Forging induces changes in the formability and microstructure of extruded $\text{Mg}_{96}\text{Zn}_2\text{Y}_2$ alloy with a long-period stacking order phase

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Abstract

We examined the effects of the processing temperature and the grain size on the ductility and flow stress of extruded Mg₉₆Zn₂Y₂ (atom%) alloy by upsettability testing at various forging temperatures and for various grain sizes of the α -Mg phase. The extruded Mg₉₆Zn₂Y₂ alloy showed a forging limit as a reduction in height of 65% at forging temperatures of 573–673 K and an initial strain rate of $3.5 \times 10^{-1} \text{ s}^{-1}$. The formability of LPSO-type Mg alloys can be improved by the production of an α -Mg phase with small grains and a LPSO phase with kink bands with a large-angle tilt. The hard LPSO phase has a bending angle of about 100° at the grain boundaries and has a large deformability. Forging of about 10% is necessary for the formation of kink bands in the LPSO phase, and these kink bands deform continuously with increasing plastic deformation.

Key words: magnesium alloy, forging, formability, microstructure, annealing, long-period stacking order phase

1. Introduction

Magnesium alloys are among the lightest practical metals and are renowned for their high specific strength, good machinability, and good shock-absorbing properties [1]. Because of their hexagonal close-packed structure, Mg alloys have few active slip systems other than basal slip; as a result, hot working is used in deformation processing of structural members [1,2]. Recent advances in grain refinement [3,4] and microstructure control [5,6], as well as the development of Mg–1.5Zn–0.2Ce alloy with good formability [7], have led to Mg alloys with better deformation-processing properties and lower processing temperatures. However, generic Mg alloys and extruded Mg–1.5Zn–0.2Ce alloy have low yield strengths and low thermal stabilities in comparison with Al alloys. In recent years, Mg–Zn–Y alloys [8–10] and Mg–Zn–Gd alloys [11,12] with long-period stacking order (LPSO) phases have been developed as high-strength heat-resistant Mg alloys [8–11], and alloys that retain a stable strength and ductility from room temperature up to high temperatures have been reported [10]. The deformation mechanism of LPSO-phase Mg alloys is known to involve the formation of kink bands in the LPSO phase and microstructural refinement of the α -Mg phase during plastic deformation; these processes result in significant increases in strength with retention of ductility [9]. Mg₉₆Zn₂Y₂ alloys subjected to various annealing temperature have been reported to elongate by a factor of up to two at temperatures of up to 573 K without a significant decrease in strength, demonstrating that the LPSO phase exerts a pinning effect on the α -Mg phase; these alloys also have good heat-forging properties [10,13].

Even though knowledge of the deformation-processing performance of high-strength, heat-resistant Mg alloys is crucial for the establishment of a technology for processing microstructure-controlled, high-strength, heat-resistant, LPSO-phase Mg alloys, the effects of structural factors and processing conditions on the deformation-processing performance of LPSO-phase Mg alloys when the microstructure of the LPSO and α -Mg phases is controlled

are not known. We therefore examined the effects of the processing temperature and the grain size on the ductility and flow stress of extruded $\text{Mg}_{96}\text{Zn}_2\text{Y}_2$ (atom%) alloy by carrying out upsettability tests for various processing temperatures and for various grain sizes of the α -Mg phase. We also investigated the upsettability ratio that results in kink-band formation in the LPSO phase.

2. Experimental Procedure

We used extruded $\text{Mg}_{96}\text{Zn}_2\text{Y}_2$ (atom%) alloy in which a second LPSO phase was dispersed in the parent phase. The cast material was synthesized by melting the elements, weighed to the target stoichiometry, to form a 200-mm-long billet with a diameter of 69 mm. The billet was extruded at 623 K to an extrusion ratio of 10 at a rate of $1.0 \text{ mm} \cdot \text{s}^{-1}$ to give a round bar with a diameter of 22 mm that was cooled with water immediately after extrusion. Fig. 1 shows scanning electron microscopy (SEM) images of the as-cast and extruded samples of the $\text{Mg}_{96}\text{Zn}_2\text{Y}_2$ alloy. The gray regions in the micrographs correspond to the LPSO phase, the black regions to the α -Mg phase, and the white regions to the intermetallic $\text{Mg}_3\text{Zn}_3\text{Y}_2$ [14]. Forging limit and flow stress curve of the $\text{Mg}_{96}\text{Zn}_2\text{Y}_2$ alloy were measured by the upsettability test [15]. In the test, a cylindrical specimen was compressed with concentrically grooved flat dies to restrict the end surfaces of the specimen, so that the influence of friction between the specimen and the die during the test was eliminated. The forging limit was defined as the reduction in height of the specimen when a crack was first observed on the surface of the forged specimen. Flow stress curve was obtained from the load-stroke data in the upsettability test [16]. From the 22 mm diameter round bar of extruded $\text{Mg}_{96}\text{Zn}_2\text{Y}_2$ alloy, specimen for the upsettability test were machined to a diameter of 16 mm and a height of 24 mm. The axis of the specimen corresponded to that of the extruded rod.

