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Effect of predeformation on semi-solid microstructure of ZK60+RE magnesium alloy

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Abstract: Microscopical techniques were used to provide the semi-solid microstructure evolutions of ZK60+RE alloys formed by compression and equal channel angular extrusion(ECAE), respectively. It is found that after compression and ECAE, as-cast microstructures exhibit an obvious directional characteristic. The predeformation exerts a significant influence on the formation of thixotropic microstructures during partial remelting. Coalescence and Ostwald ripening are operative in the semi-solid mixture for both compression and ECAE formed alloys. Furthermore, the degree of spheroidization of ECAE formed alloy is better than that of compression formed alloy in appearance.

Key words: microstructure; ZK60+RE magnesium alloy; semi-solid state; coalescence; Ostwald ripening

1 Introduction

Semi-solid processing offers numerous advantages over conventional technologies, such as the reduction of solidification shrinkage, low processing temperature and prolonged die life[1]. The key factor controlling product properties is the spheroidized, non-dendritic microstructure that behaves thixotropically and can be formed to the net shape[2−3]. Therefore, the major effort for semi-solid processing is focused on the generation of spheroidized grains.

The spheroidized structure of semi-solid processing can be achieved by the strain-induced melt activation (SIMA) process. The SIMA process involves severe hot working, producing directional grain structure in the alloy. A critical level of strain must be introduced into the alloy prior to heating to above the solidus temperature[4]. It has been demonstrated that the spheroidized microstructure of alloys suitable for subsequent semi-solid processing strongly depends on predeformation[5−8]. Therefore, it is important to study how the predeformation affects the microstructure evolution in the semi-solid state, i.e. the mechanisms of grain growth and the degree of spheroidization.

In the present work, the microstructure evolutions of ZK60+RE magnesium alloys formed by compression and equal channel angular extrusion (ECAE) during isothermal holding in the semi-solid state are examined, respectively. Furthermore, the effects of compression and ECAE on the mechanisms of grain growth and the degree of spheroidization are also discussed.

2 Experimental

High purity element Mg (99.95%) and Zn (99.9%), and Mg-30Zr (mass fraction, %) master alloy were employed in the experiment. Rare earth (RE) elements in the form of master alloy were added to the melt at 760 ℃. After complete mixing, the melt was kept at 750 ℃ for 15 min. Then, the melt was poured into a steel die kept at room temperature. Protective atmosphere was composed of SF_6 and CO_2 (mixing volume ratio was 1:100). Chemical compositions, measured with an inductively coupled argon plasma spectrometer, were 5.29%Zn, 0.77%Zr, 1.14%Nd, 0.51%La, 0.28%Pr and Mg balance (mass fraction). The solidus and liquidus temperatures of ZK60+RE magnesium alloy were examined to be 442 ℃ and 636 ℃, respectively, from a differential scanning calorimetry(DSC) experiment at 2 ℃/min.

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Some as-cast ZK60+RE ingots of *d*58 mm×60 mm were compressed into approximate 36 mm in height at 350 ℃ in a hydraulic press with the compression ratio of 40% (corresponding equivalent strain of about 0.511). As for ECAE die, both the included angle and the curvature angle of the outside corner were 90˚. Before ECAE, as-cast ZK60+RE ingots of *d*58 mm×120 mm were preheated at 350 ℃ for 60 min. During extrusion, the die was first set to 350 ℃; then the ingots were inserted into the entrance channel. The ingots were rotated counterclockwise along the exit extrusion axis by 90˚ between each pass, the so-called route Bc[9]. The ingots were extruded for four passes, which gave the cumulative equivalent strain of about 3.628[9].

The isothermal holding experiments were preformed in a vertical tube furnace under the protective atmosphere of flowing argon. Samples of *d*12 mm×15 mm were heat treated isothermally at 615 ℃ for 10−40 min. According to the Scheil equation[10], solid fraction is 0.65 at 615 ℃. To preserve the morphology and content of the unmelted fraction, in removal from the furnace, samples were immediately quenched in cold water. Heat-treated samples were polished and then etched in 4%HNO_3 aqueous solution at room temperature. The metallographic observation was carried out by optical microscope(OM).

