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The hydrolysis behaviour of Mg_2Ni and Mg_2NiH_4 in water or a 6 M KOH solution and its application to Ni nanoparticles synthesis

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ABSTRACT

Both Mg_2Ni and Mg_2NiH_4 undergo hydrolysis in water and in a 6 M KOH alkaline solution. Mg_2Ni spontaneously reacts with water to form $Mg(OH)_2$, Ni and hydrogen. Mg_2NiH_4 first dissociates into Mg_2Ni and hydrogen, and then the Mg_2Ni is further hydrolyzed into $Mg(OH)_2$ and Ni. The hydrolysis characteristics of both Mg_2Ni and Mg_2NiH_4 suggest that they are not suitable for use as electrodes in rechargeable batteries. After the $Mg(OH)_2$ in the hydrolysis product of Mg_2Ni in distilled water was carefully removed using dilute hydrochloric acid, spherical Ni particles with a size of about 10 nm were obtained. The hydrolysis of Mg_2Ni or Mg_2NiH_4 therefore provides a new and relatively simple method for the production of nickel nanoparticles.

1. Introduction

Rechargeable NiMH batteries have many advantages for use as power sources in electric and hybrid vehicles such as higher power densities and environmental friendliness [1–2]. When compared with conventional AB_5 alloys, Mg_2Ni exhibits a good hydrogen storage capacity (3.6 wt.%) and lower specific gravity. The theoretical discharge capacity of Mg_2Ni is as high as 999 mAh/g. Mg_2Ni is thus expected to be among the next generation of electrode materials. However, polycrystalline Mg_2Ni shows a very low electrochemical discharge capacity (less than 10 mAh/g) [3]. It has been reported that the electrochemical discharge capacity can be improved through the use of nano-crystalline Mg_2Ni [4–8]. In fact, a very high discharge capacity of 1082 mAh/g (exceeding the theoretically calculated value) has been reported [4].

However, in some preliminary experiments in our laboratory we found that no matter how long the milling time, the *in situ* synthesized Mg_2Ni, obtained by ball-milling Mg and Ni powders and then holding the mixture at 690 °C for 3 h, exhibited a near zero discharge capacity. When a PTFE dispersion was added as a binder to the ball-milled powder for preparing the electrode samples, heat release was observed. Our first thought was that the heat could be coming from the reaction between any unreacted Mg in the *in situ* synthesized Mg_2Ni and water in the PTFE dispersion. To further investigate the discharge capacity of Mg_2Ni, *in situ* synthesized Mg_2NiH_4 prepared in our laboratory was used to prepare the electrodes. As well as a further heat release, additional drops of the PTFE dispersion were needed or else the mixture was too “dry” to prepare the electrodes. The Mg_2NiH_4 electrode that was prepared still exhibited a near zero discharge capacity. The *in situ* synthesized Mg_2NiH_4 was then directly pressed into an electrode without any binder in order to avoid the above phenomena. This Mg_2NiH_4 electrode could not maintain its shape in a 6 M KOH solution and broke into many pieces with the generation of many gas bubbles. In some instances, the Mg_2NiH_4 electrode caught fire when the sample contacted the surface of the alkaline solution.

An arc-melted Mg_2Ni sample, instead of an *in situ* synthesized one, was then used to repeat the above experiments. The results were the same. An abnormally high amount of $Mg(OH)_2$, much higher than could be generated from the surplus Mg in the cast Mg_2Ni alloys, was observed in the XRD patterns for the tested electrode. The excess $Mg(OH)_2$ could have only come from the hydrolysis of Mg_2Ni.

A rapid degradation of Mg_2Ni alloys in alkaline solutions during the charge–discharge process has been observed by many researchers [7–11]. The degradation has generally been attributed to the corrosion of Mg_2Ni alloy in the highly corrosive electrolyte with the production of $Mg(OH)_2$ [5–10]. Kuji et al. [8], observed the hydrolysis phenomenon of Mg_2Ni in both distilled water and a KOH solution. They assumed that Mg_2Ni alloys were easily hydrogenated by simply immersing the alloys in 6 M KOH solution or in distilled water. In fact, the increase in hydrogen concentration in their samples resulted from the hydrolyzed $Mg(OH)_2$ since it was not removed from their samples [11].

