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TMS (The Minerals, Metals & Materials Society), 2012

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Processing-Microstructure-Property Relationships II

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Enhancement of Strength and Ductility of $Mg_{96}Zn_2Y_2$ Rolled Sheet by Controlling Structure and Plastic Deformation

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Abstract

Mg-Zn-Y alloys are well known to possess greatly enhanced strength during plastic deformation because of the presence of kink bands in the LPSO phase and refinement of the grains of the alpha Mg phase. On the other hand, Mg–rare earth (RE) and Mg–Zn–RE alloys with a long period stacking order (LPSO) phase show a high tensile yield strength when subjected to an extrusion process but it is not known whether the LPSO and alpha Mg phases develop during plastic deformation. We examined the effect of the finely dispersed LPSO phase and the alpha Mg phase on the development of high strength in sheets of $Mg_{96}Zn_2Y_2$ subjected to a few passes of rolling. The mechanical properties and thermal stability of the alloy were also investigated. The tensile yield strength of rolled sheets of $Mg_{96}Zn_2Y_2$ was 360 MPa and its elongation was 5% when the material was subjected to thermomechanically controlled processing at 673 K with a four-pass rolling schedule. However, the tensile yield strength decreased and the elongation increased at annealing temperature of 623 K or above, because of the presence of grain growth in the alpha Mg phase and the restoration of kink bands in the LPSO phase.

Introduction

Lightweight Mg alloys with excellent shock-absorption properties are being actively adopted for electronic information devices and automotive parts [1,2]. For use in such structural applications, Mg alloys need to have adequate ductility, thermal stability, and strength. However, Mg alloys often exhibit low ductility and low tensile yield strength at room temperature and above as a result of a scarcity of slip systems in their hexagonal close-packed structure [3]. Among the known effective ways for improving the ductility and tensile yield strength of Mg alloys are grain refinement [4] and control of the texture [5], because these promote prismatic slips and facilitate the creation of large plastic deformations [6].

Recently, Mg–Zn–Y alloy has been found to have superior mechanical properties to those of other Mg alloys [7]. Mg–Zn–Y alloy has a long period stacking order (LPSO) phase as a secondary phase in the main alpha Mg phase [7–9]. In general, Mg alloys with LPSO phases are known to have greatly enhanced mechanical properties, whereas their ductility can be maintained only by extrusion and/or plastic deformation of the cast metal. It has been suggested that kink deformations in the LPSO phase and microstructural refinement of the alpha Mg phase occur during extrusion deformation. The tensile yield strength, microstructure, fatigue properties, and thermal stability of extruded Mg–Zn–Y alloy have been reported [7–10]. On the other hand, many of the plastic deformation processes of LPSO-type Mg alloy are the result of the extrusion process, and there have been few attempts to examine the high strength developed by the rolling process and the working process. In addition, it is generally known that rolling of Mg alloys containing added rare earth (RE) element requires many working cycles in comparison with commercial Mg alloys [11–12].

In this present study, we examined the control of the microstructure of the LPSO phase and the alpha Mg phase of extruded $Mg_{96}Zn_2Y_2$ alloy by means of a short annealing treatment, and we studied the strength, elongation, and annealing properties of $Mg_{96}Zn_2Y_2$ material that had been subjected to the optimal working and rolling processes to produce rolled sheets of high strength and good ductility.

Experimental Procedures

In the present study, we used extruded $Mg_{96}Zn_2Y_2$ (atomic%) alloy in the form of the as-received material. The alloy was annealed at 673 K in an electric furnace for 3.6 ks to decrease the anisotropy resulting from increased elongation and the specimens were subsequently cooled in water. The alpha-Mg phase showed static recrystallization, grain growth. Kink bands in the LPSO phase were restored by the annealing treatment. Specimens 5 mm thick, 20 mm wide, and 50 mm long were machined from the annealed and extruded materials. The direction of rolling was perpendicular to the direction of extrusion. The rolling process was performed at a roll temperature of 493 K and a roll speed of 0.17 m s^{-1}. The specimen was subsequently heated at 673 K for 0.6 ks in an electric furnace and finally the rolled specimens were quenched in water.

Tensile specimens with a gauge section 2.5 mm in width, 1 mm in thickness, and 12 mm in length were machined from the rolled sheet and various annealed sheets in the direction parallel to the rolling direction. Tensile tests were carried out at an initial strain rate of 5×10^{-4} s^{-1}. Annealing treatment was carried out at various temperatures between 473 and 773 K in an electric furnace for 3.6 ks, and the specimens were subsequently cooled in water.

