

# 기계적 합금화법으로 제조한 $Mg_2NiH_x$ -Graphene 복합재료의 수소화 특성 평가

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## Evaluation of Hydrogen Properties on $Mg_2NiH_x$ -Graphene Composites by Mechanical Alloying

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**Abstract** >> Mg hydride has a high hydrogen capacity (7.6%), at high temperature, and is a lightweight and low cost material, thus it a promising hydrogen storage material. However, its high operation temperature and very slow reaction kinetics are obstacles to practical application. In order to overcome these disadvantages of Mg hydride, graphene powder was added to it. The addition of graphene has been shown to reduce the operating temperature of dehydrogenation.

Moreover, in this report the environmental aspects of  $MgH_x$ -Graphene composites are investigated by means of the environmental life cycle assessment (LCA) method.  $MgH_x$ -Graphene mixture was prepared by hydrogen induced mechanical alloy (HIMA). The synthesized powder was characterized by XRD(X-ray Diffraction). The hydrogenation behaviors were evaluated by using a Sievert's type automatic PCT apparatus. Such evaluation of Materials also conducted in the LCA.

From the result of P-C-T(Pressure-Composition-Temperature) curves, the  $MgH_x$ -3wt.% graphene composite was evaluated as having a 5.86wt.% maximum hydrogen storage capacity, at 523K. From absorption kinetic testing, the  $MgH_x$ -7wt.%graphene composite was evaluated as having a maximum 6.94wt.%/ms hydrogen absorption rate, at 573K. Environment evaluation results for the  $MgH_x$ -graphene composites and other materials indicated environmental impact from the electric power used and from the materials themselves.

**Key words** : Hydrogen storage materials(수소저장자료), Hydrogenation properties(수소화 특성), Reactive mechanical alloying(기계적 합금화법),  $Mg_2NiH_x$ -graphene, Life Cycle Assessment(환경전과정평가)

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## 1. Introduction

In recent years, serious environmental concerns regarding pollution have emerged, while competition for energy resources has intensified<sup>1,2)</sup>. To solve these problems, the introduction of a fuel cell system to promote the hydrogen economy is important<sup>3)</sup>. Hydrogen has attracted attention as an ideal energy resource and is expected to be applied in fuel cells. Thus, there is a need for high efficiency catalysts and for a safe technology for room temperature hydrogen storage. Many metal hydrogen systems, such as those made from Mg-based hydrides, are promising as solutions for hydrogen storage. However, the hydrogen absorption/desorption kinetics of such materials are too low for practical application.

The main reasons for the slow hydrogen sorption kinetics are an oxide layer on the magnesium surface and blocked hydrogen diffusion<sup>4)</sup>. In order to improve hydrogen sorption/desorption kinetics, ball milling is most widely used. Even though it usually takes a longer time to prepare the alloy by this method, ball milling prevent magnesium hydride from creating a solid layer on the surface.

Carbon nano-materials such as graphene, fullerenes, and carbon nanotubes have been widely studied and applied not only in the electronics and chemical fields but also in hydrogen storage. This is because carbon materials offer many advantages, such as a low mass density and large surface area<sup>5)</sup>.

In a previous paper<sup>6)</sup>, it was determined that graphene is an efficient destabilizing agent for MgH<sub>2</sub>, resulting in a decrease of the absorption temperature of this phase while still maintaining a temperature high enough for practical applications. Catalysis on the hydriding

reaction indicated that the absorption kinetics and hydrogen storage capacity increase in proportion to the concentration of the substitutional graphene. We seek fundamental knowledge of this system and understanding of the effect of graphene content on the structure and hydrogenation properties of the synthesized materials. In addition, we seek to identify the most environmentally important activities within the life cycle of MgH<sub>x</sub>-graphene composites, for which purpose an environmental life cycle assessment (LCA) was conducted.

## 2. Experimental Procedure

MgH<sub>x</sub> was prepared by ball milling Mg powder (99.9%) under a H<sub>2</sub> atmosphere at a pressure of 2MPa for 48hrs. The ball to chips weight ratio was 40:1, and the mill's rotation speed was 200rpm. The composites of 3, 7wt.% graphene (12nm flake, grade: AO-3, Graphene-Supermarket) and the MgH<sub>x</sub>. Previously obtained were than mechanically milled for 72hrs using a planetary ball mill(Pulverisette-5, FRITSCHE Co.).

