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Microstructure and mechanical properties of rapidly solidified Mg alloy powders compacted by magnetic pulsed compaction (MPC) method

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ABSTRACT

Gas atomized Mg–Zn_{4.3}Y_{0.7} (at%) alloy powders were consolidated by using a magnetic pulsed compaction (MPC) process, and obtained by rapid solidification not to lose the fine microstructure during the generally conducted thermal consolidation process. MPC is known as one of the most rapid pressing process with the GPa range. The effects of the discharging voltage and thermal pressure on the microstructure, hardness, density and compressive strength were investigated. The experimental results showed that the density increased with increasing the voltage although the sample MPCed still consists of pores especially between the powder boundaries even at the maximum pressure of this investigation. The density was improved further up to 96% to the cast value by the MPC at the maximum voltage. Uniform and fine microstructure formed in the alloy powders as atomized was almost maintained even after the thermal MPC.

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1. Introduction

Magnesium belongs to the lightest metal and has a higher specific strength than aluminum and steel, showing the unique application opportunities to the light weight structure industry [1,2]. Owing to its excellent properties, it has been widely used in automobile, aerospace, electronic components, and leisure sports industries. However, its intrinsic low strength and corrosion resistance have limited to expand the industrial application. Among various ways to overcome the problem, powder metallurgy (PM), including the powder synthesizing process using a rapid solidification method, became the strongest candidate, since it is known to be effective to produce not only high performance structures, but also a near-net-shape. As one of the approach, Kim et al. reported that Mg alloy powders having the fine grain size less than $5 \,\mu$ m. could be successfully synthesized using a gas atomization process, followed by extrusion. [3,4] However, it is also mentioned that the Mg alloy easily degrades in microstructure's properties during the extrusion that operated at a certain temperature with time. Thus, much faster time and lower energy PM processing like magnetic pulsed compaction (MPC) would be necessary for the Mg alloy powder consolidation. The MPC pressurizes the powders dynamically using a magnetic pulse with the pressures more than 2 GPa, result-

* Corresponding author. Tel.: +82 32 8500 409; fax: +82 32 8500 390. *E-mail addresses*: hjchae@kitech.re.kr (H.J. Chae), ydkim1@hanyang.ac.kr (Y.D. Kim), tskim@kitech.re.kr (T.-S. Kim). ing in the high speed movement of particles over 10-100 m/s and very short duration of an order of microsecond (μ s) [5,6], resulting in forming a segregation free and a fine grain structure. Its easy energy controllability and high energy efficiency becomes a further opportunity of application. Therefore, MPC methods are thought to be quite useful to consolidate the rapidly solidified Mg alloy powders with the fine grains and uniform structure, which is sensitive to the thermal energy exposure.

In this study, Mg– $Zn_{4.3}Y_{0.7}$ alloy powders were produced using the commercial scale gas atomizer, and consolidated using the MPC. The variation of material's properties such as relative density, hardness, compressive strength and microstructure were investigated as a function of MPC pressure and temperature.

2. Experimental procedures

Mg–Zn_{4.3}Y_{0.7} (at%) alloy powders were fabricated using a gas atomizer constructed by a boron nitride melt delivery nozzle of 5 mm in diameter and an annular Ar gas nozzle operating at a pressure of 5 MPa. For increasing of liquidity, the master alloy was melted at 1023 K which was 200 K above the liquidus temperature. The gas atomized Mg–Zn_{4.3}Y_{0.7} alloy powders distributed in a size range of 10–250 μ m were classified using a conventional sieving method. In this study, we used the powders smaller than 150 μ m. The powders consolidated using the MPC process, and the specifications of the machine as follows:

- (1) The maximum discharge energy is 30 kJ.
- (2) The pulse force is below 1000 kN.
- (3) The highest discharge voltage is 3.8 kV.

For more information, the details are well expressed in Refs. [7,8]. The $Mg\text{-}Zn_{4.3}Y_{0.7}$ alloy powder was pressed with the discharge voltage of 1.20 kV,

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Fig. 1. SEM images of atomized Mg-Zn_{4.3}Y_{0.7} alloy powders (a) morphology and (b) microstructure.

