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IMPROVEMENT OF STRENGTH AND DUCTILITY OF MG-ZN-CA-MN ALLOY BY EQUAL CHANNEL ANGULAR PRESSING

L.B. Tong [1, 2], M.Y. Zheng [1, *], S.W. Xu [2], P. Song [1], K. Wu [1], S. Kamado [2]
[1] School of Materials Science and Engineering, Harbin Institute of Technology, Harbin 150001, PR China
[2] Department of Mechanical Engineering, Nagaoka University of Technology, Nagaoka 940-2188, Japan

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Abstract

An ultrafine-grained (UFG) Mg–5.25 wt.% Zn–0.6 wt.% Ca–0.3 wt.% Mn alloy was produced by subjecting the as-extruded alloy to equal channel angular pressing (ECAP) for 4 passes at 250 and 300 °C, respectively. ECAP resulted in a remarkable grain refinement. After ECAP processing at 250°C, basal planes are oriented both parallel and inclined about 45° to the extrusion direction, while most the basal planes are oriented parallel to the extrusion direction after ECAP processing at 300 °C. Both yield strength and elongation were increased after ECAP processing. The yield strength was higher in the ECAPed alloy processed at 300 °C with larger grain size, indicating that the texture strengthening effect was dominant over the strengthening from grain refinement in the ECAPed alloy. The grain refinement may lead to dislocation slip on non-basal plane and grain boundary sliding, which improved the ductility of the ECAPed Mg–Zn–Ca–Mn alloy.

Introduction

Magnesium and its alloy are especially attractive for applications in aeronautics and automobile components due to their low density, high specific strength and good castability [1]. However, the poor ductility of Mg alloys at room temperature due to the limited number of slip systems available in the hcp crystal structure [2], restricts their widespread application. Grain refinement is an effective way to increase the strength and ductility of Mg alloys at ambient temperature [3]. During the last decade, equal channel angular pressing (ECAP) has emerged as a widely-known procedure for the fabrication of UFG metals and alloys through introducing intensive strain during processing [4].

In addition to the grain refinement, texture modification has a great influence on the mechanical properties in Mg alloys [5]. It has been reported that the yield stress of Mg alloys was decreased after ECAP processing in despite of grain refinement, exhibiting an inverse Hall-Petch relationship, because of the intensive development of texture after ECAP processing [6].

Alloying Mg with Ca increases the strength and corrosion resistance [7], while the presence of Zn in the binary Mg-Ca alloys enhances the precipitation hardening response [8], and Mn addition can improve the strength of Mg alloy effectively through grain refinement [9]. Recently, Mg–Zn–Ca alloys with high hardness and good creep resistance have been developed, due to the existence of fine stable precipitates in the alloys [10, 11], which are considered as $Ca_2Mg_6Zn_3$ [11, 12]. However, the investigation on the possibility of improving the mechanical properties in Mg-Zn-Ca-Mn alloy by using ECAP processing has not been investigated yet.

The purpose of the current research is to investigate the influence of the ECAP processing on the microstructure and mechanical properties of the as-extruded Mg–Zn–Ca–Mn alloy, with the main focus on the effect of grain size and texture on the mechanical properties.

Experimental Procedures

The alloy with a chemical composition of Mg - 5.25 wt.% Zn - 0.6 wt.% Ca - 0.3 wt.% Mn was prepared from pure Mg (99.99%), Zinc (99.98%), Ca (99.98%) and Mg–1.18 wt.% Mn master alloy, using electric resistance heating furnace in a SF_6 and CO_2 protective atmosphere. The melting alloy was held at 720 °C for 10 min and then cast into a steel mold.

The as-cast ingot was extruded at 300 °C with a reduction ratio of 10. The as-extruded alloy was machined into billets for ECAP with cross-sectional dimensions of 10 mm × 10 mm and a length of 70 mm. The ECAP die had characteristic angles $\Phi = 90°$ and $\psi = 37°$. The ECAP processing was conducted at 250 and 300 °C for 4 passes (corresponding to a total strain of $\varepsilon \approx 4.2$), using processing route Bc (the sample was rotated by 90° in the same direction between each pass) with constant displacement rate of 10 mm/s. The samples for microstructure observation and tensile tests were cut along y-plane (parallel to ECAP direction and normal direction). EBSD data were obtained from a JEOL FESEM JSM-7000F scanning electron microscopy equipped with TSL MSC-2200. The tensile tests were performed using Instron 5569 tensile machine with an initial strain rate of 1 mm/min at room temperature.