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Table 1
Chemical composition of the arc-melted Mg_2Ni pellets

Elements	Mg	Ni	O	N	C
(wt.%)	49.3	50.6	0.042	0.018	0.023

It is well known that the hydrolysis reaction between MgH_2 and water produces hydrogen [12]. In addition, Transition metal (Ni, Cu, Au and Ag) nanoparticles have been successfully synthesized by hydrolysis of transition metal magnides based on the results of this study [13–17].

In this study arc-melted Mg_2Ni was used to investigate the hydrolysis behaviour of Mg_2Ni and Mg_2NiH_4. The thermodynamics and mechanisms of the hydrolysis of Mg_2Ni and Mg_2NiH_4 are discussed in detail.

2. Materials and experimental procedures

Arc-melted Mg_2Ni pellets (MPD Technology Corporation, Wyckoff, USA) were used in this study. The chemical analysis of the arc-melted Mg_2Ni pellets (nominal formula: $Mg_{2.35}Ni$) is given in Table 1. The pellets were ball-milled under an argon atmosphere for 2 h in a laboratory high energy ball mill SPEX8000 (Spex Industries, Inc., Edison, USA) at a speed of 1200 rpm. The vial was made from tungsten carbide, and was 6.35 cm in diameter and 7.62 cm long. The milling balls, which were 1.27 cm in diameter, were made from 440C martensitic stainless steel. The weight ratio of the balls to the Mg_2Ni pellets was about 1:1. Mg_2NiH_4 particles were prepared by hydrogenating the ball-milled Mg_2Ni particles under a hydrogen atmosphere of 6 at. at 350 °C for 2 h in a tube furnace. The synthesized Mg_2NiH_4 particles were rust brown in colour.

10 g of either the ball-milled Mg_2Ni or the synthesized Mg_2NiH_4 particles were immersed in 500 ml of distilled water or 6 M KOH for 120 h. The solid particles were filtered out, and then dried on Rotavapor at 60 °C (no rinse cycle). The ball-milled Mg_2Ni particles, the synthesized Mg_2NiH_4 particles, and their hydrolysis products in distilled water or 6 M KOH were characterized by X-ray diffraction (XRD).

The paramagnetism of the hydrolysis product was examined by using a magnet. Among the initial materials, and the potential hydrolysis products, only Ni exhibits paramagnetism. Therefore, this test could be used to check whether Ni particles were present in the hydrolysis product.

The nickel particles were obtained by carefully removing the $Mg(OH)_2$ in the hydrolysis product of Mg_2Ni in distilled water by 0.5 M hydrochloric acid. The morphology of Ni particles was characterized by TEM using a Philips/FEI Technai 20 transmission electron microscope equipped with an energy dispersive X-ray analysis system.

3. Results

There was 10 wt.% surplus Mg in the initial Mg_2Ni pellets. Therefore, besides the Mg_2Ni peaks, some small Mg diffraction peaks were observed in the XRD pattern for the ball-milled particles (see Fig. 1a). Some weak MgH_2 diffraction peaks were observed in the XRD pattern for the synthesized Mg_2NiH_4 particles (Fig. 2a), together with strong Mg_2NiH_4 diffraction peaks, but no Mg_2Ni peaks were found. These results suggest that after hydrogenation under a hydrogen atmosphere of 6 at. at 350 °C for 2 h in a tube furnace, the ball-milled Mg_2Ni and the surplus Mg particles had transformed into Mg_2NiH_4 and MgH_2, respectively.

