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# Production of single phase $Al_{12}Mg_{17}$ alloy fabricated by powder metallurgy

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Porous single phase $Al_{12}Mg_{17}$ alloy was fabricated by cooling in to water after powder metallurgy sintering for use as hydrogen storage fuel cell. Because $Al_{12}Mg_{17}$ phase has more theoretical hydrogen storage capacity than the phases occurred such as $Al_3Mg_2$ and R(AlMg) in microstructure of the alloy. The pore morphologies, microstructure and phases were investigated. It was seen that phases such as $Al_{12}Mg_{17}$, $Al_3Mg_2$ and R(AlMg) occur in the microstructure of the sample fabricated by powder metallurgy sintering. The formation of $Al_3Mg_2$ and R(AlMg) phases was blocked by speed quenched into water after sintering and single phase $Al_{12}Mg_{17}$ was obtained.

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*Keywords:* Porous single phase $Al_{12}Mg_{17}$, Rapid quenching, Hydrogen storage characteristic

## 1. Introduction

Magnesium and its alloys have wide applications in automotive and aerospace industries because of their low density and high specific strength [1]. In the last few years, magnesium-based hydrogen storage alloys have attracted considerable interest as hydrogen storage materials due to their main advantages such as high H/M ratio, low specific weight and low cost [2,3]. However, their poor resistance to corrosion is a serious impediment against their wider applications. On the other hand, aluminium and its alloys have excellent corrosion resistance, high specific strength, good surface decoration characteristic and lower cost [1]. Alloying with Al adds improves heat transfer to the Mg-Al hydride bed, which is essential for fast dehydrogenation. In addition to this it has generally been found that the thermodynamics and kinetics of Mg–Al compared to Mg are improved along with the resistance towards oxygen contamination [4].

Hydrogen could be an ideal energy carrier of the future because it burns cleanly without producing pollutants and could be produced from renewable energy sources and abundant natural resources. However, the problem to store hydrogen is a bottleneck for practical use [5]. Due to the drawbacks related to $H_2$ storage in gaseous or liquid forms, interest mostly focuses on the possibilities offered by solid hydrides. First, it must absorb and desorb $H_2$ on demand under the mildest possible conditions. Second, it must be lightweight. Third, absorption and desorption must be fast, controlled and reversible [6]. Various materials have been explored as the candidates for hydrogen storage including metal hydrides, inorganic oxides, metal-organic frameworks, porous polymers and carbon materials [5].

A number of studies on the hydrogenation/dehydrogenation behaviour of Mg–Al alloys have been published. It is generally observed that upon hydrogenation the Mg–Al alloy disproportionates under formation of $MgH_2$ and Al according to the overall scheme below

$$MgxAly + xH_2 = MgH_2 + yAl$$

Hydrogen absorbed/desorbed schemes of $Mg_{17}Al_{12}$ and $Mg_2Al_3$ phases are like as showed below equations;

$$Mg_2Al_3 + 2H_2 = 2MgH_2 + 3Al$$
$$Mg_{17}Al_{12} + 9H_2 = 9MgH2 + 4Mg_2Al_3$$

Theoretical hydrogen storage capacity of $Mg_{17}Al_{12}$ phases is higher than the other phases [4]. Therefore, it is necessary to produce single phase $Mg_{17}Al_{12}$. Hydrogen storage capacity increased linearly with the increase in microporosity in graphite compacts in a study by Armandi at al. [7]. Considering this, in this study, porous single phase $Al_{12}Mg_{17}$ alloy was produced by a route including powder metallurgy sintering followed by quenching (rapid cooling).

## 2. Experimental procedure

Mg and Al powder each having 99.8% purity and an average size of -325 mesh (Alfa Aesar) were used in the experiment. The mixed powders of Al and Mg in the Al-52 at.%Mg atomic proportion were blended in a rotating container for 4 h. Then, the mixture was pressed into cylindrical compacts, approximately 15 mm in length and

10 mm in diameter at 500 MPa. These compacts were heated by a heating rate of with 20 ºC/min up to 430 ºC and sintered for 4 h in a furnace under the protection of high purity argon gas. Some sintered samples were immediately quenched into water and the others were quenched by air to room temperature separately.