Results and discussion

Microstructure

Fig. 1 shows the microstructure of the as-extruded and as-ECAPed Mg–Zn–Ca–Mn alloy from EBSD analysis. As shown in Fig. 1a, in addition to some elongated grains, fine equiaxed grains with well-defined grain boundaries was observed, with average grain size of 4 μm, which indicated that dynamic recrystallization process occurred during hot extrusion, but it was not completed. After ECAP processing for 4 passes, the homogenous structure was observed, and grain size was decreased substantially due to the dynamic rescrystallization. The average grain size of the as-ECAPed alloys prepared at 250 and 300 °C was 1.0 and 1.2 μm, respectively, as shown in Fig.1 (b) and (c). Some low angle grain boundaries (LAGBs) were observed in the as-ECAPed alloy, which resulted from the dynamic recovery during the repeating ECAP process.

Texture

Fig. 2 shows the texture of the as-extruded and as-ECAPed alloy. A basal texture was observed in the as-extruded alloy, but some basal planes were inclined about 15° to extrusion direction, which

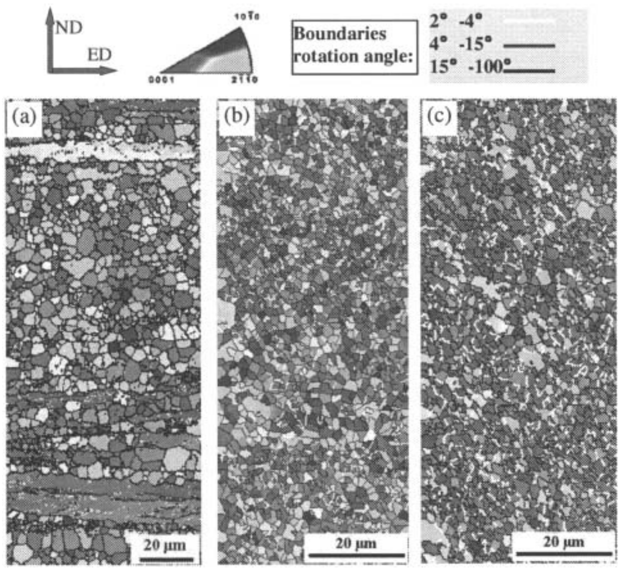

Fig. 1 The orientation imaging microscopy (OIM) maps of Mg-Zn-Ca alloy after (a) extrusion, (b) ECAP for 4 passes at 250 °C and (c) at 300 °C.

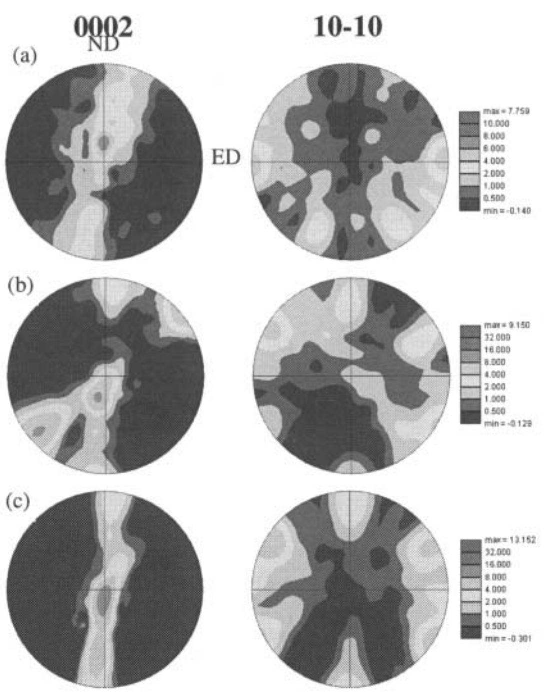

Fig. 2 Pole figures of the Mg-Zn-Ca alloy after (a) extrusion, (b) ECAP for 4 passes at 250 °C and (c) ECAP at 300 °C.

was derived from the recrystallization during the hot extrusion [13]. After ECAP for 4 passes at 250 °C, most of basal planes were inclined about 45° to extrusion direction, in addition, part of the basal planes are oriented parallel to the extrusion direction. While the ECAP was carried out at 300 °C, a distinct texture was observed with basal plane parallel to ED, which was similar with that in the as-extruded alloy. Similar texture evolution was observed in the extruded AZ31 Mg alloy after ECAP processing for 1 pass at 250 and 300 °C. The texture in the specimen processed at 250 °C, may be dominated by tensile twin and basal slip, while in the specimen processed at 300 °C, c+a pyramidal slip occurred at initial stage of deformation, the following crystal rotation dominated by basal slip and grain boundary sliding. The different textures formed during ECAE processing at the different processing temperature perhaps caused by the deformation mechanism at the initial stage of the deformation [14].