1.30 kV, 1.40 kV, 1.50 kV and 1.60 kV for 0.3 s. The compacted sample was polished and etched using a chemical solution (4.2 g picric acid + 80 ml ethanol + 20 ml distilled water). Then, the microstructures and hardness were observed by scanning electron microscope (Model JSM-5410), and measured by Rockwell hardness tester (Model Wilson-2001T), respectively. Density of the specimens was measured using Archimedes method. The compacts were cut to cylinder shape with a size of $2 \text{ mm} \times 4 \text{ mm}$ by wire cutting, and the strength-strain was tested on universal testing machime (Model MTS810) under compression mode with the constant strain rate of 10^{-3} s^{-1} at room temperature.

3. Results and discussion

Fig. 1 is SEM photos showing (a) morphology and (b) microstructure of rapidly solidified (R/S) Mg–Zn_{4.3}Y_{0.7} alloy powders. The size of powders shown here belongs to the group of 90–149 μ m, which was located in the average cumulated size distribution range among total powders produced. The powder showed near spherical morphology with a rough surface and a partial attachment of satellite particles (a). It consists of dendrite like-equixed grains of about 2–4 μ m in average diameter (b), which is much finer than that of as-cast sample [9]. In order to identify the compositional variation in powders, Energy Dispersive X-ray spectroscopy (EDS) was conducted between the grain and the grain boundaries as listed in Table 1. It indicates that the elements of Zn and Y were rich in the grain boundaries rather than in the grains. Fig. 2(a) shows the proportional relationship between the load and voltage, where the load admitted to the sample during the MPC increased as the volt-

Table 1

Composition of grain and grain boundary in Mg powders (Fig. 1) by EDS.

	Mg	Y	Zn
Grain	94.21	2.11	3.68
Grain boundary	88.26	4.92	6.82

age increased. But, the linear relationship was shown from 1.20 kV, which would present the linear compaction trend. Fig. 2(b) is a typical sample consolidated using the MPC process at the condition of 1.60 kV. The diameter and the thickness of the samples were 20 mm and 5 mm, respectively. The microstructure of Mg-Zn_{4.3}Y_{0.7} alloy showed in Fig. 3, as a function of the applied voltages of (a) 1.30 kV, (b) 1.40 kV, (c) 1.50 kV and (d) 1.60 kV. The lowest discharge voltage of 1.30 kV in the experimental condition found to consist of many pores which mostly formed between the powders. The porous structure gradually became dense as the voltage increased, but still consisted of pores even at the highest discharge voltage of 1.60 kV. Variation in density and hardness of Mg alloy powder by the MPC is as plotted in Fig. 4. The density was increased from 1.842 g/cm³ to 1.852 g/cm³, 1.863 g/cm³, 1.875 g/cm³ and 1.881 g/cm³ as the discharge voltage increased from 1.20 kV to 1.30 kV, 1.40 kV, 1.50 kV and 1.60 kV, respectively. The hardness also increased from 45.7 HR to 46.5 HR, 47.5 HR, 49 HR and 51 HR, respectively as the pressure increased. The maximum density of 1.881 g/cm³, however, is still lower even at the maximum voltage of 1.60 kV, which is about 89% to the cast density. This is well matched with the porous structure shown in Fig. 3(d). Thus, it may need to pressurize the powders with the thermal energy. Fig. 5 shows the overall (a) and magnified (b) microstructures of Mg alloy powder bulks MPCed at 1.60 kV and 573 K. It contains no pore, compared to the one MPCed only at the pressure of 1.60 kV. It is seen a little variation in the grain size from $2 \,\mu m$ to $4 \,\mu m$ in the atomized powders to $3-5 \,\mu m$ possibly due to the thermal exposure during the MPC (b). Very sound interfaces are also found to form without any pore. The resultant properties of thermally MPCed sample at the condition are as listed in Table 2. Comparing the properties obtained from the samples between only pressurized and thermally pressurized, the latter presents much improved value from 1.881 g/cm³ to 1.952 g/cm³ in density and 51.0-55.5 HR in hardness, respectively. However, the compressive



Fig. 2. Variation of load with applied voltage (a) and photograph of bulk Mg alloy after MPC.

