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ARTICLE

Grain Refinement and Texture Evolution of Mg-1Zn-1Gd Alloy in Equal Channel Angular Pressing

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Abstract: The effect of equal channel angular pressing (ECAP) on the microstructure and texture, as well as dynamic recrystallization behavior of Mg-1Zn-1Gd alloy (wt%) was investigated. The results show that after the equal channel angular pressing at 350 °C, the microstructures of these samples are composed of fine recrystallized grains, and there are a large number of homogeneously distributed equiaxed grains in the matrix. Upon 8 passes, the uniform ultra-fine grain structure with an average grain size of 3.6 μm is obtained. The mechanism of the discontinuous dynamic recrystallization (DDRX) and continuous dynamic recrystallization (CDRX) leads to grain refinement. The micro texture was analyzed through electron backscatter diffraction (EBSD) technique. It is revealed that after 4 passes of ECAP, a strong basal texture is achieved (multiple random distribution ~19.76). With increasing the extrusion passes, the grain on the basal planes of extruded alloy sheet is mainly elongated along the extrusion direction, the orientation distribution changes from concentrated state to decentralized state. The texture is weakened and the maximum is 15.66. Finally, the texture is related to the plane anisotropy of the Mg-1Zn-1Gd alloy.

Key words: Mg-1Zn-1Gd alloy; equal channel angular pressing; grain refinement; DRX; texture

The magnesium alloy containing rare earth (RE) has the advantages of high specific strength and good thermal stability, and has a broad application prospect in aerospace and automotive, because of their low density, high specific strength and great demands in aspect of energy saving and environment protection[1-3].

Unfortunately, commercial magnesium alloys such as the AZ series magnesium alloys produced by traditional techniques usually have poor ductility and strong anisotropy at room temperature, which restrict their further pressing formability and industrial applications[4]. With expansion of the application of Mg alloys, the development of new high refinement magnesium alloys have attracted great attention in recent years. Therefore, improving the formability by changing or weakening the texture is an effective method to boost a wider use of magnesium alloys in industry[5]. There are many different plastic deformation techniques such as equal channel angular extrusion[6,7], differential speed rolling[8,9], torsion extrusion[10] and cyclic extrusion[11], which have been used for investigating Mg alloys and have enhanced the mechanical properties in developing weaker or non-basal textures.

It has been reported that the equal channel angular pressing can effectively improve the properties of Mg-RE alloys by thermal mechanical processing[12]. Dynamic recrystallization (DRX) occurs easily, which can be divided into discontinuous recrystallization and continuous recrystallization upon the ECAP. The characteristic of the DDRX is nucleation and nucleus growth by high angle boundary migration. However, CDRX is characterized by the continuous absorption of dislocation into subgrain boundaries, which effectively lead to the formation of high angle boundaries and new refined grains[13].

In general, the extrusion passes and the addition of

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rare-earth can result in the microstructural refinement and strength enhancement of extruded magnesium alloys. By adding Zn/Gd in different ratio, a large number of second-phase particles are involved in Mg matrix, which will contribute to the particle-stimulated nucleation and weaken the texture. Therefore, different contents of Zn and Gd may act different effects on weakening basal texture, ductility and formability improvement in the Mg-RE alloys. A new study about room-temperature ductility and formability of extruded Mg-Zn-Gd alloy slabs as structural materials for its good forming characteristics has revealed a promising potential. However, the mechanism is not clear so far. In this study, Zn and Gd are major alloying elements (the content of Zn is equal to Gd) in the Mg alloys, and the DRX behavior and grain refinement at different extrusion passes of equal channel angular pressing were investigated.

1 Experiment

The Mg-1Zn-1Gd (wt%) alloy was used in this research. The initial material with a diameter of 120 mm was cast, which was square billet with a length of 100 mm, a width of 50 mm and a height of 50 mm after ECAP. ZG11 alloy billet was preheated to 350 °C, and then extruded by ECAP. The ECAP process was performed using a rotary die with φ=90° (inner arc of curvature) and Φ=60° (outer arc of curvature)[14]. The billets were subjected to 8 passes at 350 °C with a rate of 12 mm/min.