Some specimens were annealed in air in an electric furnace at 473–773 K for 3.6 ks and then immediately cooled in water. The upsettability test was performed on a universal tester at an initial strain rate of $3.5 \times 10^{-1} \text{ s}^{-1}$. To prevent the heated specimen from rapidly cooling on the die, the dies were heated to a temperature of 523 K when the testing temperatures were higher than 523 K. For the case of testing temperatures below 473 K, the dies were heated to the same temperature as the specimen. The as-extruded and annealed specimens were tested after heating for 1.8 ks in an electric furnace at the testing temperature. The specimens were cooled with water within 3 s of testing.

We also investigated the changes in the microstructures of the α -Mg phase and the LPSO phase with compression of a reduction in height of 5, 10, or 30% and we performed hardness test and microstructural observations in the plane perpendicular to the direction of compression. The hardness tests were performed by using a Vickers hardness meter with load of 1.96 kN and an indentation time of 20 s. The microstructural observations were conducted by optical microscopy (OM), SEM, and transition electron microscopy (TEM). The crystal orientation was determined by means of electron-backscatter diffraction (EBSD) patterns on in $250 \times 250 \text{ }\mu\text{m}$ region with a measurement step of $0.3 \text{ }\mu\text{m}$. Hardness test and microstructural observations were performed at the centers of the forged specimens.

3. Results and Discussion

3.1 Changes in the formability and microstructure of the extruded material

Optical micrographs were recorded of the as-extruded specimen [Fig. 2(a)] and of specimens annealed for 3.6 ks at 573, 673, or 773 K [Figs. 2(b)–(d), respectively]. The proportion of recrystallized region of the α -Mg phase was about 70%, and the remaining region did not recrystallize and it extended in the direction of extrusion. The content of LPSO phase in the extruded $\text{Mg}_{96}\text{Zn}_2\text{Y}_2$ rod was 25% by volume. As the annealing temperature

increased, the grains grew to a size of over 10 μm and the regions of LPSO phase became plate-shaped; however, there was no change in the volumetric content of the LPSO phase. Fig. 3 shows the forging limit and the flow stress curve of the extruded and the as-cast specimens obtained from the upsettability test at various forging temperatures. The forging limit of the extruded specimen at room temperature was a reduction in height of less than 5%; and the forging limit increased with increasing forging temperature, and reached a reduction in height of 65% for a forging temperature of 573–673 K. On the other hand, the forging limit was dropped to the reduction in height of 55% above 673 K.

High-temperature tensile-deformation studies on extruded $\text{Mg}_{96}\text{Zn}_2\text{Y}_2$ alloy showed that the yield stress and elongation were 280 MPa and 22%, respectively, for testing temperatures of up to 523 K. However, it has been claimed that the yield stress decreases to 145 MPa and the elongation increases to 60% when the testing temperature is increased to 573 K, implying that grain boundary sliding occurs in the α -Mg phase at a testing temperature of 573 K [17]. The flow stress curves of the as-extruded and annealed specimens at various forging temperatures (Fig. 3) exhibited work hardening behavior at temperatures below 573 K and work softening behavior at higher temperatures. Static recrystallization of the α -Mg phase during heating of the material and dynamic recrystallization during forging were expected to occur readily at forging temperature of 573 K, resulting in a maximum reduction in height for forging limit at this temperature. In other words, a higher forging limit can be achieved by means of static and dynamic recrystallization of the α -Mg phase to maintain the texture of the micrograins and by processing to transform kink bands in the LPSO phase into grain boundaries with a large angular tilt, as discussed in the following sections.