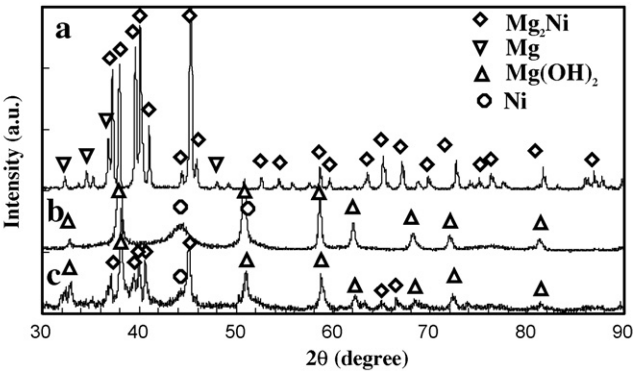

Fig. 1. Powder XRD patterns for (a) as-cast Mg_2Ni alloy, (b) its hydrolysis product in distilled water for 120 h, (c) its hydrolysis product in 6 M KOH solution.

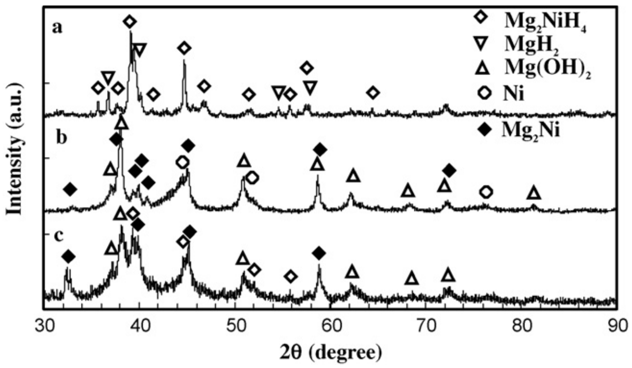

Fig. 2. Powder XRD patterns for (a) the synthetic Mg_2NiH_4, (b) its hydrolysis product in distilled water for 120 h, (c) its hydrolysis product in 6 M KOH solution.

After the ball-milled Mg_2Ni and the synthesized Mg_2NiH_4 particles were immersed in 500 ml of 6 M KOH solution or distilled water, there was initially a rapid release of hydrogen bubbles, especially for the Mg_2NiH_4 samples. The release rate of hydrogen bubbles then gradually slowed down. The pH value of the solution, determined using pH papers, rapidly reached a value of 10–11 in the samples immersed in water, and thereafter retained that level.

Brucite ($Mg(OH)_2$) and Ni peaks were observed in the XRD pattern for the hydrolysis product of the ball-milled Mg_2Ni in the distilled water (see Fig. 1b). The results indicated that Mg_2Ni had hydrolyzed into $Mg(OH)_2$ and Ni after being immersed in distilled water for 120 h. The width of the Ni peaks was fairly broad, which reflects the fact that the crystallite size of Ni was extremely small.

Besides Brucite, some weak Mg_2Ni peaks were observed in the XRD pattern for the hydrolysis product of the ball-milled Mg_2Ni in the 6 M KOH solution (see Fig. 1c). The amount of $Mg(OH)_2$, reflected by the peak intensities in Fig. 1c, is much more than that resulting from the hydrolysis of the surplus Mg. Although, no significant Ni peaks can be identified in Fig. 1c, a paramagnetism test showed that there were some Ni particles in the hydrolysis product.

Only Brucite, Ni and few Mg_2NH_4 peaks were found in the XRD pattern for the hydrolysis product of the synthesized Mg_2NiH_4 in distilled water (see Fig. 2b). No Mg_2Ni peaks were detected, which shows that Mg_2NiH_4 had hydrolyzed into $Mg(OH)_2$ and Ni after being immersed in distilled water for 120 h.

$Mg(OH)_2$, Mg_2Ni, and Mg_2NiH_4 peaks were observed in the XRD pattern for the hydrolysis product of Mg_2NiH_4 in the 6 M KOH solution (see Fig. 2c). As was the case for the Mg_2Ni sample in the alkaline solution, no significant Ni peak was observed in the XRD pattern (Fig. 2c). However, some Ni particles were detected in the hydrolysis product using the paramagnetism test.

