Effect of annealing on the microstructure and mechanical properties of Mg−**2.5Zn**−**0.5Y alloy**

*Li Zhang*1,2), *Zheng Liu*1), *and Ping-li Mao*1)

1) School of Materials Science and Engineering, Shenyang University of Technology, Shenyang 110870, China 2) School of Materials Science and Engineering, Shenyang Aerospace University, Shenyang 110136, China (Received: 5 January 2014; revised: 14 March 2014; accepted: 19 March 2014)

Abstract: The microstructure and mechanical properties of extruded Mg−2.5Zn−0.5Y alloy before and after annealing treatments were investigated. The as-extruded alloy exhibits a yield tensile strength (YTS) of 305.9 MPa and an ultimate tensile strength (UTS) of 354.8 MPa, whereas the elongation is only 4%. After annealing, the YTS and UTS decrease to 150 MPa and 240 MPa, respectively, and the elongation increases to 28%. Interestingly, the annealed alloy maintains an acceptable stress level even after a much higher ductility is achieved. These excellent mechanical properties stem from the combined effects of fine α-Mg dynamic recrystallization (DRX) grains and a homogeneously distributed icosahedral quasicrystalline phase (I-phase) in the α-Mg DRX grains. In particular, the superior ductility originates from the coherent interface of I-phase and α -Mg and from the formation of the secondary twin $\{10\overline{1}\}$ - $\{10\overline{1}\}$ $\{2\}\{38^{\circ} < 1\overline{2}\}$ $\{10>$) in the tension twin ${1012}.$

Keywords: magnesium alloys; extrusion; annealing; microstructure; mechanical properties; twinning

1. Introduction

Magnesium alloys have attracted a great deal of attention, especially in the aircraft and modern automobile fields, because of their high strength-to-density ratio, good machinability, and good damping behavior [1]. However, the moderate strength and limited ductility of Mg at room temperature, which are consequences of its hexagonal close-packed (HCP) structure [2], hinders its broader application. The addition of elemental Zn can effectively refine the grain size and improve the strength of Mg alloys [3]. Alloying with rare earth (RE) elements is another effective method to improve the strength and ductility of Mg because RE elements can form some intermetallic phases with Mg and refine the grain size through changing the crystallization surroundings [4−6]. The co-addition of Zn and Er or Y, in particular, results in the formation of quasicrystals with an unusual quasiperiodic lattice structure and unique properties in Mg alloys [2,7−8]. In such cases, more attention has been devoted to the icosahedral quasicrystalline phase (I-phase) reinforced Mg-based alloys [9−13].

The mechanical properties of the I-phase-containing Mg alloys have been reported to depend primarily on the volume fraction of the I-phase and their degree of dispersion [9]. However, most reported Mg alloys with excellent mechanical properties were fabricated through special approaches. For example, the $MgZn_{3,3}Y_{0,43}$ alloy fabricated by powder metallurgy and hot extrusion processes exhibits a yield stress of 410 MPa with an elongation of 12% [14]. Recent research has shown that a yield strength of 381.45 MPa and an ultimate tensile strength of 438.33 MPa were obtained in $Mg_{96}Y_3Zn_1$ alloy processed by extrusion and equal-channel angular pressing (ECAP) [15].

Hence, the development of a conventional method for improving the mechanical properties of these alloys efficiently is of interest. In this study, the Mg−2.5Zn−0.5Y alloy fabricated by conventional casting and hot extrusion exhibits excellent mechanical properties. The higher strength and excellent ductility are ascribed to the refined dynamic recrystallization (DRX) grains and fine intermediate phases obtained by hot-extrusion and annealing treatments. More-

 \bigcirc Springer

Corresponding author: Zheng Liu E-mail: zliu4321@vip.sina.com.cn

[©] University of Science and Technology Beijing and Springer-Verlag Berlin Heidelberg 2014

over, the secondary twin $\{10\overline{1}1\} - \{10\overline{1}2\}(38^{\circ} < 1\overline{2}10$ that developed during the tensile process plays an important role in improving the plasticity of the alloy.

2. Experimental

The Mg−2.5Zn−0.5Y (wt%) alloy was prepared from pure Mg, pure Zn, and Mg−Y master alloys using a graphite crucible in an electric resistance furnace under the protection of a cover gas. All of the raw materials were cleaned and dried before being placed into the graphite crucible. The melt was maintained at 700°C for 10 min and then cast into a 200°C graphite mold with the protection of an anti-oxidizing flux and was subsequently allowed to cool under open atmosphere.