The schematic diagram of ECAP process adopted is represented in Fig.1. After the extrusion, the mid-layer of the steady state region of the sample was sliced off for microstructure and texture analysis. For microstructure characterization, the specimens were observed by optical microscope (OM, LEICA DMI3000 M) and scanning electron microscopy (SEM) with an X-ray energy-dispersive spectrometer (EDS), etched in oxalic acid and chrysolepic acid. Texture analysis of the extruded samples was performed by an Nova NanoSEM 430 electron backscatter diffraction (EBSD) system through the electrochemical polishing, and the data were analyzed with HKL Channel5 software. To ensure the accuracy of data, more than 10 000 data points were recorded for different samples.

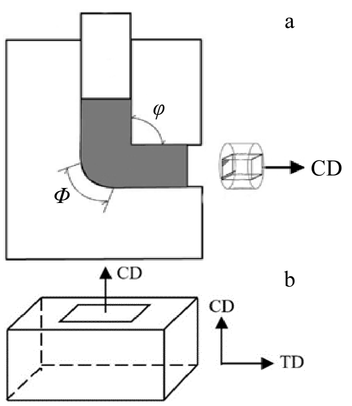

Fig.1 Schematic diagram of equal channel angular pressing (a) and sample in transverse direction (TD) and compressing direction (CD) (b)

2 Results and Discussion

2.1 Microstructure evolution

Fig.2 shows the microstructure of Mg-1Zn-1Gd alloy after the equal channel angular pressing. It is found that dynamic crystallization and grain refinement are presented in all samples during extrusion. The microstructure characteristics of all alloys have a significant change from as-cast to 8 passes, they all exhibit highly heterogeneous deformation microstructures. The initial microstructure of as-cast state is coarse, with an average grain size of about 80 μm. Most of them are isometric crystals and the degree of homogenization is high. After 1 pass, the original grain boundary becomes serrated, and some dynamic recrystallized grains are observed at the original grain boundary.

With the gradual increase of deformation and the extrusion to 4 passes, the primary grains tend to be a necklace type structure. And deformation twinning is clearly shown after 4 passes. This is easy to understand, because it is prone to induce twinning behavior and dynamic recrystallization when the grains are in a certain size. And the slight serrations are visible at some twin boundaries with new twin grains (arrowed in Fig.2c). Upon further extrusion to 8 passes, the number of the dynamically recrystallized grains increases obviously and show a typical partially recrystallized structure, because some twins need to be consumed for new born grains. According to the grain size and morphology, the recrystallized grain is distinguished from the unrecrystallized one. It is easy to discriminate because the size of the original deformed grain is obviously different from that of the recrystallized grain.

As shown in Fig.2, a large number of deformed grains are generally surrounded by new tiny grains that originate from the grain mantle regions. When new fine grains surround the original grain boundaries and twin grain boundaries, it means the start of the DRX process. In this study, there are two ways of DRX nucleation. One is twin grain boundary nucleation, such as $\{10\bar{1}2\}$ twin grain boundary refers to DRX nucleation. The other is particle stimulated nucleation (PSN) mechanism. Jiang et al[15] showed that because the twin grain boundary has a high mobility under continuous plastic deformation, the consumption and orientation of $\{10\bar{1}2\}$ twin grains indirectly promote the subsequent DRX process. $\{10\bar{1}2\}$ twin grains slowly consume the whole parent grains from the grain boundary, thus promoting the grain development of $[10\bar{1}0]$ orientation. Subsequently, the DRX process occurs in these grains.

