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Microstructure evolution and mechanical behavior of AZ31 Mg alloy processed by equal-channel angular pressing

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Abstract: AZ31 Mg alloy bar was subjected to 8-pass equal-channel angular pressing(ECAP) at 623 K. Microstructure evolution was observed by optical microscopy(OM) on cross section and X-ray diffraction analysis. The room temperature mechanical properties of the ECAP processed specimens were also investigated. A fine-grained structure with an average sub-grain size of 9 μm is obtained after 7 ECAP passes. XRD analysis indicates that after ECAP, in placing of $\{10\bar{1}0\}$, planes $\{10\bar{1}1\}$ and $\{10\bar{1}2\}$ become the dominant directions that are favourable for grain refinement. ECAP processed AZ31 Mg alloy exhibits significant improvement in elongation but decrease in strength. The elongation of the specimen increases continuously up to 2 passes and then remains stable at further passes. This improvement can be related to the evolution of crystallographic texture and the scattered orientation of the basal plane (0001).
Key words: AZ31 Mg alloy; equal-channel angular pressing(ECAP); superplasticity; microstructure

1 Introduction

Magnesium alloys are attractive for lightmass structural applications in the transportation industry because of their low density and high specific strength and stiffness[1]. However, the symmetry of the hexagonal close-packed crystal structure has the limited number of independent slip systems, resulting in poor formability and ductility near room temperature[2−3]. Fortunately, this can be resolved by using ultrafine-grained(UFG) structure. UFG materials have sufficient room temperature ductility and superplasticity at high strain rates[4−6].

Equal channel angular pressing(ECAP) is a promising technique for obtaining microstructures with ultra-fine grains in the sub-micrometer to nanometer range for bulk metallic materials[7]. In addition, the UFG structure is reasonably stable at elevated temperature and is capable of providing superplastic deformation in tensile testing[8]. An early attempt to achieve significant grain refinement in AZ91 alloy by ECAP with a grain size of 1 μm and an elongation of 660% at 6×10^{-5} s^{-1} and 200 ℃ was successful[9]. The grain size of a cast Mg-9Al-1Zn alloy was significantly refined to 0.5 μm after 8 passes of ECAP at 448 K via route A[10], and superplasticity, especially at low temperatures and high strain rates, was achieved for Mg alloys with UFG structures through ECAP[11−12]. Recently, KOIKE et al[13] reported that the ductility was improved for fine-grained AZ31 Mg alloy due to the activity of nonbasal slip, grain boundary sliding(GBS) and recovery in high strained region. However, this significant improvement of superplasticity, i.e. enhancement of elongation to fracture, is at the expense of strength by ECAP[14]. The reason is not clear .

In the present work, AZ31 Mg alloy was subjected to repetitive ECAP processing in order to improve mechanical properties of Mg alloy by grain refinement. Relationship between microstructures and mechanical properties of ECAP-processed AZ31 Mg alloy was investigated.

2 Experimental

A cast billet of AZ31 Mg alloy (Mg-2.8%Al-0.9%Zn-0.24%Mn, mass fraction) was machined into cylindrical specimens with diameter of 15 mm and

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height of 70 mm for ECAP processing after homogenization. The intersecting angle between the two channels was 90˚ and the angle of the outer arc at the intersection was 20˚. The 8-pass ECAP processing was performed at 623 K. These ECAP specimens were rotated by 90˚ along the longitudinal axis of the specimen after each pass, which was so-called route B_c, to obtain a homogeneous microstructure. The die was heated by a flexible heating blanket during the testing. For each pass, the die was heated to the designated temperature and stabilized prior to mounting the specimen. The specimen was coated with graphite and boron nitride for lubrication before being put in the entrance channel at the testing temperature. The specimen was held at the temperature for 15 min to achieve stabilization.

Tensile specimens with gauge length of 25 mm and diameter of 5 mm were machined from the initial sample and also from the rods produced by ECAP. The tensile tests were carried out by an MTS machine at a strain rate of 3 mm/min. The grain structures in the as-extruded and ECAP processed specimens were examined by optical microscope(OM). The samples were ground and polished following standard metallography procedures, and etched at room temperature for 5 s using a solution of 1% HNO_3, 24% $C_2H_6O_2$ and 75% H_2O (volume fraction).

