Effect of Extrusion Parameters on Mechanical Properties of Mg₉₇Zn₁Y₂ Alloys at Room and Elevated Temperatures

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The effects of alloy extrusion parameters, such as extrusion ratio, temperature, and speed on the mechanical properties at room and elevated temperatures and the microstructure evolution were investigated in the production of high strength Mg-Zn-Y alloys. The alloy used is a Mg₉₇Zn₁Y₂ (at%) which is engineered to acquire a long-period stacking ordered (LPSO) structure phase to increase alloy-strength. The microstructure of the extruded Mg₉₇Zn₁Y₂ alloy consists of hot-worked and dynamically recrystallized (DRXed) α -Mg grains that includes a fiber-shaped LPSO phase elongated along the direction of extrusion. Whereas an increase in average equivalent strain promotes the DRX of α -Mg matrix and the dispersion of the fiber-shaped LPSO phase, an increase in average metal flow rate is conducive to the DRX of α -Mg grains, but is not to the dispersion of LPSO phase. The mechanical properties of the extruded Mg-Zn-Y alloys are affected by changes in the area fraction of the DRXed grains and the dispersion of the fiber-shaped LPSO phase. As the extrusion ratio and extrusion speed increase, overall DRX bringing grain growth in its train in the α -Mg matrix phase decreases the tensile strength of alloys, but the dispersed fiber-shaped LPSO phase remaining in the DRXed grains region makes good the adverse effect of overall DRX followed by grain growth.

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1. Introduction

Being at the top of the list of light structural materials, magnesium alloys (Mg alloys) seem likely to find great use in automotive, railway, and aerospace applications where weight reduction is of overriding importance.^{1,2)} On the other hand, however, they also have such problems as relatively low strength and low ductility that limit their wide application. Wider application of Mg alloys dictates higher strength and plasticity. The last decade has witnessed the advent of highly strong and ductile Mg-Zn-rare earth (RE) alloys.³⁻⁷⁾ The Mg-Zn-RE (RE = Y, Gd, Tb, Dy, Ho, Er, Tm) alloys have been attracting much attention not only because of their excellent mechanical properties but also of their unique microstructure. The Mg-Zn-RE alloys are composed of an α -Mg matrix phase and a long-period stacking ordered (LPSO) structure phase.⁵⁻¹⁴⁾ The LPSO phase-containing Mg alloys that are hot-extruded exhibit high tensile yield strength (>350 MPa) and moderate elongation (>5%) at room temperature, and high tensile yield strength (>300 MPa) at 473 K.^{4-7,15)} It was reported previously that the mechanical properties of extruded alloys improved owing to α -Mg matrix grain refinement by dynamic recrystallization (DRX), LPSO phase dispersion, and kink-deformation band formation in the LPSO phase.¹⁶⁻²¹⁾ Meanwhile, feasibility studies of the LPSO phase-containing Mg-Zn-RE alloys have been under way. With these alloys, extrusion is one of the most important large-scale manufacturing method. As much as a high extrusion ratio and a high extrusion speed count much in mass production,²²⁻²⁴⁾ the microstructure evolution and strengthening mechanism in extruded alloys have not yet been elucidated in detail. The present study attempts to

clarify the influence of extrusion parameters on the microstructure evolution and mechanical properties of the LPSO phase-containing Mg₉₇Zn₁Y₂ alloys obtained at room and elevated temperatures. Changes in the mechanical properties of the alloys are expressed as functions of the average equivalent strain and intra-extrusive average metal flow rate.

2. Experimental

2.1 Materials preparation

Master ingots with a nominal composition of Mg₉₇Zn₁Y₂ (at%) were prepared by high frequency induction melting in a carbon crucible, followed by casting into steel mould. The cast ingots were cut into billets 29 mm in diameter and 70 mm in length. The billets were extruded at ratios ranging from 3 to 15. Extrusion ratio, R , is defined as follows:

$$R = \frac{A_0}{A_e}, \quad (1)$$

where A_0 is the cross-sectional area of raw material billet and A_e the cross-sectional area of extrusion bar. Die half angle, θ , is 30 degrees. Extrusion temperatures are 623 K and 723 K. In order to control average equivalent strain rate, extrusion was performed at ram speeds ranging from 1.0 mm s⁻¹ to 7.0 mm s⁻¹. The billets were soaked for 20 min at each extrusion temperature, prior to extrusion.

