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Effects of die angle on microstructures and mechanical properties of AZ31 magnesium alloy processed by equal channel angular pressing[①]

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Abstract: AZ31 Mg alloy was prepared through equal channel angular pressing (ECAP) by Bc route in two different dies, in which the intersecting angles between channels are 90° and 120° respectively. Microstructures and tensile behaviors of the processed material at room temperature were investigated. The 90° die could provide more effective grain refinement in one pass of ECAP. But after multiple passes of deformation and when the total strain reached about 8, the alloy processed by the two dies acquired tiny homogeneous equiaxed grains with a mean size of 1 - 5 μm and possessed similar mechanical properties with a large ductility of above 45% and a yield strength of 100 MPa, which are much different from the ductility of 18% and the yield strength of 200 MPa for the conventionally extruded material. The X-ray diffraction results show a great difference on peak of the basal plane (0001) between sections perpendicular and parallel to the extrusion direction for the conventionally extruded alloy but the difference lowers much for the ECAP processed alloy. The ultra-fine-grain microstructure and the texture or scattered orientation of the basal plane (0001) has the great effect on the alloy's mechanical behavior.

Key words: AZ31 magnesium alloy; ECAP; die angle
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1 INTRODUCTION

Magnesium alloys are the lightest metallic structural materials and hence they provide great potential in the weight saving of automotive and aerospace components, material handling equipment, portable tools and even sporting goods[1, 2]. Due to their hexagonal close-packed (HCP) crystal structure, magnesium alloys perform poor formability and limited ductility at room temperature, thus their products are mainly fabricated by casting, in particular, die-casting, and the applications of wrought magnesium alloys are limited. Now it is imperative to expedite the development of wrought magnesium alloys in order to facilitate the wider structural application and fabricate a variety of Mg products. As we know, the mechanical properties and the formability of magnesium alloys could be improved greatly through grain refinement by severe plastic deformation such as extrusion with large extrusion ratio, rolling, or equal channel angular pressing(ECAP)[3]. Equal channel angular pressing(ECAP) is a new technology for massive billets to produce microstructures with submicron and nanometric grain size by introducing extremely large plastic strain without change in the cross-sectional shape area during the multiple passes of shearing deformation by processing through a special die[4]. Recently, ECAP technique has been applied to refine the microstructure and enhance the mechanical properties at room temperatures or acquire superplasticity at elevated temperatures for many alloys such as aluminum, copper, titanium or magnesium alloys[4-10]. Mabuchi et al[8] reported that the ECAP processed AZ91 alloy acquired a grain size of 1 μm and exhibited a maximum elongation of 660 % at $6\times10^{-5}\ s^{-1}$ at the relatively low temperature of 200 ℃. Yamashta et al[9] reported that pure Mg and Mg-0.9Al could improve both their strength and ductility by ECAP. Very recently, Mukai et al[1] reported that the ductility of AZ31 alloy after ECAP is twice larger than that of the conventionally extruded counterpart. Kim et al[10] reported the ECAPed AZ61 alloy exhibited large elongation(50% - 60%) in tension. In this paper, the effects of die angle on microstructures and properties of AZ31 magnesium alloy processed by equal channel angular pressing were examined. Up to now, no literature has been reported about AZ31 magnesium alloy processed by ECAP in a 120° die.

2 EXPERIMENTAL

AZ31 Mg alloy was prepared by semi-continuous casting and its chemical composition is Mg-2.9%Al-1.0%Zn-0.4%Mn (mass fraction). AZ31 Mg alloy

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was homogenized at 410 ±5 ℃ for 8 h, and then extruded to rods with cross-sectional size of 22.0 mm × 22.0 mm in an extrusion ratio of 4∶1 at 350 ℃. Specimen of 22.0 mm × 22.0 mm × 120 mm were subjected to ECAP through two different dies respectively. For one die, the intersecting angle between channels Φ is 120° and the corner angle Ψ, defined as the angle subtended by the outer arc curvature, is 20°. For another die, Φ is 90° and Ψ is 20°. The rods were held at 300 ℃ for about half an hour and then pressed through the die preheated to 300 ℃ with the extrusion velocity of 20 mm/s. The rods were repeatedly pressed 1 − 12 passes in the 120° die and 1 − 8 passes in the 90° die respectively. All pressings were conducted by rotating the sample about the longitudinal axis by 90° between each consecutive passage (designated as route Bc[11]). And the mixture of graphite powder and engine oil (2∶1) was used as lubricant in the case of extrusion.