3 Results and discussion

3.1 Characteristics of compressed alloys and ECAEed alloys

The optical micrographs of compressed alloys and ECAE formed alloys are shown in Fig.1. Fig.1(a) indicates that the α-Mg grains oriented themselves in the direction vertical to the compressive direction and exhibited an obvious directional characteristic. Fig.1(b) shows an obvious directional band structures after ECAE.

3.2 Effect of predeformation on microstructures after isothermal heat treatment

Figs.2(a)−(d) show the optical micrographs of compression formed ZK60+RE alloy heat treated at 615 ℃ for 10, 20, 30 and 40 min, respectively. Compared with Fig.1(a), the compression deformed microstructures disintegrated into many strain-free grains with polygon boundaries, which proved the occurrence of recrystallization (Fig.2(a)). These strain-free grains nucleated and grew in the expense of strained grains during heating. With increasing the isothermal holding time up to 20 min, it was found that some solid particles coalesced, which led to the formation of entrapped liquid pools (Fig.2(b)). At 30 min, the grain boundary liquid film became thicker, therefore, the grain spheroidization was activated. Some small grains suspending in liquid phase gradually melted and leaved behind themselves a pool of liquid (Fig.2(c)). When the isothermal holding time was further prolonged to 40 min, most of polygonal grains evolved into near-spheroidal grains (Fig.2(d)). Figs.3(a)−(d) show optical micrographs of ECAE formed ZK60+RE alloys heat treated at 615 ℃ for 10, 20, 30 and 40 min, respectively. Grains in the ECAE formed alloy were fine and equiaxed in 10 min isothermal holding. Grains were uniformly surrounded by liquid film, which indicated that coarsening in ECAE formed alloy was mainly controlled by diffusion through the liquid rather than by solid-solid contracts. After 10 min of isothermal holding, the grain boundary liquid film became thicker and the degree of spherodization of microstructure was improved.

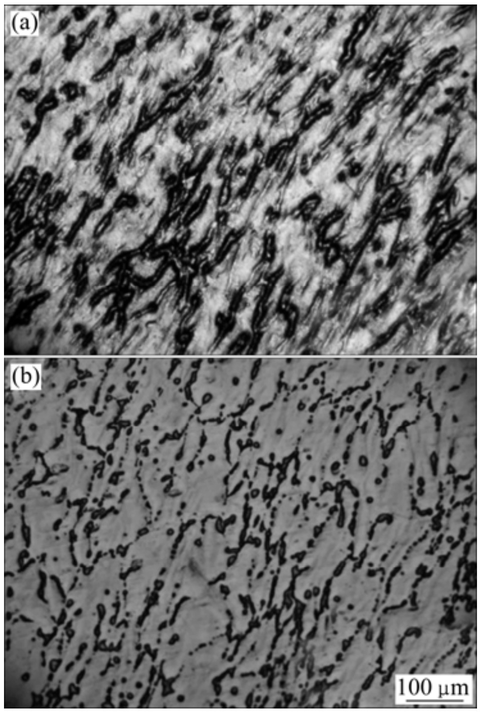

Fig.1 Optical micrographs of ZK60+RE magnesium alloy: (a) After compression; (b) After ECAE

It is found from Fig.4 that, with increasing holding time, the solid fraction decreased for both compression formed sample and ECAE formed sample. In the present work, the practical values of solid fractions measured by image analysis for compressed samples were 0.92, 0.83, 0.79 and 0.74 for 10 min, 20 min, 30 min and 40 min isothermal holding, respectively. For ECAE formed samples, the practical values of solid fractions were 0.81, 0.79, 0.76 and 0.72, respectively, when holding time increased from 10 min to 40 min. According to the Scheil equation[10], the calculated solid fraction was only 0.65. Two possible explanations are proposed for the difference between calculated solid fractions and

