Thermodynamics, Kinetics and Modeling Studies of KH- RbH- and CsH-Doped

2LiNH₂/MgH₂ Hydrogen Storage Systems

Jalaal Hayes^a and Andrew Goudy^{b*}

Department of Chemistry

Delaware State University

1200 N. DuPont Highway, DE 19901.

^a: jalaal.hayes@gmail.com, and ^b: agoudy@desu.edu

*Corresponding Author - Phone: 302-857-6534, Fax: 302-857-6539

ABSTRACT

In this study, the effects of several alkali metal hydride dopants on the thermodynamics and kinetics of the 2LiNH₂/MgH₂ system were determined. The results showed that the stabilities of the doped 2LiNH₂/MgH₂ system are in the order: KH < RbH < CsH.

Kinetics measurements showed that the absorption and desorption rates are in the order: RbH > KH > CsH, with absorption rates being about twice as fast as desorption from the corresponding materials. As expected, the activation energies for the reactions were in the order: RbH < KH < CsH with the activation energies for absorption being less than that for the corresponding desorption reaction. Modeling studies revealed that desorption reactions are controlled by diffusion during the entire process. However, for absorption reactions the rate-controlling process changed during the course of the reactions. The

rate-controlling process in the first 70% of the absorption reactions was reaction at the phase boundary whereas diffusion controlled the rate in the latter stages.

1. Introduction

Lithium amide (LiNH_2) and its modifications are promising hydrogen storage materials for on-board vehicular applications because of their relatively high theoretical hydrogen storage capacities and low cost [1-5]. It has been found that the partial substitution of Li in LiNH_2 by Mg greatly improves its thermodynamic properties [6]. Equations (1) and (2) show the chemical reactions for the $2\text{LiNH}_2/\text{MgH}_2$ system that involve hydrogen absorption and desorption.

The first step in Equation (1) produces $\text{Mg}(\text{NH}_2)_2$ irreversibly. The $\text{Mg}(\text{NH}_2)_2$ is then involved in the reversible absorption and release of hydrogen in Equation (2). Scientists have found that hydrogen can be released from this system at an onset temperature of 130 °C, which is considerably lower than that for other complex hydrides such as borohydrides. However, this operating temperature and its relatively slow kinetics are still not sufficient to meet DOE's goals for hydrogen storage. Therefore efforts have been made to develop dopants that would lower the operating temperature even further and improve kinetics. A number of dopants have been studied [7-10] but the most effective ones to date have been those based on alkali metal hydrides. P. Chen and

colleagues [11] observed that potassium hydride had a great effect thermodynamically and kinetically when introduced into the $2\text{LiNH}_2/\text{MgH}_2$ hydrogen storage system. Scientists such as Luo et al [12] and Durojaiye et al [13] also studied the KH-doped lithium amide-magnesium hydride hydrogen storage system. They found that potassium hydride is a more effective dopant for the $2\text{LiNH}_2/\text{MgH}_2$ (2:1) complex than the $\text{LiNH}_2/\text{MgH}_2$ (1:1) system for desorption studies. Durojaiye et al [14] also discovered that rubidium hydride was a very effective catalytic additive for the $2\text{LiNH}_2/\text{MgH}_2$ system. They found that the hydrogen desorption rate from the RbH-doped sample was approximately twice as fast as that from the KH-doped sample and about 60 times faster than that from the un-doped sample. They also found that the addition of 3 mol % RbH lowered the desorption temperature of the system by 94 °C. Hayes et al [15] did kinetics and modeling studies on the RbH-doped $2\text{LiNH}_2/\text{MgH}_2$ system and found that diffusion was the rate-controlling process for absorption and release of hydrogen at 160 °C. Other researchers have studied the effects of Rb-related additives such as RbF [16] and dopants RbH-KH on the $2\text{LiNH}_2/\text{MgH}_2$ system [17]. In these studies, they also saw that RbH plays a significant role in lowering the reaction temperature and speeding up the process. In a recent paper, Goudy and group members [18] reported that cesium hydride is an effective dopant for the lithium amide-magnesium hydride storage system. This was the first time that cesium hydride was reported to be an effective dopant for the $2\text{LiNH}_2/\text{MgH}_2$ system. It was found that CsH was very effective in lowering the desorption enthalpy and in improving the reaction rates. In this study, the alkali metal dopants KH, RbH, and CsH were added to the $2\text{LiNH}_2/\text{MgH}_2$ system in order to compare their effectiveness in improving the