The extruded bars were machined into tensile samples with a gauge length of 25 mm and a diameter of 5 mm and some samples were annealed at 300 ℃ for 5 h. All tensile tests were performed in a universal test machine at a strain rate of 2 mm/min. Microstructures of the experimental material were mainly examined on an optical microscope and Philips XL30 scanning electron microscope (SEM). The X-ray diffraction spectra were measured on a Philips X'pert MPD X-ray diffractometer with monochromated Cu K_α radiation operated at 50 kV and 300 mA. The recording was taken with the fixed sample width (0.01°) and scanning speed (5.00°/mm).

3 RESULTS AND DISCUSSION

3.1 Microstructures of the experimental materials

Fig. 1 shows the microstructures of the extruded and ECAPed AZ31 Mg alloy. The mean grain size of the extruded alloy is 20 − 30 μm, as shown in Fig. 1(a). After ECAP, the alloy's microstructures are effectively refined by dynamic recrystallization (DRX). From Figs. 1(b) and (e), it could be found that one pass pressing brought about some tiny subgrains near some original coarse grains, and more fine grains appeared for the processing in the 90° die than that in the 120° die. With the increasing of number passes, the amount of fine grains is increased greatly. From Figs. 1(c), (d), (g), (f) and (h), it could be seen that after eight-passes of pressing in the 120° die or four passes of pressing in the 90° die, the fine microstructure become near uniform. After twelve passes of pressing in the 120° die or eight passes of pressing in the 90° die, the alloy's microstructures consist of tiny homogeneous equiaxed grains with a mean size of 1 − 5 μm.

3.2 Mechanical properties of ECAPed AZ31 magnesium alloy

Fig. 2 shows the typical tensile behaviors of the extruded, one-passed, eight-passed, twelve-passed AZ31 Mg alloys ECAPed in the 120° die (a) and the extruded, one-passed, four-passed, eight-passed AZ31 Mg alloys ECAPed in the 90° die (b). It can be found that, compared with the extruded material, the ECAPed material processed in the 120° die or in the 90° die has the greatly improved ductility, lowered yield strength and little changed tensile strength (UTS), especially when the number of pressing passes is large. Mechanical properties of the alloy after twelve passes of pressing in the 120° die are very close to that after eight passes of pressing in the 90° die. By the time the largest ductility reaches above 45% and the yield strength is 100 MPa for the ECAPed material compared with an elongation of 18% and a yield strength of 200 MPa for the extruded counterpart.

3.3 X-ray diffraction results

Fig. 3 shows the XRD spectra of (a) the extruded AZ31 alloy, (b) the twelve-passed AZ31 alloy ECAPed in the 120° die and (c) the eight-passed AZ31 alloy ECAPed in the 90° die for sections perpendicular and parallel to the extrusion direction. It can be seen that, there exists great difference on the magnitude of the (0001) plane diffraction peak between the two sections for the as-extruded alloy, but lowered difference about which for the alloy ECAPed in the 120° die or in the 90° die. Thus it is suggested that the distribution of basal plane (0001) is inclined to parallel to the extrusion direction in the as-extruded alloy, but the texture changed and the distribution of basal plane (0001) varied, departed from the extrusion direction and became scattered or rearranged in some direction between the extrusion and the transverse direction for the alloy ECAPed in the 120° die or in the 90° die.

3.4 Discussion

Dynamic recrystallization (DRX) happened in the extruded AZ31 Mg alloy and the ECAPed AZ31 Mg alloy and made the grain size refined greatly due to the effect of the deformation temperature and the total strain (and strain rate). The extrusion brought about homogeneous equiaxed grains, the ECAP generated a great deal of small subgrains and the microstructure became finer and more uniform with the increasing number of press-ing passes to certain level.