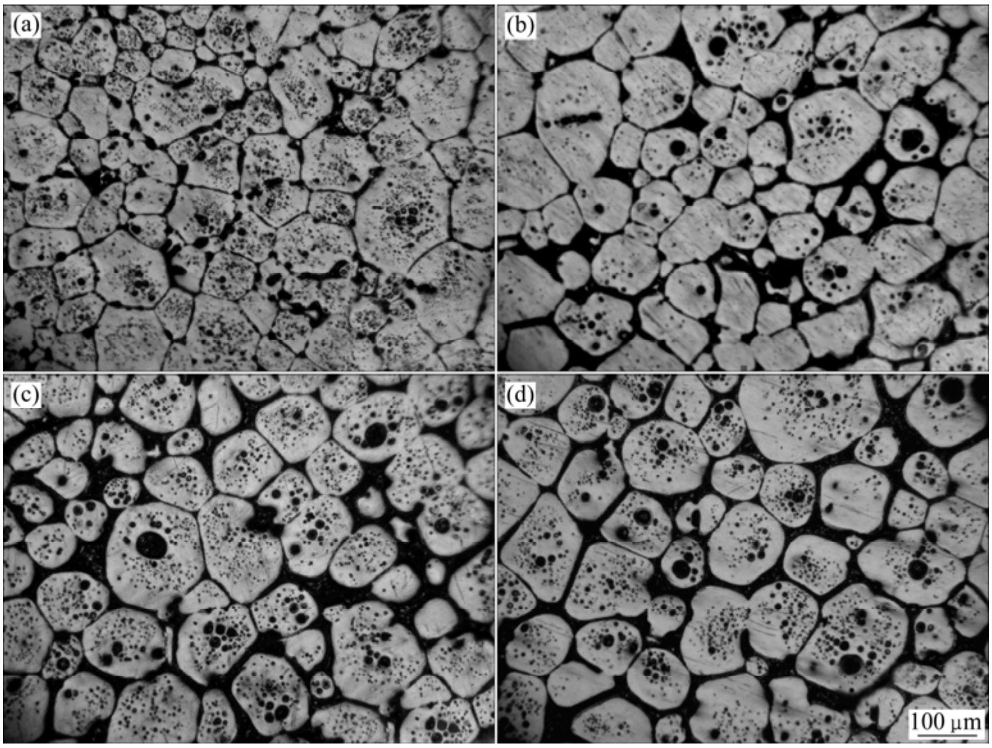

Fig.2 Optical micrographs of compression formed ZK60+RE alloys heat treated at 615 ℃ for different time: (a) 10 min; (b) 20 min; (c) 30 min; (d) 40 min

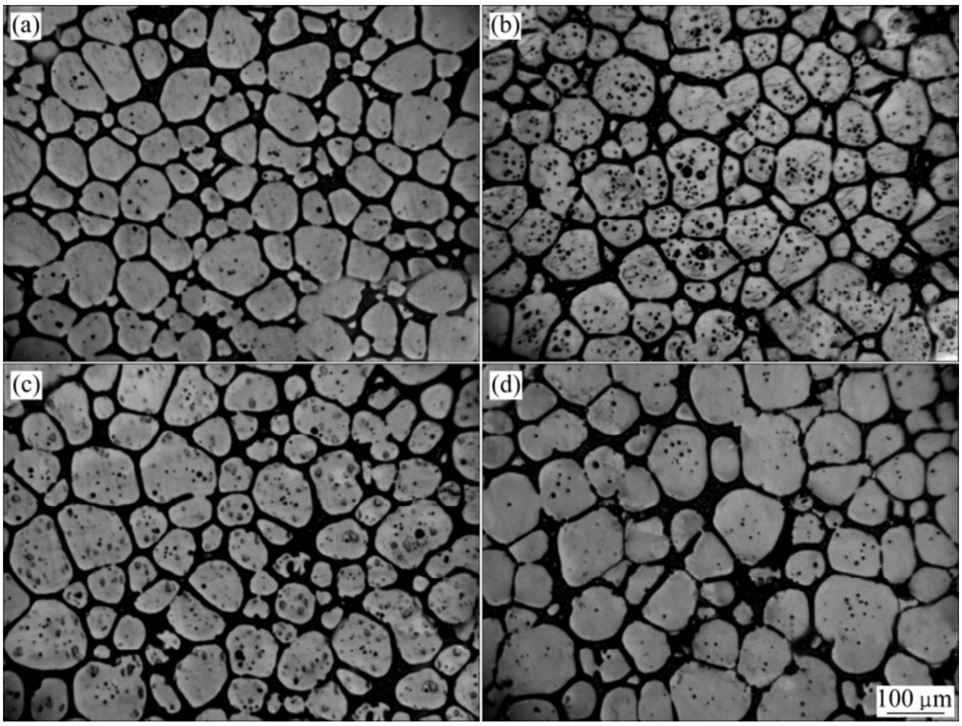

Fig.3 Optical micrographs of ECAE formed ZK60+RE alloys heat treated at 615 ℃ for different time: (a) 10 min; (b) 20 min; (c) 30 min; (d) 40 min