The general porosity of the specimens produced was determined by the formula, $f$ = 1-m/(dV), in which V and m are the volume and mass of the porous specimen respectively and d is theoretical density. The theoretical density of Al-52 at.%Mg mixture is 2.13 gr/cm$^3$. The general pore characteristics of the products were analysed using an optical microscope. To investigate the chemical composition, the surfaces of specimens were etched by a mixture of 10% HF in water. An energy-dispersive X-ray spectrometer (EDS) coupled with the scanning electron microscopy (SEM, LEO Evo-40VP) was used to locally measure the chemical composition of the specimens. The phase constituents were determined by X-ray diffraction (XRD, Rigako Rad-B D-Max 2000 XRD) analysis using CuKα radiation with 1.54046 A.

## 3. Result and discussion

Fig. 1(a) and (b) shows porous samples quenched and the samples cooled in atmosphere in the room temperature after sintering. It is seen that the samples quenched into water are more porous and have a more cancellous structure than the samples quenched by air to room temperature. This is also seen clearly from the porosity calculations. The porosity ratios of the samples quenched into water and the ones quenched by air to room temperature are %11.2 and %8.7, respectively. That is, porosity is higher in the sample quenched into water. In this study, it was seen that gas pimples emerged from the sample when the sample was quenched into water, and enlarged the pores.

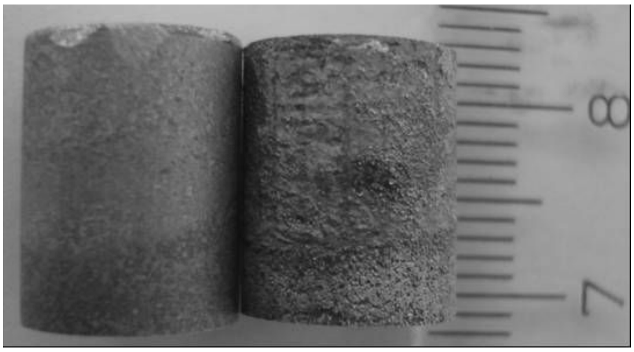
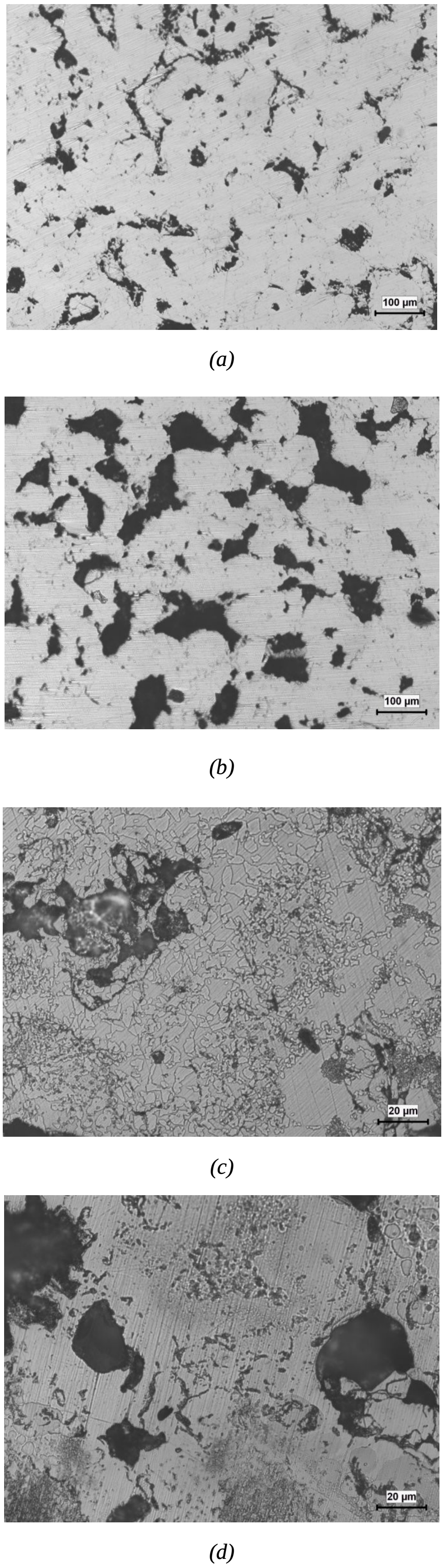