Tensile Properties

Nominal stress vs. nominal strain curves of Mg-Zn-Ca alloys in the tensile tests at room temperature were shown in Fig. 6. The ultimate tensile strength (UTS), tensile yield strength (TYS) and elongation of the as-extruded and as-ECAPed alloy were shown in Table 1. After ECAP for 4 passes at 300 °C, both the strength, and elongation were increased, while after ECAP for 4 passes at 250°C, yield strength was decreased and elongation to failure was increased.

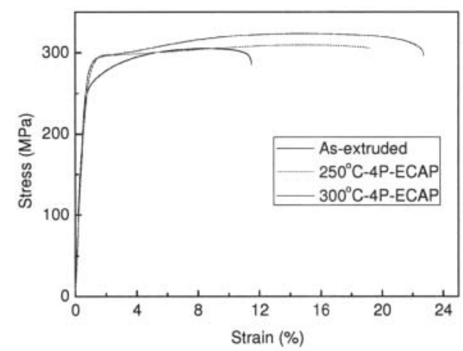

Fig. 3 Tensile stress vs. strain curves of the Mg-Zn-Ca alloy at room temperature after extrusion and ECAP processing.

Table 1 Tensile properties of the as-extruded and as-ECAPed Mg-Zn-Ca alloy.

	TYS (MPa)	UTS (MPa)	Elongation (%)
As-extruded	242	305	11.4
ECAP at 250 °C	228	309	19.1
ECAP at 300 °C	258	324	22.7

In general, the yield stress of polycrystalline materials varied with grain size, the relationship usually following the Hall-Petch equation [15]:

$$\sigma_y = \sigma_0 + kd^{-1/2} \tag{1}$$

where d was a measure of the grain diameter and σ_0 and k were experimentally derived constants. As shown in Fig.1, the alloy processed by ECAP at 300°C has larger grain size than that at 250 °C. However, the tensile strength was higher in the alloy processed by ECAP at 300°C. This indicated that such grain size dependency of yield strength did not exist in the present study. The variation of yield strength was considered to be derived from crystallographic texture modification during ECAP processing at different temperatures.

The Schmid factor for basal plane slip varied with the basal plane orientation, according to the definition of Schmid factor m:

$$m = \tau_c / c_s = \cos\lambda \cos\varphi \qquad (2)$$

where λ and φ were the angles between the stress axis and the slip direction and slip plane normal, respectively, τc was the critical resolved shear stress, σ_s was the applied stress. As shown in Fig. 4, the average Schmid factor of basal slip system was decreased to 0.136 in the as-extruded alloy processed at 300 °C.

The TYS of the Mg alloys with hexagonal close packed structure was thought to be dependent on both the grain size and the texture. Although the grain size of the Mg alloy was significantly reduced after ECAP at lower temperature, the TYS of the as-ECAPed alloy was lower than that of the as-extruded alloy. In the alloy processed by ECAP at 250°C, the basal plane was more favorably oriented for basal slip. This indicated that texture softening dominated over the strengthening from the grain refinement in the alloy processed by ECAP at 250°C, leading to a lower yield stress. While after ECAP for 4 passes at 300 °C, both Schmid factor and average grain size were decreased, therefore, both the strengthening effect from grain refinement and texture modification contribute to the increase of the TYS (258 MPa).

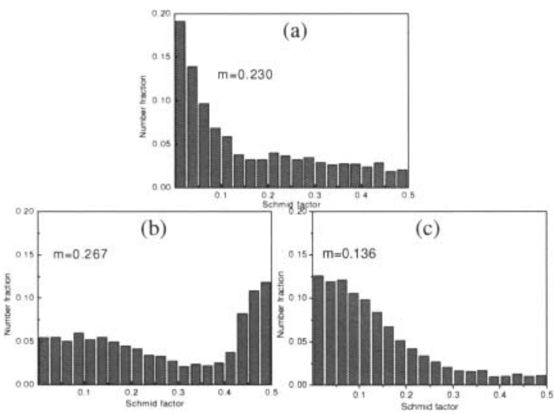

Fig. 4 Schmid factor for basal slip system of the Mg-Zn-Ca alloy after (a) extrusion, (b) ECAP at 250 °C and (c) ECAP at 300 °C.

