Microstructure Evolution and Grain Coarsening Behaviour During Partial Remelting of Cyclic Extrusion Compression Formed AZ61 Magnesium Alloy

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The effects of CEC passes, isothermal holding time and reheating temperature on the microstructure evolution and grain coarsening behaviour of AZ61 magnesium alloy produced by the recrystallisation and partial melting (RAP) process were investigated. Before partial remelting, as-cast AZ61 alloy was deformed by cyclic extrusion compression (CEC) with one pass and two pass at 330 °C. After CEC, the microstructure consisted of unrecrystallized grains and deformed eutectic compounds. Increasing isothermal holding time resulted in the formation of spheroidal grains surrounded by liquid films. With increasing the isothermal holding time, the solid grain size increased and the degree of spheroidization was improved. With increasing the reheating temperature, namely increasing liquid fraction, the solid grain size obviously decreased during the period from 560 °C to 570 °C and then slightly increased after 570 °C, while the shape factor increased monotonously. During partial remelting, increasing reheating temperature can properly shorten the isothermal holding time to obtain fine structure. Moreover, increasing the numbers of CEC passes could produce finer semi-solid microstructure. The coarsening behavior of solid grains in the semi-solid state obeys Ostwald ripening and grain coalescence mechanisms. The coarsening rate constant, K , was $80 \mu\text{m}^3 \cdot \text{s}^{-1}$ for samples partially remelted at 595 °C. After CEC plus partial remelting, the ideal and fine semi-solid state structure can be obtained, which was suitable for thixoforming.

KEY WORDS: AZ61 magnesium alloy; Cyclic extrusion compression; Microstructure evolution; Grain coarsening

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1. Introduction

Magnesium alloys have received much attention due to their high specific strength and stiffness, superior damping capacity, high thermal conductivity, high dimensional stability, good electromagnetic shielding characteristics and excellent machinability^[1–4]. Most magnesium alloy products are processed by high pressure die-casting (HPDC)^[5–7]. However, HPDC components suffer from low

mechanical properties due to the existence of inherent defects, such as porosity, hot cracks and oxide inclusions^[8–10]. Furthermore, those defects not only limit their use in safety-critical applications but also deny the opportunity for further hardening by heat treatment^[11,12]. So an effective tactic to minimize these disadvantages is to develop magnesium alloys with fine-grained microstructures. It has been recognized that semi-solid processing offers significant advantages over casting routes, such as less entrapped air, reduction on macrosegregation, high dimensional precision and prolonged die life^[13–18]. It has been demonstrated that the spheroidized microstructure of alloys suitable for subsequent semi-solid processing^[19–21] and the globular structure of solid can significantly enhance the mechanical

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properties of material, so the key factor for controlling product engineering performance is the spheroidized, non-dendritic microstructure that behaves thixotropically and can be formed to the net shape^[22,23].

The required globular microstructure can be obtained by the recrystallisation and partial melting (RAP) process. In the RAP process, material is deformed by extrusion at temperatures below the recrystallisation temperature and then reheated into the semi-solid state. During the RAP process, severe plastic deformation (SPD) processing is helpful for the formation of fine and spheroidal grains during partial melting. SPD techniques include equal channel angular pressing (ECAP), high-pressure torsion (HPT), accumulative roll-bonding (ARB), multi-axial forging (MAF) and cyclic extrusion compression (CEC), *etc.* Among these SPD techniques, the CEC process is very suitable for a RAP process. On the one hand, the interface size of billets remains constant after CEC. On the other hand, it is beneficial to get fine and spheroidal grains during partial remelting due to the high accumulated equivalent strain^[24–28].

The aim of present work is to employ the CEC process to predeform an AZ61 alloy. The effects of CEC passes, isothermal holding time and reheating temperature on microstructure evolution were investigated. Moreover, grain coarsening behaviour of CEC-formed AZ61 alloy during partial remelting was also discussed.

