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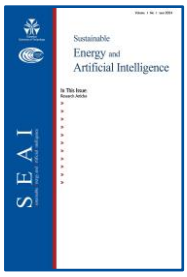

Hydrogen Storage Properties of PMMA Coated MgH_2 - Nb_2O_5 Composite Powder Prepared by High Energy Ball Milling

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Abstract

High temperature of hydrogen desorption from MgH_2 (about 350°C) and its slow kinetics are the main challenges in using this material for solid-state hydrogen storage. This research aims to study the effect of adding Nb_2O_5 on reducing the temperature of hydrogen desorption and using polymethylmethacrylate (PMMA) coating to prevent the oxidation of MgH_2 particles and improve the kinetics of hydrogen desorption. For this purpose, three samples include a: as-received MgH_2, b: MgH_2 milled for 4 h and coated with PMMA, and c: milled mixture of MgH_2 – 5wt. % Nb_2O_5 and coated with PMMA were produced. The prepared samples were analyzed using TG-DSC, XRD, EDS, and FESEM. The results showed that adding Nb_2O_5 and coating with PMMA reduces the hydrogen desorption temperature (about 40°C) compared to the as-received MgH_2. The amount of hydrogen desorption up to 310°C for samples a, b, and c was measured as 2.2, 1.6, and 1.7wt. %, respectively. The amount of hydrogen desorption after 39 minutes at 310°C was 3.5, 1, and 1wt. % for the mentioned samples, respectively. Studying the hydrogen desorption properties of the samples after 45 days showed the positive effect of the PMMA coating in preventing the oxidation of MgH_2.

Keywords: Hydrogen storage; Magnesium hydride; Ball milling; Polymethylmethacrylate; Niobium oxide.

1. Introduction

Nowadays, one of the main challenges facing the world is to provide the required energy, supplied mainly by fossil fuels. However, fossil fuels are limited in nature and will be exhausted in the future. Furthermore, fossil fuels brought serious environmental concerns to the globe. Consequently, the development and use of new and renewable energy sources have attracted much attention in recent years. One of the renewable energy sources is hydrogen, used in fuel cells. Hydrogen is considered a clean and renewable fuel since the initial material for hydrogen generation is water, accessible everywhere. Moreover, hydrogen reaction with oxygen produces water vapor that is not harmful to the environment [1-3].

On a mass basis, the energy density of hydrogen is nearly 3 times that of gasoline, cf. 120MJ/kg for hydrogen and 44MJ/kg for gasoline. In contrast,

the volumetric energy density of hydrogen is so low, i.e., 0.01MJ/L concerning 32MJ/L for gasoline, at STP conditions. Hence, significant difficulty in storing large quantities of hydrogen has become one of the main challenges of hydrogen usage. Hydrogen can be stored in three forms: solid, liquid, and gas. Storing hydrogen in liquid or gas form has received little consideration because of safety issues and requires big storage tanks. Currently, hydrogen storage investigations have been focused on the solid form of hydrogen. Some metal hydrides offer the possibility of solid-state hydrogen storage. The related process involves (i) adsorption, (ii) reaction of hydrogen with metal, alloy, or compounds, and (iii) release of hydrogen under specific conditions [4-6].

Magnesium is one of the attractive hydrogen storage materials. It is a lightweight hydride with high storage capacity (7.6wt.%) and low cost of production. However, the main concerns of using magnesium hydride as hydrogen storage material are low temperature and pressure of hydrogen release and high enthalpy of hydrogen desorption reaction. Another drawback of this metal hydride is its high affinity for air. The surface of the magnesium hydride oxidizes rapidly in contact with air, which leads to capacity reduction and low rates of hydrogen absorption/desorption [7].