3 Results and discussion

3.1 Effect of die angle on microstructures

Fig.1 shows the optical images of as-received specimen and those after 4-pass ECAP processed specimens. The original microstructure of the cast ingot is not uniform on the cross sections, as shown in Fig.1(a). Larger grains up to hundreds of micrometers are surrounded by smaller grains with tens of micrometers in diameter. During ECAP processing, their microstructures are effectively refined by dynamic recrystallization (DRX). As illustrated in Figs.1(b) and (c), it can be found that AZ31 Mg alloy after 4-pass ECAP through 120˚ die via route B_c still consists of a considerable number of large grains and shows a non-uniform grain structure. But the microstructure after 4-pass ECAP through 90˚ die via route B_c is more heterogeneous with very fine grains, about 10 μm in diameter.

DU et al[15] reported that the difference of equivalent true strain at the angles in the range of 90˚−120˚. LI et al[16] reported that DRX caused the grain size refinement due to the effect of the deformation temperature and the total strain or the strain rate occurred in AZ31 Mg alloy after ECAP. The extrusion led to homogeneous equiaxed grains and the ECAP generated many small sub-grains, so that the microstructure became finer and more homogeneous with increase of pressing passes up to a critical value. The effect of deformation temperature is significant as high temperature is favorable for grain growth. The lower the processing temperature, the finer the DRX grain size. IWAHASHI et al[17] calculated the shear strain of a single pass to be 0.68 through 120˚ die and 1.05 through 90˚ die. The strain generated by 4-pass ECAP through 90˚ die is larger than that through 120˚ die, thus more grain-refined effects took place after 4-pass ECAP through 90˚ die. Since larger strain could generate more dislocation cell walls that lead to the formation of more sub-grains during the dynamic crystallization process, it

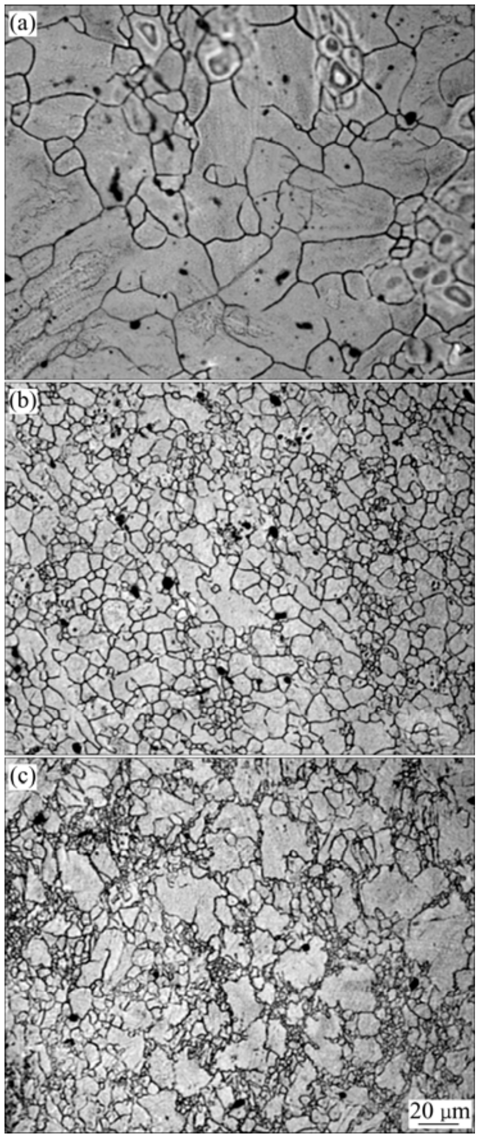

Fig.1 Typical microstructures of ECAP AZ31 Mg alloys as function of die angle via route B_c: (a) Original unpressed morphology; (b) 4-pass through 90˚ die; (c) 4-pass through 120˚ die

is apparent that the die angle significantly affects the deformation and therefore influences the microstructures of specimens after ECAP.

