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Metal Hydrides

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Metal Hydrides

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20.1 Metal Hydrides

Hydrogen storage by metal hydrides comprises an intermetallic alloy phase that has the capability to absorb and hold vast amounts of hydrogen by chemical bonding. An appropriate hydrogen storage matrix should have the capacity to absorb and release hydrogen without compromising the matrix structure. Metal hydrides are prepared by reaction between a metallic phase and hydrogen. When exposed to hydrogen at certain pressures and temperatures, these phases absorb large quantities of hydrogen gas and form the corresponding metal hydrides. If this is the scenario, the hydrogen is distributed compactly throughout the intermetallic lattice. Metal hydrides represent an exciting process of hydrogen storage, which is inherently safer than the compressed gas or liquid hydrogen storage. Additionally, some intermetallics (including metals and alloys) store hydrogen at a higher volume density than liquid hydrogen.

The qualities required to make these intermetallics useful include the facility to absorb and release large amounts of hydrogen gas, many times without damaging the storage material and with good selectivity (only hydrogen absorption). Moreover, suitable metal hydrides absorb and release hydrogen at rates that can be controlled by adjusting temperature and/or pressure.
As mentioned in the preceding text, a typical relation of metal hydrides with hydrogen can be expressed as \( M + \frac{x}{2} H_2 \leftrightarrow MH_x + \text{Heat} \), where \( M \) represents the intermetallic matrix and \( H \) is hydrogen. The effect is reversible and the direction is determined by the pressure (and temperature) of the hydrogen gas. If the pressure is above a certain level (the equilibrium pressure), the effect proceeds to the right to form the hydride, whereas below the equilibrium pressure, hydrogen is liberated and the intermetallic matrix returns to its original state. The equilibrium pressure, itself, depends upon temperature. It increases due to expansion with an increment in temperature (and vice versa). The molecular hydrogen first dissociates into hydrogen atoms due to the available metal catalysts and interacts with the host metal lattice to form metal hydride as shown in the schematics of Figure 20.1a. Similarly, the reverse reaction yields hydrogen in gaseous form as represented in Figure 20.1b.

In general, hydrides are classified according to the nature of the bonding of hydrogen to the host lattice such as covalent, saline or ionic, and metallic, all of these classes bearing different bonding characteristics. Such a classification doesn’t always clarify the characteristic features of the compound in question. For example, lithium hydride is classified as a saline hydride, where in reality, it actually exhibits some covalent characteristics. The rare-earth hydrides are normally classified in the metallic hydride group, whereas they exhibit some characteristics similar to those of volatile and saline hydrides (e.g., high heat of formation). The ionic hydrides are usually crystalline and show high heats of formation and high melting points. The ionic alkali and alkaline-earth hydrides have a higher density than the pure alkali (45%–75%) and alkaline-earth metals (20%–25%). Covalent hydrides are generally thermally unstable, and this instability goes up with

![FIGURE 20.1](image-url)

(a) Metal hydride formation during \( H_2 \) absorption in host metal lattice. (b) Hydrogen gas release during the dehydrogenation of metal hydride.
increasing atomic weight of the nonhydrogen element(s). Typical covalent hydrides are carbon hydrides, boron hydrides, germanium hydrides, etc. Covalent hydrides normally exhibit low symmetric structures. Metallic complex hydrides are normally formed by the transition metals, for example, ScH\(_2\)\(^{17}\). They generally exhibit metallic characteristic properties such as high thermal and electrical conductivity, hardness, luster, etc. Due to the wide homogeneity ranges adopted by metallic hydrides, they have occasionally been considered as solid solutions of hydrogen in the interstitials of metal, alloy, or intermetallic matrices. Yet another classification based on the carbon-based sorbents, for example, single-wall or multiwall carbon nanotubes, polymer nanostructures, zeolites, and metal organic frameworks, possesses weak van der Waals attraction toward hydrogen. The classification of hydrides on the basis of temperature of operation and thermodynamic values such as \(\Delta H\) of reaction is schematically shown in Figure 20.2.