2. Experimental

The alloy used in the present study was Mg-Al-Zn alloy, AZ61, with the measured composition (wt.%) of 5.9Al, 0.9Zn and 0.21Mn. The principle of the cyclic extrusion compression (CEC) process^[29] is represented schematically in Fig. 1. In the CEC process, a sample was contained within a chamber and then extruded repeatedly backwards and forwards. This process was invented to allow arbitrarily large strain

Fig. 1 Schematic of cyclic extrusion compression (CEC)

deformation of a sample with preservation of the original sample shape after n passes. The accumulated equivalent strain ε was approximately given by

$$\varepsilon = 4n \ln\left(\frac{D}{d}\right) \quad (1)$$

where D is the chamber diameter, d is the channel diameter and n is the number of deformation cycles. Since the billet in the CEC process was compressed from the both ends, a high hydrostatic pressure was imposed. In the present study, the CEC process was conducted at 330 °C with the ingots coated in molybdenum disulphide (MoS_2) as a lubricant and used a hydraulic press operating at a pressing of 25 mm/min, the chamber diameter (D) and the channel diameter (d) were 80 mm and 40 mm, respectively. The ingots were extruded for one-pass and two-pass, which gave accumulated equivalent strain of 2.77 and 5.54, respectively.

The isothermal holding experiments were performed in a vertical tube furnace under the protective atmosphere of flowing argon. Samples were partially remelted isothermally at various temperatures for 10–40 min. To preserve the morphology and content of the unmelted fraction, in removal from the furnace, samples were immediately quenched in cold water. Care was taken to ensure that the beginning time of isothermal holding (0 min) was defined as the time when the samples were heated to predefined temperature without holding partially remelted. The samples were sectioned at a sufficient depth from the surface to avoid errors in the microstructural analysis due to anomalous coarsening of grains at the surface^[30]. Mean grain size and shape factor of solid grains were calculated in each case by using

$$d = \frac{\sum_{N=1}^N \sqrt{4A/\pi}}{N} \quad (2)$$

$$F = \frac{\sum_{N=1}^N \frac{4\pi A}{P^2}}{N} \quad (3)$$

where A and P are area and perimeter of grains respectively and N is the number of grains. For each sample, measurements were taken from the whole sectioned area with 400–500 grains per sample^[31–34].

The approximate relationship between solid fraction f_s and temperature (T) was determined using the Scheil equation, which assumes that homogenisation of the liquid was complete, with no diffusion in the solid^[35]:

$$f_s = 1 - \left(\frac{T_M - T_L}{T_M - T}\right)^{\frac{1}{1-k_0}} \quad (4)$$

where T_M is the melting point of pure metal, T is the temperature of isothermal holding, T_L is the liquidus temperature of alloy and k_0 is the equilibrium distribution coefficient. In this study, T_M , T_L and k_0 were taken as 650 °C, 620 °C and 0.36, respectively. Therefore, the expected solid fraction (f_s) of AZ61 alloy was 0.61 at 595 °C in this study.

3. Results and Discussion

3.1 Microstructures of as-cast and CEC-formed AZ61 magnesium alloys

Fig. 2 shows the microstructures of as-cast and CEC-formed AZ61 magnesium alloys, the XRD pattern taken from as-cast AZ61 magnesium alloy is shown in Fig. 3. XRD analysis indicated that as-cast AZ61 magnesium alloy consisted of α -Mg matrix and β -Mg₁₇Al₁₂ phase. As shown in Fig. 2(a), the β -Mg₁₇Al₁₂ phase precipitated as discontinuous network at grain boundaries. The presence of eutectic in the microstructure was attributed to non-equilibrium solidification caused by the fast cooling rate of conventional casting process. As shown in Fig. 2(b), the microstructure was subjected to severe plastic deformation and the boundaries of the grain were not clear. Brittle β -Mg₁₇Al₁₂ phase were elongated and distributed over the grain boundaries like strings. With increasing numbers of extrusion passes, brittle and hard β -Mg₁₇Al₁₂ gradually ruptured and distributed along grain boundaries, the microstructures became finer obviously along extrusion direction, as shown in Fig. 2(c).

3.2 Effect of CEC passes on CEC-formed AZ61 magnesium alloy during partial remelting

Fig. 4 shows the microstructures of CEC-formed AZ61 magnesium alloy partially remelted at 595 °C ($f_s=0.61$) for 10 min. Comparing Fig. 4(a) with Fig. 4(b), we could find that the grain size of two-pass CEC-formed alloy was finer than that of one-pass CEC-formed alloy during isothermal holding, the degree of spheroidization of two-pass CEC-formed alloy was better than that of one-pass CEC-formed alloy under the same isothermal conditions. During the CEC process, a fraction of external work remains in alloys and forms distortion energy. The distortion energy stored in alloys will provide the driving force to promote recovery and recrystallization during subsequent reheating^[36]. Many researchers^[37–39] found that with increasing numbers of extrusion passes, the distortion energy stored in the alloy non-linearly in-

creased, so increased the numbers of CEC passes could produce finer semi-solid microstructure, but as a semi-solid forming process, each additional pass would lead to great cost which we must consider.