The microstructures of the as-rolled specimen and the annealed specimens were observed by optical microscopy (OM), scanning electron microscopy (SEM), transmission electron microscopy (TEM), and electron back-scattered diffraction (EBSD). EBSD was carried out in a 200 × 200 μm region with a measurement step of 0.3 μm. In our study, the cross section of the microstructure was observed from the rolling direction. Figure 1 shows SEM micrographs of an as-extruded sample of $Mg_{96}Zn_2Y_2$ alloy and a sample annealed at 673 K for 3.6 ks. The alloy contained an LPSO phase, an alpha Mg phase, and $Mg_3Zn_3Y_2$ compounds. The bright areas in both figures are believed to correspond the LPSO phase. The LPSO phase in the annealed materials appears to be more finely dispersed than that in the extruded alloy. LPSO phase does change from a plate type to a block type on heat treatment, and the plate-type LPSO phase was formed in the grains and grain boundaries.

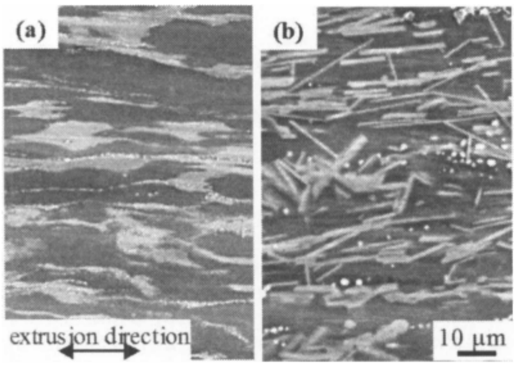

Figure 1. SEM micrographs of (a) the as-extruded $Mg_{96}Zn_2Y_2$ alloy and (b) Mg96Zn2Y2 alloy annealed at 773 K for 3.6 ks.

Results and Discussions

Anisotropy of the $Mg_{96}Zn_2Y_2$ rolled sheet

It is well known that plasticity in LPSO-type Mg–Zn–Y alloy can be generated by processing. The LPSO phase, which is the secondary phase, acts as a fiber reinforcement and is anisotropic. It has been reported [13] that in the as-extruded material, the 0.2% proof stresses in the direction of extrusion shows a difference of about 30% from that in the direction perpendicular to the extrusion direction. Figure 2 shows the results of our investigations of the anisotropy of the tensile properties of the alloy; here, the angle 0° is parallel to the rolling direction. The maximum value of the anisotropy of the 0.2% proof stresses in the rolled material was about 14%, which is less than half that of the extruded material.

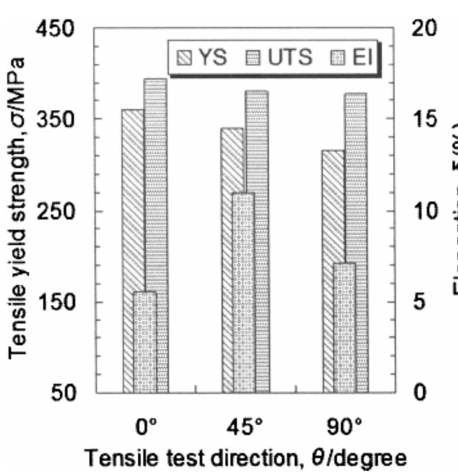

Figure 2. Relationship between mechanical properties and the tensile direction for a $Mg_{96}Zn_2Y_2$ rolled sheet. The tensile test direction of 0° was parallel to the direction of rolling. YS = yield strength; UTS = ultimate tensile strength; El =elongation.

The decrease in anisotropy may have resulted from dispersal of the LPSO phase in the grain boundary of the alpha Mg phase through the heat treatment and rolling processes. The dispersal of the LPSO phase by heat treatment, as shown in Fig. 1, is effective in reducing the anisotropy, and even low-deformation processing results in a high strength of over 360 MPa. The tensile strength was almost constant and was independent of the test direction. However, elongation showed a maximum at a tensile direction of 45°. An important factor in controlling the tensile yield strength is the fine dispersion of the LPSO phase before plastic deformation so that its anisotropy is low; this can be achieved by appropriate heat treatment and processing of the LPSO-type Mg alloy.