Portions of the cast ingots were extruded at 380°C with an extrusion ratio of 12.75:1 and at an extrusion rate of 1 mm/s. The specimens for microstructural observation and tensile tests were cut parallel to the extrusion direction (ED). The microstructures were observed by optical microscopy (OM) using an Olympus GX71 optical microscope. Phase identification was performed by X-ray diffraction (XRD) on an instrument equipped with a Cu K_{α} radiation source. The mechanical properties were tested using a MTS Landmark electro-hydraulic servo testing system. The tensile specimens with a gauge length of 15 mm and a gauge diameter of 4 mm were machined from the extruded bar to make the tensile axis parallel to the ED. The fracture surfaces were observed by scanning electron microscopy (SEM) using an electron microscope operated in secondary-electron mode. The I-phase/ α -Mg interfaces were observed by transmission electron microscopy (TEM) on a JEM-2000EX transmission electron microscope. The micro-texture was analyzed using electron backscatter diffraction (EBSD) to investigate the effect of annealing treatments on the microstructural evolution of the alloy.

3. Results and discussion

3.1. Microstructure

The microstructures of the as-cast Mg−2.5Zn−0.5Y alloys are shown in Fig. 1. The granular intermediate phases (the dark points) are distributed homogenously in α-Mg (the white area), as shown in Fig. 1(a). The morphology of the intermediate phases differs from that previously reported [9,12], possibly because of the different quantities of Zn and Y or the different cooling rate used in the present work. The intermediate phases with granular morphology improve the properties of this alloy. Zhang *et al.* [16] observed that excellent mechanical performance could be obtained with a Mg−Zn−Y alloy prepared by precipitating spherical intermediate phases via the addition of Mn. The morphology of the intermediate phases is evident in the SEM image in Fig. 1(b). Insert A shows the detailed morphology of a typical intermediate phase. The average size of the intermediate phases is approximately 10 µm.

Fig. 1. Microstructures of as-cast Mg-2.5Zn-0.5Y alloy: (a) optical microstructure and (b) SEM image (insert A is a high-magnification image of one intermediate phase).

The microstructures of the extruded and annealed specimens are shown in Fig. 2. The as-cast α -Mg dendritic structure develops large aggregates distributed homogeneously along the extruded direction after hot extrusion. Extensive mechanical (deformed) twins are observed in the banding α-Mg grains. The large α-Mg grains are caused by partly DRX, and the twins are related to the severe plastic deformation during hot extrusion. The intermediate phases also become finer and still distribute homogenously in α -Mg (see the black points in Fig. 2(a)). These deformed twins and the fine intermediate phases may contribute to the mechanical properties of the Mg−2.5Zn−0.5Y alloy.

As shown in Figs. 2(b)–2(d), the α -Mg grains are evidently refined after the annealing treatment at 425°C. The average size of the α-Mg grains is approximately 40 μ m after the annealing treatment at 425°C for 10 min. 20% of the α-Mg grains exist as twins, which indicates that this annealing temperature is not sufficient to release the deformed twins within 10 min. Deformed twins disappear when the annealing time is increased to 15 min, and the α -Mg grains are more even. However, when the annealing time is increased to 20 min, some annealing twins appear and the α-Mg grains tend to grow. The tensile test results show that the specimen annealed at 425°C for 15 min exhibits good performance. An annealing temperature of 425°C and an annealing time of 15 min are the optimal annealing conditions for Mg−2.5Zn−0.5Y alloy to obtain even grains and good mechanical properties.

Fig. 2. Microstructures of as-extruded and annealed Mg−**2.5Zn**−**0.5Y alloy: (a) as-extruded, (b) annealed at 425°C for 10 min, (c) annealed at 425**°**C for 15 min, and (d) annealed at 425°C for 20 min.**