3.3 Effect of isothermal holding times on CEC-formed AZ61 magnesium alloy during partial remelting

Fig. 5 shows the microstructures of one-pass ($\varepsilon_e = 2.77$) CEC-formed AZ61 magnesium alloy partially remelted at 595 °C ($f_s = 0.61$) for different isothermal holding times. According to Fig. 2(b), the microstructure of deformed AZ61 magnesium alloy was unrecrystallised. However, when the alloy has been heated up to 595 °C, recrystallisation occurred entirely. The microstructure in the semi-solid state consisted of fine and almost perfectly globular recrystallised grains. Grain boundaries were gradually penetrated by liquid due to the dissolution of the grain boundary eutectic phases, as shown in Fig. 5(a). As soon as liquid phase has occurred at grain boundaries, grain spheroidization and coarsening were activated simultaneously, as shown in Fig. 5(b). Note that significant solid coarsening had occurred in the semi-solid state after 30 min and the thickness of liquid film remarkably increased, as shown in Fig. 5(c). When holding time was extended to 40 min, the globular solid grains became more rounded, as shown in Fig. 5(d). Comparison of Fig. 5(a) and Fig. 5(d) indicated that recrystallised grains boundary liquid

Fig. 3 XRD pattern of as-cast AZ61 magnesium alloy

Fig. 2 Microstructures of as-cast and CEC-formed AZ61 magnesium alloys: (a) as-cast; (b) one-pass CEC; (c) two-pass CEC

Fig. 4 Microstructures of CEC-formed AZ61 magnesium alloy partially remelted at 595 °C ($f_s = 0.61$) for 10 min: (a) one-pass CEC; (b) two-pass CEC

Fig. 5 Microstructures of one-pass ($\varepsilon_e = 2.77$) CEC-formed AZ61 magnesium alloy partially remelted at 595 °C ($f_s = 0.61$) for 10 min (a), 20 min (b), 30 min (c) and 40 min (d)

film was thicker, excessive solid particle coarsening and the recrystallised grains became more spheroidal with increasing isothermal holding time. Grains were uniformly surrounded by liquid film, which indicated that coarsening in CEC formed alloy was mainly controlled by diffusion through the liquid rather than by solid-solid contracts.

Fig. 6 shows the effects of holding time on the size and the shape factor of solid grain as a function of holding time at 595 °C. As shown in Fig. 6, with increasing holding time from 10 min to 40 min, for both the solid grain size and the shape factor increased, which indicated that increasing holding time increased the equivalent diameter and promoted the degree of spheroidization.

For much diffusion controlled coarsening systems,

including solid/liquid mixtures, coarsening kinetics can be described by the Lifshitz, Slyozov and Wagner (LSW) theory^[24]:

$$L^n - L_0^n = Kt \quad (5)$$

where L is the grain size after time t and L_0 is the initial grain size, K is the coarsening rate constant, n is the coarsening exponent and t is the holding time. To evaluate coarsening kinetics of CEC formed AZ61 magnesium alloy, it was assumed in the present study that coarsening was controlled by diffusion in the liquid and was cubic, *i.e.* $n = 3$. The coarsening rate constant, K , in Eq. (5) was given experimentally by the gradient of the best fit straight lines shown in Fig. 7, and was $80 \mu\text{m}^3 \cdot \text{s}^{-1}$ for samples partially remelted at 595 °C ($f_s = 0.61$).

Fig. 8 shows the coarsening rate constant K vs. solid fraction f_s for AZ61 produced by the CEC process, together with results from some coarsening studies on other magnesium alloys and magnesium matrix composite. As shown in Fig. 8, slightly lower

Fig. 6 Plots of solid grain size and shape factor as a function of holding time for one-pass ($\varepsilon_e=2.77$) CEC-formed AZ61 magnesium alloy partially remelted at 595 °C ($f_s = 0.61$)

Fig. 7 Plot of equivalent grain size cubed as a function of holding time for one-pass ($\varepsilon_e = 2.77$) CEC-formed AZ61 magnesium alloy partially remelted at 595 °C ($f_s = 0.61$)