In addition, twin grain boundaries can effectively hinder the dislocation movement and promote the formation of subgrain along the boundary. In the same way, as one of the factors of

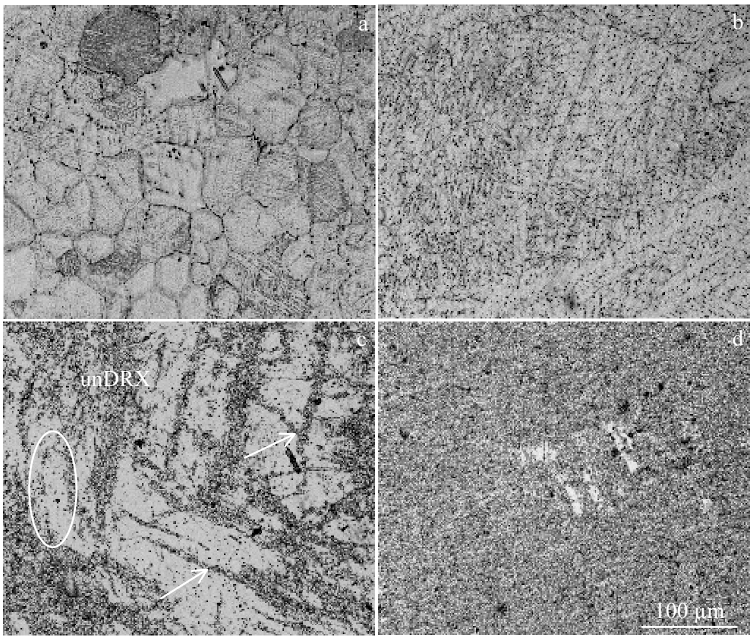

Fig.2 Microstructural evolution of Mg-1Zn-1Gd alloy after ECAP at 350 °C with a rate of 12 mm/min: (a) as-cast, (b) 1 pass, (c) 4 passes, and (d) 8 passes

recrystallizing mechanism, particles can effectively affect the process of recrystallization. Doherty et al[16] showed that in the process of plastic deformation, particles will affect the deformation microstructure and texture by effects such as increase in dislocation density, the production of large deformation heterogeneity of slip system. It is certain that with the increase of the extrusion pass, the residual unrecrystallized region and the twinning form a dynamic recrystallization along the boundary. At the same time, the large particles promote the nucleation of grains through pining grain boundaries (Zener pinning). Unfortunately, it is hard to prove deformation bands from unrecrystallized region or the twins by the optical micrographs.

2.2 Dynamic recrystallized behavior

The orientation maps from EBSD data was investigated for obtaining more information about DRX operation mechanisms. In the EBSD maps, the high-angle boundaries (>15°) were represented by thick black, and the low-angle boundaries conform to thin black (10°~15°) and thin white lines (2°~10°).

Dynamic recrystallization mechanism is divided into continuous recrystallization and discontinuous recrystallization[17]. Discontinuous DRX refers to the gradual transformation of the stable small angle grain boundary into a large angle grain boundary during extrusion, which is started at a high angle boundary. As shown in Fig.3, a large number of low angle grain boundaries (LAGB) occur in coarse grains and twin grains, which can determine the activation of basal slips, prismatic slips and <*c*+*a*> pyramidal slips. Some researches reveal that the Schmid factor of prismatic slips and pyramidal slips is much higher than that of basal slips, and the critical resolved shear stress (CRSS) of prismatic and pyramidal slips decreases rapidly when the temperature increases[18]. With the activation of non-basal slips and the interaction of slip system and grain boundaries, great quantities of LAGBs form. With increasing the passes, a large number of dislocations are absorbed by LAGBs, which transform to high angle grain boundaries (HAGB). Then, new fine grains are born along grain boundaries.