3.2 Change in formability of extruded material by annealing

Fig. 4 shows the forging limit of the annealed specimen determined from the upsettability tests. Fig. 4(b) is the flow stress curve of the specimen annealed at 773 K; this specimen showed the largest elongation in a room-temperature tensile test [10]. Fig. 4 shows that the forging limit of the as-extruded specimen and that of specimens annealed at 573 K and 673 K were the same, whereas the forging limit for the specimen annealed at 773 K was identical to that determined by the upsettability test on the cast $\text{Mg}_{96}\text{Zn}_2\text{Y}_2$ alloy, and the forging limit was about three times higher than that of the as-extruded specimen. However, the formability of the specimen annealed at 773 K was worse than that of the other annealed specimens when the forging temperature was increased. As can be seen in Figs. 1 and 2, the internal morphology of the cast specimen and the specimen annealed at 773 K showed an absence of kink bands in the LPSO phase. The specimens with a morphology in which the LPSO phase lacked kink bands (which increase the strength of this alloy) showed greater reduction in the height of the forging limit around room temperature compared with the corresponding specimens containing kink bands; however, the formability at elevated forging temperatures was better for the specimens with kink bands in the LPSO phase. A comparison of the forging flow stress curve of the as-extruded specimen [Fig. 4(b) and Fig. 3] shows that there is no point at which work softening becomes significant as the forging temperature increased. It has been reported [18] that the forging properties of AZ-type Mg alloys, in general, deteriorate as the grain size increases and that work softening in the flow stress curve is caused by dynamic recrystallization. The results for the α -Mg phase in the alloy used in this work matched those of AZ-type Mg alloys. The α -Mg phase of the specimen annealed at 773 K had a large grain size of over 10 μm , which is considered to be responsible for the reduce in the reduction in height of the forging limit compared with the other annealed and extruded materials, as shown in Figs. 1 and 4. The upsettability tests on the extruded and annealed specimens demonstrated that to give a reduction in height of 65% at 573–673 K in one step

without crack formation, the α -Mg phase in $\text{Mg}_{96}\text{Zn}_2\text{Y}_2$ alloy must have a refined microstructure. Furthermore, in our study the formation of kink bands in the LPSO phase was found for the first time to contribute to the achievement of a formability of about 5% in one step, as shown in Fig. 5.

3.3 Morphology changes on increasing height reduction for forging limit

Optical micrographs of the extruded specimens and specimens annealed at 773 K and forged at 573 K to a reduction in height of 5, 10, or 30% are shown in Fig. 5; Fig. 6 shows the corresponding inverse pole figure (IPF) and pole figure maps for the as-extruded and annealed specimens. As can be seen in Fig. 5, the LPSO phase became bent on increasing the reduction in height of the extruded specimen, and that of the specimen forged with height reduction of 5% was bent to a similar extent to the LPSO phase of the as-extruded specimen, as reported previously. However, forging of 10% or more resulted in the formation of a continuously bent structure in the LPSO phase, and no gaps were visible at the phase boundary between the α -Mg and the LPSO phases. Furthermore, the grain size of the α -Mg phase of the extruded specimen was over 10 μm . The grain size grew to 5 μm in the specimen forged with a height reduction of 5%, and in the specimen forged with height reduction of 30% the grains aggregated to form 20 μm grains in the α -Mg phase. The strength of the aggregate increased correspondingly from 7.9 to 48.7. On the other hand, as shown in Fig. 5, the LPSO phase was plate-shaped in the specimen annealed at 773 K, indicating that the LPSO phase was bent less than that in extruded specimens with forging to the same height reduction. On the other hand, twinning was observed in the α -Mg phase, and the grain size in the α -Mg phase decreased slightly from 10.8 μm to 8.8 μm (Fig. 6). The twins formed in forging with a height reduction of 5% were not observed after forging with a height reduction of 30%; therefore, twins that are formed during the early stages of deformation in the α -Mg

phase are considered to decrease the crystal grain size with increased deformation. The strength of the aggregate increased from 5 to 7.3.

We also investigated kink bands in the LPSO phase in extruded specimen forged to a reduction in height of 30% at 573 K, because the grains in this specimen were large and aggregated; the relevant low- and high-magnification TEM micrographs are shown in Fig. 7. We identified steps of continuous kink bands formed in the LPSO phase and a large tilt between adjacent grains to an angle not found in previous reports [8–9, 11]. This implies that refinement of the LPSO phase is driven by an increase in the tilt angle of the kink bands because of continuous formation of kink bands. Kink bands in the LPSO phase observed after extrusion generally have a bending angle of 5° to 10° against the adjacent LPSO phase [8-9]. However, the high-magnification image in Fig. 7 shows the presence of a bending angle of the kink bands against the adjacent LPSO phase of about 100° ; it also shows that the kink bands were continuously deformed. These results imply that the LPSO phase, which is known to be a hard phase [19], has a large deformability.

