Microstructure and texture characteristics of ZK60 Mg alloy processed by cyclic extrusion and compression

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Abstract: The microstructure and crystallographic texture characteristics of an extruded ZK60 Mg alloy subjected to cyclic extrusion and compression (CEC) up to 8 passes at 503 K were investigated. The local crystallographic texture, grain size and distribution, and grain boundary character distributions were analyzed using high-resolution electron backscatter diffraction (EBSD). The results indicate that the microstructure is refined significantly by the CEC processing and the distributions of grain size tend to be more uniform with increasing CEC pass number. The fraction of low angle grain boundaries (LAGBs) decreases after CEC deformation, and a high fraction of high angle grain boundaries (HAGBs) is revealed after 8 passes of CEC. Moreover, the initial fiber texture becomes random during CEC processing and develops a new texture.

Key words: ZK60 Mg alloy; cyclic extrusion and compression; electron back scatter diffraction (EBSD); microstructure; texture

1 Introduction

Magnesium alloys have attracted considerable interest from the automobile industry because of their low density and high specific strength. Nevertheless, as structural components in automobiles, the material should exhibit both sufficient ductility and attractive specific strength since components may fail by fracture due to shear or tensile forces[1–5]. Thus, the relatively low strength and ductility of Mg alloys due to the hexagonal close packed (HCP) structure with limited slip systems is a major difficulty that hinders their widespread application[1].

Recently, it has been demonstrated that the plastic deformation of Mg alloys is strongly influenced by their texture as well as their grain size. The ductility of Mg alloys may be significantly enhanced by texture control and grain refinement through severe plastic deformation (SPD) such as equal channel angular pressing (ECAP)[2, 6], cyclic extrusion and compression (CEC)[1, 7–10] and accumulative roll bonding (ARB). As a continuous SPD processing, CEC seems more adaptable for industrial applications. Moreover, it is very suitable to refine grains of hard-to-deform metals such as Mg alloys since it imposes three-dimensional compression stresses during processing[3, 11–13].

Our previous results demonstrated that the tensile ductility of ZK60 alloy increased with the increase of CEC accumulated strain. However, the strength shows an opposite trend though the grain size is reduced[1, 3], i.e. exhibiting a reverse or inverse Hall-Petch relation. Similar results in CEC AZ31 alloy[12] and ECAP AZ31 alloy[2] were also reported. Its underlying mechanism is complicated and maybe relevant to grain boundary sliding, texture evolution, grain refinement, twinning, dislocation density and so on. Much more research is needed to investigate the microstructure characteristics of SPD processed metal materials to reveal the relationship between microstructure and mechanical properties. Therefore, the aim of the present study is to investigate the microstructure and crystallographic
texture characteristics of an extruded ZK60 alloy subjected to CEC processing using high-resolution EBSD.

2 Experimental

The alloy used in the present study was commercial ZK60 alloy. It was received in the form of an extruded bar with a diameter of 29.5 mm and then cut into specimens with dimensions of \( d = 29.5 \text{ mm} \times 42 \text{ mm} \) for CEC processing. The CEC processing was carried out by pushing a billet from one cylindrical chamber with diameter \( D \) to another chamber with equal dimensions. The inter-chamber can be considered as an extrusion die with a smaller diameter \( d \), as illustrated in Fig.1[1, 13]. The die was lubricated using graphite and preheated to 503 K before processing. During the final extrusion pass, the opposite ram B was removed in order to release the rod. The ram speed was 8 mm/s. All specimens were quenched in water immediately after deformation. In the present study, \( d \) and \( D \) are 20 mm and 30 mm, respectively.

![Fig.1 Schematic illustration of CEC facility and procedure: (a) Initial state; (b) Extruding by ram A; (c) End of ram A involvement; (d) Reverse extruding employing ram B; (e) End of ram B involvement; (f) Final extruding to obtain rod](image)

EBSD samples were prepared by mechanical grinding and mechanical polishing, final mechanical polishing was performed with a diluted OPS solution (10%, volume fraction). Thereafter, electropolishing was achieved with an AC2 solution at a voltage of 15 V for 10–20 s at –30 °C[8]. The index quality profiles obtained from the EBSD measurements (four scans for each state on zones of 150 µm×150 µm again with a scan step of 1 µm). The electron diffraction analyses were performed in a Zeiss 55VP FEG-SEM equipped with a Nordif EBSD detector and the TSL OIM EBSD software. Scan steps of 0.1 µm for CEC samples and 0.3 µm for as-extruded samples were employed.