It is clearly understood that the metal or complex hydrides operate in the region of moderate temperatures 0°C–100°C with appropriate \(\Delta H\) of \(\sim 30–55\) kJ/mol H\(_2\). Whereas the ionic or covalent hydrides require high temperature, \(\Delta H\) of \(\sim 70–80\) kJ/mol H\(_2\) and the carbon-based sorbent systems mandate cryogenic operating temperatures with \(\Delta H\) < 20 kJ/mol H\(_2\).

Hydrogen can be packed and stored in a solid state by forming metal hydride\(^{4–12}\). During the formation of the metal hydride, hydrogen molecules are dissociated into hydrogen atoms, which insert themselves into interstitial spaces inside the lattice of intermetallic compounds and/or alloys (Figure 20.1).

At a large distance from the metal surface, the energy difference between a hydrogen molecule and two separate hydrogen atoms is the dissociation energy (H\(_2\) \(\rightarrow\) 2H, \(E_D = 435.99\) kJ/mol). The first attractive interaction of the hydrogen molecule approaching the
metal surface is the van der Waals force leading to the physisorbed state ($E_{phys} \approx 10 \text{ kJ/mol}$) more or less one hydrogen molecule radius ($\approx 0.2 \text{ nm}$) from the metal surface. Closer to the surface, the hydrogen has to overcome an activation barrier in order to dissociate and create the hydrogen metal bond. The height of the activation barrier depends on the surface elements involved. Hydrogen atoms sharing their electrons with the metal atoms at the surface are in the chemisorbed state ($E_{Chem} \approx 50 \text{ kJ/mol \cdot H}_2$). The chemisorbed hydrogen atoms may have a high surface mobility, interact with each other, and form surface phases at considerably high coverage. In the following step, the chemisorbed hydrogen atoms can jump into the subsurface layer and finally diffuse to interstitial sites through the host metal lattice as depicted in Figure 20.3. An interstitial hydrogen atom contributes its electron to the band structure of the metal.

In the $\alpha$-phase solid solution, the hydrogen to metal ratio is small ($H/M < 0.1$), and hydrogen is exothermically dissolved in the metal. The metal lattice expands relative to the hydrogen concentration by approximately 2–3 Å$^3$ per hydrogen atom$^{13}$ At greater hydrogen concentrations in the host metal ($H/M > 0.1$), a strong H–H interaction due to the lattice expansion becomes significant, and the hydride phase ($\beta$-phase) nucleates and grows. The hydrogen concentration in the hydride phase is commonly found to be $H/M = 1$.

The volume expansion between the coexisting $\alpha$- and the $\beta$-phase$^{14}$ corresponds in many cases 10%–20% of the metal lattice. Therefore, at the phase boundary, large stresses are created that frequently lead to a decrepitation of brittle host metals including intermetallic compounds resulting in a final hydride powder with a representative particle size of 10–100 μm.

An idealized representation of pressure–concentration–temperature (PCT) isotherms for the $\alpha$-phase solid solution and $\beta$-phase hydride is shown in Figure 20.4. A plateau is observed on the pressure versus hydrogen/metal ratio for fixed temperature. When the reaction is complete, another sharp pressure rise is seen if more hydrogen is added.

![Figure 20.3](image_url)

**Figure 20.3**
Schematic representation of the dissociation of hydrogen gas molecule.
At a certain temperature, the plateau pressure reflects one point on a pressure–temperature (van’t Hoff) plot. The log of pressure versus reciprocal temperature is linear with a negative slope and is related to the heat of reaction (heat of hydriding); the plateau pressures must increase with temperature due to the linear relationship between pressure and temperature.

An increment in isothermal temperature causes the plateau pressure to increase and, at the same time, reduces the width of the plateau that represents the miscibility regime of the \(\alpha\) - and \(\beta\) -phase. This narrowing process of the plateau with increasing of temperature continues until, eventually, at a certain critical temperature, \(T_c\), the plateau disappears totally, reducing the miscibility of the two phases to zero, and the \(\alpha\) -phase converts continuously into the \(\beta\) -phase. The slope and length of the equilibrium plateau are of particular importance for hydrogen storage application; a flat plateau enables the reversible absorption and desorption of hydrogen from a metal simply by raising or lowering the surrounding hydrogen pressure above or below the plateau pressure.