Fig. 8 Plot of coarsening rate constant as a function of solid fraction for AZ61 magnesium alloy in this study and other Mg alloys in other studies^[41,33,42].

coarsening rate ($80 \mu m^3 \cdot s^{-1}$ at $f_s = 0.61$) was observed for AZ61 produced by the CEC process in the present study than that for AZ61 produced by the SIMA process ($94.7 \mu m^3 \cdot s^{-1}$ at $f_s = 0.612$). This is because the values of accumulated equivalent strain are higher during the CEC process in the present study than that for AZ61 produced by the SIMA process, so the distortion energy during the CEC process is higher than the SIMA process. During the reheating process, liquid phase appears in very short time, the solid grains are surrounded by liquid films which inhibit the coalescence, so the coarsening rate constant is relatively lower during the CEC process than the SIMA process for AZ61 alloy. However, a relatively lower coarsening rate was observed for mechanical stirring process SiCp/AZ61 alloy ($27.6 \mu m^3 \cdot s^{-1}$ at $f_s = 0.695$) than for RAP process AZ61 alloy obtained in the present study. The significant difference in the coarsening rate constant must be attributed to the presence of SiC grains in AZ61 alloy. SiC grains with a relatively large radius enriched at the solid/liquid interface are responsible for hindering the mobility of solute atoms and the welding of contact grains and therefore reduce the coarsening rate constant. From Fig. 8 we can find that with increasing temperature, the coarsening rate constant of SIMA formed ZK60-RE and RAP-formed AZ91D-RE were both incremental. For SIMA-formed ZK60-RE, with the temperature increased from 600 °C to 615 °C, the coarsening rate constant, K , increased from 255 to $371 \mu m^3 \cdot s^{-1}$. For RAP-formed AZ91D-RE, with the temperature increased from 553 °C to 566 °C, the coarsening rate constant, K , increased from 167 to $201 \mu m^3 \cdot s^{-1}$. As for ZK60-RE and AZ91D-RE alloys, coarsening rate constants increased as the solid fraction decreased. So coarsening rate can be promoted by the decrease of solid fraction (increasing temperature). This finding is consistent with those of Atkinson and Liu^[40]. Atkinson and Liu^[10] suggested that coarsening rate constant not only to temperature but also to solid fraction, because diffusion is faster at higher temperature and liquid provides a much faster diffusion path than solid and once a continuous liquid path is present around the spheroids coarsening could accelerate.

3.4 Effect of reheating temperature on microstructure evolution of CEC-formed AZ61 magnesium alloy in the semi-solid state

Fig. 9 shows the semi-solid microstructures of CEC-formed AZ61 alloys after partial remelting at various temperatures of 560 °C, 570 °C, 580 °C and 590 °C with the holding time for 10 min. As shown in Fig. 9, 10 min isothermal holding at 560 °C was not enough for spheroidization. Solid grains were still stick together and the separation did not occur. This can be attributed to low liquid fraction and therefore solid grains were not completely penetrated by liquid

Fig. 9 Microstructures of one-pass ($\varepsilon_e=2.77$) CEC-formed AZ61 magnesium alloy partially remelted at 560 °C (a), 570 °C (b), 580 °C (c) and 590 °C (d) for 10 min

film. Increasing temperature to 570 °C resulted in the liquid fraction increased and the separation of contacted solid grains, some individual solid grains were subjected to spheroidization. However, a lot of solid grains still stucked together. When the reheating temperature increased to 580 °C, contacted solid grains were separated and solid grains were uniformly surrounded by liquid film. At 590 °C, solid grains were almost perfectly spheroidal, distribute in the liquid phase. The shape changed from polygonal to globular with increasing temperature.

Fig. 10 shows the variation of solid grain size and shape factor with reheating temperatures for CEC-formed AZ61 magnesium alloy during partial remelting. As shown in Fig. 10, the grain size decreased initially before 570 °C, and then a slight increasing as the isothermal reheating temperatures increased after 570 °C. When reheated the AZ61 magnesium alloy up to 560 °C just above the solidus, it was in its initial stage of recrystallisation process, so the amount of liquid was limited and some solid grains were still stick together. With increasing reheating temperature up to 570 °C, the amount of liquid phase much more and gradually infiltrated into grain boundaries, so the unrecrystallised structures touched liquid and the liquid tended to penetrate unrecrystallised structures causing fragmentation. Fragmentation resulted in the decreasing of average grain sizes below 570 °C. As shown in Fig. 10, the shape factor monotonously increased with reheating

