Anisotropy Of Texture-Controlled Powder Metallurgy Magnesium Alloys Via Roll-Compaction Process

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Abstract— Powder Roll-Compaction Process (RCP), consisting of twin rolls and fragmentation using a cutting mill, was employed to assist in the grain refinement and texture control of coarse AZ31B magnesium alloy powder by repeated severe plastic deformation from all directions. It was verified that numerous twins were densely induced into the matrix, and orientation randomization was significantly carried out. Wrought AZ31B alloys consolidated by hot extrusion show a good balance of high yield stress and elongation by employing RCPed powder. For example, in the case of 50 cycles using RCP, the extruded alloy indicated exhibited homogeneous and fine grains less than 1 µm, with superior mechanical properties of 280 MPa yield stress and 15% elongation. SEM-EBSP analysis showed the randomized (0001) basal orientation of the wrought alloys via RCP process and a drastic increase of the mean Schmid factor with increase in the number of cycles in RCP process. The anisotropy of yield stress was greatly improved due to both of the grain refinement and uniform texture formation of the extruded magnesium powder alloys via RCP process.

Keywords—Magnesium; powder; cold rolling; grain refinement; orientation, tensile strength

I. INTRODUCTION

Magnesium (Mg) alloys promise a remarkable lightweight effect due to its low density of about 1.74 g/cm³ [1], and results in both a fuel consumption improvement and CO₂ emission gas saving by their application to structural components in transformation industries such as automotives, airplanes, motorcycles and railways [2-5]. When strengthening Mg alloys, grain refinement is significantly effective compared to aluminum alloys. This is because YS (σ) depends on the Hall-Petch equation ($\sigma = \sigma_0 + k \cdot d^{-0.5}$, k; Hall-Petch constant, d; grain size) [6, 7] and the k value of magnesium allovs is at least twice that of aluminum alloys [8]. The conventional hot rolling process is useful to refine grains of Mg alloys by a dynamic recrystallization. For example, the repetition of hot rolling on AZ61 alloy slabs easily produces refined grains 5 µm or less [9]. However, the difference of tensile strength comparing the rolling and transverse directions becomes large due to their strong texture. That is, the conventional hot rolled Mg alloy sheets have an isotropy of mechanical properties [10], which causes a limitation in employing them as industrial materials. From a viewpoint of their industrial applications,

wrought Mg alloys with both a high tensile strength and superior mechanical anisotropy are desired. In this study, a new powder metallurgy (PM) process for the grain refinement and texture control has been developed and applied to machined AZ31B alloy chips by using a Roll Compaction (RCP) process [11, 12]. The isotropic tensile strength of extruded AZ31B alloys, consolidated using the RCPed powder via a hot extrusion process, was investigated. For the texture analysis, the orientation evaluation of the RCPed powder and its extruded materials by SEM-EBSP was carried out. The effect of the number of RCP cycles on mechanical properties, microstructures and textures of extruded alloys was discussed.

II. EXPERIMENTAL PROCEDURE

The principle of the RCP process is schematically illustrated in Fig.1. Coarse magnesium alloy chips, having a mean particle size of 0.72 mm, and their composition is shown in Table 1.



Fig.1 Schematic illustration of Roll-compaction (RCP) equipment in using Mg alloy powder.

Table 1 Chemical compositions of AZ31B raw powder (in wt.%).

AI	Zn	Mn	Si	Cu	Ni	Fe	Mg
3.05	0.82	0.40	0.020	0.003	0.0006	0.0023	Bal.

They were prepared by machining the conventionally extruded AZ31B alloy shown in (Fig. 1a), were used as the input raw powder. In this process, the raw powder was first consolidated as sheet materials shown in (Fig. 1b) by twin rolls at room temperature,

where a roll diameter was 200 mm in this study. Then, the green sheet was fractured to the coarse powder as shown in (Fig. 1c) by the cutting mill. They were cycled into the powder feeder for twin-rolling repeatedly. The repetition of this process was carried out from 10 to 50 times, and supplied a severe plastic deformation on AZ31B powder in all directions. A 4.4 kN/mm load was applied to powder materials between the rolls, and the rotation speed of 313 mm/s was selected. In this process, a constant rolling ratio is significantly important to provide a uniform plastic deformation to Mg powder every working cycle. The clearance between the twin rolls was kept rigid using hydraulic pressure. Therefore, the particle size distribution is one of the important parameters in RCP process. By optimizing the fracturing conditions of as-rolled green sheets using the cutting mill, the particle size distribution of RCPed AZ31B powder was kept as a constant and steady value by repeating the number of cycles on the RCP up to 50 times as shown in Fig.2.



Fig.2 Particle size distribution of AZ31B raw powder and RCPed ones.