The equilibrium pressure as a function of temperature is related to the changes of \(\Delta H\) and \(\Delta S\), in that order, by the van’t Hoff equation: \(^{15-17}\)

\[
\ln \left[ \frac{p(H_2)}{p^\circ} \right] = \frac{\Delta H}{RT} - \frac{\Delta S}{R} \tag{20.1}
\]

where
- \(p(H_2)\) is the hydrogen equilibrium pressure at \(\alpha\) - to \(\beta\) -phase hydride conversion, determined from PCT isotherms
- \(p^\circ\) is the standard pressure
- \(R\) is the gas constant
- \(T\) is the temperature

The \(\Delta H\), of hydride formation is an important parameter characterizing the alloy as a proper hydrogen absorber for various applications. In general, the difference in standard \(\Delta S\) between a metal hydride alloy and its hydride is small and is on the order of...
10 J/mol/K. The modification in $\Delta S$ with hydride formation is mainly provided by the loss of the standard $\Delta S$ of hydrogen gas (130.858 J/mol/K at 298 K), which means that $\Delta S$ can be assumed to be a constant and does not depend on the nature of the metal hydride alloy. The knowledge of $\Delta H$ particularly is significant to the heat management needed for practical engineering devices and is a fundamental measure of the M–H bond strength. The van’t Hoff plot ($\ln P$ vs. $1/T$) is a practical way to compare hydrides due to their thermal stabilities.

An overview of hydrogen storage alloys has been discussed from the solid–gas reaction point of view. There are a number of important properties that must be considered in metal hydride storage. Some of the most important ones are (1) ease of activation, (2) heat transfer rate, (3) kinetics of hydriding and dehydriding, (4) resistance to gaseous impurities, (5) cyclic stability, (6) safety, (7) weight, and (8) cost. Although metal hydrides can theoretically store large amounts of hydrogen in a safe and compact way, the practical gravimetric hydrogen density is limited to <3 mass%. It is still a challenge to explore the properties of lightweight metal and complex hydrides.

Hydrogen is a highly reactive element and has been found to form hydrides and solid solutions with thousands of metals and alloys. A hydride family tree of the elements, alloys, and complexes is shown in Figure 20.5.

The majority of the 91 natural elements below hydrogen in the periodic table will form hydrides under appropriate conditions such as $\text{VH}_2$, $\text{NaH}$, $\text{LaH}_2$, and $\text{ZrH}_2$. Unfortunately, the PCT properties are not very suitable for the 1–10 atm and 0°C–100°C range used for practical hydrogen storage. Only V is in the range, and there has been extensive study of solid solutions of V and other metals. Pd has been used for over 100 years for H storage,
but it is very costly, it doesn’t hold much hydrogen, and it requires heating temperatures above 100°C to release atomic hydrogen.

### 20.1.1 Intermetallic Alloys

To prepare useful reversible hydrides, strong hydride-forming (A) elements are combined with weak hydriding (B) elements to form alloys (especially intermetallic compounds) that have the desired intermediate thermodynamic affinities for hydrogen. A good example of this characteristic is the combination of La (forming LaH$_2$ with 25°C, $P_d = 3 \times 10^{-29}$ atm, and $\Delta H_f = -208$ kJ/mol H$_2$) and Ni (NiH with 25°C, $P_d = 3400$ atm, and $\Delta H_f = -8.8$ kJ/mol H$_2$) to form the intermetallic compound LaNi$_5$ (LaNi$_5$H$_6$ with 25°C, $P_d = 1.6$ atm, and $\Delta H_f = -30.9$ kJ/mol H$_2$). This ability to interpolate between the extremes of elemental hydriding behavior opened the door to the modern world of reversible hydrides.