Employing catalyst materials and mechanical milling are appropriate solutions to improve the hydrogen absorption/desorption characteristics of hydrides. In previous investigations, various elements and compounds such as Al, Cr, Nb, Ni, Ti, V, Zr and their oxides, $Mg_2Ni/TiH_{1.5,}$ Ni MOF, VTiMn, 2D layered WS_2 and MoS_2, AlCuFeNiTi high antropy alloy, and $Co_3V_2O_8$ have been used as catalysts to increase the absorption/desorption features of hydrogen [8-20]. However, the mentioned catalysts do not prevent surface oxidation. A novel approach for avoiding surface oxidation of magnesium hydride is to confine it in the polymer matrix with selective permeability for hydrogen [21-23]. The addition of niobium oxide as a catalyst agent to magnesium hydride reduces the release temperature of hydrogen and improves the reaction kinetics [12,24-27]. Polymethylmethacrylate (PMMA), as a coating or matrix for magnesium hydride, is selectively permeable to hydrogen and prevents the passage of oxygen. Therefore, it prevents oxygen and water vapor molecules in the air from reaching the magnesium hydride surface. Thereby, PMMA avoids oxidation of magnesium hydride and enhances hydrogen release properties [22].

The aim of this research is to investigate the effect of mechanical milling, niobium oxide addition as a catalyst, and PMMA as a surface oxidation avoiding agent on the hydrogen sorption properties of MgH_2 such as desorption temperature, kinetics and chemical phases, from magnesium hydride.

2. Materials and Methods

The starting materials used in this research were magnesium hydride, niobium oxide, polymethyl methacrylate (PMMA), and acetone. Table 1 shows the relevant specifications of these materials.

Table 1. Specifications of the starting materials used in this study

Material	Purity (wt.%)	Particle Size (μm)	Supplier
MgH_2	> 98	100	Sigma Aldrich
Nb_2O_5	> 99	1	Alfa Aesar
PMMA (MW=120000)	> 98	---	Sigma Aldrich
Acetone	High Purity	---	Merck

Mechanical milling was performed using a planetary ball mill machine (model PM-200) containing two milling vials manufactured by Asia Sanat Rakhsh, Iran. The milling process used wear-resistant chromium steel balls with diameters of 7, 10, and 13mm, a milling ball-to-powder weight ratio of 10:1, and hardened chromium steel vials with a volume of 250cm^3.

80mL of acetone and 2g of magnesium hydride were poured into milling vials. Then, 5wt.% of the magnesium hydride was added to the niobium oxide powder in one of the vials to prepare the samples. The other vial contains magnesium hydride without any addition of niobium oxide. Since the magnesium hydride powder and acetone mixture are highly pyrophoric, mechanical milling was performed under an argon atmosphere, and the vial lid was sealed carefully with silicone paste. After milling, the content of each vial was transferred to a laboratory flask, and 0.5g PMMA (equivalent to 25wt% of magnesium hydride) was added and stirred magnetically until complete dissolution of PMMA in acetone. Then, solutions were discharged into a beaker and placed in an ultrasonic bath at 50-60°C until evaporation of acetone and then polymerization of PMMA. The milled powders were embedded in the PMMA matrix using the mentioned procedure.

XRD analysis using Philips Xpert pro diffractometer (40kV, 100mA, Cu-Kα radiation of wavelength 1.54Å and step size of 0.02°) was

utilized to identify the initial and resulting phases during milling of magnesium hydride and niobium oxide. XRD data analysis was performed using X'Pert High Score Plus software. Morphology of the samples, coating conditions of magnesium hydride powders with PMMA, and niobium oxide distribution in magnesium hydride were investigated by Field-Emission Scanning Electron Microscope (FESEM) using an FEI NanoSEM 450 instrument. For FESEM analysis, the samples were placed on an Al-support using double-sided carbon tape and then coated with gold. Image J software was used to measure particle size, mean particle size, and size distribution. For each sample, 40 to 50 measurements were carried out, and the average value was reported. FTIR (Fourier Transform Infrared) spectroscopy (Thermo-Electron, model AVATAR, USA) was also employed to identify the PMMA polymer and PMMA re-polymerization from acetone. The dehydrogenation behavior of the samples was evaluated by Thermo-Gravimetric analysis (TGA) using PerkinElmer, model STA 6000 instrument. The TGA method was carried out under an argon atmosphere (5N purity). In TGA experiments, the temperature of the samples was raised from room temperature to 310°C with a heating rate of 10°C/min and maintained for 10 min at 310°C. To ensure accurate dehydrogenation results, the TGA experiment was performed at the same conditions for PMMA polymerized from acetone to determine the mass reduction of milled PMMA. Subsequently, the mass reduction of PMMA was subtracted from the obtained TGA curves of the samples mixed with PMMA. Hence, the resulting TGA curves indicate mass reduction of magnesium hydride. TGA experiments were performed for coated magnesium hydrides immediately after sample preparation and once again after 45 days of air exposure to investigate the coating effect on preventing oxidation of magnesium hydrides and the efficiency of the polymeric coating.

