Dynamically Recrystallized Structure and Mechanical Properties of $\text{Mg}_{96}\text{Zn}_2\text{Y}_2$ Alloys Deformed by ECAP

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$\text{Mg}_{96}\text{Zn}_2\text{Y}_2$ alloys were subjected to equal channel angular pressing (ECAP) to systematically investigate changes in microstructure and tensile properties over eight passes. $\text{Mg}_{96}\text{Zn}_2\text{Y}_2$ as-cast specimens consisted of three phases: α -Mg; LPSO (long period stacking ordered), and $\text{Mg}_3\text{Zn}_3\text{Y}_2$. The phases proved stable against induced strain, enduring eight passes of ECAP, resulting in high-strain deformation. Deformation microstructure by ECAP can be divided into two regions: dynamically recrystallized (DRXed) α -Mg grain regions; and deformed regions including LPSO phase with kink deformation. The mean grain size of α -Mg grains decreased up to four passes of ECAP. However, the mean grain sizes in 4-pass and 8-pass specimens were 1.44 μm and 1.85 μm , indicating that mean grain size saturates due to dynamic recrystallization (DRX) after four ECAP passes. Basal texture of α -Mg grains inclined from tensile direction after one pass of ECAP; the intensity increased with increasing number of ECAP pass. LPSO texture also formed but with low intensity even after eight passes of ECAP. ECAP suppressed greatly DRX compared to extrusion under comparable strain. 0.2% proof stress increased up to four passes of ECAP and subsequently saturated due to DRX. On the other hand, elongation increased with increasing volume fraction of DRXed α -Mg region.

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1. Introduction

Magnesium (Mg) alloys are very attractive candidate materials for use in lightweight technology due to their light weight and high strength-to-weight ratio. As a result of the energy efficiency these alloys offer, their applications are expected to expand into fields of structural materials for transport components such as automobiles, trains, and airplanes. Therefore, new Mg alloys with high strength are being studied and developed. In 2001, Kawamura *et al.*¹⁾ developed extremely high strength $\text{Mg}_{97}\text{Zn}_1\text{Y}_2$ alloys using rapidly solidified powder metallurgy processing (RS P/M) and reported high yield strength of 610 MPa and an elongation of 5%. The $\text{Mg}_{97}\text{Zn}_1\text{Y}_2$ RS P/M alloys consisted of nano-crystalline α -Mg grains with long-period stacking ordered (LPSO) phase.²⁾ The LPSO phase has been found in both gravity-cast Mg–Zn–rare earth (RE) and RS P/M alloys.^{3,4)} Yoshimoto *et al.*⁵⁾ investigated microstructure and mechanical properties of extruded $\text{Mg}_{100-x-y}\text{Zn}_x\text{Y}_y$ alloys (at%, $x = 0\text{--}2.5$, $y = 0\text{--}3$) with different quantities in alloy elements, finding that $\text{Mg}_{96}\text{Zn}_2\text{Y}_2$ alloys have three phases: α -Mg, LPSO, and $\text{Mg}_3\text{Zn}_3\text{Y}_2$. Among Mg–Zn–Y alloys studied, extruded $\text{Mg}_{96}\text{Zn}_2\text{Y}_2$ alloys showed 5% elongation and the highest yield strength of 390 MPa at room temperature. Yamasaki *et al.* have reported microstructural evolution and strengthening mechanism of extruded $\text{Mg}_{96}\text{Zn}_2\text{Y}_2$ ⁶⁾ and $\text{Mg}_{97}\text{Zn}_1\text{Y}_2$ ⁷⁾ alloys. Their previous paper regarding Mg–Zn–Y alloys reported that a multimodal microstructure develops during extrusion. The α -Mg matrix phase is bimodally grained; dynamically recrystallized

(DRXed) α -Mg fine-grains (mean grain size $\sim 4\mu\text{m}$) with random crystallographic orientation and hot worked α -Mg coarse-grains with strong $\langle 10\bar{1}0 \rangle$ fiber texture parallel to extrusion direction (ED). The LPSO phase grains develop $\langle 10\bar{1}0 \rangle$ //ED fiber texture. While DRXed α -Mg fine-grains contribute to improvement in ductility, the textured coarse-grains included in the LPSO phase contributes to the mechanical strength of the alloy. In other words, formation of bimodal microstructure that consists of the fiber-textured and DRXed grains can realize an improvement in both strength and ductility of the alloy simultaneously.