The AB$_5$ family has an exceptional versatility because many different elemental species can be substituted (at least partially) into the A and B lattice sites. The A elements tend to be one or more of the lanthanides (elements 57–71), Ca, or other elements such as Y and Zr. The B elements are based on Ni with many other possible substitutional elements such as Co, Al, Mn, Fe, Cu, Sn, Si, and Ti. Modern commercial AB$_5$ hydriding alloys are for the most part based on the use of the lanthanide mixture or mischmetal (Mm = Ce + La + Nd + Pr) for the A site and Ni + Al + Mn + Co + … for the B site.

Hydrogen storage capacity is on the uncomfortably low side, not passing 1.3 wt.% on the plateau basis. Alloy raw material cost is high; at least by comparison to other systems (AB$_2$ and AB) that are discussed in the following, the AB$_5$ alloys are easy to activate, hardly requiring any heating. They decrepitate on the first H/D cycle to fine powder, which is mildly pyrophoric if suddenly exposed to air, a well-known property that must be included in safety considerations. Both easy activation and pyrophoricity indicate the AB$_5$ alloys do not form defensive oxide layers. Intrinsic kinetics of the AB$_5$ alloys is generally very good. In an attempt to increase the kinetics and hydrogen storage capacity in AB$_5$-type alloys such as MmNi$_{4.6}$Fe$_{0.4}$, mechanochemical pulverization at room temperature was carried out. Figure 20.6 represents the x-ray diffraction (XRD) profiles of MmNi$_{4.6}$Fe$_{0.4}$ prepared using radio-frequency induction melting followed by mechanochemical pulverization.

The nanocrystallization of peaks was observed with broadening of full width at half maximum (FWHM), thus enhancing the kinetics of hydrogen release as shown in Figure 20.7.

The scanning electron microscope (SEM) micrographs (see Figure 20.8) further justify the smaller and homogeneous crystallites with well-defined grains that enable for the higher hydrogen absorption and desorption cycling capacity with twofold increase of kinetics at room temperature.

Like the AB$_5$ compounds, the AB$_2$ intermetallics represent a large and versatile group of hydriding materials with good PCT properties at ambient temperature. The A elements are often from the IVA group (Ti, Zr, Hf) and/or rare-earth series (no. 57–71). The B elements can be a variety of transition or nontransition metals with somewhat of a preference for atomic numbers 23–26 (V, Cr, Mn, Fe). A large variety of substitutions are possible for both A and B elements, thus providing a high degree of fine tuning of PCT properties.

The decisive advantages in regard to hydrogen storage and related thermal applications are high hydrogen capacity, ease of activation, very rapid rates of absorption and desorption, long cycling life, and low cost of materials. The main disadvantages for hydrogen storage are high $\Delta H$ values (moderate stabilities of hydrides). These properties make these compounds good for hydrogen compression applications. An excellent example is ZrMn$_2$ that
FIGURE 20.6
X-ray diffractograms of (a) as-synthesized MmNi$_{4.6}$Fe$_{0.4}$ using radio-frequency induction melting and (b) induction melting followed by mechanical pulverization.

FIGURE 20.7
(a) Hydrogen desorption kinetic curves and (b) PCT of as-synthesized and mechanically pulverized MmNi$_{4.6}$Fe$_{0.4}$. 
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3.6 atoms of hydrogen and $\Delta H = -53.0 \text{ kJ/mol } \text{H}_2$, showing a high hydrogen content and a high $T_{\text{dec}}$ related to the high $\Delta H$. The high $T_{\text{dec}}$ and the high hydrogen content make this compound good for hydrogen compression.

Method of preparing an alloy of zirconium and manganese suitable for storing hydrogen consists in two steps $^{21}$: make an intimate mixture of Zr and Mn in correct stoichiometric ratio and heat the sample between 900°C and 1150°C. The intimate mixture is done using a straightforward, repeatable, and inexpensive procedure, ball milling under a H$_2$ atmosphere. The annealing was applied for 10 h in a helium atmosphere at 1100°C.