3.4 Forging ratio that forms kink bands in the LPSO phase

In Mg–Zn–Y alloys, kink bands are known to form in the LPSO phase and the proof stress is known to increase through refinement of the α -Mg phase during plastic deformation [9]. However, there has been no report on the deformation threshold where kink bands start to form in the LPSO phase to increase the strength of the alloy. We therefore investigated the relationship between a reduction in the height of the forging limit and the increase in strength by means of hardness tests for various reductions in the height. Kink bands in the LPSO phase are known to form through compressive deformation; therefore, the simple compression deformation is the minimum threshold for deformation.

On the basis of the micrographs shown in Fig. 2, the as-extruded specimen and a specimen annealed at 773 K were subjected to a height reduction of 30% at 473 K or 673 K. The results of the hardness tests after compression are shown in Fig. 8. Microstructural (Fig. 2) and crystal-orientation analyses showed that the α -Mg phase in the extruded specimen consists of micrograins and uncrystallized grains; the strength of the corresponding aggregate had a high value of 11. We also showed (Fig. 4) that the as-extruded specimen cannot be forged by height reduction of 20% at a forging temperature of 473 K. Forging with height reduction of 30% is, however, possible at forging temperatures of 673 K and 773 K, Kink bands were present in the LPSO phase after forging with a height reduction of 30% (Figs. 5 and 6). Therefore, changes in the LPSO and α -Mg phases are thought to occur when the reduction in height is 5 or 10%. We found, therefore, that forging with height reduction of 10% is necessary for the formation of kink bands in the LPSO phase.

3.5 Effects of kinking and kink bands in the LPSO phase on the formability of the alloy

We also investigated the effects of the formation of kink bands and structural changes involving kink deformation, kink band formation, and kink boundary formation in the LPSO phase on improvements in formability. The specimen that we used to investigate changes in kink deformation in the LPSO phase were extruded specimen annealed at 773 K for 3.6 ks and forged at 573 K to a reduction in height of 5, 10, or 30%. TEM micrographs of these specimens are shown in Fig. 9.

Fig. 5 shows that the bending angle of the LPSO phase increased with increasing reduction in height by forging. On the other hand, the IPF maps and pole figures in Fig. 6(b) show that the grain size of the α -Mg phases was refined by about 2 μm , but no marked randomization of texture occurred as a result of dynamic recrystallization [3,6]. We therefore believe that the formability improved as a result of continuous kink deformation in the LPSO phase. The

LPSO phase appeared as a flat and slow curve at a reduction in height of 5% on forging at 573 K. The interface of the kink bands was distorted and wide [Fig. 9 (a)] and did not form a sharp kink boundary, [Fig. 7]. Kink bands in the LPSO phase were not converted into definite boundaries, even when the reduction in height by forging was increased to 10%. However, it is clear from Fig. 9(b) that when an external force acts, the LPSO phases show sharper and larger curves than those produced by the introduction of kink deformation by piling up of harder LPSO phase [19]. With a reduction in height of 5%, the inclination showed a greater increase than did the diffraction pattern, obtained from the interface of the kink bands. When the reduction in height by forging reached 30%, the diffraction pattern, [Fig. 9(c)] showed a larger difference in inclination from the interface of the kink bands, and a clear boundary of unevenness was formed by the LPSO phases. It is well known that, LPSO phases are harder than α -Mg phases and that major sliding is limited to the (0001) plane and the $\langle 11\bar{2}0 \rangle$ direction [20]. In other words, it is believed that microvoids are present at the interface between the LPSO phase and the α -Mg phases whether it breaks promptly that LPSO phase does not do deformation [19]. However, it is thought that formability improved as a result of refinement of the LPSO phase, because the inclination increased continuously as kink deformation was introduced into the LPSO phase. Shao et al. [19] and Hagihara et al. [20] reported that TEM studies on cast $\text{Mg}_{97}\text{Zn}_1\text{Y}_2$ and $\text{Mg}_{88}\text{Zn}_5\text{Y}_7$ crystals subjected to directional solidification (DS) showed formation of kink deformations, kink bands, and kink boundaries after compression. In particular, Shao et al. reported that kink boundaries, sub-kink boundaries, and kink deformations are formed in the LPSO phase, which can achieve a high strength; they also clarified that dislocation is introduced during kinking when kink bands are formed by an LPSO phase [19]. In the present study, we were unable to clarify whether kink bands were formed or to elucidate the role of dislocation in the kink deformation mechanism. However, the TEM micrographs shown in Fig. 9, closely resemble

those reported by Shao et al. [19], suggesting that the inclination increased with increasing reduction in height by forging. Previous reports have shown that a high strength is produced when kink bands and kink deformations are introduced into an LPSO phase [8–12]. In the present study, it was clear that the LPSO phase displayed good formability.