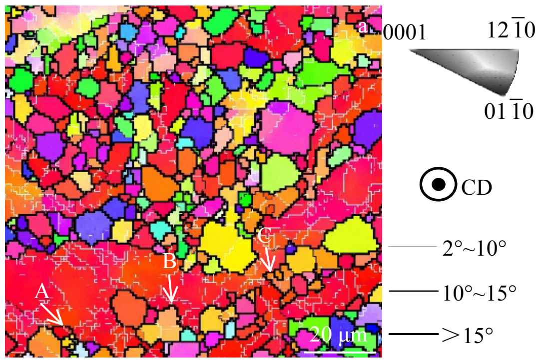

Fig.3 Orientation map of Mg-1Zn-1Gd alloy deformed after extrusion to 4 passes

As arrowed by A in Fig.3, a new born fine grain is obvious which forms along the original boundary after higher extrusion pass. The bulging behavior usually occurs before the crystallization. The performance of the bulging is indicated by arrow B in Fig.3, and the grain boundary is serrated into a bulge, where a new grain probably appears. The example of the development between bridging low-angle boundary and the vicinity original boundary is marked by arrow C in Fig.3.

As illustrated in Fig.4, new exiguous born grains primarily form from grain mantle regions and recrystallized twin areas upon extrusion from 4 passes to 8 passes. However, it can be seen from orientation maps that after the large extrusion pass, dynamic recrystallization occurs more frequently, and more fine grains form. With increasing the deformation and the continuous development of DRX, it results in a typical microstructure composed of small new DRXed grains and coarse unDRXed grains (marked in Fig.4a).

At the 8 passes of extrusion, the DRXed grain volume fraction increases obviously. As can be seen from Fig.4b, all the unDRXed zones are almost consumed by the new fine born grains, with an average grain size about 3.6 μm. Fig.4c illustrates the particles and EDS results of point marked as circle in Fig.4a. The phases of particle may be $Mg_{15}Zn_5Gd_4$ according to the result of EDS. In this study, there are other mechanisms influencing the completed DRX microstructure evolution, which is related to the presence of $Mg_{15}Zn_5Gd_4$ phase. At the 4 passes of extrusion, the $Mg_{15}Zn_5Gd_4$ phases are broken into fine particles with the size about 3 μm. The forced strain gradient near $Mg_{15}Zn_5Gd_4$ phase particles forms a region of dislocation density and large orientation gradient, which promotes the nucleation of recrystallization[19]. The process is called particles-stimulated nucleation (PSN). Through these different microstructure characteristic, it is shown that twinning mechanism, PSN and DRX process play the dominant role in microstructure evolution in ECAP process.

So as to achieve the effect of grain refinement (typically shown in Fig.5), Fig.5 presents the EBSD observation results of the Mg-1Zn-1Gd alloy grain boundary misorientation map at extrusion 8 passes. Upon 8 passes, there are a large number of low angle grain boundaries adjacent to the large angle grain boundaries, which indicate that massive volume fraction of grains are refined during ECAP. They are rarely found serrated, and most of them are smooth.

2.3 Texture evolution

The grain orientation information about the Mg-1Zn-1Gd alloy by ECAP is characterized by EBSD, as shown in Fig.6. Fig.6a and 6b show the microstructure evolution of the Mg-1Zn-1Gd alloy. The high angle grain boundaries and the low angle grain boundaries are shown by black and white color lines, respectively. In the map, some bulky coarse unDRXed grains and fine equiaxial dynamic recrystallized grains are observed. A large number of low angle grain boundaries are distributed around the unrecrystallized zones at 4 passes, but massive equiaxial crystal grains appear in Fig.6b. It can be seen from the IPF map of extrusion from 4 passes to 8 passes that all coarse original grains are converted into fine grains in the cone die during extrusion. From the (0001) pole figure, a single peak basal texture with a basal pole tilted to TD is obtained, which results in the activation of <*c*+*a*> pyramidal slip systems[20]. At 8 passes, owing to CRSS concentrated at twin boundaries and grain boundaries, it provides a driving force for DRX. Therefore, the non-basal dislocations are consumed and LAGBs transform into HAGBs, which means that new fine grains form, eventually changing the basal pole to TD.