2.2 Calculation of extrusion parameters

In this study we attempt to estimate the average equivalent strain and the average metal flow rate (average equivalent strain rate) during extrusion. The average equivalent strain, $\bar{\epsilon}$, is given by

$$\bar{\epsilon} = \ln R. \quad (2)$$

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Fig. 1 Average equivalent strain dependence of the tensile yield strength at room and elevated temperature for the Mg₉₇Zn₁Y₂ alloys extruded with average metal flow rates of 0.6–0.66 s⁻¹ at (a) 623 K and (b) 723 K.

The average metal flow rate, $\dot{\bar{\epsilon}}$, is calculated using the following equation:

$$\dot{\bar{\epsilon}} = \left\{ \frac{6V_e \ln R \cdot \tan\theta}{D(1 - 1/R\sqrt{R})} \right\}, \quad (3)$$

where V_e is extrusion ram speed, θ is die half angle, and D is billet diameter. The calculations are described in greater detail in appendix.

2.3 Materials characterization

Tensile tests were carried out using an Instron Model 5584 tensile testing machine at room and elevated temperatures with an initial strain rate of 5×10^{-4} s⁻¹. The dimensions of tensile specimens were 2.5 mm in diameter and 15 mm length. The tensile axis was laid off along the direction of extrusion. The microstructure of the extruded alloys was observed by confocally optical microscopy with a Lasertec Model C-130 and field-emission scanning electron microscopy (SEM) with a JSM-7001F. SEM images were taken in the secondary electron mode and the back-scattered electron mode.

3. Results

3.1 Mechanical properties

Figure 1 shows changes in the tensile yield strength of extruded Mg₉₇Zn₁Y₂ alloys at 623 K and 723 K determined as a function of average equivalent strain induced during extrusion. Average metal flow rate is $0.6 \text{ s}^{-1} \sim 0.66 \text{ s}^{-1}$. The alloy that was extruded at 623 K with average equivalent strain ranging from 1.6 to 2.7 exhibited high tensile yield strength at room temperature, but the tensile yield strength at elevated temperatures decreased with increasing average equivalent strain (Fig. 1(a)). In extrusion at 723 K, on the other hand, tensile yield strength was constant at ≤ 523 K regardless of an increase in average equivalent strain. Tensile yield strength at ≥ 573 K decreased with increasing average equivalent strain (Fig. 1(b)).

Figure 2 shows changes in the tensile yield strength of extruded Mg₉₇Zn₁Y₂ alloys with average equivalent strain of 2.3 (R10) at 623 K and 723 K determined as a function of

Fig. 2 Average metal flow rate dependence of the tensile yield strength at room and elevated temperature for the Mg₉₇Zn₁Y₂ alloys extruded with average equivalent strain of 2.3 at (a) 623 K and (b) 723 K.

Fig. 3 Optical micrographs of the longitudinal section of Mg₉₇Zn₁Y₂ alloys extruded with average equivalent strain of (a) 1.1, (b) 1.6, (c) 2.3, and (d) 2.7. Extrusion temperature and average metal flow rate are 623 K and 0.6–0.66 s⁻¹, respectively.

average metal flow rate. Tensile yield strength decreased with increasing average metal flow rate. In addition, the average metal flow rate had a decreasing effect on the tensile yield strength with increasing testing temperature.

3.2 Microstructure

Figure 3 shows optical micrographs of a longitudinal section of the Mg₉₇Zn₁Y₂ alloys extruded at 623 K with various equivalent strain of 1.1 (R3), 1.6 (R5), 2.3 (R10), and 2.7 (R15). Extrusion speed was varied to keep the metal flow rate constant at $0.6 \sim 0.66$ s⁻¹. The billet grain size before extrusion was approximately 400 μm , the crystal grains were refined bimodally into the worked and the DRXed grains during extrusion. Some original old grains remained and were elongated in the direction of extrusion, while some were

Fig. 4 Optical micrographs of the longitudinal section of $\text{Mg}_{97}\text{Zn}_1\text{Y}_2$ alloys extruded with various average metal flow rates at different temperatures: of (a) 0.3 s^{-1} , (b) 0.9 s^{-1} , and (c) 1.6 s^{-1} at 623 K; (d) 0.3 s^{-1} , (e) 1.2 s^{-1} , (f) 1.9 s^{-1} at 723 K. Average equivalent strain is 2.3.

observed to be undergoing dynamic recrystallization (DRX). As the average equivalent strain increased, DRX were promoted. When the average equivalent strain was 2.7, area fraction of the DRX grains reached $>55\%$.