measured solid fractions. One is that the solid-liquid diffusion equilibrium was not reached because there was not long enough holding time in the present work. According to Fig.4, the solid fraction was still changed after the samples were held for 30 min, which demonstrated that liquid and solid phase were in the state of dynamic non-equilibrium. The other is that Zr and RE elements were not taken into account by the Scheil equation.

Fig.5 shows the effects of holding time on the equivalent diameter and the shape factor of solid particles. As shown in Fig.5, with increasing holding time from 10 min to 40 min, for both compression formed

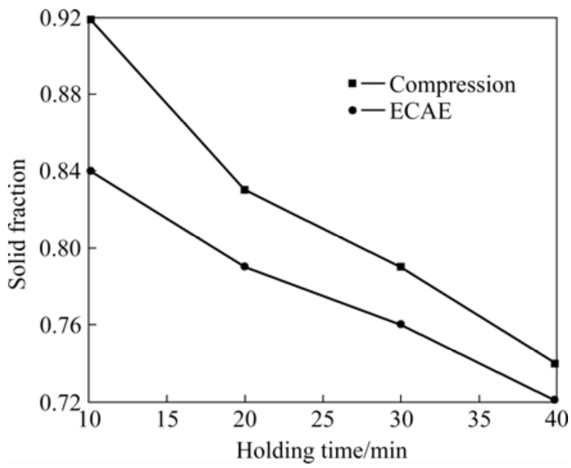

Fig.4 Plot of solid fraction vs holding time for compression formed alloys and ECAE formed alloys

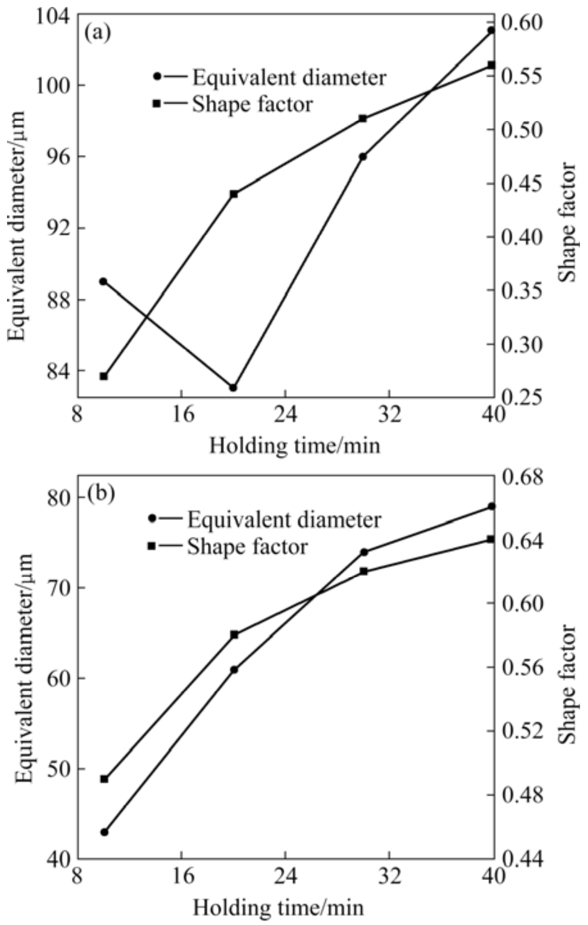

Fig.5 Effects of holding time on equivalent diameter and shape factor of solid particles in semi-solid ZK60+RE alloys: (a) After compression; (b) After ECAE