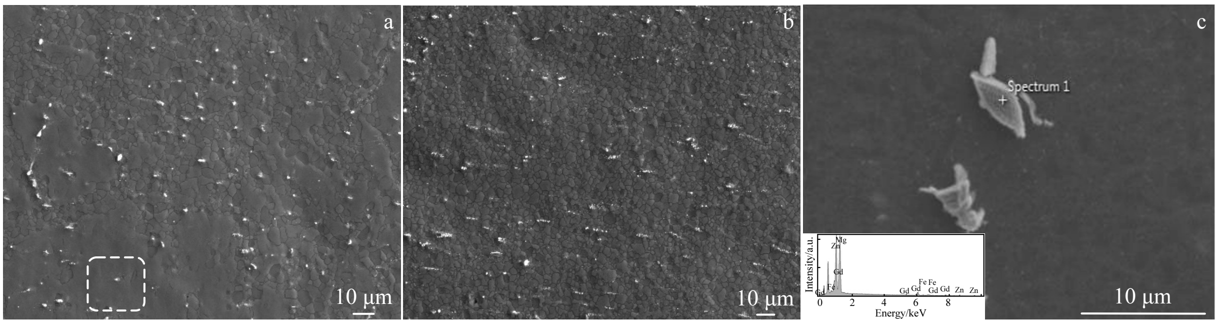

Fig.4 SEM images of Mg-1Zn-1Gd alloy deformed in extrusion to 4 passes (a) and 8 passes (b); (c) particles and EDS results of point marked in Fig.4a

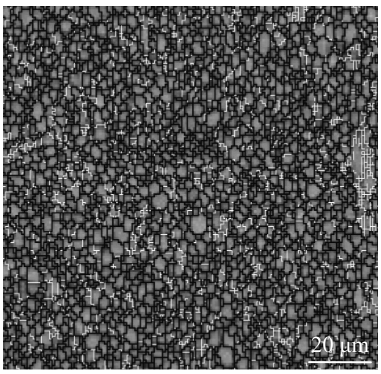

Fig.5 EBSD results of Mg-1Zn-1Gd at extrusion 8 passes corresponding TO boundary misorientation map

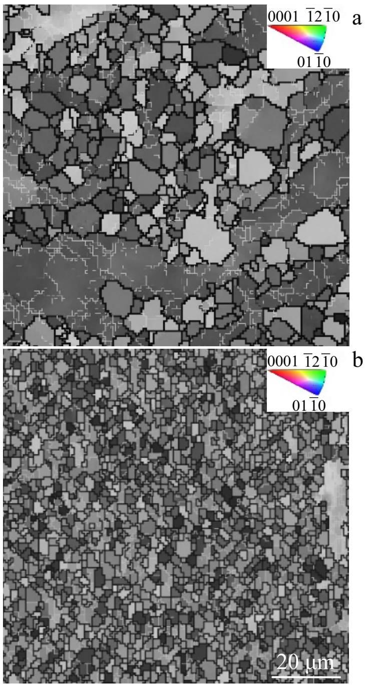

Fig.6 EBSD maps of Mg-1Zn-1Gd alloy after 4 passes (a) and 8 passes (b)

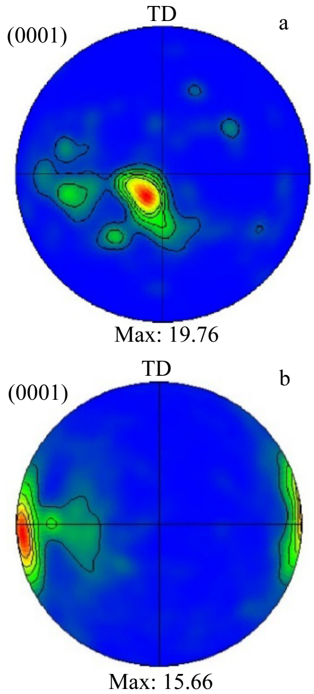

Fig.7 (0001) pole figures of dynamic recrystallized grains after 4 passes (a) and 8 passes (b)