X-ray diffraction analysis(XRD, D8 Advance, Bruker) was performed using a Cu K $\alpha$  radiation of 1.5405 Å (scanning speed: 3deg/min, scanning angle: 20~80°).

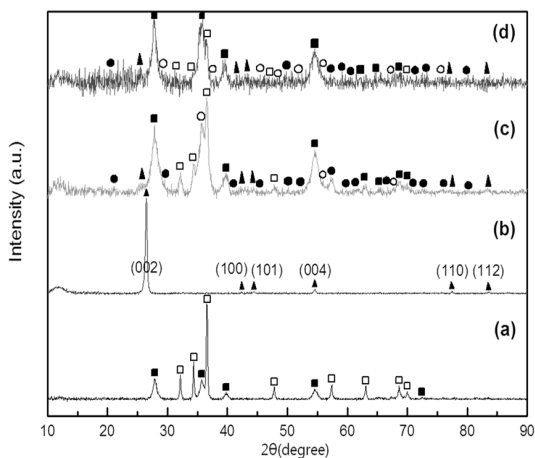
The hydrogen sorption properties of the composites were determined using an automated Sievert's apparatus. The composites were activated under an activation treatment condition. After activation, the Pressure-Composition-Isothermal(PCI) curves were measured at 473K, 523K, 573K and 623K with an H<sub>2</sub> pressure of 5MPa. Absorption and desorption kinetics of the MgH<sub>x</sub>-graphene composites were evaluated under the same temperature for 1hours, under 5MPa hydrogen pressure.

The present LCA is especially worthy of attention as a preliminary indication of the future environmental impact of the production and evaluation processes for MgH<sub>x</sub>-graphene composites. The material requirements and environmental emissions are inventoried and the energy requirements are discussed, based on actual production data. The analysis is performed according to the recommendations of ISO norms 14040<sup>7)</sup>, and updates thereof. The performance of the analyzed MgH<sub>x</sub>-graphene composite is also compared to other examples of Mg<sub>2</sub>Ni based on different materials.

### 3. Results and Discussion

#### 3.1 X-ray diffraction(XRD) analysis

Fig. 1 presents XRD analysis results of the as-prepared samples (Graphene, MgH<sub>x</sub>, MgH<sub>x</sub>-graphene composites). The graphene flakes exhibit a very sharp characteristic peak at  $2\theta=26.55^\circ$ <sup>5)</sup>, whereas the MgH<sub>x</sub>-graphene composites showed a broader peak than that of the graphene. MgC<sub>2</sub> phase and, Mg<sub>2</sub>C<sub>3</sub> phase are



**Fig. 1** X-Ray Diffraction (XRD) of MgH<sub>x</sub>-graphene composites: (a) MgH<sub>x</sub>, (b) graphene, (c) MgH<sub>x</sub>-3wt.% graphene, and (d) MgH<sub>x</sub>-7wt.% graphene (□:Mg, ■:MgH<sub>2</sub>, ▲:graphene, △:graphite oxide, ○:MgC<sub>2</sub>, ●:Mg<sub>2</sub>C<sub>3</sub>)

present in the MgH<sub>x</sub>-graphene composites. All diffraction peaks of MgH<sub>x</sub>-graphene composites correspond to the JCPDS. This indicates that a composite MgH<sub>x</sub>-graphene material suitable for use as a target sample was produced successfully. In previous study<sup>6)</sup>, two phases were also significantly observed in MgH<sub>x</sub>-5wt.% graphene and MgH<sub>x</sub>-10wt.% graphene.