3.2 Effect of passes on microstructures

Fig.2 shows the OM images of 8-pass ECAP processed specimens. After 1 pass of ECAP at 623 K, the microstructure is more heterogeneous with very fine grains about 9−11 μm in diameter. With further ECAP processing at the same temperature, the volume fraction of fine grains increases while that of large grains decreases. A homogeneous fine microstructure with an average grain size of 9 μm after 7 passes of ECAP can be obtained. The average grain size is further homogeneous after 8 passes of ECAP. Some grains are refined to a few micrometers with little residual strain whereas other large grains undergo distortion with traces of plastic deformation. Such a heterogeneous microstructure is rarely observed in ECAP processed cubic structured alloy such as Al alloys and steels[18−19]. This heterogeneity hcp packed Mg alloy is believed to result from the different orientations of the grains in relation to the shear direction imposed by ECAP. The easy slip systems in the hcp structured materials such as Mg are limited, compared with those in cubic materials. As a result, the Schmid factors for these slip systems differ significantly in grains with different orientations. A large number of grains are initially in the so-called hard orientations with small Schmid factors where deformation is difficult to occur. On the other hand, those soft oriented grains with large Schmid factors can deform easily. LIN et al[20] reported a high Schmid

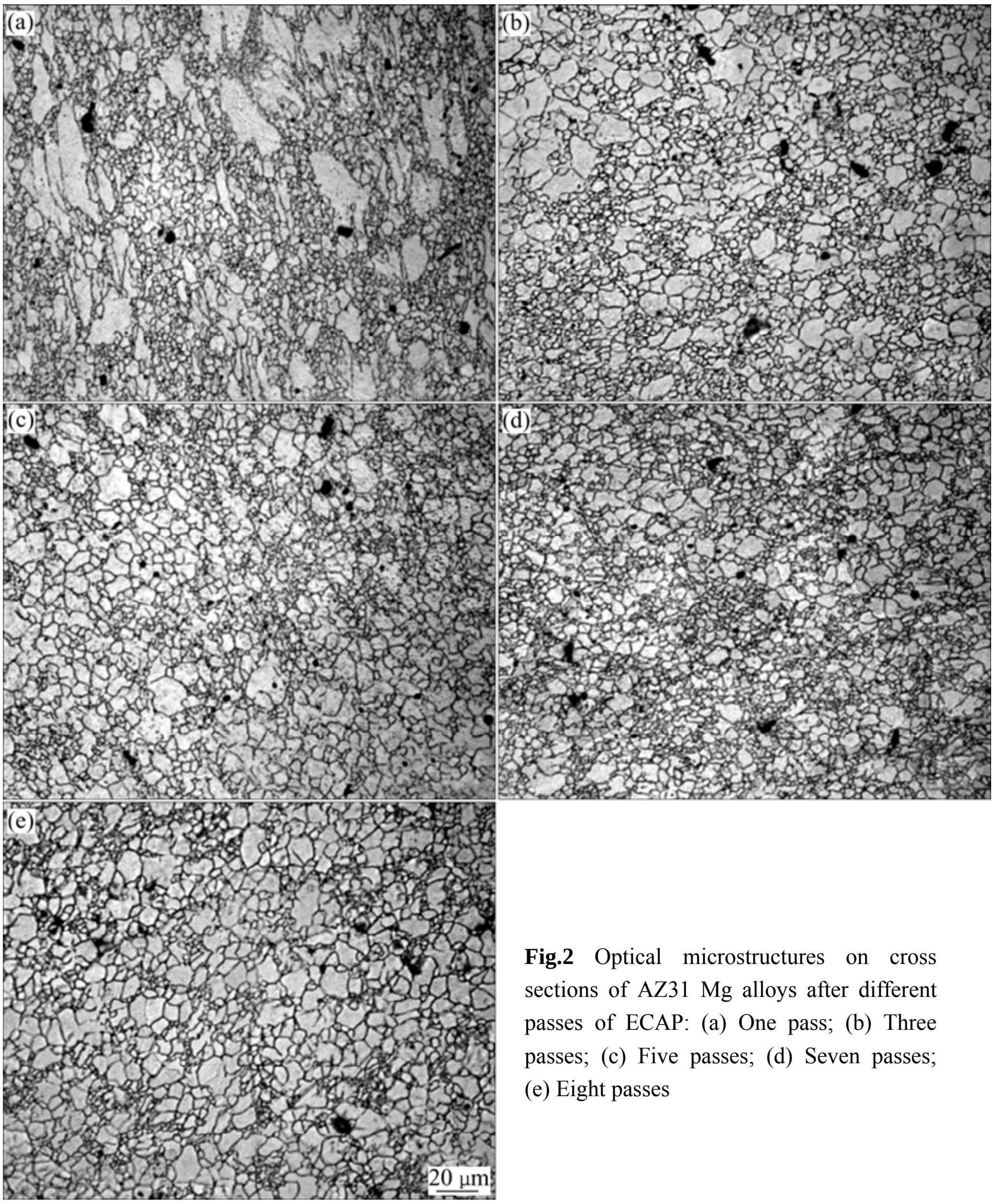

Fig.2 Optical microstructures on cross sections of AZ31 Mg alloys after different passes of ECAP: (a) One pass; (b) Three passes; (c) Five passes; (d) Seven passes; (e) Eight passes

factor of 0.27 for Mg alloy under the ECAP condition and zero under the extruded condition, leading to the non-uniform microstructures. This heterogeneity in grain structure is reduced as the deformation increases with increase of pass.