Figure 4 shows optical micrographs of the $\text{Mg}_{97}\text{Zn}_1\text{Y}_2$ alloys extruded at 623 K and 723 K with various metal flow rate of 0.3 s^{-1} , 0.9 s^{-1} , 1.2 s^{-1} , 1.6 s^{-1} , and 1.9 s^{-1} . Though average equivalent strain did not vary (extrusion ratio was 10), an increase in average metal flow rate precipitated DRX bringing grain growth in its train.

Figure 5 shows SEM images of the $\text{Mg}_{97}\text{Zn}_1\text{Y}_2$ alloys extruded at 623 K with varying equivalent strain of 1.1, 1.6, 2.3, and 2.7. SEM images revealed that the LPSO phase region was elongated in the direction of extrusion. Furthermore, many kink deformation bands were seen to be formed in the LPSO phase, as reported previously.^{5,6,16–21,25} The dispersion of the LPSO phase seems to increase with increasing average equivalent strain.

Figure 6 shows SEM images of the $\text{Mg}_{97}\text{Zn}_1\text{Y}_2$ alloys extruded at 623 K with varying average metal flow rate. The LPSO phase region was elongated along the extrusion direction, but the dispersion of the LPSO phase did not seem to be affected by the variation of average metal flow rate.

4. Discussion

The microstructure of extruded Mg-Zn-Y alloys with the LPSO phase is characterized by the bimodal microstructure evolution in the α -Mg matrix region and the dispersion of the

Fig. 5 SEM images of the longitudinal section of $\text{Mg}_{97}\text{Zn}_1\text{Y}_2$ alloys extruded with average equivalent strain of (a) 1.1, (b) 1.6, (c) 2.3, and (d) 2.7. Extrusion temperature and average metal flow rate are 623 K and $0.6\text{--}0.66 \text{ s}^{-1}$, respectively.

fiber-shaped LPSO phase. Namely, the extruded $\text{Mg}_{97}\text{Zn}_1\text{Y}_2$ alloys consist of randomly oriented DRXed α -Mg grains, worked α -Mg grains with strong basal texture ($[10\bar{1}0]//$

Fig. 6 SEM images of the longitudinal section of Mg₉₇Zn₁Y₂ alloys extruded with average metal flow rates of (a) 0.3 s⁻¹, (b) 0.9 s⁻¹, (c) 1.6 s⁻¹. Extrusion temperature and average equivalent strain are 623 K and 2.3 (R10), respectively.

Fig. 7 Changes in area fraction and average grain size of the DRXed grains in Mg₉₇Zn₁Y₂ extruded alloys as a function of average equivalent strain. Extrusion temperature and average metal flow rate are 623 K and 0.6–0.66 s⁻¹, respectively.

Fig. 8 Changes in area fraction and average grain size of the DRXed grains in Mg₉₇Zn₁Y₂ extruded alloys as functions of average metal flow rate. Average equivalent strain is 2.3.

E.D.), and fiber-shaped LPSO phase grains with kink deformation band.^{19–21)} The microstructural characterization of extruded Mg–Zn–Y alloys awakened our interest in the mechanism whereby each region contributes to the improvement of their tensile strength at room and elevated temperatures.