3. Results and Discussions

3-1. Morphology of Initial Powders

Fig. 1 shows the FESEM image of the starting magnesium hydride powder. This figure shows that the particles do not have a uniform shape. Fig. 2 depicts the size distribution of the particles, which is in the range of 30 to 100µm.

3-2. Effect of Milling on Particle Size

Fig. 3 displays the FESEM image of magnesium hydride embedded in PMMA after 4 hours of mechanical milling. The average particle size decreased from 80µm (for starting magnesium hydride powder) to 1.8µm. Fig. 4 shows the size distribution of magnesium hydride after mechanical milling. Based on this figure, the size of the particles was mainly in the range of 1 to 4µm, and the average particle size was 1.8µm. Mechanical milling changed the morphology of the particles from a lumpy shape (Fig. 1) to an almost flat-shaped form (Fig. 3). This flat-shaped form is beneficial for hydrogen release because of its freer surface area. Furthermore, particles have smaller thicknesses in the flat-shaped form (according to Figs. 1 and 3 and their scale bar), and released hydrogen atoms would travel a shorter distance to reach the surface and combine as hydrogen molecules.

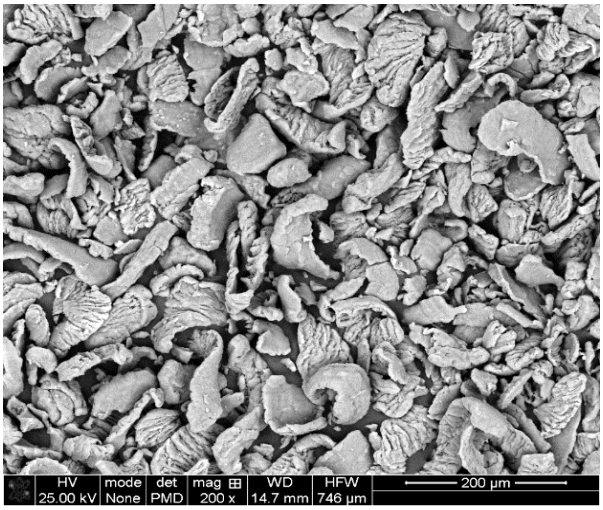

Fig. 1. FESEM image of the initial magnesium hydride powder.

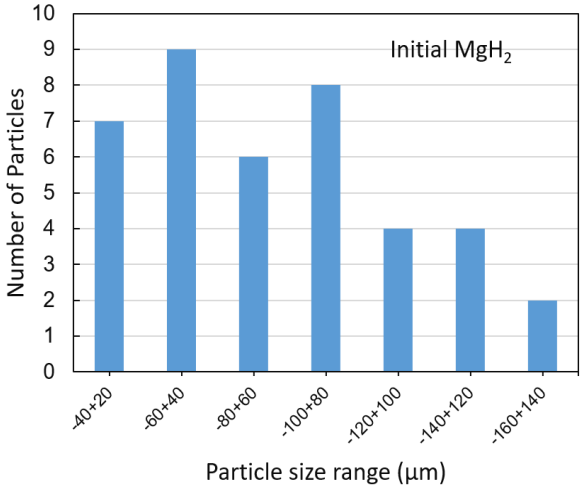

Fig. 2. Size distribution of the initial MgH_2 powder

Fig. 5 shows the FESEM image of PMMA-embedded magnesium hydride containing 5wt%

niobium oxide milled for 4 hours. Fig. 6 illustrates the corresponding size distribution measured with Image J software. Mechanical milling has a considerable effect on the size reduction of magnesium hydride containing 5wt% niobium oxide. Niobium oxide is a much harder phase than magnesium hydride, and its presence facilitates the size reduction of magnesium hydride during mechanical milling [8,28].