3.2. Mechanical properties

Fig. 3 shows the tensile strain−stress curves of the as-extruded and annealed Mg−2.5Zn−0.5Y alloy. The ultimate tensile strength (UTS) and yield tensile strength (YTS) of the as-extruded alloy reach 354.8 MPa and 305.9 MPa, respectively, whereas the elongation is only 4%. The UTS and YTS of the annealed alloy are approximately 240 MPa and 150 MPa, respectively, whereas the elongation can reach 28%. Interestingly, the annealed alloy maintains an acceptable stress level even after achieving a much higher ductility. These excellent mechanical properties stem from the combined effects of fine α-Mg DRX grains and a homogeneously distributed I-phase in the α -Mg DRX grains, as shown in Fig. 4. The I-phase exhibits good thermal stability during the annealing process. Thus, after the annealing treatment, the homogenous distribution of the I-phase in the α-Mg DRX grains would still provide a substantial strengthening effect for the Mg alloys. However, the remarkably enhanced ductility after the annealing process suggests that the bonding nature of the interface between the I-phase and the α-Mg matrix is strong. Bae *et al.* [8] confirmed this viewpoint by establishing the orientation relationship of atomic-scale bonding between the I-phase and the hexagonal Mg matrix using high-resolution TEM. The SEM micrographs of the fracture (see Fig. 5) reveal more uniform dimples in the fractured surfaces of the annealed alloys (Figs. 5(b)−5(d)) than in the fractured surfaces of the extruded alloy (Fig. 5(a)). This improved uniformity suggests that the I-phase might contribute to the enhancement of ductility because of its coherent characteristic with the α-Mg matrix.

Fig. 3. Tensile strain–stress curves of as-extruded and annealed Mg−**2.5Zn**−**0.5Y alloy.**

Fig. 4. TEM micrograph of the interface between the I-phase and α-Mg (the inset shows the SAED pattern of the I-phase).

*782 Int. J. Miner. Metall. Mater***.,** *Vol. 21***,** *No. 8***,** *Aug. 2014*

In addition, the formation of the secondary twin ${101 \over 1}$ $-{101 \over 2}$ $(38^{\circ} < 1\overline{2}$ 10>) significantly improved the plasticity of the alloy. As shown in Fig. 6(a), some twins are distributed in the grain, others are across the grain, and some twins form in some of the other twins. The tension twin $\{10\bar{1}2\}(86^{\circ}\leq 1\bar{2}10^{\circ})$ indicated by the blue line and the contraction twin $\{10\bar{1}1\}(56^{\circ} < 12\bar{2}10 >)$ indicated by the yellow line were identified using the EBSD technique, as shown in Fig. $6(b)$. As shown in Fig. $6(c)$, another twin exists according to the disorientation angle map: the secondary twin ${1011} - {1012} (38^o < 1210^o)$ formed in the tension twin $\{10\bar{1}2\}$ during the tensile test, which may significantly improve the plasticity of the alloy. In addition, the low-angle grain boundary (see the peak in the misorientation angle at < 10° in Fig. $6(c)$) indicates the dislocation glide during the tensile test. Furthermore, the fact that the tension twins and the contraction twins formed interactively, coordinately inducing the formation, also effectively improves the elongation.

4. Conclusions

(1) The as-extruded Mg−2.5Zn−0.5Y alloy exhibits excellent strength and poor plasticity. The ultimate UTS and YTS of the alloy can reach 354.8 MPa and 305.9 MPa, respectively, whereas the elongation is only 4%. The UTS and YTS decrease to 240 MPa and 150 MPa, respectively, and

Fig. 5. SEM fractographs of tensile specimens: (a) as-extruded, (b) annealed at 425°**C for 10 min, (c) annealed at 425°C for 15 min, and (d) annealed at 425°C for 20 min.**

L. Zhang et al., **Effect of annealing on the microstructure and mechanical properties of Mg**−**2.5Zn**−**0.5Y alloy** *783*

the elongation increases to 28% after the annealing treatment.

(2) The excellent mechanical properties of the annealed Mg–2.5Zn–0.5Y alloy is ascribed to fine α -Mg grains, a fine quasicrystalline I-phase distributed homogeneously in the α-Mg matrix, the coherence of the I-phase and $α$ -Mg, as well the formation of the secondary twin ${1011}$ – ${1012}$ $(38^{\circ} < 12^{\circ}10$) in the tension twin $\{1012\}$ during the tensile test.

Acknowledgements

This work was financially supported by the Program for Science and Technology Development of Liaoning Province, China (No. 2013201018) and the Program for Liaoning Innovative Research Team in Universities. The author Li Zhang thanks Ph. D. candidate G.S. Duan (Northeastern University) for his assistance with EBSD tests.