4. Conclusion

Forging limit of extruded $\text{Mg}_{96}\text{Zn}_2\text{Y}_2$ alloy showed a reduction in height of 65% at forging temperatures of 573–673 K and an initial strain rate of $3.5 \times 10^{-1} \text{ s}^{-1}$ in the upsettability test. The formability of LPSO-type Mg alloys can be improved through forging to produce an α -Mg phase with small grains and an LPSO phase with kink bands with a large-angle tilt. The hard LPSO phase has a bending angle of about 100° at the grain boundaries and has a large deformability. Upsetting with a reduction in height of about 5% is necessary for the formation of kink bands in the LPSO phase, and these kink bands deform continuously with increasing deformation.

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Figure Captions

Fig. 1 SEM micrographs of $\text{Mg}_{96}\text{Zn}_2\text{Y}_2$ alloys: (a) as-cast and (b) extruded alloy.

Fig. 2 Optical micrographs of extruded and annealed $\text{Mg}_{96}\text{Zn}_2\text{Y}_2$ alloys. As-extruded alloy (a) and alloy specimens annealed at 573 K (b), 673 K (c), or 773 K (d) for 3.6 ks

Fig. 3 Forging limit and flow stress curves of as-extruded $\text{Mg}_{96}\text{Zn}_2\text{Y}_2$ alloys at room and elevated temperatures, obtained by the upsettability test.

Fig. 4 Forging limit and flow stress curves at room and elevated temperatures of extruded $\text{Mg}_{96}\text{Zn}_2\text{Y}_2$ alloys heat-treated at 773 K for 3.6 ks, obtained by the upsettability test.

Fig. 5 Optical micrographs of extruded (a–c) and annealed (d–f) $\text{Mg}_{96}\text{Zn}_2\text{Y}_2$ alloys forged to reductions in height of (a) and (d) 5%, (b) and (e) 10%, or (c) and (f) 30%. The direction of forging is shown in the figure.

Fig. 6 (a) Inverse pole-figure and pole-figure maps of as-extruded specimens of $\text{Mg}_{96}\text{Zn}_2\text{Y}_2$ alloys with forging to a reduction in height of 5, 10, or 30%. Forging temperature 573 K; initial strain rate of $3.5 \times 10^{-1} \text{ s}^{-1}$.

Fig. 6 (b) Inverse pole-figure and pole-figure maps of extruded specimens of $\text{Mg}_{96}\text{Zn}_2\text{Y}_2$ alloy annealed at 773 K for 3.6 ks with forging to a reduction in height of 5, 10, or 30%. Forging temperature 573 K; initial strain rate of $3.5 \times 10^{-1} \text{ s}^{-1}$.

Fig. 7 TEM micrographs of 30% forged $\text{Mg}_{96}\text{Zn}_2\text{Y}_2$ alloy at 573 K and an initial strain rate of $3.5 \times 10^{-1} \text{ s}^{-1}$. Figure (b) is a high-magnification image.

Fig. 8 Vickers hardness of extruded $\text{Mg}_{96}\text{Zn}_2\text{Y}_2$ alloy samples, with or without heat treatment, measured in the central region after the upsettability test.

Fig. 9 TEM micrographs of kink bands and kink deformation in the LPSO phase in a specimen. The specimens were forged to a reduction in height of 5% (a), 10% (b), or 30% (c) at 573 K and initial strain rate of $3.5 \times 10^{-1} \text{ s}^{-1}$.

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Fig. 6 (b) Inverse pole-figure and pole-figure maps of extruded specimens of Mg96Zn2Y2 alloy annealed at 773 K for 3.6 ks with forging to a reduction in height of 5, 10, or 30%. Forging temperature 573 K; initial strain rate of $3.5 \times 10^{-1}\ \text{s}^{-1}$.

Fig. 7 TEM micrographs of 30%-forged $\text{Mg}_{96}\text{Zn}_2\text{Y}_2$ alloy at 573 K and an initial strain rate of $3.5 \times 10^{-1} \text{ s}^{-1}$. Figure (b) is a high-magnification image.

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