Figures 3 and 4 reveal that as the average equivalent strain and average metal flow rate increase, DRX is precipitated during extrusion. The relationship between the area fraction of DRXed grains and two extrusion parameters, average equivalent strain and average metal flow rate, is shown in Figs. 7 and 8, respectively. These results reveal that as the average equivalent strain and average metal flow rate increase, DRX of the α -Mg matrix bringing grain growth in its train is precipitated during extrusion. Figure 9 exhibits the relationship between the area fraction of the DRXed grains and the tensile yield strength of the extruded Mg₉₇Zn₁Y₂ alloys. An increase in area fraction of the DRXed grains

decreased alloy strength at room and elevated temperatures. Though the micrometer-sized grains that are randomly oriented are quite effective in enhancing alloy ductility because magnesium show strong plastic anisotropy of hcp crystal structure,²⁶⁾ they may play a role in the reduction of alloy strength at room and elevated temperatures. Conversely, when the tensile direction is parallel to the direction of extrusion, the worked grains with a strong basal texture act in favor of alloy strength because of a low average Schmid factor for $\langle 11\bar{2}0 \rangle \langle 0002 \rangle$ basal slip and the suppression of $\langle 10\bar{1}\bar{1} \rangle \{10\bar{1}\bar{2}\}$ twinning.²⁷⁾ The extruded α -Mg grains embedded in the DRXed α -Mg matrix are effective for alloy strengthening.

Let us turn our attention to the LPSO phase that is conducive to alloy strengthening. It was reported that the LPSO phase itself also helped increase alloy strength as a fiber-like reinforcement.¹⁹⁾ Figures 5 and 6 exhibit how the dispersion of LPSO phase changes with increasing average equivalent strain and average metal flow rates, respectively.

Fig. 9 Relationship between tensile yield strength of $\text{Mg}_{97}\text{Zn}_1\text{Y}_2$ extruded alloys at room and elevated temperatures and area fraction of the DRXed grains.

For purposes of this study the level of LPSO phase dispersion, D_L (μm^{-1}), was defined by the following expression to determine it quantitatively:

$$D_L = \frac{\sum N_v}{\sum L_v}, \quad (4)$$

where L_v (μm) is the length of a vertical-line segment plotted vertically against the direction of extrusion in the SEM images (Figs. 5 and 6) and N_v is the number of LPSO phases intersected by segment L . The total length of segments, $\sum L_v$, is longer than 5 mm. Furthermore, SEM images show that the fiber-shaped LPSO phase is not uniformly distributed. As the density of the LPSO phase in the extruded $\text{Mg}_{97}\text{Zn}_1\text{Y}_2$ alloys varies from region to region because the extruded $\text{Mg}_{97}\text{Zn}_1\text{Y}_2$ alloys consist of bimodal α -Mg grains, it is necessary to estimate the dispersion of the LPSO phase in each region. Assuming that the worked grain region is extruded axisymmetrically in the direction of extrusion in a state of invariable worked grain volume, the LPSO phase dispersion in the worked grain region, D_L^{worked} , is estimated by the following expression:

$$D_L^{\text{worked}} = \sqrt{R} D_L^{\text{cast}}, \quad (5)$$

where D_L^{cast} is the LPSO phase-dispersion in as-cast state. The LPSO phase-dispersion in the DRXed grains region, D_L^{DRX} , is calculated by distributing an apparent LPSO phase-dispersion (D_L) according to the area fraction of each region,

$$D_L^{\text{DRX}} = \frac{D_L - \sqrt{R} D_L^{\text{cast}} (1 - A_f^{\text{LPSO}} - A_f^{\text{DRX}})}{A_f^{\text{DRX}}}, \quad (6)$$

where A_f^{DRX} and A_f^{LPSO} are area fractions of the DRXed α -Mg grains and the LPSO phase, respectively. Figure 10 shows

Fig. 10 Changes in the LPSO phase-dispersion as functions of (a) average equivalent strain and (b) average metal flow rate.