In order to clearly understand the texture evolution, the (0001) pole figures with DRX regions are presented in Fig.7a and 7b. The weakening phenomenon of texture is observed during the dynamic recrystallization. In this case, increasing the extrusion passes results in faster recrystallization kinetics. When the Mg-1Zn-1Gd alloy is extruded from 4 passes to 8 passes at 350 °C, it has a recrystallized area fraction of about 98%, which is much higher than 63% of sample at 4 passes. The higher degree of dynamic crystallization and the larger recrystallized area fraction lead to weaker texture. The sample at 8 passes is fully recrystallized with a large number of fine grains (shown in Fig.6). Therefore, it exhibits a stronger basal texture in the alloy upon ECAP 4 passes. The maximum texture intensity decreases from 19.76 mrd to 15.66 mrd with increasing the extrusion passes. There is another possibility that the strength of texture is related to the grain size. The grain size decreases from 9.8 μm to 3.6 μm on average upon extrusion from 4 passes to 8 passes, leading to weak texture. From Fig.7a, it can be seen that the (0001) basal planes are mainly vertical to CD (center of the circle). With increasing the extrusion passes, the (0001) basal planes change apparently. In Fig.7b, basal plane of the DRX grains is mainly parallel to the CD, and it appears with larger range. There is a possibility to explain the strong texture evolution: the plastic deformation is mainly the activation of slip system, i.e. prismatic compensation, leading to formation of distinct texture. This means that the more extrusion passes containing suitable imposed strain, the more homogeneous the deformation, and the weaker the development of anisotropy and texture.

From the misorientation angle distribution in Fig.8a and 8b, after the 4 passes, the sharp peak represents a large number of low angle boundaries (<5°) and a few high angle boundaries are characterized in the misorientation distribution map. The high peak fraction at the low angles of 5° implies that a large number of subgrain structures exist in grains in Fig.8a. Meanwhile, there are also some apparent intensity peaks about the angles from 30° to 60° in Fig.8b, which indicate that the angle peaks are related to the volume fraction of recrystallization, since the about 30° peak is a near-coincidence site lattice boundary ∑15a with a misorientation angle about 30° in the magnesium[21]. This is related to the weakening of texture in the process of grain growth. As the extrusion pass increases, the fraction of high angle boundaries increases effectively and that of low angle boundaries decreases from 0.3% to 0.06%. The low misorientation angles of <10° are the indication of subgrain structure[22]. The subgrain boundary of Mg-1Zn-1Gd alloy at 4 passes is more than at the 8 passes, indicating that with increasing the extrusion passes, the release of strain energy can promote the grain transformation effectively, and the new grains emerge by the transformation of low angle boundaries into high angle boundaries. It can also prove that the DRX grains in Mg-1Zn-1Gd alloy at 8 passes have more random orientations. The result corresponds with the analysis of original microstructures evolution.

The typical distribution line charts of grain size for Mg-1Zn-1Gd alloy is shown in Fig.9. Upon 4 passes, there is a

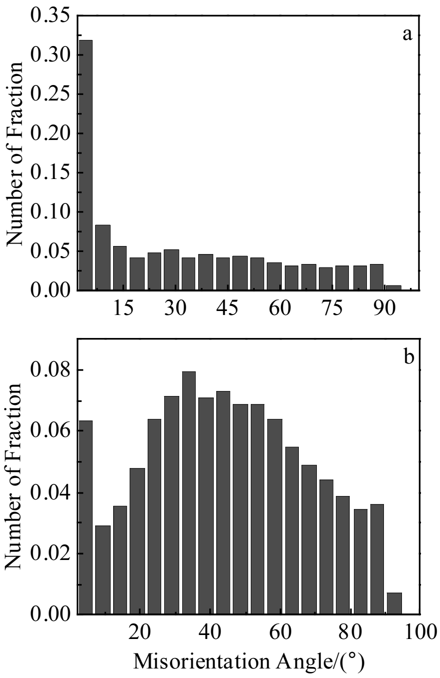

Fig.8 Misorientation angle distributions after 4 passes (a) and 8 passes (b)