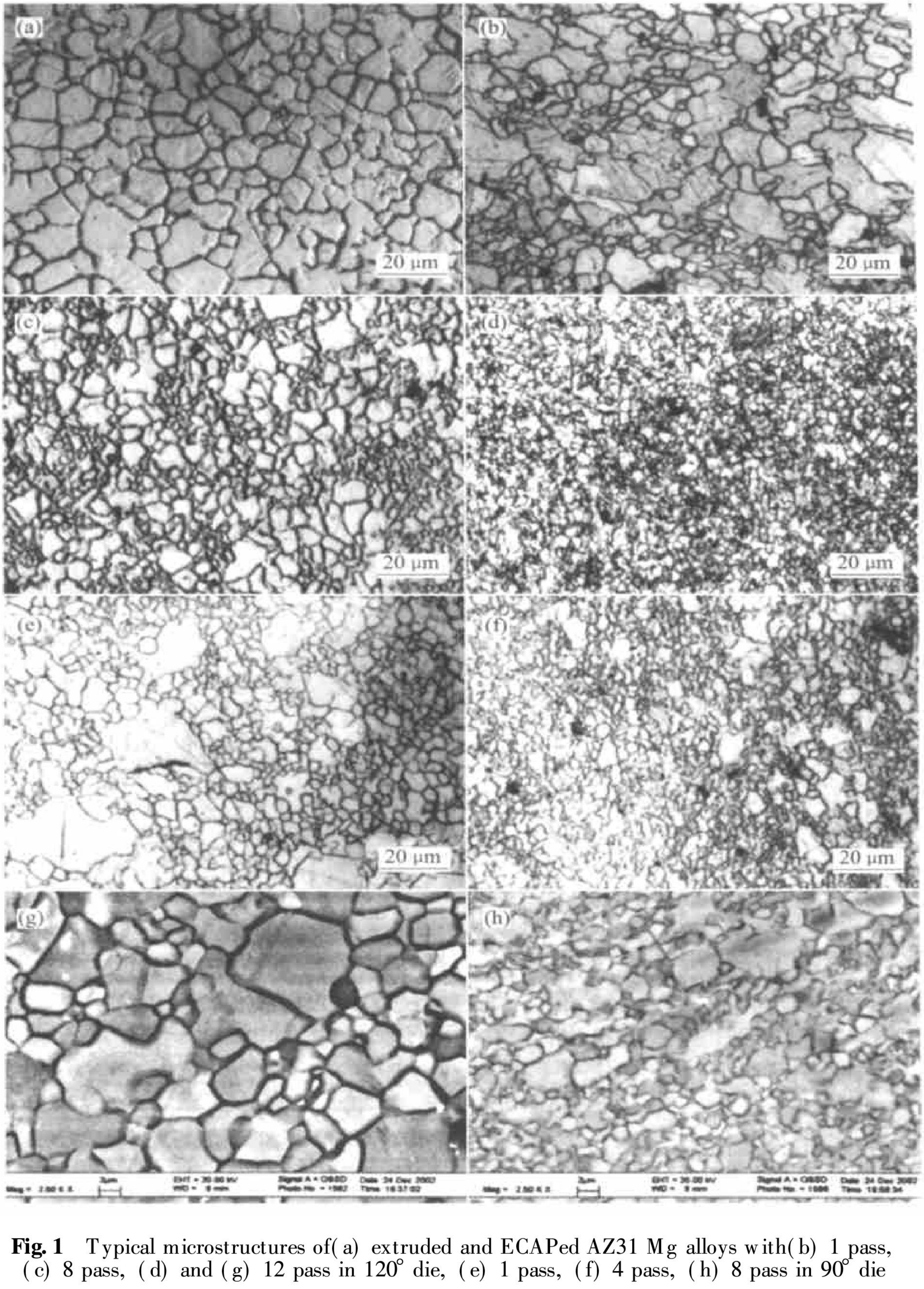

Fig. 1 Typical microstructures of (a) extruded and ECAPed AZ31 Mg alloys with (b) 1 pass, (c) 8 pass, (d) and (g) 12 pass in 120° die, (e) 1 pass, (f) 4 pass, (h) 8 pass in 90° die

The effect of deformation temperature is very large, because high temperature is favorable for the grain growth. So the lower the processing temperature, the finer the DRX grain size. According to Iwahashi et al[12], the shear strain of a single pass of pressing is calculated to be 0.68 through the 120° die and 1.05 through the 90° die. The extrusion in this experiment generated a strain of about 1.4, which was larger than one pass of ECAP, but less than multi-pass of ECAP did. The strain generated by one pass of processing in the 90° die is larger than that in the 120° die, thus more valid grain-refined effect took place after one pass of processing in the 90° die. Because more strain would bring about more dislocation cell walls, leading to the formation of more fine subgrains during the dynamic crystallization process. After a