changes in the apparent dispersion of LPSO phase as functions of average equivalent strain and average metal flow rate. The estimated dispersions in the worked grains region and the DRXed grains region are also plotted in the figures. As is clear from Fig. 10(a), whereas the apparent LPSO phase-dispersion increased monotonously with increasing average equivalent strain, the estimated dispersion in the DRXed grains decreased with increasing average equivalent strain when average equivalent strain was ≤ 2 and then increased. As Fig. 10(b) shows, on the other hand, that though apparent LPSO phase-dispersion did not seem to depend on average metal flow rate, the estimated dispersion in the DRXed grains increased with decreasing average metal flow rate. The alloy extruded with lower average metal flow rate exhibited high tensile yield strength at room and elevated temperatures (Fig. 2). Adequate retention of the worked grains and high dispersion of the fiber-shaped LPSO phase in the DRXed grains region seem to account for the high strength of the alloys. Judging from the shapes of LPSO phases, the strengthening effect of the LPSO phase may be obtained by a mechanism of fiber-like reinforcement^{19,28)} in the $\text{Mg}_{97}\text{Zn}_1\text{Y}_2$ alloy. To corroborate this assumption it is necessary to evaluate the morphological effect of the LPSO phase as well as its dispersion. Combination of a higher extrusion ratio with a higher extrusion speed, which is required for mass production helps generate a great deal of heat during extrusion. A very high temperature eventually brings about overall DRX in the α -Mg matrix phase. In this study, α -Mg grains for the most part were completely DRXed in extrusion with an average metal flow rate of $\geq 1.2 \text{ s}^{-1}$ and an average equivalent strain of ≥ 2.3 at a high temperature of $\geq 723 \text{ K}$. Under the extrusion conditions mentioned above, two types of model material with the overall DRXed grains were prepared to evaluate the morphological effect of the LPSO phase. Figure 11 shows SEM images of two typical $\text{Mg}_{97}\text{Zn}_1\text{Y}_2$ alloys extruded at 723 K with the overall DRXed grains. Though both the alloys were equally about $10 \mu\text{m}$ in grain size and had much the same LPSO phase dispersion, the morphology of the LPSO phase differed from one alloy to the other with respect to aspect ratio. Figure 12 shows the aspect ratio distribution in the LPSO phase of $\text{Mg}_{97}\text{Zn}_1\text{Y}_2$ alloys

Fig. 11 SEM images of the longitudinal section of Mg₉₇Zn₁Y₂ alloys extruded at 723 K with: (a) average equivalent strain of 2.3 and average metal flow rates of 1.9 s⁻¹, and (b) 2.7 and 2.1 s⁻¹.

extruded at 723 K with average equivalent strain of 2.3 (R10) and 2.7 (R15). Obviously enough, the alloys extruded with average equivalent strain of 2.3 were found to have large amount of the fiber-shaped LPSO phase with a high aspect ratio. Intense equivalent strain reduced the aspect ratio of the LPSO phase during extrusion. Figure 13 shows tensile yield strength of the Mg₉₇Zn₁Y₂ alloys extruded at 723 K with average equivalent strain of 2.3 and 2.7 and various metal flow rates. Figures 12 and 13 show that the alloy with a high aspect ratio LPSO phase exhibits higher yield strength, especially, at elevated temperatures. These extruded Mg₉₇Zn₁Y₂ alloys owe their high elevated-temperature tensile strength to the textured α -Mg grains and the fiber-shaped LPSO phase embedded in the DRXed α -Mg matrix region. The dispersion and aspect ratio of the LPSO phase as well as the area fraction of the worked grains with strong basal texture are parameters of crucial importance for strengthening of the Mg₉₇Zn₁Y₂ alloys.

5. Conclusions

The following conclusions have been drawn from this study: in which changes in the elevated temperature-mechanical properties and the microstructure evolution of the extruded Mg₉₇Zn₁Y₂ alloys with LPSO phase were referred to as functions of the average equivalent strain and average metal flow rate.

(1) Under conditions of constant average metal flow rate of 0.6 s⁻¹–0.66 s⁻¹, the extruded alloys exhibited high strength at <500 K regardless of an increase in the average equivalent

Fig. 12 LPSO phase-aspect ratio distribution of Mg₉₇Zn₁Y₂ alloys extruded at 723 K with: (a) average equivalent strain of 2.3 and average metal flow rates of 1.9 s⁻¹, and (b) 2.7 and 2.1 s⁻¹.

Fig. 13 Average metal flow rate dependence of the tensile yield strength at room and elevated temperatures for the extruded Mg₉₇Zn₁Y₂ alloys.

strain. An increase in average equivalent strain precipitated not only the DRX of α -Mg matrix grains followed by grain growth but also dispersion of the fiber-shaped LPSO phase. It deserves special mention that the highly dispersed fiber-shaped LPSO phase in the DRXed grain region seems likely to compensate alloy strength reduction due to DRX bringing grain growth in its train.