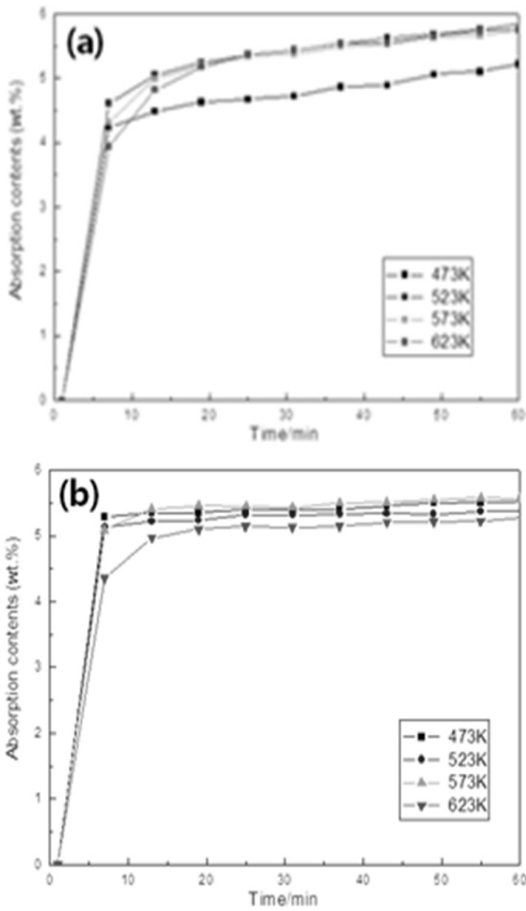
#### 3.2 Hydrogen sorption

##### 3.2.1 Hydrogen absorption kinetics

Fig. 2 shows the absorption curves which illustrate the rate increases as the graphene composites at 473K, 523K, 573K and 623K under 50bar H<sub>2</sub>. The absorption rate increases as the graphene content rises. In particular, hydriding at 523K and 573K occurs at a higher rate than it does at 623K. This effect can be ascribed to the fact that at 523K hydriding is accompanied by formation of MgH<sub>2</sub>. In addition, the magnesium hydride is formed more easily, which means that a large number of nuclei appear on the particle surfaces. This fact determines the higher rate of hydriding at the above temperature.

Comparison with MgH<sub>x</sub>-graphene composites shows that both composites have a similar kinetic behavior when hydride at 523K, 573K, and 623K. However, the presence of Mg<sub>2</sub>C<sub>3</sub> or MgC<sub>2</sub> phases behavior when hydriding at 473K. In the case of MgH<sub>x</sub>-7wt.% graphene, the MgC<sub>2</sub> phase is more dominant than Mg<sub>2</sub>C<sub>3</sub>.

A preceding study shows that MgH<sub>x</sub>-10wt.% graphene was 2.1 and 1.6wt.% at 573 and 673K. MgH<sub>x</sub>-7wt.% graphene and MgH<sub>x</sub>-10wt.% graphene shows a similar trend. In the same previous experiment, it was shown that have a positive effect on both its absorption kinetics and hydrogen capacity. Additionally, shortening

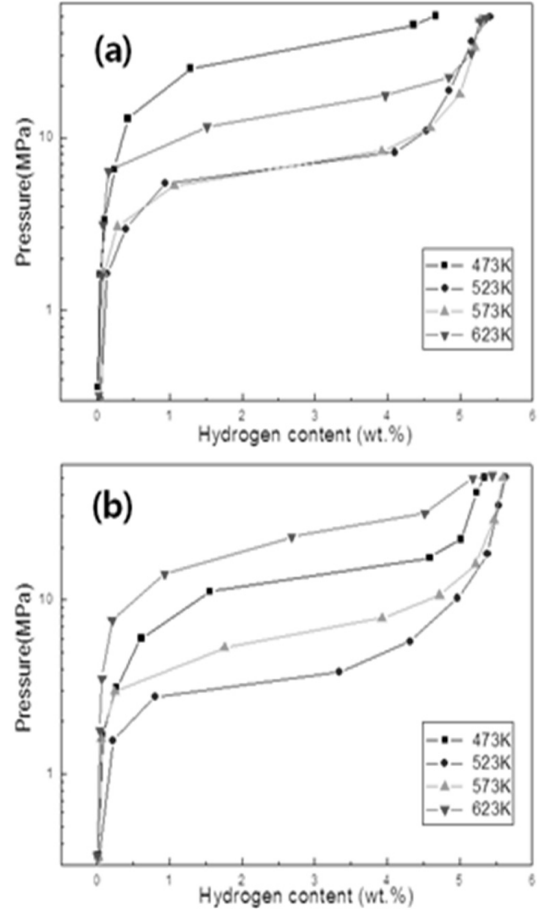


**Fig. 2** Hydrogenation kinetic on (a) MgH<sub>x</sub>-3wt.% graphene and (b) MgH<sub>x</sub>-7wt.% graphene composites under 5MPa of H<sub>2</sub>

of the hydrogenation time is due to formation of a carbon coating layer between particles during ball milling. The carbon coating layer prevents particle agglomeration and restoration of the oxide layer on the fresh cracked surface<sup>4)</sup>.