Table 2Summary of Mg-Zn4.3Y0.7 alloy properties gas atomized and MPCed at 1.60 kV at room temperature and at 573 K.

	Density (g/cm ³)	Hardness (HR)	Compressive strength of failure (MPa)	Compressive elongation at failure (%)
Room temparature	1.881	51	A lack of reproducibility	A lack of reproducibility
573 K	1.952	55.5	186.22	3.21

strength of 186.22 MPa and strain of 3.21% failure are quite lower than not only those of 370 MPa and 17.2% by hot rolling [9], but also 325 MPa and 16% in the extruded sample [10] at the same composition. The lower mechanical properties may correspond to still low density of about 95% to the one of cast sample. Existence of prior powder boundaries marked as arrows in Fig. 5(a) becomes another proof of low density. They are able to summarize the reason why MPC could not consolidated the Mg powders fully as follows:

(1) Strong oxide layer formed on Mg powder.

(2) Limited slip system of Mg.

It was reported that the compaction of powder shape materials using MPC with the high velocity was much effective for ductile and easily slipped one due to an easy diffusion of atoms. In order to consolidate the powders, the atoms in the powder have to diffuse through the powder interface after breaking the hard surface usually formed as thin oxide layer. The layer formed in Mg alloy powders has a composition of MgO which is a little bit porous but has a melting point of 3073 K [11]. The oxide layer generally formed during atomization is about 45 nm in thickness, and can be breakable and separated into Mg and O₂ by heating during pressurizing for consolidation in the temperature range of 373–573 K. Another condition for its consolidation is the time under the pressure and temperature, since the Mg alloy is belong to the HCP structural group which has very limited slip system as (0001, 11 $\overline{2}$ 0). Thus, the lower densification of these powders might be due to the lack of time under the thermal pressurizing process as well as relatively



Fig. 3. Microstructures of the samples compacted in different voltage at room temperature (a) 1.30 kV, (b) 1.40 kV, (c) 1.50 kV and (d) 1.60 kV.



Fig. 4. Density and hardness of MPCed Mg alloy powder as a function of voltages at room temperature.

low temperature. Kim et al. reported that the alloy powders could be consolidated fully with the strength more than 350 MPa by the thermal extrusion at the temperature of 673 K [3,10]. In order to obtain further improved mechanical properties, it is necessary to perform the pressing and heating time dependent investigation in the future. The phase variation between the powders as solidified and the powder bulk MPCed only by pressure and by a combination



Fig. 5. SEM micrograph image of the polished cross section of the Mg– $Zn_{4,3}Y_{0,7}$ alloy consolidated at 1.6 kV–573 K.



Fig. 6. X-ray diffraction patterns of (a) initial powder, (b) MPCed at 573 K.

of pressure and thermal energy was characterized using an X-ray diffraction as shown in Fig. 6. It is not found any discernable change in the phases formed as a-Mg and Mg₇Zn₃ and the grain size.

4. Conclusion

Gas atomized Mg–Zn_{4.3}Y_{0.7} alloy powders showed a fine grained microstructure less than 4 μ m in diameters corresponding to the effect of rapid solidification. The powder bulk consolidated by MPC method initially formed a porous structure at the lowest pressure of 1.20 kV. As the pressure increased from 1.20 kV to 1.60 kV, the pores gradually became dissipate and the density increased from 1.842 g/cm³ to 1.881 g/cm³, respectively. However, it still consisted of fine pore even at the highest voltage of 1.60 kV in the experimental condition. An addition of thermal condition of 573 K to the maximum pressure of 1.60 kV resulted in forming the poreless structure as well as increasing the density more than 3.9%. The hardness was also improved more than 13% from 45 HR to 51 HR. The strength was also improved, but it is necessary to develop the further consolidation process possibly by increasing the temperature.

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