Fig.2 also indicates that the grain size changes with the number of ECAP passes up to 3 and then remains stable. The evolution of the grain size with as the number of ECAP passes is presented in Fig.3.

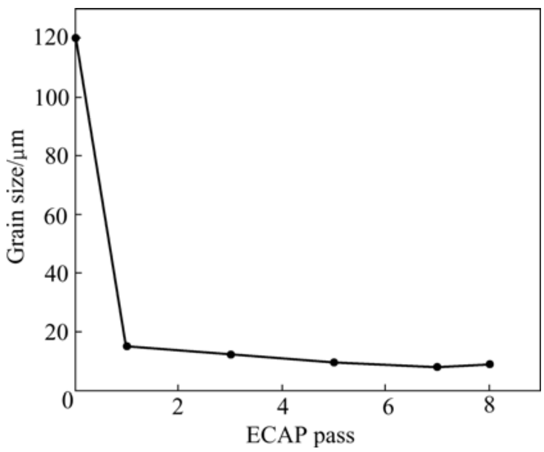

Fig.3 Grain size evolution during ECAP

Fig.4 shows XRD patterns of AZ31 Mg alloy after ECAP. It is apparent that the planes $\{10\bar{1}0\}$ for the extruded specimens show a strong tendency to lie perpendicular to the extrusion axis while no such tendency occurs under the ECAP condition. Only the basal slip system $\{0001\}\langle 11\bar{2}0\rangle$ is operated. After ECAP, the intensity of planes $\{10\bar{1}1\}$ and $\{10\bar{1}2\}$ increases. By contrast, the intensity of planes $\{10\bar{1}0\}$ decreases significantly. LIU et al[21] considered that the change of deformation mode was attributed to the grain refinement caused by the ECAP. Accordingly, it can be

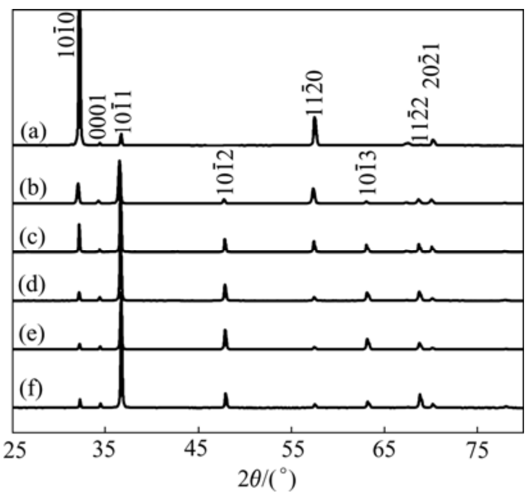

Fig.4 XRD patterns of AZ31 Mg alloy before and after ECAP: (a) Before ECAP; (b) One pass; (c) Three passes; (d) Five passes; (e) Seven passes; (f) Eight passes

concluded that during the primary stage of ECAP, the main deformation mode of α phase is $\{10\bar{1}1\}\langle 10\bar{1}2\rangle$ twinning, while during the following passes the main deformation mode is dislocation slip, which is close to the results reported by LIU et al [21].

In general, the crystallographic texture is an important strengthening mechanism, especially in the hcp metals with a low symmetry. This structural factor can be changed considerably during the process of plastic deformation. As can be seen clearly in Fig.4, a strong texture is initially observed in the as-extruded Mg alloy. The intensity of this extruded texture gradually decreases during ECAP processing. In the as-extruded material, the {0001} basal planes and $\langle 10\bar{1}0\rangle$ directions in most grains are preferentially parallel to the extrusion direction(ED). That is, the as-extruded alloy exhibits an ED//$\langle 10\bar{1}0\rangle$ fibre texture. The shear deformation during ECAP will destroy the existing fibre texture from extrusion. Consequently, the intensity of the fibre texture decreases with increasing the number of ECAP passes, and after a certain number of passes, the original fibre texture from extrusion can be completely removed. In the meanwhile, a new texture associated with the shear deformation of ECAP is gradually built up with the subsequent ECAP processing[22].

