energy than a ball mill) the mixed metal hydride NaMgH$_3$ does not form, which suggests that the energy and/or heat associated with ball milling is required for this reaction to take place.

3c. Mixtures of LiAlH$_4$ and binary hydrides:

Mixtures of LiAlH$_4$, 4 mol% Ti, and the binary hydrides 1) KH, 2) MgH$_2$ and 3) CaH$_2$ were investigated. Ball milling the mixture with KH produced two ion exchange compounds, KAlH$_4$ and K$_3$AlH$_6$, and left unreacted LiAlH$_4$ which continued the ion exchange with MSP. Ball milling with MgH$_2$ causes the LiAlH$_4$ to completely decompose to form Li$_3$AlH$_6$ which is a good indicator that the MgH$_2$ has a catalytic effect on LiAlH$_4$ decomposition. The combination containing CaH$_2$ produced starting materials, Li$_3$AlH$_6$, and Al with ball milling, indicating that the LiAlH$_4$ partially decomposes while the CaH$_2$ does not react. When heated with MSP, this mixture forms CaH$_2$, Al, LiCl and LiH, which are expected products from the decomposition of LiAlH$_4$ and reaction of the chloride from TiCl$_3$ with Li decomposition products.

3d. Mixtures of Mg$_2$NiH$_4$ and binary hydrides:

The mixed metal compound Mg$_2$NiH$_4$ was ball milled with 1)LiH and 2)NaH. In both cases, the mixtures yielded starting materials, MgH$_2$, and Mg$_2$NiH$_{2.6}$, and in the case of LiH, some Mg was present as well.

4. Conclusion

A wide variety of hydride mixtures, with and without light transition metal additives, were investigated. This work provides insight into the chemical reactions that take place between four hydride materials that are possible contenders for transportation

applications, if the systems can be fine tuned to overcome temperature, kinetic or weight percent limitations.

The addition of transition metal elements to $NaAlH_4$ does not create mixed metal alanates - no reaction is seen with ball milling, and MSP generally produces a decomposition product, Na_3AlH_6. However, the addition of elements or other hydrides destabilizes $LiAlH_4$ – with the elements Al is seen in the XRD pattern, while mixtures with binary hydrides produce the hexahydride, Li_3AlH_6 (or K_3AlH_6 in the case of KH).

$NaAlH_4$ in combination with Mg materials often produces $NaMgH_3$. When the Mg compound used is Mg_2NiH_4, $NaMgH_3$ forms with just ball milling. Mixtures with MgH_2 do not form the mixed metal hydride unless heated above 60 °C in the MSP step. Two explanations for this are likely, first that the presence of Ni catalyzes the decomposition of $NaAlH_4$, and second that Mg_2NiH_4 is less kinetically stable, and therefore more reactive, than MgH_2. The temperature effect is seen in the hexahydride product as well, but for a different reason. Mixtures containing $LiAlH_4$ and $NaAlH_4$ form mixed metal Na_2LiAlH_6 with MSP of 170 °C or higher, while Li_3AlH_6 is formed with MSP at 60 °C rather than the mixed metal product. The difference in products is likely due to the fact that solid state diffusion is relatively slow and $LiAlH_4$ melts at 125 °C so the higher temperature reactions have one molten component, greatly aiding diffusion.

Quaternary mixtures lead to known mixed metal products Na_2LiAlH_6 and $NaMgH_3$. When the transition metal additive is Cr or Mn, no residual $LiAlH_4$ reactant remains but residual MgH_2 does, while the transition metal additive $VH_{0.81}$ leaves $LiAlH_4$ reactant remaining in the product mixture. This indicates that the limiting $NaAlH_4$

reagent preferentially reacts with LiAlH$_4$ in the presence of Cr and Mn, which are active transition metal catalysts, relative to VH$_{0.81}$.

5. Acknowledgement

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Table 1: Mixtures of materials investigated.