High strength and elongation of $Mg_{96}Zn_2Y_2$ rolled sheet

Nominal stress–strain curves of as-rolled and annealed $Mg_{96}Zn_2Y_2$ alloys are shown in Fig. 3. It is well known that for extruded materials, the 0.2% proof stress falls to 200 MPa whereas the elongation increases to 20% on annealing at 673 K for 3.6 ks. In our present study, a 1-mm-thick rolled sheet was produced by a four-pass rolling process. This four-pass schedule is the minimum number of passes for rolling LPSO-type Mg alloy, and the strain ratio introduced by the rolling processing is 1.8, which is less than that produced by the extrusion process. However, the 0.2% proof stress and elongation of $Mg_{96}Zn_2Y_2$ rolled sheet material were 360 MPa and 5.5% respectively, and these values did not show any marked decrease on annealing at 573 K (Fig. 3). In other words, the Mg alloy regains its high strength even if its LPSO phase is not subject to severe plastic deformation. However, the 0.2% proof stress of rolled sheet materials was lower than that of extruded alloys by about 40 MPa, because the grain size of the alpha Mg phase of the rolled sheet materials was 5 μm, which is 3.6 times larger than that of the extruded alloy.

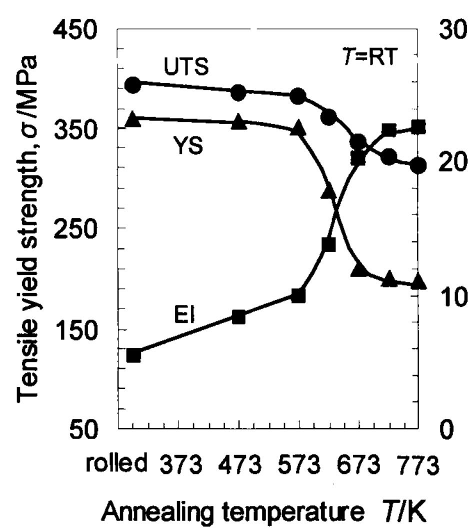

Figure 3. Relationship between the tensile yield strength and annealing temperature for a rolled sheet of $Mg_{96}Zn_2Y_2$ alloy. Tensile tests were performed at room temperature with the tensile direction parallel to the rolling direction.

When annealing was performed for 3.6 ks at 573 K, above which temperature the strength of the specimen decreased, the 0.2% proof stress was 351 MPa, the tensile strength was 381 MPa, and the elongation was 10%. It has been reported [6] that annealing of conventional Mg alloys at 523 K for 3.6 ks disrupts the crystalline orientation and causes a reduction in strength. On the other hand, the alloy that we investigated exhibited small reductions in the 0.2% proof stress and tensile strength of 10 and 12 MPa, respectively, at annealing temperature of up to 573 K, whereas the elongation was markedly improved from 5.5 to 10%. The ductility of the alloy can therefore be improved while maintaining a high strength, despite the fact that it is a Mg alloy that has a low ductility at room temperature.

Microstructure of $Mg_{96}Zn_2Y_2$ as rolled sheet

Optical micrograph and inverse pole figure (IPF) maps are show in Fig. 4 and in Fig. 4(b) indicated the orientation key of Mg alloy. In Figures 4(b) and (c), the darkest regions correspond to the LPSO phase. The LPSO phase has a large curvature in the rolling direction and shows considerable deformation. Furthermore, the formation of kink bands can be recognized in the LPSO phase. Normally, the LPSO phase does not show a large curvature in extruded materials, and we suggest that the large deformation of the LPSO phase observed in this case is responsible for the development of high strength in the rolling process. From Fig. 4(b), we estimated that the grain size of the alpha-Mg phase in the extruded material was 1.4 μm, and that in the rolled material was 5.0 μm. In addition, from IPF map and orientation key were shows the microstructure of rolled sheet were random and the proportion of recrystallized region of the alpha Mg phase was more than 70% in both the extruded and the rolled alloy. These materials therefore consisted of an alpha Mg phase (containing recrystallized and nonrecrystallized regions), an LPSO phase (25% by volume), and $Mg_3Zn_3Y_2$ compounds.

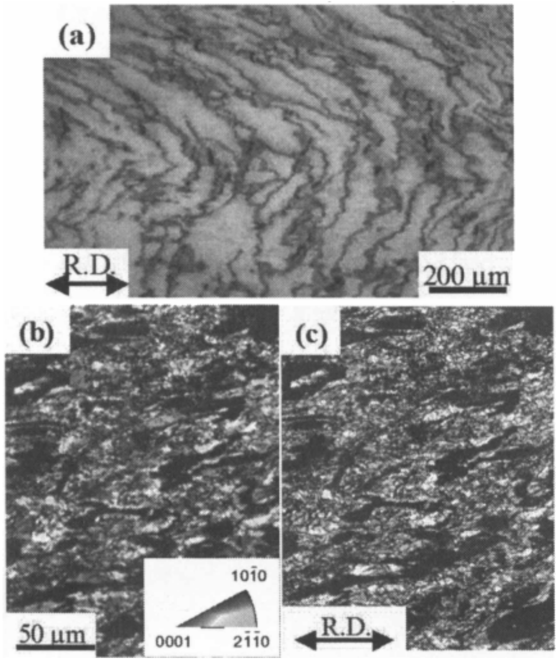

Figure 4. Optical micrograph, IPF maps, and grain-boundary maps determined by EBSD for samples of rolled $Mg_{96}Zn_2Y_2$ alloy. The black region in (b) and (c) correspond to the LPSO phase.