A is typically of the group IVA elements Ti, Zr, or Hf, and B is a transition metal, typically Ni. Another family is based on Mg$_2$Ni. $^{22}$ Unfortunately, the A$_2$B’s offer little in the 0°C–100°C and 1–10 atm range, at least with the present state of the art. They are definitely more stable. H storage capacity and cost properties of Mg$_2$Ni are attractive, but desorption temperatures are too elevated for most applications. Mg$_2$Ni is not very amenable to modification of PCT properties by ternary and higher-order substitutions. Various attempts to lower desorption temperatures have not been especially successful.

20.1.2 Solid Solutions

The first example of a reversible intermetallic hydride was demonstrated with the AB compound, ZrNi, by Libowitz in 1957 $^{23}$ The hydride ZrNiH$_3$ has a 1 atm desorption temperature of about 300°C, too high for hydrogen storage applications but suitable for hydrogen compression.

These intermetallic alloys show good volumetric and gravimetric reversible H storage capacities, competitive with the best of the AB$_3$’s and AB$_2$’s. Activation is pretty slow and hard for the ZrNi-based alloys. In any event, it may take a day or so and high pressures (50+ atm) for total activation. The passive oxide films that can form on ZrNi (and its derivatives) don’t result in a high degree of sensitivity to gaseous impurities in the H$_2$ used; hence, these alloys are resistant to impurities. Cyclic stability of the lower plateau is great, but the upper plateau tends to drift higher with H/D cycling. Although there is vast information available about ZrNi alloys, the intermediate compositional alloys ZrNi 70/30 and ZrNi 30/70 have been sparsely reported for hydrogen storage. $^{24,25}$ The XRD analysis of ZrNi70/30 before and after hydrogenation is shown in Figure 20.9. The XRD spectra show a significant change in the intensity of the peaks in ZrNi 70/30 hydride compared with the unhydrided sample (33.2°, 34.2°, 36.5°, and 43°) and the presence of a new peak (36.7°) attributed to the ZrNi 70/30 hydride phase. The crystallite
sizes of the ZrNiH$_3$ (∼48 nm) and ZrH$_2$ (∼39 nm) are calculated and compared with the unhydrided sample. The scanning micrographs of the ZrNi 70/30 after a number of hydrogenation cycles show the development of microcracks due to hydrogen interaction with the host lattice at high temperatures (Figure 20.10).

Figure 20.11 illustrates the absorption–desorption PCT for the ZrNi 70/30 alloy, giving evidence of a typical metal hydride behavior. The plateau region is the most important segment of a PCT plot, which represents the pressure of hydrogen in equilibrium with the metal–metal hydride (α + β) phases, representing the dissociation pressure of the metal hydride at the desired temperature; the temperature indicates the thermal stability of the metal hydride. Moreover, when the temperature goes from 300°C, 325°C, 350°C, 375°C, and 390°C, it causes the plateau pressure to increase and, at the same time, reduces the width of the plateau that represents the miscibility regime of the α- and β-phase. As an example, at 375°C, hydrogen content of 1 wt.% is reached at 13 bars. According to the investigations, ZrNi 70/30 alloy presents a metal hydride behavior: low kinetics, moderate to high equilibrium pressures, and moderate to high hydrogen content, conditions that make this alloy the most convenient for hydrogen compression applications. From the PCT isotherms, the ∆H of reaction has been calculated to be ∼39 kJ/mol H$_2$ for the ZrNi70/30.

In addition to the AB$_5$, AB$_2$, AB, and A$_2$B intermetallic compounds discussed previously, various other families of intermetallics have been shown capable of reversible hydriding/dehydriding reactions. Examples include AB$_3$, A$_3$B$_7$, A$_6$B$_{23}$, A$_3$B$_{17}$, and A$_3$B. Most structures involve long-period AB$_5$ and AB$_2$ stacking sequences and are thus crystallographically related to these two classic families. Most have narrow plateaux with long sloping upper legs (e.g., GdFe$_3$) or multiple plateaux (e.g., NdCo$_3$ or Pr$_2$Ni$_3$).
SEM micrographs of (a) pristine, (b) hydrogenated, and (c) hydrogenated in higher magnification showing microcracks of ZrNi 70/30 material.
20.1.3 Composites