thermodynamics and kinetics of hydriding and dehydriding from the $2\text{LiNH}_2/\text{MgH}_2$ system. Each doped system was studied at several temperatures in order to determine activation energies. Modeling studies were used to determine the rate-controlling process for hydrogen absorption and desorption.

2. Materials and methods

The materials used in this research were obtained from the Sigma Aldrich Corporation. The LiNH_2 was 95% pure whereas the MgH_2 powder was hydrogen storage grade containing less than 0.1% trace metal contaminants. The KH dopant was obtained commercially whereas the RbH and CsH dopants were prepared in the laboratory using a procedure that was described in a previous publication [18]. The concentration of dopants used in all the reactions was 3.3 mol%. Sample handling was performed in a Vacuum Atmospheres argon-filled glove box that was capable of achieving less than 1 ppm oxygen and moisture. The $\text{LiNH}_2/\text{MgH}_2$ doped mixtures were prepared by milling each sample mixture for up to 10 h in a SPEX 8000M Mixer/Mill. This mill had an argon-filled stainless steel pot containing four small stainless steel balls. A Perkin Elmer Diamond TG/DTA was used to determine the thermal stability of the mixtures. Pressure Composition Isotherm (PCI) and Temperature Programmed Desorption (TPD) analyses were done in a gas reaction controller unit to determine the hydrogen desorption properties of the mixtures. This fully automated apparatus was manufactured by the Advanced Materials Corporation in Pittsburgh, PA. The TPD analyses were done in the 30–450 °C range at a temperature ramp of 4 °C/min. Kinetics experiments were performed in an all stainless steel Sievert's apparatus. It contained ports for adding

hydrogen, venting and evacuating and it also contained pressure regulators for controlling the hydrogen pressure applied to the sample. The kinetics was monitored by measuring the rate of pressure change in remote reservoirs as gas was absorbed or released from the sample. High purity hydrogen gas of 99.999% purity was used throughout the analyses. Further details about the procedures described here can be found elsewhere [18 - 20].

3. Results

3.1 Pressure-composition isotherms (PCIs) and van't Hoff plots

Figs. 1 and 2 contain pressure-composition absorption and desorption isotherms for the KH-, RbH-, and CsH-doped systems at 180°C. Isotherms were also determined in the 160 – 180 °C range for each system. Desorption isotherms have been reported for these systems [18] but this is the first time that absorption isotherms have been reported for the CsH-doped system. The plots in Figs. 1 and 2 show that the plateau pressures at 180°C for the doped materials are in the order: KH > RbH > CsH. According to Equation (3), a plot of $\ln P$ vs $1/T$ should be linear.

$$\ln P = -\Delta H/RT + C \quad (3)$$

P represents the mid-plateau pressure, ΔH is the enthalpy, R is the gas constant and T is the absolute temperature. The plateau pressures obtained at the various temperatures were used to construct the van't Hoff plots shown in Figs. 3 and 4. The slopes of these plots were used to determine enthalpies of absorption and desorption. The enthalpy values are shown in Table 1 along with other thermodynamic and kinetic data. The

results indicate that the enthalpies are in the order: CsH > RbH > KH. It can also be seen that the absorption enthalpies are smaller than the corresponding desorption enthalpies.