Severe plastic deformation (SPD) processes for grain refinement strengthening have been actively studied in various kinds of metallic materials since the late 1990s.^{8–11)} In equal channel angular pressing (ECAP), a typical SPD, a billet specimen is repeatedly pressed through a characteristic ECAP die with two channels of equal cross-section intersecting at a high angle, with large shear strain $> \varepsilon_{\text{eq.}} = 4.0$ induced to the billet specimen. ECAP has been specifically known to improve both strength and ductility of Mg alloys due to ultra-grain refinement and texture development.^{12–15)} ECAP has been applied to various types of Mg–Y–Zn alloys with different compositions; their microstructure and mechanical properties have been reported in $\text{Mg}_{97}\text{Y}_2\text{Zn}_1$ ^{16,17)} and $\text{Mg}_{96}\text{Y}_3\text{Zn}_1$.¹⁸⁾ These alloys showed high yield strengths of more than 350 MPa due to grain refinement through ECAP.^{16,18)} However, while elongations decreased to less than 7% at room temperature after four passes of ECAP,^{16,18)} ECAPed Mg–4.3 mass%Zn–0.7 mass%Y alloys showed superplasticity at both 523 K and 623 K.¹⁹⁾ Conversely, $\text{Mg}_{96}\text{Y}_2\text{Zn}_2$ display increases in both yield strength and elongation after extrusions, as described in the previous paragraph.⁶⁾ Therefore, $\text{Mg}_{96}\text{Y}_2\text{Zn}_2$ alloys are

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Fig. 1 Schematic illustrations of (a) ECAP die and (b) tensile specimen.

expected to show both higher strength and ductility after ECAP. In this study, ECAP was applied to $\text{Mg}_{96}\text{Zn}_2\text{Y}_2$ alloys, and microstructure and mechanical properties were investigated.

2. Experimental Procedures

$\text{Mg}_{96}\text{Zn}_2\text{Y}_2$ alloy ingots were prepared using a high frequency induction furnace at 973 K in an Ar atmosphere and were cut into round bar billets with a diameter of 8.9 mm and a length of 85 mm. Figure 1 shows schematic illustrations of (a) ECAP die and (b) tensile specimen. Channel (Φ) and die corner (Ψ) angles of the ECAP die used in this study were respectively 90° and 20° . Using the ECAP die, the equivalent strain of 1.05 was induced into specimens by a single pass of ECAP. Here, some processing routes are in ECAP.²⁰ In this study, ECAP was executed over eight passes using route B_C; the specimen was rotated 90° clockwise between each pass. Billets were coated in a MoS_2 lubricant and were subsequently kept within the ECAP die at 673 K for 1.2 Ks before each ECAP pass. ECAP was carried out at 673 K with a ram speed of 2.5 mm/s. Billets were quenched in water after each ECAP pass. Tensile tests were performed using specimens with a gage length of 10 mm and a gage diameter of 2.0 mm. The loading axis in tensile tests was parallel to extrusion direction (ED) shown in Fig. 1 and the initial strain rate was 5.0×10^{-4} /s. Microstructures were observed from ED using optical microscopy, X-ray method, and FE-SEM/EBSD method.

3. Results and Discussion

Figure 2 shows XRD profiles of $\text{Mg}_{96}\text{Zn}_2\text{Y}_2$ alloys before and after ECAP. $\text{Mg}_{96}\text{Zn}_2\text{Y}_2$ as-cast and ECAPed specimens

Fig. 2 XRD profiles of $\text{Mg}_{96}\text{Zn}_2\text{Y}_2$ alloys before and after ECAP.

consisted of three phases: α -Mg; $\text{Mg}_3\text{Zn}_3\text{Y}_2$; and Mg_{12}ZnY (LPSO). No changes in phases and no generations of new phases were observed even after eight passes of ECAP, showing that the three phases were stable under high strain. Figure 3 shows (a) optical micrograph and (b) backscattered electron (BSE) image of the as-cast specimen. The as-cast specimen displayed a dendrite microstructure and a mean grain size of α -Mg of $46.5 \mu\text{m}$. BSE image clearly shows that the as-cast specimen certainly had three phases: α -Mg in black, LPSO phase with lamellar structures in gray, and $\text{Mg}_3\text{Zn}_3\text{Y}_2$ in white. Figure 4 shows confocal images of ECAPed specimens when observed from transverse direction (TD) of specimens. Microstructures of each specimen were bimodally grained into two regions: DRXed α -Mg grain region in gray, and deformed region including LPSO phase in black. The volume fraction of both DRXed α -Mg grain region and non-DRXed α -Mg grain region (deformed region) are summarized in Fig. 5. With increasing number of ECAP passes, the volume fraction of DRXed α -Mg grain region increased while that of the deformed region decreased. The

Fig. 3 (a) Optical micrograph and (b) BSE image of the as-cast specimen.