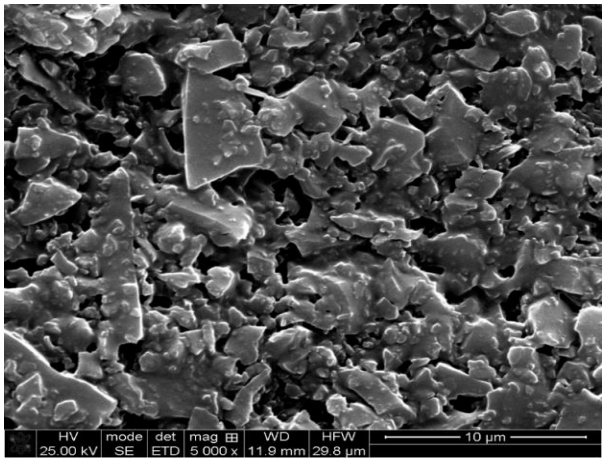

Fig. 3. FESEM image of PMMA-embedded MgH_2 powder after 4h of ball milling

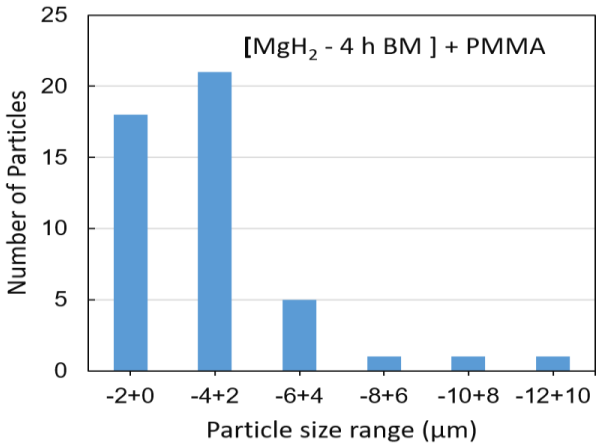

Fig. 4. Size distribution of PMMA-embedded MgH_2 powder after 4h of ball milling

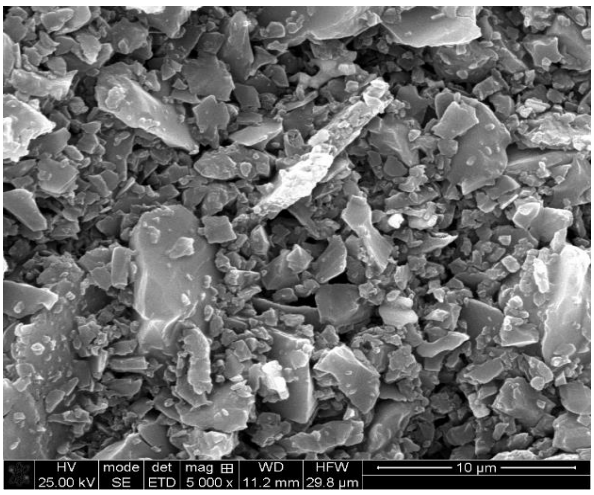

Fig. 5. FESEM image of PMMA-embedded MgH_2 - 5wt% Nb_2O_5 powder after 4h of ball milling

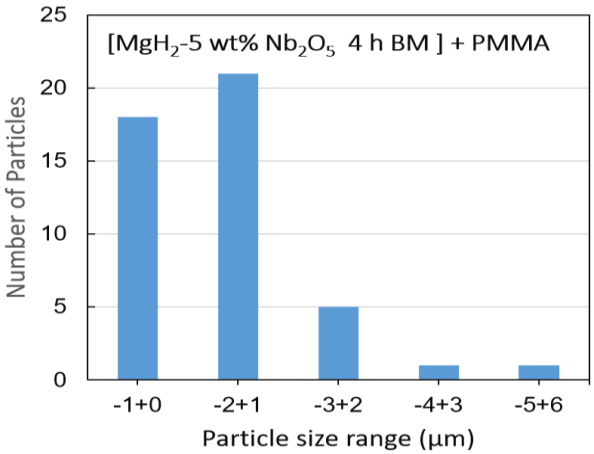

Fig. 6. Size distribution of PMMA-embedded MgH_2 - 5wt% Nb_2O_5 powder after 4h of ball milling

3-3. Effect of Bball Milling on the Powder Composition

Fig. 7 shows the EDS map analysis of PMMA-embedded magnesium hydride powder containing 5wt.% niobium oxide after 4 hours of mechanical milling. As can be seen from Fig. 7, red and yellow color regions indicate elemental magnesium and niobium, respectively. It is obvious that Mg and Nb were distributed properly on the examined surface. However, accumulation of either Mg or Nb can be identified in some regions.