3.2 Kinetics

The hydrogen desorption kinetics of the KH- RbH- and CsH-doped 2LiNH₂/MgH₂ hydrogen storage materials were investigated using constant pressure thermodynamic driving forces. In order to achieve a constant pressure thermodynamic driving force for desorption kinetics the ratio of the equilibrium plateau pressure at midpoint (p_m) to the opposing pressure (p_{op}) was set to the same value for all the reactions studied. This ratio (p_m/p_{op}) has been denoted the N-Value. In the case of absorption the N-Value is (p_{op}/p_m). For all reactions in this study, the N-Value was set to 3. Fig. 5 contains the rate curves for absorption of hydrogen by the KH-, RbH-, and CsH-doped systems at temperature of 180°C. The results show that, under the conditions used, the RbH doped material absorbs hydrogen the fastest whereas the CsH doped material absorbs the slowest. The times required for 90% hydrogen absorption at 180 °C and N = 3, by each material, are given in Table 1. Fig. 6 contains rate curves for desorption of hydrogen from the KH-, RbH-, and CsH-doped materials under the same conditions. A comparison of the times required for 90% absorption and desorption in Table 1 reveals that absorption occurs faster than desorption. For both absorption and desorption, the reaction rates are in the order: RbH > KH > CsH.

3.3 Modeling Studies

It was also of interest to determine what process controls the rates of hydriding and dehydriding from these materials. In order to do this, two theoretical equations based on the shrinking core model were used. These equations are shown below:

$$\frac{t}{\tau} = 1 - (1 - X_B)^{1/3} \quad (4)$$

$$\frac{t}{\tau} = 1 - 3(1 - X_B)^{2/3} + 2(1 - X_B) \quad (5)$$

In these equations t is the reaction time and X_B is the fraction reacted. The quantity τ is a collection of constants related to the initial radius of the hydride particles, the gas phase concentration of reactant, the effective diffusivity of hydrogen atoms in the hydride and the density of the metal hydride. A model based on Equation (4) will have chemical reaction at the boundary between the hydrided and dehydrided phases controlling the reaction rate. Based on Equation (2), the hydrided phase will consist of $\text{Mg}(\text{NH}_2)_2 + 2\text{LiH}$ whereas the dehydrided phase consists of $\text{Li}_2\text{Mg}(\text{NH})_2$. A model based on Equation (5) is one in which diffusion controls the overall reaction rate. In the case of absorption diffusion will occur through the hydrided phase. For desorption, diffusion would be through the dehydrided phase. Both models were used to determine what process controls the reaction rate in the reactions. Equations (4) and (5) were used to generate theoretical data that were fitted to the kinetic data for each of the reaction mixtures. Figs. 7 and 8 each contain three curves. One is an experimental curve taken from the kinetics curves in Figs. 5 or 6, a second curve was calculated from Equation (4) and a third curve was calculated from Equation (5). In order to determine the theoretical curves, it was first necessary to determine a value for τ , which appears in Equations (4) and (5). The determination was accomplished through a series of statistical data analyses in which the

value of τ necessary to minimize the standard deviation between the experimental and theoretical data was calculated.

The results in Fig. 7 are for hydrogen absorption along the two-phase plateau region of the isotherm by the KH doped mixture. A comparison of the curves shows that the reaction controlled model fits the experimental data better than the diffusion controlled model during the first 70% of the reaction. Beyond 70% the experimental data begins to fit the diffusion controlled curve better. This behavior is also typical of that which was found for the RbH- and CsH-doped samples. The modeling curves for the RbH- and CsH-doped samples are shown in the supplementary materials, Figs. S1 and S3. A possible explanation for the two step modeling process seen here is that when hydrogen is absorbed by a dehydrided particle, a layer of hydrided product is formed on the surface. As hydrogen continues to be absorbed, the product layer becomes thicker and the core of dehydrided material shrinks. During this process, hydrogen must diffuse from the outer surface of the particle to the phase boundary between the two layers. Thus, there are two processes going on simultaneously: diffusion through the outer product layer and reaction at the phase boundary. The slower of these two processes will be the rate-controlling step. Initially, the outer product layer is very thin and diffusion through it does not take much time. Reaction at the phase boundary is the slow step at this point. When 70% of the reaction is complete, the product layer has become much thicker than it was in the initial stages and diffusion through it takes a relatively long time. At this point, diffusion becomes the rate-controlling step. Near the end of the reaction, the edge of the two-phase plateau region is reached and the N-Value (ratio of P_{op}/P_m) changes. At this point the

thermodynamic driving force changes and neither process controls the rate. This causes the experimental curve to diverge from the theoretical modeling curve.