The mean particle size of RCPed AZ31B powder in this study was about 2.4~2.6 mm. The RCPed AZ31B powder was consolidated under 400 MPa at room temperature. The green compacts with a diameter of 43 mm and 86 mm were heated at 473K for 300 s in a nitrogen atmosphere, and immediately extruded as rod and plate specimens, respectively, where the extrusion ratio was 32 ~ 37 and the extruding speed was 1 m/s in both. Figure 3 shows the appearance of extruded plate specimens by consolidation of AZ31 alloy powders via RCP process, having 40 mm width, 8 mm thickness and 1,400 mm length. The tensile specimen taken parallel to the extrusion direction (ED) was machined from these extruded plate materials, which is indicated as " $\theta = 0^{\circ}$ ", while the transverse direction specimen is as " θ =90°" in this study. Tensile testing of each extruded P/M AZ31B alloys was carried out at room temperature with a strain rate of 5*10⁻⁴ /s. The microstructures were observed with optical microscopy (OM) and SEM (JEOL: JSM-6400F). For the OM observation, each specimen was etched by a mixture of 75 ml H₃PO₄ + 25 ml H₂O after a mechanical polishing. The texture evaluation by Electron Backscatter Diffraction Pattern (EBSP) analysis on the electrolytic polished specimens was carried out with the TSL orientation imaging microscopy

system (OIM version 4.6). In the electrolytic polishing, the mechanically polished specimen was etched for 30 s by a mixture of 50ml H_3PO_4 + 30ml C_2H_6O under a voltage of 10 V.



Fig.3 Appearance of extruded P/M AZ31B plate materials.

III. RESULTS AND DISCUSSION

Figure 4 shows the optical microstructures of AZ31B raw powder and after RCP processing with N=10, 30 and 50 cycles.



Fig.4 Optical microstructures of AZ31B powder with induced deformation twins via RCP process; as-received raw powder (a), N=10 (b), 30 (c) and 50 cycles (d).

As shown in (a), many twins are induced in the matrix due to a large strain during machining of the AZ31B extruded billet to prepare the raw powder. With increases in the number of cycles in RCP, the macroscopic changes of the matrix, which are similar to the deformation during a mechanical alloying (MA) process [12], are observed. The primary powder

boundaries and some pores at the boundaries gradually disappear with increasing the number of cycles in RCP process. A light microscope observation at high magnification was carried out on RCPed AZ31B powder with N=10 and 50 cycles, and its results were also shown in Fig.4. It indicates that the number of twins remarkably increases with increasing its cycle number and smaller twins become more uniform and much dense in the matrix of RCPed AZ31 alloy powders. This indicates a repetition of the plastic deformation by RCP induces numerous twins into the AZ31B powder from all directions. Figure 5 shows EBSP analysis results on AZ31B raw powder (a) and RCPed with 50 cycles (b).



Fig.5 Image quality mapping and inverse pole figures by SEM-EBSP analysis on raw powder (a) and RCPed one with N=50 cycles (b).

As mentioned above, raw powder obviously contains some deformation twins, and a very strong texture due to hot extrusion. On the other hand, as a distinguished morphology, RCPed powder has a flat surface shape, which corresponds to the surface of the as-rolled sheet materials shown in Fig.1 (b). Roll lines are also obvious on the flat surface of RCPed powder. Therefore, the rolling direction (RD), transverse direction (TD), and normal direction (ND) can be clearly determined on the RCPed powder surface. The image quality (IQ) mapping (Fig 1a) shows fine grains about 200~500 nm. In considering that the mean grain size of AZ31B raw powder is 16 µm, the repetition of RCP process was significantly effective to refine grains by a severe plastic deformation. Concerning the textures in TD, RD and ND of RCPed powder (Fig. 1b), the color distribution is remarkably uniform compared with the raw powder shown in (Fig. 1a-2), that is, the biased textures could be completely reduced by RCP process. Figure 6 shows a change in the inverse pole figures (IPF) and the intensity of RCPed powder by increasing the number of the repetition cycles. As mentioned in Fig.5, the raw powder has a strong texture on the (0001) basal plane because it was prepared from an extruded AZ31B alloy by cutting. Figure 6 (a) also shows the same result, and has a high Imax value of 45.4. With an increase in the RCP cycle number, as shown in Figure 6 (b) through (d), each IPF randomly changes and the Imax value gradually decreases. When comparing the Imax of RCPed powder with N=50 with raw powder, it is 10% or less, that is, the texture is significantly randomized by the repetition of RCP process.



Fig.6 Changes in pole figures of AZ31 raw powder (a), N=1 (b), 10 (c) and 50 cycles (d) via RCP process.