As noted, no significant Ni peaks were observed in the XRD patterns for both samples immersed in an alkaline solution. However, some Ni particles were detected using the paramagnetism test. This may be explained as follows: The hydrolysis products ($Mg(OH)_2$ and Ni particles) are extremely fine. Hence, their diffraction peaks are of low intensity, and are broad. The amount of $Mg(OH)_2$ is relatively higher than Ni because of the surplus Mg in the initial material. Some Ni peaks can also overlap with peaks from $Mg(OH)_2$ and Mg_2Ni.

After the $Mg(OH)_2$ in the hydrolysis product in distilled water was carefully removed using dilute hydrochloric acid, the XRD results show that the final product consists of Ni and a small amount

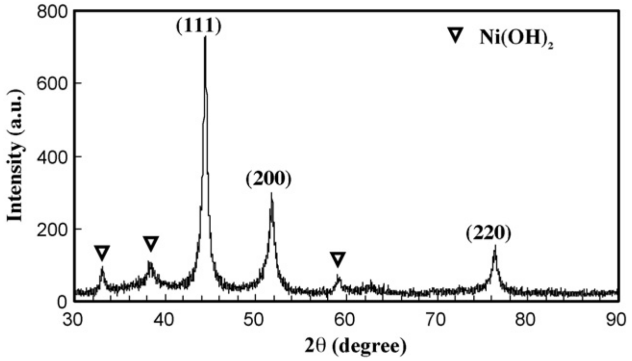

Fig. 3. XRD powder patterns for Ni nanoparticles.

of $Ni(OH)_2$ (see Fig. 3). TEM image shows that the final product nanoparticles was a roughly spherical shape with a size of about 10 nm (see Fig. 4a). The result of EDS (see Fig. 5) shows that some Mg and oxygen impurities exist in the Ni nanoparticles. These impurities might come from some remaining $Mg(OH)_2$ and the oxides of the Ni nanoparticles. High-resolution TEM image (see Fig. 4b) shows that these nanoparticles are Ni nanoparticles because the spacing of the lattice fringes was about 0.208 nm, close to 0.204 nm (the spacing of the (1 1 1) planes in Ni).

4. Discussion

Mg can react with water and form $Mg(OH)_2$ thereby releasing hydrogen gas. The reaction can be written as follows:

$$Mg(s) + 2H_2O(l) \rightarrow Mg(OH)_2(s) + H_2(g) \tag{1}$$

where, s, l, and g in brackets denote the solid, liquid and gas state, respectively. The standard free energy change for $Mg(OH)_2$ and H_2O are −833.7 kJ/mol and −237.14 kJ/mol, respectively [18]. The standard free energy change, $\Delta G_1{}^{o}$, of Reaction (1) is about −359.42 kJ/mol. The free energy change ΔG_1 of Reaction (1) is given as

$$\Delta G_1 = -359.42\,\text{kJ/mol} + RT\ \ln \frac{\alpha_{Mg(OH)_2} P_{H_2}}{\alpha_{Mg} \alpha^2_{H_2O}} \tag{2}$$

where, α is activity, and P is partial pressure.

Under the conditions of hydrolysis (abundant water and a limited amount of Mg_2Ni), two possible reactions for the hydrolysis of Mg_2Ni are as follows:

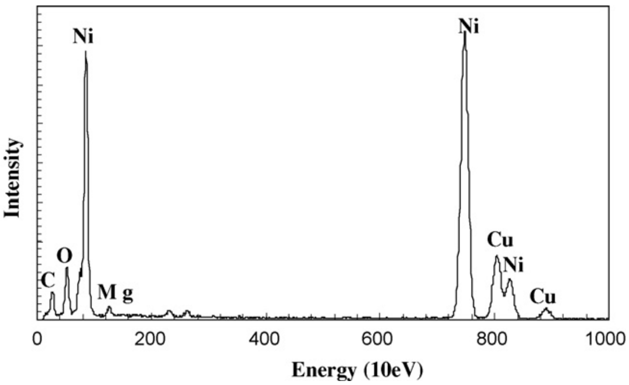

Fig. 5. The EDS spectrum of Ni particles produced through hydrolysis of Mg_2Ni in distilled water.