Fig. 4 Confocal images of (a) 1-pass, (b) 2-pass, (c) 4-pass and (d) 8-pass specimens when observed from TD.

Fig. 5 Volume fraction of dynamically recrystallized (DRXed) α -Mg grain region and deformed region, as a function of equivalent strain.

volume fraction of the regions significantly changed between two and three passes of ECAP, meaning that a critical point for affecting microstructure may exist in $\text{Mg}_{96}\text{Zn}_2\text{Y}_2$ alloys. BSE images of (a–c) ECAPed specimens and (d) kink deformation in 8-pass specimen when observed from ED are shown in Fig. 6. After one pass of ECAP, both α -Mg grain size and width of LPSO phase decreased; as seen in Fig. 3(b), $\text{Mg}_3\text{Y}_3\text{Y}_2$ were subdivided into particles in white for comparison with the as-cast specimen. Kink deformation, sharp bends indicated by arrows in Fig. 6, were observed in LPSO phase and was found to occur even by a single pass of ECAP ($\epsilon_{\text{e.q.}} = 1.05$). Such kink deformation plays a role in strengthening in LPSO type Mg alloys.²¹⁾

Figure 7 shows (0002) and (10 $\bar{1}$ 8) pole figures of the as-cast and ECAPed specimens. Here, (10 $\bar{1}$ 8) was selected to investigate LPSO texture since XRD peaks of (0002), (10 $\bar{1}$ 0),

(10 $\bar{1}$ 1) and (11 $\bar{2}$ 0) in LPSO phase overlap those in α -Mg. The as-cast specimen displayed random orientation. Basal texture of α -Mg grains and LPSO texture formed after one pass of ECAP. Basal texture became more evident with increasing number of ECAP passes; *c*-axes of α -Mg grains inclined by approximately 57° from ED toward ND (normal direction). Similar textures with *c*-axes inclined from ED have been reported in AZ31B^{14,22)} and AZ61²³⁾ after ECAP passes using route Bc. Here, shear direction differs in each pass using route Bc; however, the highest pole intensity maintained at almost the same position up to eight passes of ECAP. Such stability has been also reported in ECAPed pure Mg using route Bc.¹⁵⁾ Therefore, α -Mg grains were found to form basal texture even in ternary alloys after ECAP, similar to pure magnesium. On the other hand, LPSO phase also evidenced this texture after ECAP. LPSO texture perpendicular to ED strengthened under four passes of ECAP and subsequently split into three in 8-pass specimen. Here, (10 $\bar{1}$ 8) and (0 0 0 18) in LPSO phase with 18R structure correspond to (10 $\bar{1}$ 1) and basal planes in α -Mg with 2H structure. Thus, (10 $\bar{1}$ 8) pole figures displayed indicate that basal planes in LPSO phase slightly inclined from ED toward ND up to four passes of ECAP and subsequently was perpendicular to ED in 8-pass specimen. Also, the change in LPSO texture means that LPSO phase deforms up to eight passes of ECAP.

Figure 8 shows inverse pole figure (IPF) maps of ECAPed specimens, with image quality (IQ) map overlays. Here, IQ maps reflect the quality of Kikuchi lines; bright areas in Fig. 8 correspond to α -Mg, while dark areas correspond to either LPSO phase or $\text{Mg}_3\text{Zn}_3\text{Y}_2$ or grain boundaries since obtaining clear Kikuchi-line diffractions is difficult in these regions. Mean grain size of 46.5 μm in the as-cast specimen significantly decreased by the first pass of ECAP. 1-pass

Fig. 6 BSE images of ECAPed specimens (a)–(c); arrows indicate LPSO phase with kink deformation and (d) kink deformation in 8-pass specimen observed from ED.