alloys and ECAE formed alloys, the shape factor increased, which indicated that increasing holding time promoted the degree of spheroidization. In addition, the degree of spheroidization for ECAE formed alloys was better than that for compression formed alloys under the same isothermal holding condition. Fig.5 also shows the variations of the equivalent diameter with the holding times for the compression and ECAE formed alloys. As shown in Fig.5(a), in the compression formed alloys, the equivalent diameter decreased initially, and then increased, as the holding time increased. However, the ECAE formed alloys showed a continuous increase of the equivalent diameter as the holding time increased. For the compression formed alloys, in the early stage of isothermal holding (10 min), because of relatively lower rate of liquid formation, solid particles were not totally wetted and surrounded by liquid films, which resulted in the solid-solid contacts and the formation of aggregates. With increasing holding time, liquid phase gradually infiltrated into grain boundaries and aggregates became separated into individual solid particles. Therefore, the equivalent diameter decreased initially during the holding. For the ECAE formed alloys, solid particles were uniformly surrounded by liquid films in the early stage of isothermal holding (10 min). With increasing holding time, solid particles were coarsened by grain coalescence and Ostwald ripening mechanisms.

In SIMA alloys, the final equiaxed microstructure is the result of fragmentation of a heavily deformed initial dendritic microstructure by liquid penetration at incipient melting in the high-energy grain boundaries formed in the alloy during the process[11−12]. The degree of predeformation exerts a significant influence on the initial microstructure during isothermal holding in the semi-solid state. According to the above results, the initial grain size of ECAE formed alloys is finer than that of compression formed alloy in the semi-solid state. This can be attributed to the different amount of energy stored in the alloys. In this work, the ECAE formed alloys have experienced the four-pass ECAE process and therefore much strain energy is stored in ECAE formed alloys, compared with compressed alloy with only 40% compression ratio. The stored energy provides the driving force for recrystallization. The initial recrystallized grain size during partial remelting depends mainly on the amount of stored energy. The more the amount of stored energy, the smaller the recrystallized grain size. By comparing Fig.2(a) with Fig.3(a), it can be found that the rate of liquid formation of ECAE formed alloy is faster than that of compression formed alloy. The reason might be analyzed as follows. During reheating, the liquid of eutectic firstly occurs at grain boundaries with high distortion energy. Compared with compression formed alloys, because of severe plastic deformation during ECAE, ECAE formed alloys have higher distortion energy, which enhances the rate of liquid formation. The rate of liquid formation determines the thickness of liquid film, which further determines the mechanism of grain growth in the semi-solid state. It is well established that two mechanisms are generally considered to control grain coarsening in the semi-solid state: Ostwald ripening and grain coalescence[13−15]. Grain coalescence is dominant in short time after liquid is initially formed. When liquid film is thin, coarsening in alloys is mainly controlled by coalescence, which leads to an increase in grain growth rate. With prolonging isothermal holding time, liquid film becomes thicker and therefore coarsening in alloys is mainly controlled by diffusion through the liquid rather than by coalescence. In this case, Ostwald ripening is activated. Because the rate of liquid formation is relatively low and liquid film is thin, the grain growth in compression formed alloy follows coalescence in the early stage of isothermal holding and then Ostwald ripening in further isothermal holding. However, as for ECAE formed alloys, because of high rate of liquid formation, relatively thick liquid films surrounding solid particles reduce the contribution of coalescence ripening to the total coarsening mechanism. According to Ref.[11], Ostwald ripening mechanism driven by the presence of variable curvature on the surface of each grain contributes to a large extent to grain spheroidization. In other words, Ostwald ripening mechanism promotes the spheroidization of grains. Therefore, the grains of ECAE formed alloy are more spheroidal in appearance than those of compression formed alloy under the same isothermal holding condition.

4 Conclusions

1) After the compression process and the ECAE process, as-cast microstructures exhibit an obvious directional characteristic.

2) With increasing holding time from 10 min to 40 min, for both compression and ECAE formed alloys, the shape factor increases. The degree of spheroidization for ECAE formed alloys is better than that for compression formed alloys under the same isothermal holding condition. Furthermore, the rate of liquid formation of ECAE formed alloy is faster than that of compression formed alloy.

3) In the compression formed alloys, the equivalent diameter decreases initially, and then increases, as the holding time increases. However, the ECAE formed alloys show a continuous increase of the equivalent diameter as the holding time increases.

4) The ECAE formed alloys are better suited for semi-solid forming than the compression formed alloys.

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