References

- [1] X.Y. Lou, M. Li, R.K. Boger, S.R. Agnew, and R.H. Wagoner, Hardening evolution of AZ31B Mg sheet, *Int. J. Plast.*, 23(2007), No. 1, p. 44.
- [2] J. Bohlen, S.B. Yi, D. Letzig, and K.U. Kainer, Effect of rare earth elements on the microstructure and texture development

Fig. 6. EBSD results of specimens: (a) grain mapping and inverse pole figure (IPF) image, (b) distribution of twins, and (c) misorientation angle distribution.

in magnesium–manganese alloys during extrusion, *Mater. Sci. Eng. A*, 527(2010), No. 26, p. 7092.

- [3] C.J. Boehlert and K. Knittel, The microstructure, tensile properties, and creep behavior of Mg-Zn alloys containing 0−4.4 wt.% Zn, *Mater. Sci. Eng. A*, 417(2006), No. 1-2, p. 315.
- [4] X.F. Guo, S. Remennik, C.J. Xu, and D. Shechtman, Development of Mg−6.0%Zn−1.0%Y−0.6%Ce−0.6%Zr magnesium alloy and its microstructural evolution during processing, *Mater. Sci. Eng. A*, 473(2008), No. 1-2, p. 266.
- [5] N. Stanford, Micro-alloying Mg with Y, Ce, Gd and La for texture modification: a comparative study, *Mater. Sci. Eng. A*, 527(2010), No. 10-11, p. 2669.
- [6] N. Stanford and M. Barnett, Effect of composition on the texture and deformation behaviour of wrought Mg alloys, *Scripta Mater.*, 58(2008), No. 3, p. 179.
- [7] H. Li, W.B. Du, S.B. Li, and Z.H. Wang, Effect of Zn/Er weight ratio on phase formation and mechanical properties of as-cast Mg−Zn−Er alloys, *Mater. Des.*, 35(2012), p. 259.
- [8] D.H. Bae, S.H. Kim, D.H. Kim, and W.T. Kim, Deformation behavior of Mg−Zn−Y alloys reinforced by icosahedral quasicrystalline particles, *Acta Mater.*, 50(2002), No. 9, p. 2343.
- [9] J.Y. Lee, H.K. Lim, D.H. Kim, W.T. Kim, and D.H. Kim, Effect of volume fraction of quasicrystal on the mechanical properties of quasicrystal-reinforced Mg−Zn−Y alloys, *Mater. Sci. Eng. A*, 449-451(2007), p. 987.
- [10] A. Singh, H. Somekawa, and T. Mukai, High temperature processing of Mg−Zn−Y alloys containing quasicrystal phase

*784 Int. J. Miner. Metall. Mater***.,** *Vol. 21***,** *No. 8***,** *Aug. 2014*

for high strength, *Mater. Sci. Eng. A*, 528(2011), No. 21, p. 6647.

- [11] A. Singh, Y. Osawa, H. Somekawa, and T. Mukai, Ultra-fine grain size and isotropic very high strength by direct extrusion of chill-cast Mg−Zn−Y alloys containing quasicrystal phase, *Scripta Mater.*, 64(2011), No. 7, p. 661.
- [12] S.Q. Luo, A.T. Tang, F.S. Pan, K. Song, and W.Q. Wang, Effect of mole ratio of Y to Zn on phase constituent of Mg−Zn−Zr−Y alloys, *Trans. Nonferrous Met. Soc. China*, 21(2011), No. 4, p. 795.
- [13] M.X. Xia, S. Mitra, B. Dhindaw, G.J. Liu, and Z.Y. Fan, Melt conditioned, high-pressure die casting of Mg−Zn−Y alloy,

Metall. Mater. Trans. B, 41(2010), No. 1, p. 209.

- [14] E. Mora, G. Garcés, E. Oñorbe, P. Pérez, and P. Adeva, High-strength Mg−Zn−Y alloys produced by powder metallurgy, *Scripta Mater.*, 60(2009), No. 9, p. 776.
- [15] B. Chen, C. Lu, D.L. Lin, and X.Q. Zeng, Characterization of microstructure in high strength $Mg_{96}Y_3Zn_1$ alloy processed by extrusion and equal channel angular pressing, *J. Rare Earths*, 29(2011), No. 9, p. 902.
- [16] J.S. Zhang, H.W. Du, W. Liang, C.X. Xu, and B.F. Lu, Effect of Mn on the formation of Mg-based spherical icosahedral quasicrystal phase, *J. Alloys Compd.*, 427(2007), No. 1-2, p. 244.