3.3 Effect of passes on mechanical properties

Fig.5 shows the mechanical properties of the AZ31 Mg alloy before and after ECAP at 623 K for different passes. The uniform elongation of AZ31 Mg alloy increases continuously and the strength decreases slightly after ECAP. Particularly, the elongation increases from 28% of as-extruded alloy to 50% of 2-pass ECAP processed specimen and then remains stable. However, the ultimate strength decreases from 321.2 MPa of as-extruded alloy to 283.9 MPa of 2-pass ECAP processed specimen and then remains stable also. The results indicate that the strength reduction with the

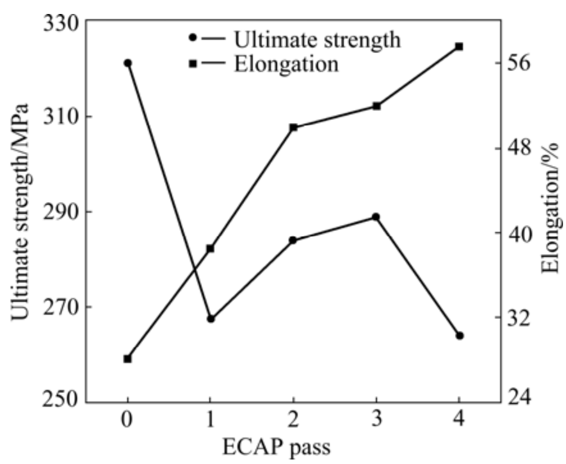

Fig.5 Mechanical properties of AZ31 Mg alloy as function of passes of ECAP

decrease of grain size is consistent with the results on AZ61 alloy fabricated by ECAP[22].

The reason why the strength of ECAP alloy with increasing passes of ECAP is lower than that of the extruded alloy must be related to the texture transition occurring during ECAP. Because the critical resolved shear stress(CRSS) for basal plane slip in Mg single crystal exhibits 100 times lower than that for nonbasal plane slip near room temperature[23]. So the distribution of basal plane (0001) in Mg alloy will have great effects on mechanical properties. As we know, the extruded Mg alloy exhibits a strong texture with the majority of basal plane arranged parallel to the extruded direction[24]. Thus, their tensile properties especially the yield strength in direction parallel to the extrusion direction are high. As to the ECAP processed Mg alloy, the distribution of basal plane (0001) has been modified to some degree or became more scattered, so it is in favor of the dislocation slip during deformation in the tension experiment. This is the reason why the elongation of AZ31 Mg alloy increases and the strength decreases after ECAP. In other words, ECAP results in the increase of the tensile strength at room temperature as a consequence of the reduction of the grain size as well as the increase of the dislocation density. Simultaneity, at high temperature, the recovery process becomes important, the tensile strength decreases and the ductility increases owning to ECAP for Mg alloy.

From the microstructural analysis and the mechanical testing, it can be deduced that at high temperatures the large elongation and the low strength of the ECAP processed specimens compared with the initial state are most probably caused by the relatively small grain size, which enables the material to deform by the mechanism of grain boundary sliding. Grain boundary sliding plays an important role in superplasticity. This phenomenon has been confirmed by CHUVILDEEV et al[25].

4 Conclusions

1) The effect of high temperature ECAP processing on the microstructure and the mechanical properties of AZ31 Mg alloy is studied by means of optical microscopy(OM), X-ray diffraction analysis and mechanical testing. ECAP is successfully used to achieve a reasonably homogeneous ultrafine-grained micro-structure for the AZ31 Mg alloy. With increasing the number of ECAP passes, the initial coarse grained structure in the as-extruded material is transformed gradually into an ultrafine-grained microstructure with an average grain size of 9 μm. XRD analysis indicates that after ECAP, the planes $\{10\bar{1}1\}$ and $\{10\bar{1}2\}$ hold the ascendancy directions replacing the planes $\{10\bar{1}0\}$ and become favourable for the grain refinement.

2) The ECAP processing leads to relatively small changes of the tensile strength, but significant improvement of the uniform elongation. Ultimate strength of AZ31 Mg alloy reaches 321.1 MPa. The strength decreases after ECAP compared with that of as-extruded alloy. The uniform elongation of the alloy increases continuously up to 2 passes and then remained unchanged.

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