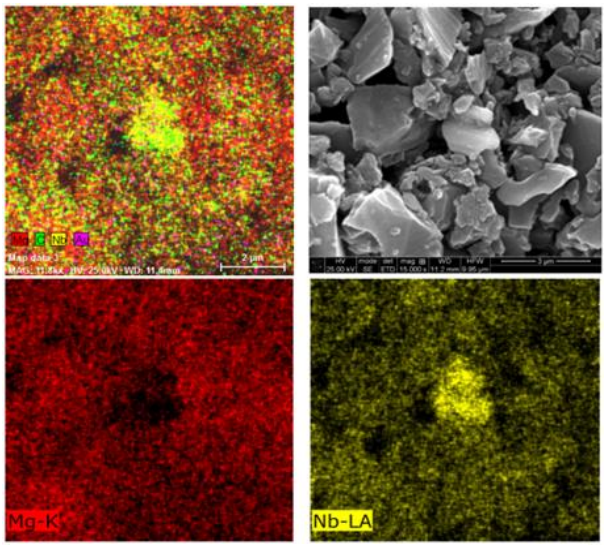

Fig. 7. EDS map analysis of PMMA-coated MgH_2 - 5wt% Nb_2O_5 powder.

3-4. Structural Analysis with XRD

Fig. 8a shows the XRD pattern of milled magnesium hydride powder embedded in PMMA. A weak diffraction peak related to the PMMA phase was observed at about 20° in this figure. Since PMMA coating was used as a thin layer on the surface, it was detected as a minor peak. All the major diffraction peaks in Figure 8a are associated with magnesium hydride. Several minor peaks related to magnesium were also observed that may be related to the magnesium content of the initial powder.

Fig. 8b illustrates the XRD pattern of milled magnesium hydride powder containing 5wt% niobium oxide coated with PMMA. Based on this figure, no diffraction peaks related to magnesium oxide or hydroxide were detected, which implies that no oxide layer was formed in the magnesium hydride powders. Therefore, magnesium hydride particles were protected sufficiently against oxygen molecules and air humidity during the milling, coating, and handling of the prepared samples.

Based on the obtained XRD results, intermetallic compounds containing Mg and Nb were not observed, in contrast to the findings of other researchers [8,29]. This could be due to the milling method used in this work, i.e., wet milling conditions using acetone, concerning dry milling. In other words, the accumulated energy of the wet milling process was insufficient to initiate the reaction between magnesium hydride and niobium oxide.

3-5. Hydrogen Desorption Properties

Fig. 9 shows the results of hydrogen desorption tests as a function of temperature up to 310°C for the three samples, which are (i) initial magnesium hydride, (ii) PMMA-coated milled magnesium hydride, and (iii) PMMA-coated milled magnesium hydride containing 5wt% niobium oxide. Fig. 10 also illustrates the results of hydrogen desorption tests for the three samples as a function of time at 310°C. Referring to Fig. 9, the dehydrogenation amounts up to 310°C was about 2.2wt% for the initial magnesium hydride and around 1.6wt% for the two PMMA-coated samples. The initial powder of magnesium hydride was kept under an argon atmosphere, and its surface oxidation was partially prevented. Therefore, its dehydrogenation amount is higher than the other two samples (i.e. PMMA coated samples). However, due to the thin oxide layer that might be formed during the handling and carrying of the samples, the initial desorption temperature of the initial magnesium hydride was about 40°C higher than that of the coated sample. It means that PMMA coating led to a slight reduction in the starting desorption temperature. It can be concluded from Fig. 10 that at 310°C, the dehydrogenation amount was 3.5wt% and 0.5wt% for the initial magnesium hydride and the two PMMA-coated samples, respectively. Furthermore, the presence of a Nb_2O_5 catalyst has a minor effect on the dehydrogenation amount, possibly due to the lack of compound formation between magnesium hydride and niobium oxide during ball milling.