Fig. 10 Plot of solid grain size and shape factor as a function of reheating temperature for one-pass ($\varepsilon_e=2.77$) CEC-formed AZ61 magnesium alloy

temperatures increased. The maximum value of shape factor was obtained at 590 °C, the minimum value of shape factor was obtained at 560 °C. It can be concluded that the degree of spheroidization strongly depends on liquid fraction. Therefore increasing reheating temperatures was beneficial for the formation of liquid, which promoted the degree of spheroidization. This attributed to the dissolution-reprecipitation mechanism driven by the presence of variable curvature on the surface of each solid grain. According to the results from some coarsening studies on other magnesium alloys and magnesium matrix composite, a similar tendency between reheating temperatures and shape factor can be obtained: the

Fig. 11 Plot of shape factor as a function of reheating temperature for AZ61 magnesium alloy in this study and other Mg alloys in other studies^[11,18,31,30]

shape factor consistently increased with increasing the reheating temperatures, as shown in Fig. 11.

The comparison of Fig. 5 with Fig. 9 indicated that prolonging isothermal holding time and increasing the reheating temperature were favorable for obtaining the finer solid grains due to the fragmentation of blocky structure. Furthermore, comparing Fig. 6 with Fig. 10, it was also found that both the longer holding time and the higher reheating temperature can promote the degree of spheroidization, and the reheating temperature should be as high as possible to shorten isothermal holding time for complete recrystallization. However, if the isothermal holding time was too long and the reheating temperature was too high, it was difficult for holding semi-solid feed stocks and the coarse solid grains were achieved^[11].

It is well established that two mechanisms are generally considered to control grain coarsening in the semi-solid state: Ostwald ripening and grain coalescence. Ostwald ripening is defined as two spheroidal grains which are very different in size merge into a larger spheroidal grain^[14]. Coalescence is usually defined as the formation of one large grain upon contact of two smaller spheroidal grains with similar size^[26]. When the reheating temperature was relatively low (560 °C) and the holding time was relatively short, liquid film was initially formed, the thickness of grain boundary liquid film was relatively thin and the amount of liquid phase was low, the distance of neighboring solid grains was short and the rate of atom diffusion was relatively low, which resulted in the low rate of fragmentation. Therefore, coarsening in the early stage of partial remelting was mainly controlled by coalescence, which lead to an increase in grain growth rate and resulted in the irregular solid grains with larger size (Fig. 5(a), Fig. 9(a) and Fig. 9(b)). With prolonging isothermal holding time and increasing temperature (decreasing solid faction), liquid film became thicker, therefore coarsening in alloys was mainly controlled by diffusion through the liquid rather than by coalescence. In this case, Ostwald ripening was activated

(Fig. 5(c) and Fig. 5(d), Fig. 9(c) and Fig. 9(d)). At the same time, the corners and edges of solid grains would be melted, which resulted in the improvement of the degree of spheroidization^[30]. In summary, the results indicated that the microstructure evolution could be divided into four stages: the recrystallisation of deformed microstructure, the fragmentation of recrystallized grains caused by liquid penetration, spheroidization due to partial remelting and final solid grain coarsening.

4. Conclusions

(1) The microstructure of AZ61 magnesium alloy during partial remelting could be improved effectively by CEC process. After CEC process, the microstructure consisted of unrecrystallised grains. After CEC plus partial remelting, the ideal and fine semi-solid state structure can be obtained.

(2) Increasing isothermal holding time resulted in the formation of spheroidal grains surrounded by liquid films. With increasing the isothermal holding time, the solid grain size and the degree of spheroidization monotonously increased. With increasing the reheating temperature, namely increasing liquid fraction, the solid grain size obviously decreased during the period from 560 °C to 570 °C and then slightly increased after 570 °C, while the shape factor increased monotonously. In the present study, increasing the reheating temperature was much better than prolong the isothermal holding time to obtain fine solid grain, so we can increase the reheating temperature properly to shorten the isothermal holding time. Moreover, increasing the numbers of CEC passes could produce finer semi-solid microstructure.

(3) The coarsening behavior of solid grains in the semi-solid state obeys Ostwald ripening and grain coalescence mechanisms. The coarsening rate constant, K , obtained was $80 \mu\text{m}^3 \cdot \text{s}^{-1}$ for samples partially remelted at 595 °C ($f_s = 0.61$).

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