Fig. 7 (0002) and $(10\bar{1}8)$ pole figures of the as-cast and ECAPed specimens.

specimen consisted of coarse- and fine-grained α -Mg regions, with mean grain sizes of $13.7\mu\text{m}$ in coarse-grained α -Mg regions and $2.16\mu\text{m}$ in fine-grained α -Mg regions. Dynamic recrystallization (DRX) occurred in local areas during one pass of ECAP, evidenced by observation of fine and equiaxed grains. Such DRX has been reported in pure Mg,¹⁵⁾ AZ61,²³⁾ AZ31,²⁴⁾ $\text{Mg}_{97}\text{Y}_2\text{Zn}_{17}$ ¹⁷⁾ and $\text{Mg}_{96}\text{Y}_3\text{Zn}_{18}$ ¹⁸⁾ after ECAP. 2-pass specimen maintained heterogeneous microstructure. Although α -Mg grains ranged between $0.83\mu\text{m}$ and $9.17\mu\text{m}$, the mean grain size was measured to be $1.48\mu\text{m}$, smaller than that in 1-pass specimen. Homogeneous microstructure was obtained after four passes of ECAP. The mean grain size decreased with increasing number of ECAP passes; however, it saturated after four passes of ECAP: $1.44\mu\text{m}$ in 4-pass specimen and $1.85\mu\text{m}$ in 8-pass specimen. Saturation likely results from grain growth caused by deformation heat during ECAP, i.e., DRX.

Figure 9 shows typical stress-strain curves of the as-cast and ECAPed specimens. 0.2% proof strength, tensile strength and elongation of all ECAPed specimens used in this study were summarized in Fig. 10. The as-cast specimen had 0.2% proof stress of 156 MPa and 3.6% elongation. 0.2% proof stress ($\sigma_{0.2\%}$) and ultimate tensile strength (σ_{UTS}) increased up to four passes of ECAP and then saturated. Elongation significantly increased between one and four passes of ECAP and reached 23.5% in 8-pass specimen. Results clearly show that ECAP increases both strength and elongation of $\text{Mg}_{97}\text{Y}_2\text{Zn}_2$ alloys. To discuss the effects of ECAP on tensile properties of $\text{Mg}_{96}\text{Zn}_2\text{Y}_2$ alloys, microstructure and mechanical properties of ECAPed $\text{Mg}_{96}\text{Zn}_2\text{Y}_2$ alloys were compared with those of $\text{Mg}_{96}\text{Zn}_2\text{Y}_2$ alloys extruded at an extrusion ratio of 10 ($\varepsilon_{\text{eq.}} = 2.3$) at 623 K,⁶⁾ revealing that induced strains are approximately equivalent. Figure 11 shows changes in (a) microstructure and (b) tensile properties as a function of equivalent strain induced by ECAP. Open symbols indicate

Fig. 8 IPF maps of ECAPed specimens, with image quality (IQ) map overlays observed from ED.

Fig. 9 Nominal stress-strain curves of the as-cast and ECAPed specimens.

Fig. 10 Changes in tensile properties as a function of equivalent strain induced by ECAP.

data of an extruded $\text{Mg}_{96}\text{Zn}_2\text{Y}_2$ alloy with the highest yield stress and elongation reported in Ref. 6); all data are shown in Fig. 12. ECAP was carried out at 673 K, 50 K higher than the 623 K extrusion temperature. However, the volume fraction of DRXed α -Mg grain regions in 2-pass specimen was much lower than that in the extruded specimens, as shown in Fig. 11(a). Therefore, ECAP was found to suppress

Fig. 11 Changes in (a) microstructure and (b) tensile properties as a function of equivalent strain induced by ECAP. Open symbols indicate data when extruded with $R = 10$.⁶⁾

greatly DRX compared to extrusion when under approximate equivalent induced strain. Figure 11(b) shows changes in tensile properties as a function of equivalent strain induced

Fig. 12 Relationship between the volume fraction of DRXed α -Mg grain regions and tensile properties. Closed symbols indicate data when extruded with $R = 10$.⁶⁾