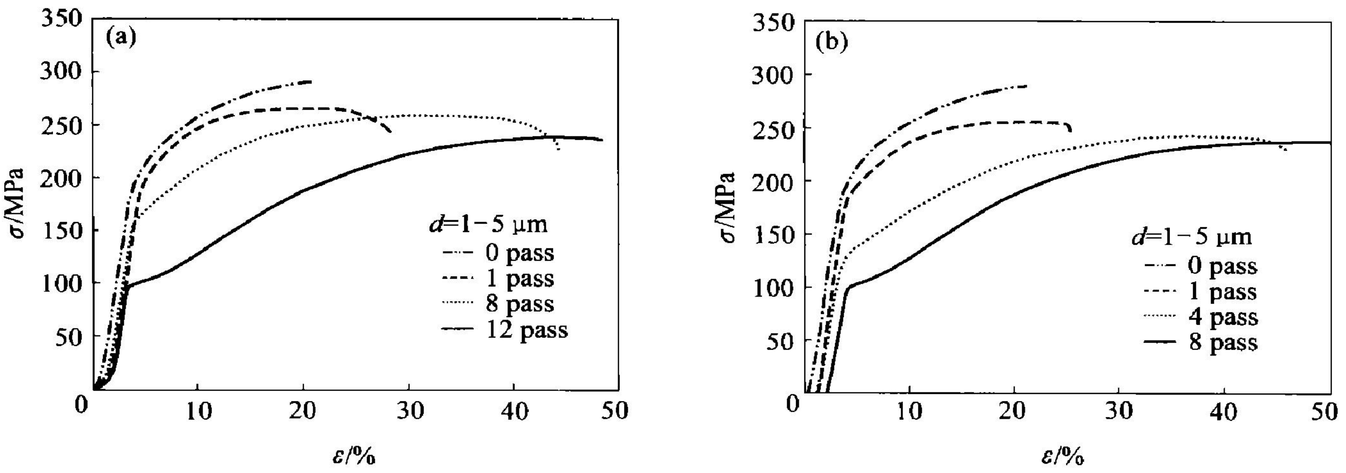

Fig. 2 Typical tensile behaviors of (a) extruded and ECAPed AZ31 alloys with one-pass, eight-pass, twelve-pass in 120° die; (b) extruded, ECAPed AZ31 alloys with one-pass, four-pass, eight-pass in 90° die after annealing

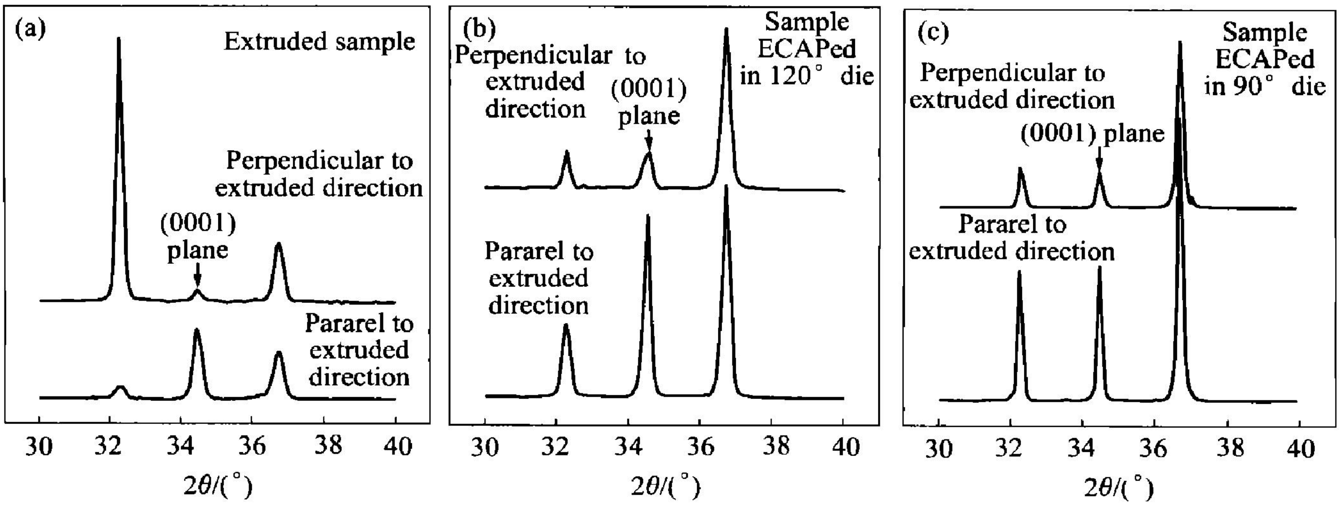

Fig. 3 XRD diffraction spectra of (a) extruded AZ31 alloy, (b) AZ31 alloy ECAPed in 120° die and (c) AZ31 alloy ECAPed in 90° die for sections perpendicular and parallel to extrusion direction