(2) Under conditions of a constant average equivalent strain of 2.3 (R10), the tensile yield strength of the extruded alloys at room and elevated temperatures decreases with increasing average metal flow rate. An increase in average metal flow rate precipitated the DRX of α -Mg matrix grains followed by grain growth, but did not promote at all the dispersion of the LPSO phase.

(3) The multimodal microstructure that consisted of worked α -Mg grains, DRXed α -Mg grains and fiber-shaped LPSO phase grains produced high strength at room and elevated temperatures. With increasing extrusion ratio and extrusion speed overall DRX took place in the α -Mg matrix and the fiber-shaped LPSO phases and continued, exerting a reinforcing effect, thereby helping maintain relatively high alloy strength.

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Appendix A: Average Equivalent Strain^{29,30)}

Assuming an axisymmetric extrusion, plastic strain components, ε_x , ε_y , ε_z , have the following relationships:

$$\varepsilon_x + \varepsilon_y + \varepsilon_z = 0, \quad (\text{A} \cdot 1)$$

$$\varepsilon_x + \varepsilon_y = -\ln R, \quad (\text{A} \cdot 2)$$

$$\varepsilon_x = \varepsilon_y = -\frac{1}{2} \ln R, \quad (\text{A} \cdot 3)$$

$$\varepsilon_z = \ln R, \quad (\text{A} \cdot 4)$$

where z axis is parallel to the direction of extrusion. Considering the second invariant of the plastic strain increment tensor, an equivalent plastic strain increment, $d\bar{\varepsilon}$, is defined by

$$d\bar{\varepsilon} = \left[\frac{2}{9} \left\{ (d\varepsilon_x - d\varepsilon_y)^2 + (d\varepsilon_y - d\varepsilon_z)^2 + (d\varepsilon_z - d\varepsilon_x)^2 \right. \right. \\ \left. \left. + \frac{3}{2} (d\gamma_{xy}^2 + d\gamma_{yz}^2 + d\gamma_{zx}^2) \right\} \right]^{1/2}, \quad (\text{A} \cdot 5)$$

where γ is shear strain. The equivalent strain, $\bar{\varepsilon}$, can be written as

$$\bar{\varepsilon} = \int d\bar{\varepsilon}, \quad (\text{A} \cdot 6)$$

where the integral is taken along the strain path. The combination of eqs. (A.2)–(A.6) yields

$$\bar{\varepsilon} = \ln R. \quad (\text{A} \cdot 7)$$

Appendix B: Average Metal Flow Rate

Average metal flow rate (average equivalent strain rate), $\dot{\bar{\varepsilon}}$, is defined by

$$\dot{\bar{\varepsilon}} = \frac{\bar{\varepsilon}}{t}, \quad (\text{B} \cdot 1)$$

where t is defined as the time required for the material to receive the average equivalent strain ($\bar{\varepsilon}$). Time, t , is obtained by a geometrical calculation,

$$V_e t \cdot \pi \left(\frac{D}{2} \right)^2 = \frac{\pi}{3} \left(\frac{D}{2} \right)^2 \frac{D}{2 \tan \theta} - \frac{\pi}{3} \left(\frac{D}{2\sqrt{R}} \right)^2 \frac{D}{2\sqrt{R} \tan \theta}. \quad (\text{B} \cdot 2)$$

Equation (B.2) becomes

$$t = \frac{D}{6V_e \tan \theta} \left(1 - \frac{1}{R\sqrt{R}} \right). \quad (\text{B} \cdot 3)$$

Therefore, average metal flow rate ($\dot{\bar{\varepsilon}}$) is determined from extrusion ram speed, V_e , die half angle, θ , billet diameter, D , and extrusion rate, R , as follows:

$$\dot{\bar{\varepsilon}} = \left\{ \frac{6V_e \ln R \cdot \tan \theta}{D(1 - 1/R\sqrt{R})} \right\}. \quad (\text{B} \cdot 4)$$

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