From 1h absorption times, it can be clearly seen that the hydriding rate of MgH<sub>x</sub>-graphene at all temperatures increased with the rise in graphene content. However for longer absorption times, MgH<sub>x</sub>-7wt.% graphene shows a larger final hydrogen storage capacity.

Hydrogen absorption pressure-composition isotherms



**Fig. 3** The absorption P-C-I curves of (a) MgH<sub>x</sub>-3wt.% graphene and (b) MgH<sub>x</sub>-7wt.% graphene composites from 0.3MPa H<sub>2</sub> to 5MPa H<sub>2</sub>

(PCI) at 473K, 523K, 573K and 623K are shown in Fig. 3. As absorption temperature increased from 473K to 523K to 573K to 623K, the amounts of absorbed hydrogen for MgH<sub>x</sub>-3wt.% graphene were 4.66wt.%, 5.41wt.%, 5.27wt.% and 5.35wt.%, respectively.

### 3.2.2 P-C-I curves

The amounts for MgH<sub>x</sub>-7wt.% graphene were 5.43 wt.%, 5.63wt.%, 5.6wt.%, and 5.45wt.%. It is also notable that the equilibrium pressure for hydrogen absorption of these composites decreases as the amount

of graphene increases, which indicates that graphene tends to support more stable hydrides.

Except at 473K, absorption plateaus increased with increasing temperature and this feature along with the hysteresis result conforms to the classic behavior of metal-hydride systems<sup>8)</sup>.

**Table 1** Hydrogen absorption capacity and Hydrogen absorption rate of MgH<sub>x</sub>-graphene composites

Sample	Temp. (K)50atm	Hydrogen absorption capacity (wt.%)	Hydrogen absorption rate (wt.%/ms)
MgH <sub>x</sub> -3wt.% graphene	473K	4.66	5.74
	523K	5.41	6.47
	573K	5.27	6.4
	623K	5.23	6.18
MgH <sub>x</sub> -7wt.% graphene	473K	5.43	6.86
	523K	5.63	6.69
	573K	5.60	6.94
	623K	5.45	6.37

### 3.3 Environmental life cycle assessment (LCA) of MgH<sub>x</sub>-graphene composites

Conducting experiments is basic to science, and experiments are necessary for the scientific investigation. However, there is also a need to increase our knowledge of the environmental consequences of experiments in order to be able to make improvements that promote sustainability in experimental processes. And during the recent years, No information was available about the environmental consequences of experiments involving materials based on Mg hydride. And research on hydrogen has been focused on life cycle stages about production, storage, transport and use<sup>9)</sup>.

The scope of our environmental impact assessment was the identification of the most environmentally important activities within the life cycle of MgH<sub>x</sub>-graphene composites. The method chosen for the study was an environmental life cycle assessment (LCA).

**Table 2** Environmental impact categories (CML 2001 and Eco-indicator 99)

CML 2001 category	Unit	Eco-indicator 99 category	Unit
Abiotic resource depletion(ARD)	Kg Sb eq	Fossil fuels	MJ
		Minerals	MJ
Acidification potential (ACP)	Kg SO <sub>2</sub> eq	Acidification/Eutrophication	PDF*m <sup>2</sup> *yr
Eutrophication potential (EUP)	Kg PO <sub>4</sub> eq		
Global warming potential (GWP)	Kg CO <sub>2</sub> eq	Climate change	DALY
Ozone layer depletion potential (ODP)	Kg CFC-11eq	Ozone depletion	DALY
Photochemical oxidation potential (POCP)	Kg C <sub>2</sub> H <sub>4</sub> eq	Respiratory(organic)	DALY
Fresh-water aquatic ecotoxicity potential (FAETP)	Kg 1,4 DCBeq	Ecotoxicity	PDF*m <sup>2</sup> *yr
Marine aquatic ecotoxicity potential (MAETP)	Kg 1,4 DCBeq		
Terrestrial ecotoxicity potential(TETP)	Kg 1,4 DCBeq		
Human toxicity potential (HTP)	Kg 1,4 DCBeq	Carcinogenic effect	DALY
		Respiratory(inorganic)	DALY
		Ionizing radiation	DALY
		Land-use	PDF*m <sup>2</sup> *yr

### 3.3.1 Environmental impact categories Impact categories quantitatively present

resource and energy consumption. As shown in Table 2, the categories specified include the key parameters for environmental impact<sup>10,11</sup>. The key parameters are carbon dioxide ( $CO_2$ ), ethylene ( $C_2H_4$ ), chloro fluoro carbon (CFC), sulphur dioxide ( $SO_2$ ), and phosphate ( $PO_4$ )<sup>12,13</sup>.