Figure 7 shows the optical microstructure changes along the extrusion direction of hot extruded AZ31B powder plate materials via RCP process. Even in the case of the raw powder, the wrought alloy has a small mean grain size of about 2.4 µm. This is because a grain growth after dynamic recrystallization during extrusion was obstructed due to the low extrusion temperature of 473K. However, its microstructure, grains. consisting of fine and coarse was heterogeneous. When increasing the number of RCP cycles, the mean grain size drastically decreases; for example, it is 0.8 µm or less in the case of N=50 cycles. The microstructural uniformity is also remarkably improved by severe plastic deformation during RCP process [14-16].



Fig.7 Optical microstructures of wrought P/M AZ31B alloys extruded at 473K in using raw powder (a), and RCPed ones with N=10~50 cycles (b)~(d).

Figure 8 shows pole figures of specimens taken along the extrusion direction (θ =0°) of the hot extruded AZ31B powder alloys. In using raw powder (Fig. 7a) and RCPed one with N=10 cycles (b), both figures indicate strong orientations of (0001) basal plane along ND and (11-20) non-basal along TD, which are typical textures

of the conventional extruded Mg alloys. When increasing the number of RCP cycles to over 30, the above orientation is gradually randomized, and the Imax value also decreases [17, 18]. In the case of N=50 cycles shown in (d), both the (0001) and [11-20] planes are completely random, and quite different from those in using raw powder. Based on the EBSP results, a dependence of the mean Schmid factor [19] of (0001) basal slip on the number of RCP cycles is shown in Fig.9. The tensile test direction corresponds to the extrusion direction (ED) and slip plane and direction, which is (0001) and [1120] respectively. A lower value means an increase in the number of grains, which is parallel or transverse to the tensile direction. That is, a higher value of the extruded Mg alloys indicates a relative randomization of (0001) basal orientation. At N=0 cycles, thus indicating the use of raw powder, the mean Schmid factor along ED (b-0) of 0.258 is larger than that of TD (b-90) with 0.194.





Fig.8 Pole figures of hot extruded AZ31B alloys via RCP (T=473K, θ =0°).

Fig.9 Dependence of Schmid factor of basal slip on number of cycles in RCP process in use of hot extruded AZ31B alloy powder materials (T=473K, θ =0°).

As shown in Fig.8 (a), the orientations of both (0001) along tensile strength and that [1120] perpendicular to tensile direction are very strong when consolidating raw powder by hot extrusion. Therefore, the mean Schmid factor of (0001) basal of b-90 is much smaller compared to b-0 specimen. Schmid factors in both directions gradually increase with an increase in the number of cycles. It means that the texture randomization of Mg powder due to the severe plastic deformation induced

from all directions by the repetition of RCP process is still effective on that of hot extruded materials. Figure 10 shows the dependence of yield stress (YS) on the anisotropy of each tensile specimen and on the number of cycles of RCP.



Fig.10 Anisotropy of yield stress of large-scale wrought AZ31B alloy extruded at 473 K, in employing RCPed powder with various number of cycles.

The YS gradually increases regardless of the tensile direction because of the grain refinement by the RCP process. However, in the comparison of the increase of YS for each direction, that of $\theta=0^{\circ}$ (in ED) with about 74MPa is larger than that of θ =90° (TD) with 11MPa when increasing the cycle number up to 50 cycles. The difference significantly indicates that YS strongly depends on not only grain size but also textures of the extruded alloys [20]. It is necessary to consider the effects of the grain refinement and basal/non-basal plane orientations on the YS anisotropy of extruded Mg alloys processed via the RCP process. As shown in the IPF mapping in Fig.8 and Schmid factor changes of Fig.9, the increased rate of change in the Schmid factor at $\theta=0^{\circ}$ specimen is much smaller than that at $\theta=90^{\circ}$ specimen in the repetition of RCP up to 50 cycles. It means that a strong orientation of (0001) basal plane of the former is gradually randomized, and causes the increase of YS. On the other hand, in the latter specimen. the number of basal orientations perpendicular to tensile direction, which cause a higher yield stress, remarkably decreases. Therefore, the increase of YS at θ =90° after RCP process with 50 cycles is smaller than that at $\theta=0^{\circ}$ specimen.

IV. CONCLUSIONS

Roll compaction process was applied to AZ31B magnesium alloy powders to progress their grains refinement and texture control for orientations randomization to improve the anisotropic microstructural and mechanical properties of extruded AZ31B alloys via RCP. With increase in the cycle numbers of RCP process, the grain refinement remarkably progressed and the more uniform grain orientation was obtained of the extruded AZ31B alloys.

As a result, the anisotropy of their tensile strength was significantly improved by using RCP process.

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