When the mass of the metal or alloy is taken into account, the metal hydride gravimetric storage density is comparable to storage of pressurized hydrogen. The best achievable gravimetric storage density is about 0.07 kg of H\textsubscript{2}/kg of metal, for a high-temperature hydride such as MgH\textsubscript{2} as shown in Table 20.1, which gives a comparison of some hydriding substances with liquid hydrogen, gaseous hydrogen, and gasoline.\textsuperscript{28,29} MgH\textsubscript{2} can effectively store hydrogen due to its thermodynamic stability; however, reaction kinetics are too slow and the decomposition temperature is high, at approximately 330\textdegree{}C.\textsuperscript{20} A possible way to achieve Mg-like storage capacity but with reversible
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Hydrogenation characteristics is to form composites with Mg as one of the components as discussed earlier. The other component may be one of the known hydrogen storage intermetallic alloys.\textsuperscript{30}

Since the MmNi\textsubscript{4.6}Fe\textsubscript{0.4} alloy discussed in Section 20.1.1 exhibited hydrogen storage capacity of less than 2.0 wt.\%, a novel strategy of forming a composite phase Mg-X wt.\% MmNi\textsubscript{4.6}Fe\textsubscript{0.4} was carried out.\textsuperscript{31} This system has dual advantages in a sense that it reduces the weight penalty of the system for hydrogen storage applications and also enhances the hydrogen storage capacity >5 wt.\% at moderate temperatures. The flowchart for the synthesis and characterization of Mg-X wt.\% MmNi\textsubscript{4.6}Fe\textsubscript{0.4} (X = 10, 20, 30, 40, and 50) is shown in Figure 20.12.

The dehydrogenation kinetic and PCT curves as demonstrated in Figure 20.13a and b reveal the high hydrogen storage capacity (~5 wt.\%) at temperatures >300°C.

The XRD pattern of the composite material shows the presence of both Mg and MmNi\textsubscript{4.6}Fe\textsubscript{0.4} phases; additional peak pertaining to MgH\textsubscript{2} was observed in the hydrogenated materials (see Figure 20.14). The effective hydrogenation and dehydrogenation in these composite materials have been confirmed through the hydrogen-induced microchannels as exhibited in SEM micrographs and are shown in Figure 20.15. The Mg in the host matrix of MmNi\textsubscript{4.6}Fe\textsubscript{0.4} thus facilitates lower desorption temperatures due to the available catalytic nanoparticles of Ni and Fe, which surpassed the hydrogen storage characteristics of Mg\textsubscript{2}Ni or other Mg-based intermetallic systems.

**Magnesium (purity 99.99%)**

Pre synthesized AB\textsubscript{5} (MmNi\textsubscript{4.6}Fe\textsubscript{0.4}/CFMmNi\textsubscript{5}) Alloy

Sieved to achieve a particle size of 100 μm

Mechanically alloyed the composite mixture using high-energy attritor ball mill

By optimizing the milling parameters

- Medium of milling: Hexane
- Ball to powder ratio: 20:1
- Speed of milling: 400 rev/min
- Milling durations: 5 h

After mechanically alloying, the milled powder (composite materials) was immediately transferred to argon-filled glove box

As-milled composite material Mg-X wt.\% AB\textsubscript{5}

Structural characterization (XRD) (Philips PW 1710)

Microstructural characterizations (SEM) (Philips XL-20 Series)

Hydrogenation/dehydrogenation behavior (Severts-type apparatus)

**FIGURE 20.12**
Flowchart diagram of synthesis and characterization of Mg-X wt.\% MmNi\textsubscript{4.6}Fe\textsubscript{0.4} composite material.
**FIGURE 20.13**
(a) Hydrogen desorption kinetics and (b) PCT characteristics of Mg-Xwt.% MmNi_{4.6}Fe_{0.4} at moderate temperatures.

**FIGURE 20.14**
XRD pictures of Mg-Xwt.% MmNi_{4.6}Fe_{0.4} before and after hydrogenation at moderate temperatures.
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References


FIGURE 20.15
SEM pictures of Mg-Xwt.% MmNi$_4$.Fe$_{0.4}$ before and after hydrogenation at moderate temperatures.