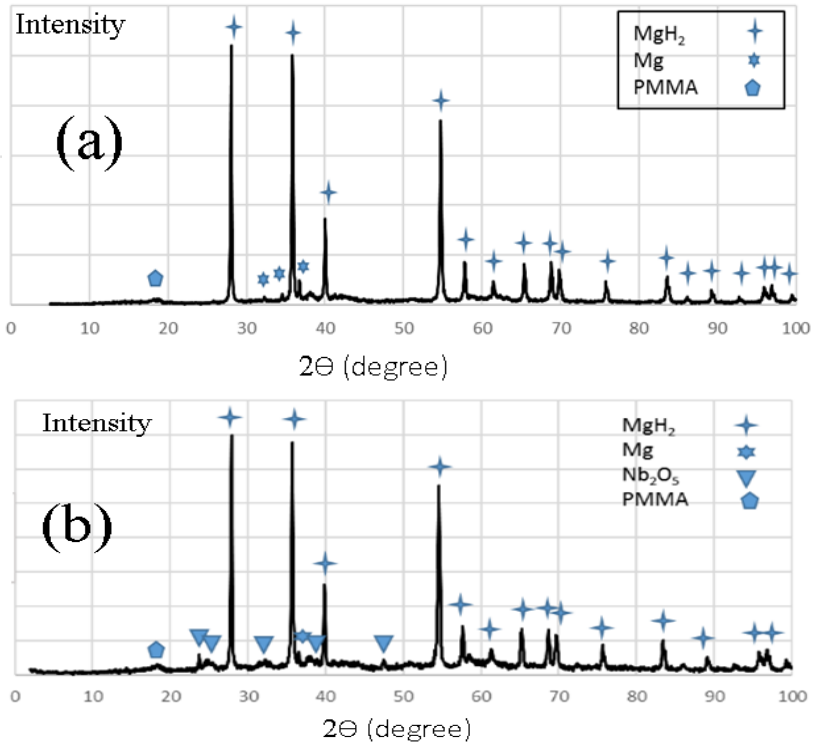

Fig. 8. XRD pattern of (a) Milled MgH_2 powder embedded in PMMA, (b) PMMA-coated MgH_2 - 5wt% Nb_2O_5 powder

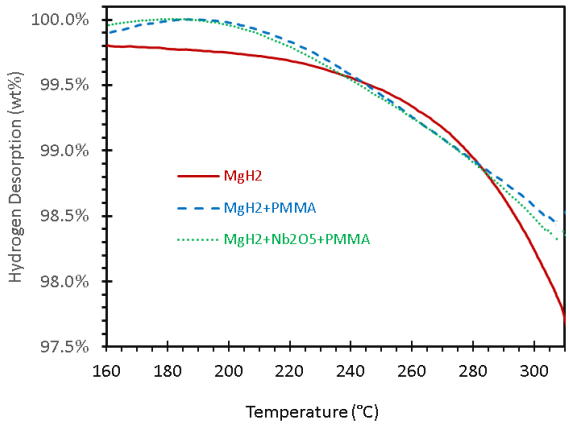

Fig. 9. Hydrogen desorption properties of the samples versus temperature up to 310°C

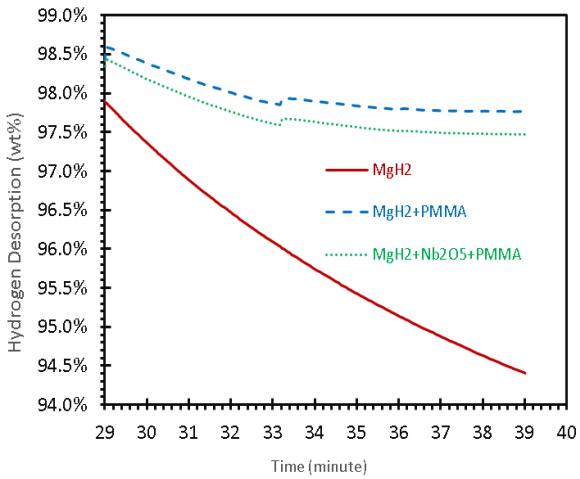

Fig. 10. Hydrogen desorption properties versus time at a constant temperature of 310°C