*Fig. 1. Macrographs of samples, (left) sample quenched by air to room temperature, (right) sample quenched into water.*



*(a)*

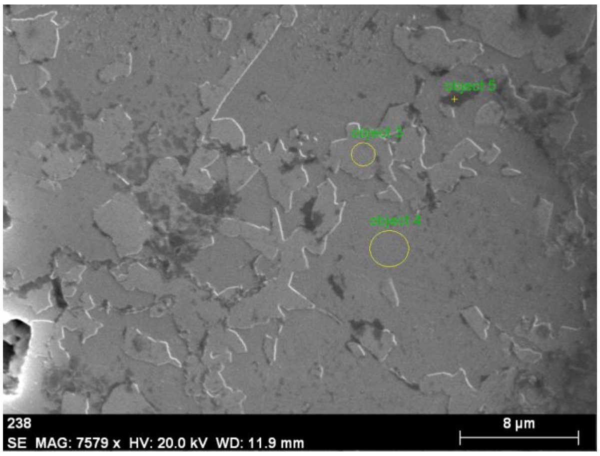
*(b)*

*(c)*

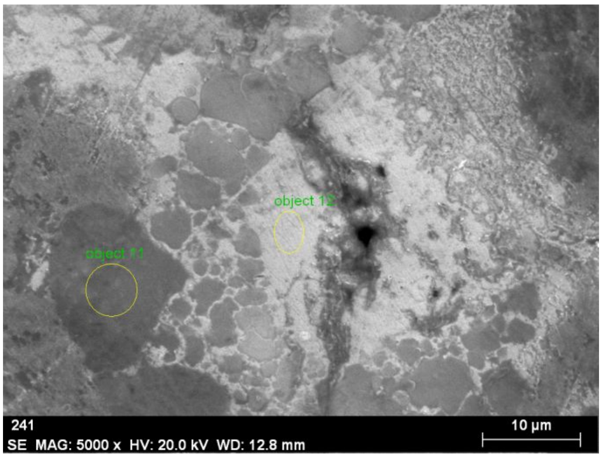
*(d)*

*Fig. 2. Micrographs of samples quenched after sintering; (a) the unetcehd sample quenched by air to room temperature; (b) the unetched sample quenched into water; (c) the etched sample quenched by air to room temperature, (d) the etched sample quenched into water.*

Fig. 2(a) and (b) show micrographs of porous samples sintered before etching (c) and (d) show micrographs after etching. There are pores in different shapes (a-b), and amount of pores in Fig. 2(b) increases with cooling in water. The phases existing (blistered layers) in micrograph of sample quenched by air to room temperature (c) disappeared in the specimens quenched into water (d). SEM micrographs of samples show clearly phases in Fig. 3. The EDS result of atomic composition was Mg–56.44 at.%Al for the blistered layer pointed with small circle (object 3) in Fig. 3(a), This phase was identified as $Mg_2Al_3$ phase. The atomic composition of bigger circle shown as object 4 in Fig. 3(a) is Mg–51.99 at.%Al, corresponding to R(MgAl) phase. The similar composition (Mg-51.22 at.%Al) is seen for the object 5 in Fig. 3 (a). The EDS compositions of grey (object 11) and light (object 12) areas are Mg-49.88 at.%Al and 49.47 at.% Al, respectively in Fig. 3 (b). These areas were identified as $Mg_{17}Al_{12}$ phase from the combination of EDS, XRD results (Fig. 4) and phase diagram (Fig. 5).



(a)



(b)

*Fig. 3. SEM micrographs and EDX regions of samples sintered, (a) quenched by air to room temperature, (b) quenched into water.*

Fig. 4 shows XRD patterns of the sample quenched by air to room temperature (curve-a) and sample quenched into water (curve-b) after sintering. The phases such as $Al_{12}Mg_{17}$, R(MgAl) and $Al_3Mg_2$ are seen in the Fig. 4 (curve-a). These phases are expected considering binary Al-Mg phase diagram in Fig. 5. Only $Al_{12}Mg_{17}$ phase was detected (in Fig. 4. curve-b) in the sample quenched into water after sintering at high temperature due to the rapid cooling.