by ECAP. 0.2% proof stress of the extruded specimen was higher than that of 2-pass specimen, though 2-pass specimen had finer grains and higher volume fractions of deformed regions, resulting in effects of textures on 0.2% proof stress to be larger in the extruded $\text{Mg}_{97}\text{Y}_2\text{Zn}_2$ alloy. The strong basal texture of α -Mg grains parallel to the tensile direction has been reported in extruded $\text{Mg}_{97}\text{Y}_2\text{Zn}_2$ alloys;⁶⁾ i.e., the Schmid factor for basal slip is close to zero in tensile tests. As a result, basal texture of α -Mg grains strongly affects 0.2% proof stress in extruded specimens. On the other hand, 2-pass specimen also displayed basal texture of α -Mg grains; however, the c -axes inclined by approximately 57° from ED toward ND, as shown in Fig. 7. Since basal slips would be easily activated in tensile tests, 2-pass specimen showed lower strength compared to extruded specimens. Conversely, the elongations of the extruded specimen and 2-pass specimen were approximately the same. The extruded specimen showed low elongation even though the volume fraction of DRXed α -Mg grain region was 42%, resulting from that the orientation of basal texture was parallel to the tensile direction. On the other hand, 2-pass specimen displayed basal texture inclined from the tensile direction; however, the volume fraction of DRXed α -Mg grain region was 8.4%. Thus, both the extruded and 2-pass specimen showed low elongation. The volume fraction of DRXed α -Mg grain and deformed regions significantly changed in the equivalent strain range between 2.2 and 3.3 as shown in Fig. 11(a); however, such changes were not observed in tensile properties as shown in Fig. 11(b). Causes behind this will be discussed in the near future.

Figure 12 shows the relationship between the volume fraction of DRXed α -Mg grain region and tensile properties. Closed symbols indicate data of $\text{Mg}_{96}\text{Zn}_2\text{Y}_2$ alloys extruded at an extrusion ratio of 10 with different microstructures; four types of starting billets were prepared with different cooling rates.⁶⁾ Also, both $\sigma_{0.2\%}$ and elongation have been reported to increase with increasing volume fraction of DRXed α -Mg grain region.⁶⁾ On the other hand, ECAPed data show that both $\sigma_{0.2\%}$ and σ_{UTS} are almost constant and independent of the volume fraction of DRXed α -Mg grain region. The different tendencies likely depend on the orientation relationship between basal texture and tensile direction. Conversely, elongation increases with increasing volume fraction of

DRXed α -Mg grain region; the results are in agreement with extrusion data. Therefore, the DRXed α -Mg grain region appears to play a role in elongation and LPSO texture in tensile properties. (0 0 0 18) in LPSO texture, basal plane, slightly inclined from ED toward ND up to four passes of ECAP and possibly affect elongation up to four passes. In 8-pass specimen with high elongation, LPSO texture split into three shown in Fig. 7, meaning that (0 0 0 18) was parallel to the tensile direction and may affect strength. However, both texture intensity and volume fraction of the deformed region including LPSO phase were low, showing that the effects of LPSO texture on strength were likely small in 8-pass specimen. Thus, grain refinement by plastic deformation is effective for strengthening of $\text{Mg}_{97}\text{Y}_2\text{Zn}_2$ alloys; however, grain size saturated due to DRX when induced strain exceeded 3.15 (3-pass). On the other hand, elongation increased with increasing volume fraction of DRXed α -Mg grain region. This study found that ECAP improves both strength and elongation of $\text{Mg}_{97}\text{Y}_2\text{Zn}_2$ alloys; in particular, elongation significantly increased due to basal texture and volume fraction of DRXed α -Mg grain region. Therefore, controlling volume fraction of DRXed α -Mg grain region is a key to increasing elongation of $\text{Mg}_{97}\text{Y}_2\text{Zn}_2$ alloys.

4. Conclusions

$\text{Mg}_{96}\text{Zn}_2\text{Y}_2$ alloys were deformed by equal channel angular pressing (ECAP), and the relationship between microstructure and mechanical properties were investigated. $\text{Mg}_{96}\text{Zn}_2\text{Y}_2$ alloys consisted of α -Mg, long period stacking ordered (LPSO) and $\text{Mg}_3\text{Zn}_3\text{Y}_2$ phases. Microstructure of ECAPed $\text{Mg}_{96}\text{Zn}_2\text{Y}_2$ alloys were divided into two regions: dynamically recrystallized (DRXed) α -Mg grain and deformed regions. 0.2% proof stress increase with increasing mean grain size. However, both grain size and 0.2% proof stress saturated after four passes of ECAP due to dynamic recrystallization (DRX). Microstructure and mechanical properties of ECAPed $\text{Mg}_{96}\text{Zn}_2\text{Y}_2$ alloys were compared with those of $\text{Mg}_{96}\text{Zn}_2\text{Y}_2$ alloys extruded at an extrusion ratio of 10. As a result, ECAP was found to suppress greatly DRX compared to extrusion when the induced strain is approximately the same. ECAP improves especially elongation rather than strength in $\text{Mg}_{97}\text{Y}_2\text{Zn}_2$ alloys. Elongation increases with increasing volume fraction of DRXed α -Mg grain region.

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