Ductility

The increased elongation was obtained in the ECAPed Mg-Zn-Ca alloy. It was reported that non-basal slip was induced in fine grained Mg alloys by compatibility stress that operates to maintain continuity at grain boundary [16]. Thus, the activity of non-basal slip near grain boundaries would be helpful for the improvement of ductility of the present ECAPed Mg alloy.

Grain boundary sliding was observed to occur at room temperature in AZ31 alloy with average grain size of 8 μm, and the ratio of the strain by grain boundaries (GBs) to total strain is about 8% at room temperature [17]. The UFG structure in the current study might enhance the grain boundaries sliding at high strain, leading to the improved ductility.

The improved tensile elongation in the ECAPed Mg-Zn-Ca alloy may be resulted from non-basal slip and grain boundary sliding, in addition to basal slip of dislocations.

Conclusion

1. The obvious grain refinement of the as-extruded Mg-Zn-Ca alloy was obtained through ECAP processing at 250 and 300 °C, respectively.
2. After ECAP for 4 passes at 250 °C, most of basal planes were inclined about 45° to ED, but the basal planes in the as-ECAPed alloy processed at 300 °C were parallel to ED.
3. The yield strength was higher in the alloy processed by ECAP at high temperature than that that at low temperature, which indicated that the texture strengthening was dominant over the strengthening from grain refinement.
4. Dislocation slip on non-basal plane and grain boundary sliding may lead to the improved the elongation of the as-ECAPed alloy with fine grains.

Acknowledgements

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References

[1] S.E. Ion, F.J. Humphreys, S.H. White, Acta Metall. 30 (1982) 1909-1919
[2] K. Matsubara, Y. Miyahara, Z. Horita, T.G. Langdon, Acta Mater. 51 (2003) 3073-3084
[3] M.Y. Zheng, S.W. Xu, X.G. Qiao, K. Wu, S. Kamado, Y. Kojima, Mater. Sci. Eng. A 483-484 (2008) 564-567
[4] R.Z. Valiev, T.G. Langdon, Prog. Mater. Sci. 51 (2006) 881-981
[5] W.M. Gan, M.Y. Zheng, H. Chang, X.J. Wang, X.G. Qiao, K.Wu, B. Schwebke, H. G. Brokmeier, J. Alloys Compd. 470 (2009) 256.
[6] W.J. Kim, S.I. Hong, Y.S. Kim, S.H. Min, H.T. Jeong, J.D. Lee, Acta Mater. 51 (2003) 3293-3307.
[7] Erlin Zhang, Lei Yang, Mater. Sci. Eng. A 497 (2008) 111–118.
[8] J.F. Nie, B.C. Muddle, Scripta Mater. 37 (1997) 1475–1481.
[9] S. A. Khan, Y. Miyashita, Y. Mutoh, Z. B. Sajuri, Mater. Sci. Eng. A 420 (2006) 315–321.
[10] J.C. Oh, T. Ohkubo, T. Mukai, K. Hono, Scripta Mater. 53 (2005) 675-679
[11] J.F. Nie, B.C. Muddle, Scripta Mater. 37 (1997) 1475-1481
[12] G. Levi, S. Avraham, A. Zilberov, M. Bamberger, Acta Mater. 54 (2006) 523-530
[13] M. Shahzad, L. Wagner, Mater. Sci. Eng. A 506 (2009) 141–147.
[14] Yu Yoshida, Lawrence Cisar, Shigeharu Kamado, Jun-ichi Koike, Yo Kojima, Mater. Sci. Forum 419–422 (2003) 533–538.

[15] N.J. Petch, J. Iron Steel Inst. 174 (1953) 25–28.
[16] J. Koike, T. Kobayashi, T. Mukai, H. Watanabe, M. Suzuki, K. Maruyama, K. Higashi, Acta Mater. 51 (2003) 2055–2065.
[17] J. Koike, R. Ohyama, T. Kobayashi, M. Suzuki, K. Maruyama, Mater. Trans. 44 (2003) 445-451.