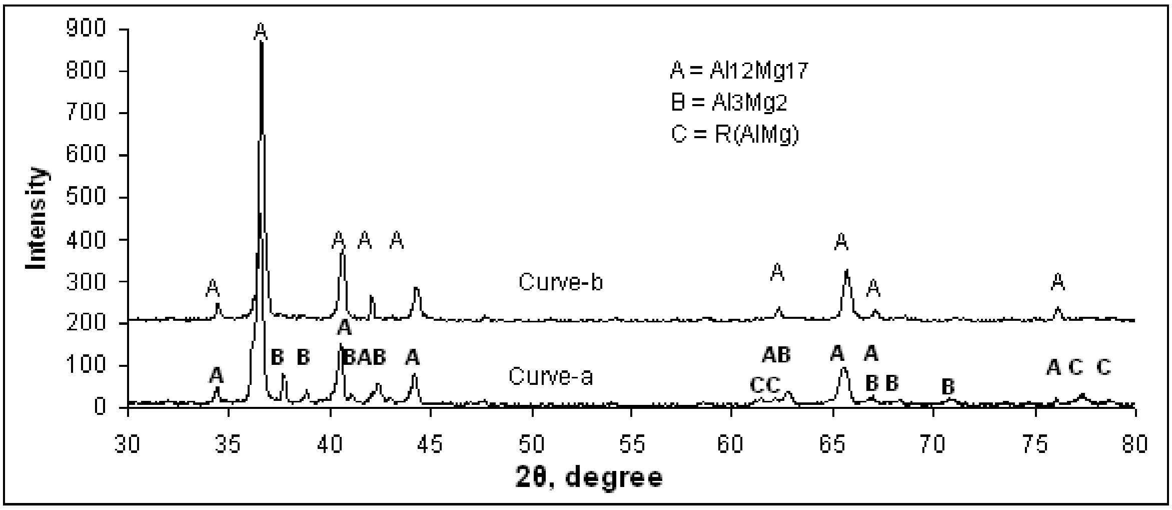


*Fig. 4. XRD patterns of the samples sintered, (Curve-a) the sample quenched by air to room temperature, (Curve-b) quenched into water.*

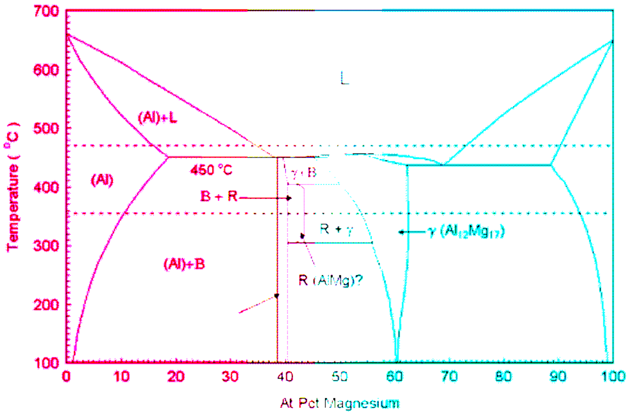


*Fig. 5. Phase diagram for the Al-Mg binary system [8].*

## 4. Conclusions

In this study, a porous alloy to be used as hydrogen storage fuel cell was fabricated by powder metallurgy sintering and the following results were obtained:

1. Porosity increased with quenching into water and the pores in different shapes distributed uniformly within the structure.

2. The phases such as $Al_{12}Mg_{17}$, R(AlMg) and $Al_3Mg_2$ are formed in the structure of porous MgAl alloy fabricated by powder metallurgy sintering.

3. A structure consisting of only single phase $Al_{12}Mg_{17}$ can be achieved by rapid cooling into water. No other phase transformation occurs by rapid cooling.

4. Rapid cooling after sintering has a great potential in producing a microstructure for a high hydrogen storage capacity due to single phase $Al_{12}Mg_{17}$ and a higher porous structure.

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