### 3.3.2 Goal and scope definition

The first component of the goal and scope definition phase is the depiction of the goal of the LCA. This includes the description of the analysed system and boundaries.

In this study the goal of the analysis is to quantify and to evaluate the impact of the  $MgH_x$ -graphene composites production system.

### 3.3.3 System Boundaries and Characteristics

The system can be defined as everything involved in the production of  $MgH_x$ -graphene composites, including the analysis for the composites. Fig. 4 shows a description of the system and each phase.

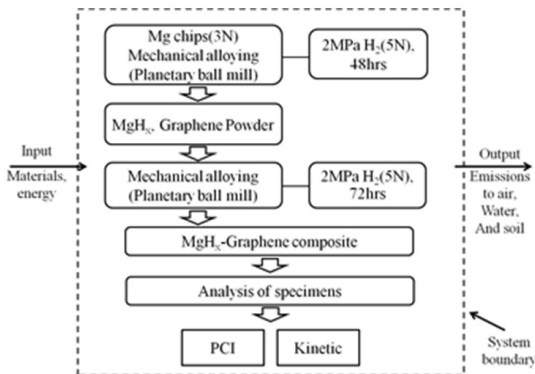


Fig. 4 Discription of  $MgH_x$ -graphene composites system

### 3.3.4 Inventory Data and Information Collection

For inventory data, the European ecoinvent inventory database (v 2.2) was used for this paper Cycle Impact Assessment (LCIA) method developed at the University of Leiden, the so called CML (Centrum voor Milieuwetenschappen (Institute of Environmental Sciences)) method, and key performance indicators were used. The CML method defines several impact categories for emissions and for resource consumption. This method groups by-products, emissions and resource consumption caused by these processes, into specific environmental impacts<sup>14</sup>.

### 3.3.5 Study Limitations

Due to the lack of available information about graphene, we used alternate data (on graphite) in the GaBi databases. In order to obtain the environmental performance of the studied processes, a combination of a Life

### 3.3.6 Life Cycle Inventory results

For the Life Cycle Inventory, the data collected were used in “Gabi” software. Energy input through the production of  $MgH_x$ -graphene,  $Mg_2NiH_x$  and  $Mg_9Ti_1Ni$  are as follows. Three samples of the PCI and kinetic experiments were used at an equal power value. Electricity use is increased when the duration of planetary ball milling is long. For  $MgH_x$ -graphene composites, the ball milling time is 120hours, for  $Mg_2NiH_x$  it is 48hours, and for  $Mg_9Ti_1Ni$  it is 96 hours.

### 3.3.7 Life Cycle Impact assessment results

In this section, the environmental performance of the investigated  $MgH_x$ -graphene,  $Mg_2NiH_x$ , and  $Mg_9Ti_1Ni$  composites is presented. All results presented represent

relative environmental impacts per functional unit.

3.3.7.1 E<sub>i</sub>' 99 methods

Fig. 5 shows the results of the environmental impacts for all three samples. MgH<sub>x</sub>-graphene has the highest impacts, followed by Mg<sub>9</sub>Ti<sub>1</sub>-Ni, and Mg<sub>2</sub>NiH<sub>x</sub>. Energy consumed during the life cycle stage constituted a majority of the impacts for many of the impact

Table 3 Scope Definition

System	Production and analysis of MgH <sub>x</sub> -5wt.% graphene
Function	Storage of hydrogen
Function unit	Hydrogen content (wt.%)
Reference flow	Powder of MgH <sub>x</sub> -graphene (9.5g)

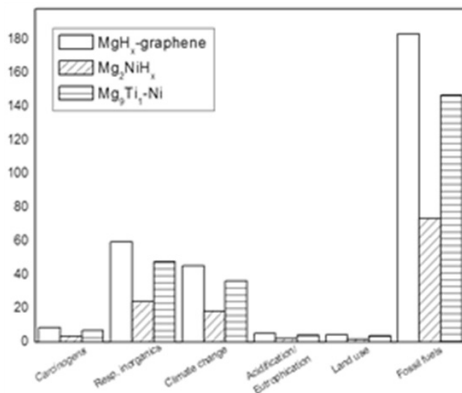


Fig. 5 Comparison of the environmental impacts assessed by applied E<sub>i</sub>'99 methods for three composites.