The modeling curves for desorption are shown in Fig. 8. The results show that the diffusion controlled curve fits the experimental curve along the entire plateau region. In this case hydrogen must diffuse through a dehydrided product layer instead of a hydrided layer, as was the case for absorption. Apparently diffusion through the dehydrided layer is much slower than reaction at the interface during the entire reaction along the two-phase plateau region. This makes it the rate-controlling step during the entire reaction. This is consistent with the fact that desorption rates are slower than absorption rates. As was seen for absorption, the theoretical and experimental curves diverge near the end of the reaction because the thermodynamic driving force changes as the reaction moves off of the plateau region. The modeling results shown in Fig. 8 are typical of those obtained for the RbH- and CsH-doped systems. The modeling curves for these are shown in the supplementary materials, Figs. S2 and S4.

It should be noted that the type of behavior observed for the $2\text{LiNH}_2/\text{MgH}_2$ system has also been reported for some borohydride systems. Ibikunle et al [21] did desorption kinetics and modeling studies on $\text{LiBH}_4/\text{MgH}_2$ and $\text{LiBH}_4/\text{CaH}_2$. They found that reaction at the phase boundary controlled reaction rates initially whereas diffusion controlled rates in the latter stages. In another study, Ibikunle and Goudy [22] reported that in a mixture of $\text{Mg}(\text{BH}_4)_2/\text{Ca}(\text{BH}_4)_2$ the rate also appeared to be under the mixed control of two processes. It was found that the reaction the rate was controlled by chemical reaction at the phase boundary in the early stages whereas diffusion was the rate-controlling process in the latter stages. A somewhat different result was obtained by

Hayes et al [15] who modeled the hydriding and dehydriding kinetics of a RbH-doped $2\text{LiNH}_2/\text{MgH}_2$ system at 160°C . They found that diffusion was always the rate-controlling process for both absorption and desorption reactions. It appears that, at the relatively low temperature of 160°C , diffusion through the product layer always takes longer than chemical reaction at the interface even in the early stages of the reaction.

3.4 Activation Energy

It was also of interest to determine the activation energies for each of the doped $2\text{LiNH}_2/\text{MgH}_2$ systems. In order to do this, the reaction order had to be established. If a reaction is first order it can be described by Equation (5), where F is the reacted fraction, k is the first order rate constant and t is the time.

$$\ln(1-F) = -kt \quad (5)$$

Thus one would thus expect a plot of $\ln(1-F)$ vs t to be linear for a first order reaction. Fig. 9 contains plots of $\ln(1-F)$ vs t for hydrogen absorption by the KH doped system. It can be seen that the plots are generally linear. These plots are typical of those obtained for the other doped mixtures. Since the slopes of these plots are equal to the rate constants, these could be used in Equation (6) to determine activation energies. Equation (6) is generally referred to as the Arrhenius equation, where E_a is the activation energy, T is the absolute temperature and R is the gas constant.

$$\ln k = -E_a/RT + \text{constant} \quad (6)$$

Based on this equation the plots of $\ln k$ vs $1/T$ in Figs. 10 and 11 were constructed and the slopes were used to determine the activation energies listed in Table 1. It can be seen that the activation energies for the doped systems are in the order $\text{RbH} < \text{KH} < \text{CsH}$. As

expected, the activation energies for absorption are less than those for the corresponding desorption reactions. This is consistent with the fact that absorption reactions were faster than desorption reactions. It should also be noted that the desorption activation energies are in close agreement with those obtained in an earlier study [18]. Those were determined using the Kissinger method [23].