Compounds	mol ratios	prep cond	MSP cond (°C-psi H_2-time)	BM Results	MSP Results
$NaAlH_4$:$LiAlH_4$:Cr	1:1:1	Spex BM 60 min	170-4500-2h	SM + Al	$NaAlH_4$, Cr, Na_2LiAlH_6, Al, NaH
$NaAlH_4$:$LiAlH_4$:Mn	1:1:1	Spex BM 60 min	190-4500-2h	SM + Al	Mn, Na_2LiAlH_6, Al, NaH
$NaAlH_4$:$LiAlH_4$:Mn	1:1:1	Spex BM 60 min	170-4500-2h	SM + Al	Mn, Na_2LiAlH_6, Al, NaH
$NaAlH_4$:$LiAlH_4$:MgH_2:$VH_{0.81}$	1:1:1:1	Spex BM 60 min	170-4500-2h	SM + Al	$LiAlH_4$:MgH_2:$VH_{0.81}$, Al, Na_2LiAlH_6, $NaMgH_3$
$NaAlH_4$:$LiAlH_4$:MgH_2:Cr	1:1:1:1	Spex BM 60 min	170-4500-2h	SM + Al	MgH_2, Cr, Al, Na_2LiAlH_6, $NaMgH_3$, NaH
$NaAlH_4$:$LiAlH_4$:MgH_2:Cr	1:1:1:1	Spex BM 60 min	60-4500-3h	SM + Al	SM + Al, Li_3AlH_6
$NaAlH_4$:$LiAlH_4$:MgH_2:Cr	1:1:1:1	cold ball mill	60-4500-3h	SM	SM + Al, Li_3AlH_6
$NaAlH_4$:$LiAlH_4$:MgH_2:Cr	1:1:1:2	Spex BM 60 min		SM + Al	
$NaAlH_4$:$LiAlH_4$:MgH_2:Cr	1:1:1:4	Spex BM 60 min		$NaAlH_4$, MgH_2, Cr, Al, Li_3AlH_6	
$NaAlH_4$:$LiAlH_4$:MgH_2:Cr	1:1:2:2	Spex BM 60 min		SM + Al	
$NaAlH_4$:$LiAlH_4$:MgH_2:Cr	1:1:2:1	Spex BM 60 min		SM + Al	
$NaAlH_4$:$LiAlH_4$:MgH_2:Mn	1:1:1:1	Spex BM 60 min	170-4500-2h	$NaAlH_4$:$LiAlH_4$:MgH_2 $MnH_{0.07}$, Al	MgH_2, Mn, Na_2LiAlH_6, $NaMgH_3$, Al, NaH
$NaAlH_4$:$VH_{0.81}$	1:1	Spex BM 60 min		n/a - outgasses	
$NaAlH_4$:Cr	1:1	Spex BM 60 min	190-4500-2h	SM	SM, Na_3AlH_6
$NaAlH_4$:Mn	1:1	Spex BM 60 min	190-4500-2h	SM	SM, Na_3AlH_6
$LiAlH_4$:Cr	1:1	Spex BM 60 min	170-4500-2h	SM + Al	SM + Al, Li_3AlH_6
$LiAlH_4$:Mn	1:1	Spex BM 60 min	170-4500-2h	SM + Al	Mn, Al
$LiAlH_4$:KH:$TiCl_3$	1:2:.04	BM 40 min		$LiAlH_4$, $KAlH_4$, K_3AlH_6	$LiAlH_4$, $KAlH_4$, K_3AlH_6, KCl,
$LiAlH_4$:MgH_2:$TiCl_3$	1:1:.04	BM 40 min		MgH_2, Li_3AlH_6, Al, LiCl, Mg	MgH_2, Al, LiCl
$LiAlH_4$:CaH_2:$TiCl_3$	1:1:.04	BM 40 min		SM, Li_3AlH_6, Al	CaH_2, Al, LiCl, LiH
$LiAlH_4$:Mg_2NiH_4	1:1			MgH_2, Mg_2NiH, $Mg_2NiH_{0.3}$, $Al_{1.1}Ni_{0.8}$	
$NaAlH_4$:Mg_2NiH_4	1:1			SM, $NaMgH_3$, $Mg_2NiH_{0.3}$	
$NaAlH_4$:Mg_2NiH_4:$TiCl_3$	1:1:.04	mortar pestle		SM, MgH_2, $Mg_2NiH_{0.3}$	
$NaAlH_4$:Mg_2NiH_4:$TiCl_3$	1:1:.04	BM 40 min		SM, Al, $NaMgH_3$, $Mg_2NiH_{0.26}$	
Mg_2NiH_4:NaH	1:1.2	mortar pestle		SM, MgH_2, $Mg_2NiH_{.26}$	
Mg_2NiH_4:LiH	1:9.9	BM 40 min		SM, MgH_2, $Mg_2NiH_{.26}$	