certain strain of deformation, the former coarse grains disappeared and the uniform microstructure was acquired. For ECAP, twelve passes of pressing in the 120° die almost generated the same large total strain (~ 8) as eight passes of pressing in the 90° die did. It could be concluded that the total strain had great influence on obtaining the tiny microstructure, which consisted of equiaxed grains with a mean size of 1 − 5 μm.

The Hall-Petch relation revealed that the finer grain size would lead to the greater yield strength. But in this experiment, the ECAPed Mg AZ31 alloy processed in the 120° die or in the 90° die had finer grain size but a lower yield strength than the as-extruded alloy did. This does not correspond with the Hall-Petch relation. Even at a given grain size, the yield stress of the annealed ECAPed alloy was much lower than that of the extruded alloy. On the other hand, the ECAPed alloy's ultimate tensile strength (UTS) was almost comparative with that of the extruded alloy as if it had nothing to do with the grain size. Why this happened? We have no clear explanation about it now. But we can conclude that it must be related to the texture transition occurring during ECAP. Because the critical resolved shear stress (CRSS) for basal plane slip in magnesium single crystal exhibits 100 times lower value than that for non-basal plane slip near room temperature[1]. So the distribution of basal plane (0001) in magnesium alloys will have great effects on mechanical properties, especially the yield strength. As we know, the extruded magnesium alloys exhibited a strong texture with the majority of basal plane arranged parallel to the extruded direction[13]. Thus their tensile properties especially the yield strength in direction parallel to the extrusion direction was high. As to the ECAPed magnesium alloy, the distribution of basal plane (0001) was modified to some degree or became more scattered, so it was in favor of the dislocation slip during deformation in the tension experiment.

The fact that the ECAPed AZ31 Mg alloy processed by the Bc route after twelve passes of pressing in the 120° die or eight passes of pressing in the 90° die has a large ductility (above 45%) must be related to the ultra-fine grain size and the distribution of basal plane (0001) also. After multiple passes of pressing was performed and when the total strain reached ~ 8, the material acquired much fine microstructures. It is easy to understand the ultra-fine-grained microstructure is beneficial to the elongation, because the ultra-fine-grained microstructure has more area of grain boundary, which can act as barriers for dislocation movement, play the role of reducing concentration stress during plastic deformation, and improve the ability to resist break. Even at a given grain size, the ductility of the ECAPed AZ31 Mg alloy was much larger than that of the extruded counterpart. XRD results show different information on distribution of basal plane (0001) between the ECAPed and extruded material. Although the easy basal plane slip could have done much contribution to the fine grains' cooperative deformation, the activation of the basal planes only is not sufficient to explain the significant increase of ductility since the von Miss criterion cannot be satisfied. Perhaps for the ECAPed alloy, some prismatic slip planes' distribution is likely to orient at about 45° along the tension direction and easier to be activated due to the higher Schmid factor for the ultra-fine-grained microstructure after the initial activation of the basal planes.

4 CONCLUSIONS

The AZ31 Mg alloy's grain size could be refined by ECAP through both the 120° die and the 90° die effectively. The refining effect of a single pass of processing in the 90° die is larger than that in the 120° die. When the large total deformation strain of ~ 8 was reached, the alloy processed by the two ECAP dies respectively acquired tiny homogeneous equiaxed grains with a mean size of 1 − 5 μm and similar mechanical properties with a large ductility of above 45%, a yield strength of 100 MPa. This is much different from the ductility of 18% and the yield strength of 200 MPa for the extruded material. X-ray diffraction results show a great difference in the basal plane's orientation between sections perpendicular and parallel to the extrusion direction for the extruded material, but lowered difference in which for the ECAPed material processed by the two different dies. It could be concluded that the ultra-fine grain size and the texture or scattered orientation of the basal plane (0001) have taken great effect on the alloy's mechanical behavior.

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