Conclusions

The results of this research have shown that the stabilities of the alkali metal hydride doped $2\text{LiNH}_2/\text{MgH}_2$ mixtures are in the order: $\text{CsH} > \text{RbH} > \text{KH}$. Kinetics measurements showed that the absorption and desorption rates are in the order: $\text{RbH} > \text{KH} > \text{CsH}$, with absorption rates being faster than desorption from the corresponding material. As expected, the activation energies for the reactions were in the order: $\text{RbH} < \text{KH} < \text{CsH}$ with the activation energies for absorption being less than that for the corresponding desorption reaction. Modeling studies revealed that the absorption reactions are controlled by reaction at the phase boundary during the first part of the process and by diffusion in the latter stages. However, for desorption reactions the rates are controlled by diffusion during the entire reaction along the two-phase plateau region. These results indicate that diffusion through the dehydrided product layer, during desorption, occurs much slower than diffusion through the hydrided layer, during absorption.

Table 1 Summary of absorption and desorption parameters for thermodynamics and kinetics results

Parameter	KH	RbH	CsH
Abs P_m	32.5	30.2	26.0
Des P_m	25.4	21.5	19.5
Abs ΔH (kJ/mol)	34.3	39.7	40.98
Des ΔH (kJ/mol)	40.9	43.0	46.10
Ea Abs (kJ/mol)	83.9	81.7	105.9
Ea Des (kJ/mol)	89.3	86.9	110.5
T₉₀ Abs (min)	183	107.5	301
T₉₀ Des (min)	297	207	455

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Fig. Captions

Fig. 1 PCI absorption curves for $2\text{LiNH}_2/\text{MgH}_2$ mixtures at 180°C

Fig. 2 PCI desorption curves for $2\text{LiNH}_2/\text{MgH}_2$ mixtures at 180°C

Fig. 3 Van't Hoff plots for hydrogen absorption by the $2\text{LiNH}_2/\text{MgH}_2$ mixtures

Fig. 4 Van't Hoff plots for hydrogen desorption from the $2\text{LiNH}_2/\text{MgH}_2$ mixtures

Fig. 5 Absorption kinetics for the doped mixtures at 180°C and $N = 3$.

Fig. 6 Desorption kinetics for the doped mixtures at 180°C and $N = 3$.

Fig. 7 Modeling plots for hydrogen absorption by the KH-doped mixture at 180°C

Fig. 8 Modeling plots for hydrogen desorption from the KH-doped mixture at 180°C

Fig. 9 First order plot for hydrogen absorption by the KH-doped mixture at 180°C and $N = 3$.

Fig. 10 Arrhenius plots for hydrogen absorption by the doped mixtures

Fig. 11 Arrhenius plots for hydrogen desorption from the doped mixtures

Table Caption

Table 1 Summary of absorption and desorption parameters for thermodynamics and kinetics results

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Figure 1

Figure 2

Figure 3

Figure 4

Figure 5

Figure 6

Figure 7

Figure 8

Figure 9

Figure 10

Figure 11

Table 1

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List of 5 suggested reviewers

Kirk L Shanahan
Savannah River National Laboratory
Bldg. 999-2W
Aiken, SC 29808
Phone: 803-507-8540
E-mail: kirk.shanahan@srnl.doe.gov

Chris Wolverton
Northwestern University
Dept. of Materials Sci. and Eng.
2220 Campus Drive
Evanston, IL 60208
Phone: 847-467-0593
Email: c-wolverton@northwestern.edu

Jacques Huot
University Du Quebec A Trois-Rivieres
3351 Des Forges
Trois-Rivieres, PQ G9A 5H7 Canada
Phone: 819-376-5011
E-mail jacques.huot@uqtr.ca

Isaac Jacob
Ben Jurion University
Department of Nuclear Engineering
Beer Sheva, 84105 Israel
Phone: 972-8-646-1316
Email: izi@bgu.ac.il

Ewa Ronnebro
Pacific Northwest National Laboratory
902 Battle Boulevard
PO Box 999, MSIN K2-03
Richland, WA 99352
Phone: 509-372-6877
E-mail: ewa.ronnebro@pnl.gov