Figure 1: XRD pattern of the 170 °C reaction of the quaternary mixture
$NaAlH_4$:$LiAlH_4$:MgH_2:Cr

[2006091401-UT1b.RAW] NaAlH4:LiAlH4:MgH2:Cr 1:1:1:1 MSP 170C 2h 4500psi Scott

00-004-0787> Aluminum - Al
00-012-0697> MgH₂ - Magnesium Hydride
00-006-0694> Chromium - Cr
00-042-0848> AlH₆LiNa₂ - Lithium Sodium Aluminum Hydride
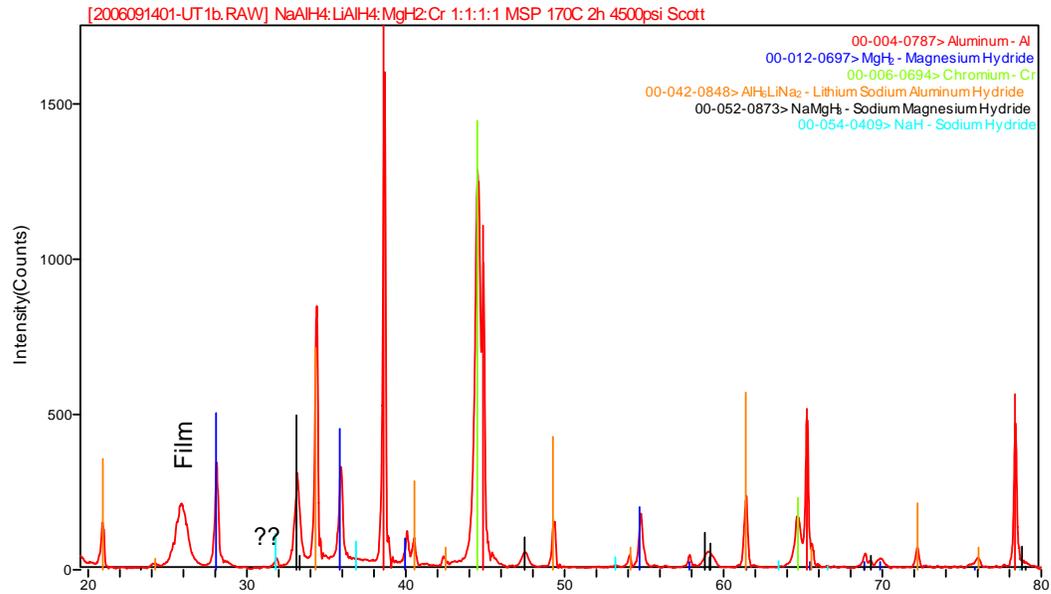00-052-0873> NaMgH₃ - Sodium Magnesium Hydride
00-054-0409> NaH - Sodium Hydride

Figure 2: XRD pattern of the 170 °C reaction of the quaternary mixture
$NaAlH_4$:$LiAlH_4$:MgH_2:$VH_{0.81}$

[2006102003-UT.xrdml] NaAlH4:LiAlH4:MgH2:VHx 1:1:1:1 MSP 170C 2H 4500 psi Scott

04-003-2522> MgH₂ - Magnesium Hydride
04-002-6895> Al - Aluminum
00-042-0848> AlH₆LiNa₂ - Lithium Sodium Aluminum Hydride
01-070-6365> NaMgH₃ - Sodium Magnesium Hydride
00-039-1171> VH₀.₈₁ - Vanadium Hydride
00-047-1156> LiAlH₄ - Lithium Aluminum Hydride