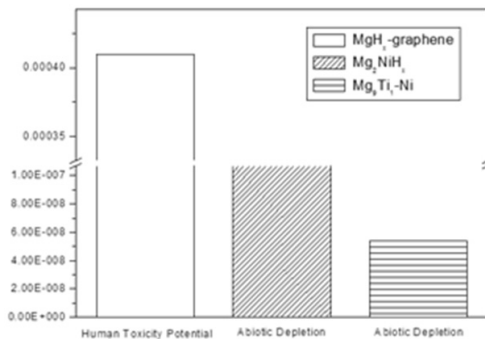


Fig. 6 Comparison of the environmental impacts assessed by applied CML 2001 methods for three composites

categories evaluated. Due to the large influence of electricity, the fossil fuels category is the highest impact category. Also the climate change and Respiratory (inorganic) categories are high because of energy consumption. Other factor is materials like to graphite, nickel and titanium. These materials have been concerned resource depletion. In addition as shown in Table 2, Fossil fuels, Minerals categories in Ecoindicator 99 same to the Abiotic resource depletion (ARD) at CML 2002. But In this method, Impact of Energy consumption is more than higher materials.

3.3.7.2 CML 2002 methods

In this method, Energy consumption is excepted so the total environmental impacts of the three samples are dominated by the materials. MgH<sub>x</sub>-graphene shows higher values for the Human Toxicity Potential (HTP) due to graphite powder in the production process. Because graphite powder is very small size, it is bad influenced by human<sup>15)</sup>. However, the other two samples show abiotic resource depletion (ADP) due to the materials involved, namely nickel and titanium<sup>16,17)</sup>. In particular, the environmental load value of nickel is higher than that of titanium. Mg<sub>9</sub>Ti<sub>1</sub>-Ni has the lowest relative impacts of all evaluated composites because the amount of nickel and titanium in that composite is smaller than in the other two. In addition, the amount of magnesium in Mg<sub>9</sub>Ti<sub>1</sub>-Ni is relatively high, and magnesium does not have an influence on the environment in the measured categories. Likewise, hydrogen has little impact on the environment.

4. Conclusion

In this paper, the hydrogen storage properties of

MgH<sub>x</sub>-graphene composites have been studied systematically. And Life cycle assessments are reported for MgH<sub>x</sub>-graphene composites from production, evaluation experiment until disposed.

- 1) The results of X-ray powder diffraction show that MgH<sub>x</sub>-3wt.% graphene consists mainly of the MgH<sub>2</sub> and Mg<sub>2</sub>C<sub>3</sub> phases, while MgH<sub>x</sub>-7wt.% graphene consist of MgH<sub>2</sub> and MgC<sub>2</sub> phases.
- 2) The kinetic experimental results show that both composites have a similar kinetic behavior when hydride at 523K, 573K, and 623K. However, the presence of the Mg<sub>2</sub>C<sub>3</sub> and MgC<sub>2</sub> phases affects the kinetic behavior when hydriding at 473K.
- 3) P-C-T curves indicate that the equilibrium pressure for hydrogen absorption/desorption of these composites decreases with increasing amounts of graphene, which indicates that graphene tends to support more stable hydrides.
- 4) Analysis of the MgH<sub>x</sub>-graphene composites production system indicates that, in the case of EI'99 methods, MgH<sub>x</sub>-graphene has the highest environmental impacts, followed by Mg<sub>9</sub>Ti<sub>1</sub>-Ni, and then Mg<sub>2</sub>NiH<sub>x</sub>. Due to the large influence of electricity consumed in the process, the fossil fuels category is the highest impact category. For CML 2002 methods, MgH<sub>x</sub>-graphene shows higher values of HTP (Human toxicity potential) due to graphite powder in the production process. However, the other two samples show ADP (a biotic resource depletion) due to the materials used, namely nickel and titanium. The Fossil fuels categories in Eco-indicator 99 can be considered similar to the Abiotic resource depletion (ARD) at CML 2002. So the two methods show similar result. The main

factor is energy consumption and minor factor is materials. But this study didn't account for life cycle of samples. Experiment for hydrogen storage of powder limited to number of times. Although electricity consumption for production is big, if the experiment can be possible to longer, this can be an economical and environment-friendly. Because of this, future studies should be considered to life cycle of samples.

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