In other words, the introduction of deformation of the LPSO phase is important in producing a high-strength rolled sheet by the rolling process. From Fig. 4(c), the frequencies of low-angle and high angle-grain boundaries in the $Mg_{96}Zn_2Y_2$ rolled sheet were determined to be 19.3% and 80.7%, respectively. The rolling process was able to developed high frequency of high angle grain boundary and a random crystal orientation in $Mg_{96}Zn_2Y_2$ alloy.

TEM micrographs of the LPSO phases in an as-rolled sheet of $Mg_{96}Zn_2Y_2$ alloy are shown in Fig. 5. The LPSO phase contained both an LPSO structure and a 2H–Mg structure. In the rolled sheet, the LPSO phase was continuously deformed, but boundaries were observed in some regions, suggested that the LPSO phase recovered, at least partially, from the kink deformation introduced during rolling process. Although it is difficult to compare results of a previous study [14] directly with those of the present study, for FCC metals, cellular microstructures at the boundary between the deformation band and the deformation matrix acts as recovery nuclei during subsequent reheating. We assume that the kinks acted as recovery nuclei in the Mg–Zn–Y alloy and led to the formation of a static structure when edge dislocations accumulated as a result of reheating of the kink deformation that formed within the LPSO phase. The continuous deformation of kink bands introduced in the LPSO phase explains why the LPSO phase, which is a hard phase, showed large deformations in the rolling process. It is clear from Fig. 5 that the LPSO phase with continuous kink deformations was formed after the rolling process and showed a large curvature. The presence of microvoids at the interface between the LPSO phase and the alpha Mg phase was not confirmed. The present study clarified for the first time that continuous deformation of kink bands is necessary to permit the LPSO phase to show large deformations and that recovery of kink bands during reheating is important.

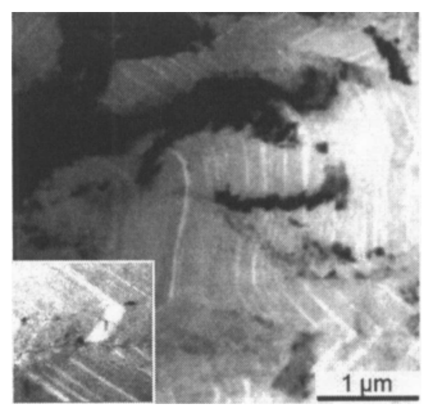

Figure 5. TEM micrographs of the as-rolled sheet of $Mg_{96}Zn_2Y_2$ alloy. The inset figure shows a high-magnification image of a kink deformation.

Grain growth in annealed $Mg_{96}Zn_2Y_2$ materials

Optical micrographs of samples of rolled alloy annealed at various temperatures are shown in Fig. 6. In Figs. 6(a)–6(c), the darker regions correspond to the LPSO phase and the brightest regions are alpha Mg phases. The grains of alpha Mg phase grew as the annealing temperature was increased, but up to an annealing temperature of 623 K, both the LPSO and alpha Mg phase showed little difference compared with those before annealing [Fig. 6(a)]. The alpha Mg phase grains became coarser as the annealing temperature was increased, but this occurred very slowly because the grain growth was controlled by the LPSO phase. The structure of the LPSO phase of extruded $Mg_{96}Zn_2Y_2$ alloy changes from a block type to a plate type as the annealing temperature is increased [10], whereas the structure of the LPSO phase in rolled $Mg_{96}Zn_2Y_2$ alloy did not change, suggesting that structure of the LPSO in rolled $Mg_{96}Zn_2Y_2$ alloy is thermally stable. Figure 6 also shows how the slight changes in the mechanical properties and microstructure of the rolled $Mg_{96}Zn_2Y_2$ alloy on annealing at 573 K might have occurred. In the tensile tests (Fig. 3), annealing at 623 K caused marked decreases in the 0.2% proof stress and tensile strength of about 50 MPa and 30 MPa, respectively. From Fig. 3 and Fig. 6, it is apparent that the static recrystallization temperature of the alpha Mg phase of this alloy is 623 K or higher. When the alloy was annealed at above 673 K, its 0.2% proof stress and tensile strength fell to about 200 MPa and 330 MPa, respectively, whereas the elongation was improved to more than 20%. Su et al. [15] reported that AZ31B Mg alloy, processed by equal channel angular pressing and annealed at 773 K for 3.6 ks, showed a 0.2% proof stress of 50 MPa, a mean alpha Mg grain size of as much as 20 μm, and an improved elongation of 15%. The alloy investigated in our study showed even better mechanical properties.