3 Results and discussion

3.1 Microstructures

Fig.2 presents the EBSD mapping in form of an inverse pole figure (IPF) map of as-extruded sample and sample after 8 passes CEC at 503 K. Different gray scales represent different orientations of the grains. The stereographic triangle in the left of the maps gives color-code employed in these maps. The nearly identical gray grains mean that the disorientations between these grains are not large[14]. Fig.2(a) shows that the microstructure of the as-extruded ZK60 alloy is rather heterogeneous and typically incompletely recrystallized. Coarse grains with 50–100 µm in size are elongated along the extrusion direction and fine recrystallized grains with 5–15 µm in size are distributed among coarse

![Fig.2 Inverse pole figure maps of ZK60 alloy before (a) and after (b) 8 passes CEC processing](image)
Most grains in Fig.2(a) exhibit dark gray, which means that the {0001} basal planes of these grains are nearly parallel to the paper. After 8 passes of CEC processing (Fig.2(b)), the grains are dramatically refined and uniformly distributed due to the dynamic recrystallization[15]. The much more randomly distributed grain gray scale indicate that the orientations of grains are changed.

### 3.2 Grain boundaries misorientation

Fig.3 shows the grain boundary map of as-extruded and CEC processed alloy. The low angle grain boundaries (LAGBs) are marked by dark gray lines (2°−5°) and light gray lines (5°−15°), respectively. The high angle grain boundaries (HAGBs) with misorientation angles larger than 15° are marked by dark lines. As shown in Fig.3(a), there are numerous LAGBs in the as-extruded material. With the increase of CEC pass number, the grains are gradually refined. The statistics of grain boundary misorientation are shown in Fig.4. It can be seen that the number fraction (NF) of LAGBs decreases and that of HAGBs increases with increasing CEC strain. The NF of HAGBs in the as-extruded ZK60 alloy is relatively low (58.1%). After CEC 8 passes, the HAGBs NF dramatically increased to 95.6%, indicating the fact that most of the LAGBs were evolved into HAGBs during CEC deformation due to dynamic recrystallization process. In addition, two NF peaks could be distinguished around 30° and 90°, respectively (Fig.4(a)). Similar results were earlier reported for AZ31 Mg alloy after CEC[8] and ECAP[14]. It could be a strain-induced result of HCP lattice alloy and its formation associated with the plastic deformation mechanism of Mg alloy, especially twinning deformation.

### 3.3 Texture analysis

Fig.5 shows the EBSD determined {0002} and \{0110\} pole figures of ZK60 alloy before and after CEC processing. As shown in Fig.5(a), the as-extruded ZK60 alloy exhibit a strong \langle 0110 \rangle fiber texture, which means that {0002} basal planes and \langle 0110 \rangle directions in most grains distribute parallelly to the extrusion direction (ED), that is why most grains in Fig.2(a) exhibit dark gray. Similar result was observed in as-extruded ZK60 alloy by XRD analysis[3]. After 2−8 passes CEC, the initial fiber texture vanishes and evolves
into a new \(\{10\bar{1}3\}\) \(\langle 30\bar{3}2\rangle + \{10\bar{1}1\}\) \(\langle 15\bar{4}3\rangle\) texture[3]. The dominant texture does not vary with the number of CEC passes ranging from 2 to 8. On the other hand, the maximum intensity of both \(\{0002\}\) and \(\{10\bar{1}0\}\) pole figures decline with increasing pass number of CEC, which indicates that the orientation of the grains gradually becomes more random.

4 Conclusions

1) The microstructure of ZK60 can be effectively refined by CEC processing. Moreover, the crystallographic orientations are uniformly distributed by CEC deformation.

2) In the as-extruded alloy, many LAGBs are observed. After CEC deformation, the number fraction of LAGBs decreases and a high fraction of HAGBs (95%) is revealed after 8 passes of CEC.

3) The initial fiber texture of as-extruded ZK60 alloy becomes disintegrated and changes into a new texture during CEC deformation. The texture type does not vary with increasing CEC pass number, but the pole intensity decreases.

References


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