Fig. 11 shows the dehydrogenation curves as a function of temperature up to 310°C for the three samples exposed to ambient air for 45 days. The dehydrogenation value of the non-coated magnesium hydride powders up to 310°C declined from 2.2wt% to 1.4wt%. The dehydrogenation value of PMMA-coated samples was not altered significantly. Fig. 12 shows the dehydrogenation value of the samples at a constant temperature of 310°C. Based on Table 2 (hydrogen desorption data of the samples) and considering the three samples exposed to ambient air for 45 days, the dehydrogenation value at 310°C was 0.5, 1.2, and 1.5wt% for non-coated magnesium hydride, PMMA coated magnesium hydride and PMMA coated magnesium hydride containing niobium oxide, respectively. It is observed that PMMA coating thoroughly prevented oxidation of magnesium hydride powder after exposure to ambient air for 45 days.

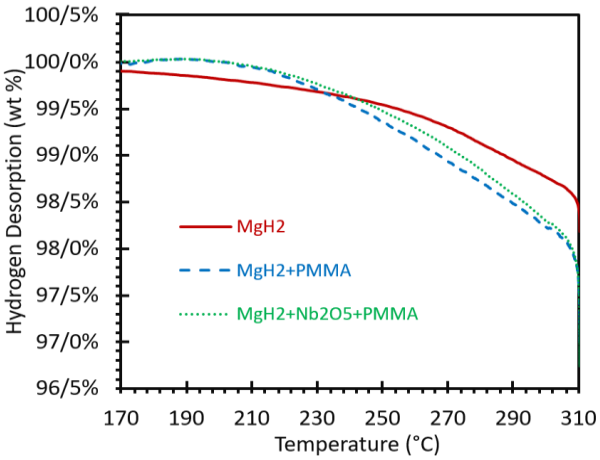

Fig. 11. Hydrogen desorption properties versus temperature for the samples exposed to ambient air for 45 days

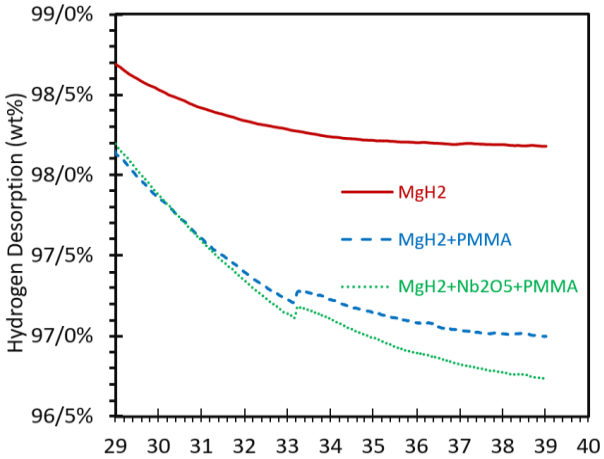

Fig. 12. Hydrogen desorption properties versus time for the samples exposed to ambient air for 45 days

Table 2. Hydrogen desorption data of the samples.

Sample		MgH_2	MgH_2 + PMMA	MgH_2 + Nb_2O_5 + PMMA
H_2 desorption on-set temperature (°C)	As-prepared	240	200	200
	After 45 days	240	200	200
Desorption up to 310°C	As-prepared	2.2	1.6	1.7
	After 45 days	1.4	1.9	1.9
Desorption at 310°C	As-prepared	3.5	1.0	1.0
	After 45 days	0.5	1.2	1.5

4. Conclusions

The following results were obtained from this research:

1) Although the energy of the mechanical ball milling performed in acetone was low, the size of the ball-milled MgH_2 particles was quite small. The corresponding average size was reduced from 100µm to 2µm. However, the energy of the milling process was insufficient for the reaction between magnesium hydride and niobium oxide.
2) The onset dehydrogenation temperature of MgH_2 was reduced from 240°C to about 200°C by applying PMMA coating to prevent surface oxidation.
3) The hydrogen desorption amount diminished from 2.2wt% to 1.6wt% up to a temperature of 310 °C and from 3.5wt% to 1wt% at a constant temperature of 310°C because of the barrier of PMMA coating on MgH_2 to hydrogen movement.
4) Thermogravimetric analysis of the samples exposed to ambient air for 45 days revealed that the hydrogen desorption amount was not changed concerning the initial samples (i.e., non-exposed to air). This observation implies that PMMA coating completely protected MgH_2 powders from oxygen and humidity contact.

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