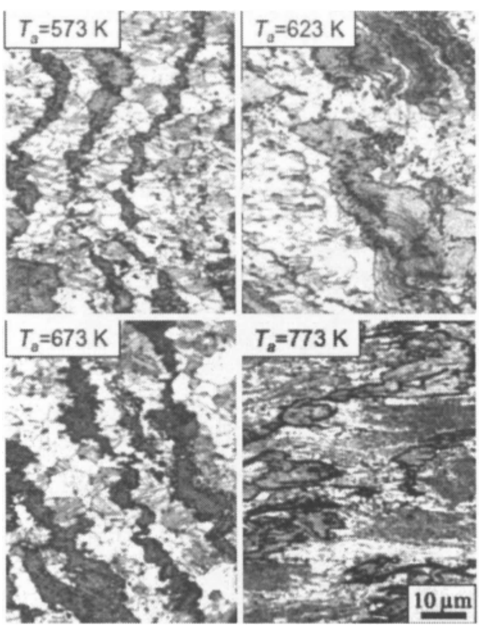

Figure 6. Optical micrographs of samples of rolled $Mg_{96}Zn_2Y_2$ alloys annealed for 3.6 ks at (a) 573 K, (b) 623 K, (c) 673 K, and (d) 773 K.

Conclusion

We investigated the effects of annealing at various temperatures for 3.6 ks on the mechanical properties and microstructure of rolled samples of Mg96Zn2Y2 alloy. The anisotropy of the rolled sheet alloy was low compared with that of the extruded alloy when rolling was performed at 673 K after annealing treatment. The important factors in the development of Mg96Zn2Y2 alloy with a high strength and a good elongation are as follows: 1. Fine dispersion of the LPSO phase in the grain boundary of the alpha Mg phase; 2. Continuous kink deformation in the LPSO phase; and 3. Large curvature in the LPSO phase after processing by rolling. The presence of microvoids at the interface between the LPSO phase and the alpha Mg phase could not be confirmed. The high tensile yield strength was retained at an annealing temperature of 573 K, with no change in the LPSO phase and slow growth in the grain size of alpha Mg phase, as well as an increasingly randomized texture.

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References

[1] Kinner. W.K., Mater. Eng., **90** (1979), 49-51.

[2] H. Friedrich, S. Schumann, J Mater. Process. Technol. **117** (2001), 276-281.

[3] G.S. Rao, Y.V.R.K. Prasad, Metall. Trans., **13A** (1982) 2219-2226.

[4] S. Mueller, K. Mueller, H. Tao, W. Reimers, Int. J. Mater. Res. **97** (2006) 1384-1391.

[5] Agnew S.R, Horton J.A, Lillo T.M, Brown D.W, Scr. Mater., **50** (2004), 377-381.

[6] N. Stanford, M. Barnett, Mater. Sci. Eng., A **496** (2008) 399-408.

[7] Y. Kawamura, K. Hayashi, A. Inoue, T. Masumoto, Mater. Trans., **42** (2001) 1172-1176.

[8] S. Yoshimoto, M. Yamasaki, Y. Kawamura, Mater. Trans., **47** (2006) 959-965.

[9] X. Zeng, Y. Zhang, C. Lu, W. Ding, Y. Wang, Y. Zhu, J. Alloy. Compd., **395** (2005) 213-219.

[10] M. Noda, T. Mayama, Y. Kawamura, Mater. Trans., **50** (2009) 2526-2531.

[11] H.K. Lim, S.W. Sohn, D.H. Kim, J.Y. Lee, W.T. Kim, D.H. Kim, J. Alloy. Compd., **454** (2008) 515-522.

[12] B. Wang, Y. Liu, A. Su, Z. Cao, Mater. Trans., **50** (2009) 261-265.

[13] T. Mayama, M. Noda, R. Chiba, M. Kuroda, Int. J. Plasticity, doi: 10.1016/j.ijplas.2011.02.007.

[14] K. Higashida, J. Takamura, N. Narita, Mater. Sci. Eng., **81** (1986) 239-258.

[15] C.W. Su, L. Lu, M.O. Lai, Mater. Sci. Technol., **23** (2007) 290-296.