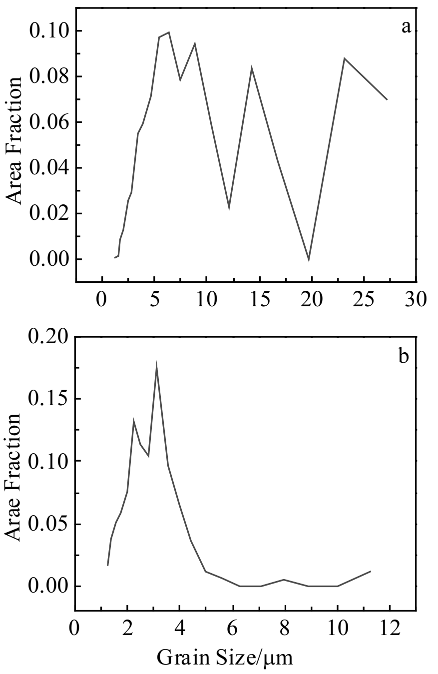

Fig.9 Grain size distribution line charts after 4 passes (a) and 8 passes (b)

bimodal distribution in the grain size fraction diagram of microstructure. The area fraction of fine grains is only 10% of 6.5 μm and 9.4% of 9.4 μm. After 8 passes (in Fig.9b), the average grain size can be refined to 3.6 μm, the fine grain fraction is improved to the maximum, and the peak distribution is between 2 and 4 μm. It means that after the 8 passes, the sample is completely recrystallized. With increasing the ECAP passes, the samples from the coarse to the fine grains are efficiently refined and the number of the fine grains ascends steeply. It can be seen in Fig.6b that the grains are more homogenous and become equiaxed grains.

3 Conclusions

1) The microstructure of Mg-1Zn-1Gd (wt%) alloy can be effectively refined by ECAP with increasing the extrusion passes. After 8 passes, the best result of grain refinement can be obtained, which is attributed to grain fragmentation and nucleation in dynamic recrystallization taking place during the extrusion.

2) The discontinuous dynamic recrystallization (DDRX) and continuous dynamic recrystallization (CDRX) are the most important mechanisms for grain refinement. The DRX degree of sample increases with increasing the extrusion passes, and the second phase is more dispersed with the increase of passes.

3) The highly refined microstructures lead to the weakening of texture (the basal texture intensity decreases with the grain refinement). All the samples have different texture evolution characteristics. At 4 passes, the (0001) basal planes are mainly vertical to CD (center of the circle). While at 8 passes, basal plane of the DRX grains are mainly parallel to the CD, and its distribution in the CD direction is greater. This is mainly the result of synergistic effect of dynamic recrystallization and cumulative strain of dynamic crystallization in the process of ECAP.

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Mg-1Zn-1Gd 合金在等径角挤压下的晶粒细化机理及织构演变

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摘 要：研究等径角挤压(ECAP)对 Mg-1Zn-1GD 合金组织、织构和动态再结晶行为的影响。结果表明，在 350 ℃等径角挤压后，试样的显微组织由细小的再结晶晶粒组成，基体中有大量均匀分布的等轴晶。8 道次后获得平均晶粒尺寸为 3.6 μm 的均匀超细晶粒结构。不连续动态再结晶(DDRX)和连续动态再结晶(CDRX)导致晶粒细化。利用电子背散射衍射（EBSD）技术对织构进行了分析，发现经过 4 道次 ECAP 后，形成了较强的基面织构(多重随机分布~19.76)。随着挤压道次的增加，挤压合金板材基面上的晶粒主要沿挤压方向拉长，取向分布由集中状态向分散状态转变。织构减弱，最大值为 15.66，且织构与 Mg-1Zn-1GD 合金的平面各向异性有关。

关键词：Mg-1Zn-1GD 合金；等径角挤压；晶粒细化；动态再结晶；织构

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