$$Mg_2Ni(s) + 3H_2O(l) \rightarrow \tfrac{3}{2}Mg(OH)_2(s) + \tfrac{1}{2}MgNi_2 + \tfrac{3}{2}H_2(g) \tag{3}$$

$$Mg_2Ni(s) + 4H_2O(l) \rightarrow 2Mg(OH)_2(s) + Ni + 2H_2(g) \tag{4}$$

Reaction (3) can be thought as an intermediate step of Reaction (4) if the further hydrolysis of $MgNi_2$ can take place. This reaction is given as follows:

$$MgNi_2(s) + 2H_2O(l) \rightarrow Mg(OH)_2(s) + 2Ni + H_2(g) \tag{5}$$

The standard free energy change for Mg_2Ni and $MgNi_2$ are −51.9 kJ/mol and −61.1 kJ/mol, respectively [19]. The free energy changes ΔG_3, $\Delta G_4$and $\Delta G_{5,}$ are given as follows:

$$\Delta G_3 = -517.78\,\text{kJ/mol} + RT\ \ln \frac{\alpha^{\frac{3}{2}}_{Mg(OH)_2} \alpha^{\frac{1}{2}}_{MgNi_2} P^{\frac{3}{2}}_{H_2}}{\alpha_{Mg_2Ni} \alpha^3_{H_2O}} \tag{6}$$

$$\Delta G_4 = -666.94\,\text{kJ/mol} + RT\ \ln \frac{\alpha^2_{Mg(OH)_2} \alpha_{Ni} P^2_{H_2}}{\alpha_{Mg_2Ni} \alpha^4_{H_2O}} \tag{7}$$

$$\Delta G_5 = -298.32\,\text{kJ/mol} + RT\ \ln \frac{\alpha_{Mg(OH)_2} \alpha_{Ni} P_{H_2}}{\alpha_{MgNi_2} \alpha^2_{H_2O}} \tag{8}$$

The hydrolysis conditions in this study are room temperature and one atmosphere pressure. Therefore, the free energy changes for the reactions can be approximately considered as the standard free energy changes, if the activities are neglected. The negative standard free energy changes for Reactions (1), (3)–(5) indicate that

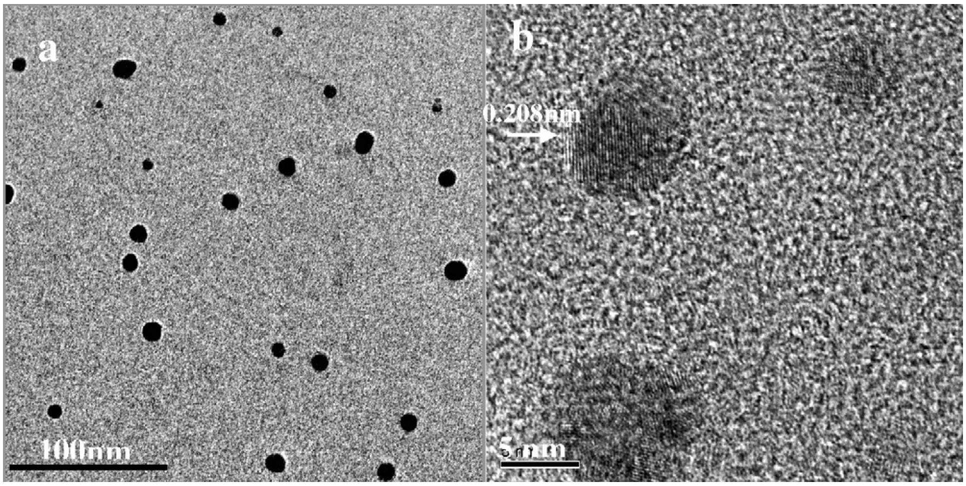

Fig. 4. Morphology of Ni nanoparticles after removal of $Mg(OH)_2$ (a) TEM image, (b) high-resolution TEM image.

these reactions are spontaneous. The chemical affinities for Reactions (3)–(5) are in the following order:

$$\Delta G_4 < \Delta G_3 < \Delta G_5 \tag{9}$$

Therefore, Reaction (4) is more favourable than Reaction (3). In addition, if there is an intermediate Reaction (3), $MgNi_2$ should not start to hydrolyze until the hydrolysis is complete for all Mg_2Ni since Mg_2Ni is more active than $MgNi_2$. Both Mg_2Ni and Ni, but no $MgNi_2$ were found in the hydrolysis product. Thus, Mg_2Ni probably directly hydrolyzed into Ni.

When the ball-milled Mg_2Ni particles were immersed in the distilled water, a small amount of the surplus Mg and a larger amount of Mg_2Ni gave rise to many small cells with small cathodes and large anodes. Thus, Reaction (1) is enhanced, and the hydrolysis reaction for Mg_2Ni was inhibited until all the Mg was consumed. At that stage, the hydrolysis of Mg_2Ni would begin.

There is not a large difference in the thermodynamics for hydrolysis between the ball-milled Mg_2Ni particles in water and in the 6 M KOH solution. However, the activities of $Mg(OH)_2$ and H_2O are significantly affected.

Reaction (4) can be written as two ionic reactions:

$$Mg_2Ni(s) + 4H^+ \rightarrow 2Mg^{2+} + Ni + 2H_2(g) \tag{10}$$

$$Mg^{2+} + 2OH^- \leftrightarrow Mg(OH)_2(s) \tag{11}$$

The solubility of product constants of $Mg(OH)_2$ is 5.6×10^{-12}. The OH^- concentration is estimated as 2.237×10^{-4}. The corresponding pH value is 10.4, which agrees with the pH value (10–11) of the solution in the samples immersed in distilled water. Kuji et al. [8], reported that the pH value is 11.2. The pH value of the 6 M KOH solution is higher than 14. Hence, the concentration of the H^+ ions in the samples immersed in the 6 M KOH solution is much lower than that of the samples immersed in water. Therefore, the hydrolysis rate of Mg_2Ni in the 6 M KOH solution is much slower that that in water. In other words, if the pH value of the solution is reduced (adding some acid), the hydrolysis rate of Mg_2Ni will be greatly increased.

MgH_2 and Mg_2NiH_4 in the hydrogenated samples can react with water and form $Mg(OH)_2$, and release hydrogen gas. The reaction can be written as follows:

$$MgH_2(s) + 2H_2O(l) \rightarrow Mg(OH)_2(s) + 2H_2(g) \tag{12}$$

The XRD patterns for the hydrolysis product of Mg_2NiH_4 in water and the alkaline solution suggest that Mg_2NiH_4 was first dissociated into Mg_2Ni and hydrogen. The dehydrogenation of Mg_2NiH_4 can be written as:

$$Mg_2NiH_4(s) \rightarrow Mg_2Ni(s) + 2H_2(g) \tag{13}$$

The Mg_2Ni will further hydrolyze into $Mg(OH)_2$ and Ni in water and in an alkaline solution (Reaction (4)). The overall reaction for the Mg_2NiH_4 hydrolysis is:

$$Mg_2NiH_4(s) + 4H_2O(l) \rightarrow 2Mg(OH)_2(s) + Ni + 4H_2(g) \tag{14}$$

The standard free energy change for MgH_2 and Mg_2NiH_4 are −35.9 kJ/mol, −64.4 kJ/mol, respectively. The free energy changes $\Delta G_{12,}$ ΔG_{13} and ΔG_{14} for Reactions (12)–(14) are given as:

$$\Delta G_{12} = -323.52\,\text{kJ/mol} + RT\ \ln \frac{\alpha_{Mg(OH)_2} P^2_{H_2}}{\alpha_{MgH_2} \alpha^2_{H_2O}} \tag{15}$$

$$\Delta G_{13} = 12.5\,\text{kJ/mol} + RT\ \ln \frac{\alpha_{MgNi} P^2_{H_2}}{\alpha_{MgNiH_4}} \tag{16}$$

$$\Delta G_{14} = -654.44\,\text{kJ/mol} + RT\ \ln \frac{\alpha^2_{Mg(OH)_2} \alpha_{Ni} P^4_{H_2}}{\alpha_{Mg_2NiH_4} \alpha^4_{H_2O}} \tag{17}$$

From a thermodynamics point of view, the hydrolysis of Mg is easier than MgH_2 since $\Delta G_1 < \Delta G_{12}$. However, it was observed that the hydrolysis of MgH_2 is more severe than for Mg regardless of whether it is in water or in an alkaline solution. Possible reasons for this behaviour as follows: There is a volume expansion of 32% accompanying the transition from hcp Mg to rutile MgH_2. Hence, the bonds in MgH_2 are greatly weakened. In addition, MgH_2 is an ionic compound, which is more easily affected by the ionic water.

Mg_2NiH_4 has a monoclinic antiflurite structure, built of an irregular tetrahedral $[NiH_4]$ complex surrounded by a distorted cube of magnesium ions [20]. H atoms prefer to be located in the neighbourhood of the Ni atoms and are covalently bonded with Ni forming a complex of nominal composition NiH_4. The NiH_4 is ionically bonded to magnesium [21]. The ionic characteristic of Mg_2NiH_4 may be the reason for the release of hydrogen on immersion in water.

The hydrolysis characteristics of Mg_2Ni and Mg_2NiH_4 in water and in alkaline solutions may be the reason that Mg_2Ni exhibited a rapid degradation and a poor cycle life in alkaline solution. Hence, Mg_2Ni is not suitable for use as electrodes in rechargeable batteries. In addition, their electrochemical discharge capacities cannot be improved through generating nano-crystalline Mg_2Ni since reducing the Mg_2Ni particle size will accelerate its hydrolysis rate.

After removal of $Mg(OH)_2$ in the hydrolysis product obtained in distilled water using dilute hydrochloric acid, Ni nanoparticles can be obtained. Therefore, the hydrolysis of Mg_2Ni and Mg_2NiH_4 provides with a new and relatively simple method for producing nickel nanoparticles. Compared with other more conventional synthesis methods for Ni nanoparticles, this method offers a potential to produce Ni nanoparticles on a large scale. In addition, there are many similar binary magnesium intermetallic compounds, such as Mg_2Cu, Mg_3Ag, Mg_3Au, Mg_3Pt and Mg_3Pd that had been successfully used to produce these transition metal nanoparticles by a similar hydrolysis technique [22].

5. Conclusions

The main conclusions from this study are as follows:

1. When Mg_2Ni is immersed in water or in an alkaline solution, it will spontaneously react with water to form $Mg(OH)_2$, Ni and hydrogen.
2. When Mg_2NiH_4 is immersed in water or in an alkaline solution, it will spontaneously first dissociate into Mg_2Ni and hydrogen. The Mg_2Ni will then further hydrolyze into $Mg(OH)_2$ and Ni.
3. Reducing the pH value of the solution (adding an acid) will accelerate the hydrolysis of Mg_2Ni and Mg_2NiH_4.
4. The hydrolysis characteristics of Mg_2Ni and Mg_2NiH_4 suggest that they are not suitable for use as electrodes in rechargeable batteries.
5. Ni particles resulting from hydrolysis of Mg_2Ni are roughly spherical in shape and have a very small particle size (nano range).
6. The hydrolysis of Mg_2Ni and Mg_2NiH_4 provides a new and relatively simple method for producing nano-size nickel particles.

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