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ScienceDirect
Trans. Nonferrous Met. Soc. China 20(2010) 178−182
Transactions of Nonferrous Metals Society of China
www.tnmsc.cn

Reheating and thixoforging of ZK60+RE alloy deformed by ECAE

ZHAO Zu-de(赵祖德)[1], CHENG Yuan-sheng(程远胜)[2], CHEN Qiang(陈 强)[1],
WANG Yan-bin(王艳彬)[1], SHU Da-yu(舒大禹)[1]

1. Southwest Technique and Engineering Institute, Chongqing 400039, China;
2. School of Materials Science and Engineering, Harbin Institute of Technology, Harbin 150001, China

Received 26 December 2008; accepted 16 April 2009

Abstract: The two-pass equal channel angular extrusion (ECAE) process was introduced into strain-induced melt activation (SIMA) to predeform a ZK60 alloy with rare earth (RE) addition. Microstructure evolution of ECAE-formed ZK60+RE alloy during reheating was investigated. Furthermore, tensile properties of thixoforged components were determined. The results show that the SIMA process can produce ideal microstructures, and spheroidized solid particles with little entrapped liquid can be obtained. With prolonging holding time, the size of solid particles increases and the degree of spheroidization is improved. The tensile properties of the thixoforged ZK60+RE samples are close to those of two-pass ECAE-formed samples.
Key words: magnesium alloy; reheating; thixoforging; equal channel angular extrusion (ECAE)

1 Introduction

Semi-solid processing has significant advantages over conventional casting, forging and powder metallurgy techniques[1]. Thixoforging, which belongs to thixoforming, is a semi-solid processing route. One main requirement of this process is that the starting material must be treated in such way that the microstructure of the remelted alloy is spheroidal rather than dendritic. The thixoforging process involves the preparation of a feedstock material with thixotropic characteristics, reheating the solid feedstock material and shaping the semi-solid billet to components[2].

There are various routes to achieve alloys with thixotropic microstructure, which are summarized in Ref.[3]. Strain-induced melt activation (SIMA) is one of solid state routes. This route involves working above the recrystallisation temperature followed by reheating to the semi-solid state[4]. On the formation of the liquid phase, high angle boundaries between solid grains will be penetrated, causing fragmentation; and therefore, globular solid particles can be achieved. Recently, JIANG and LUO[5] developed a new SIMA method, in which equal channel angular extrusion(ECAE) was used as the strain step. Compared with the common SIMA process, the new SIMA process could generate more spherical and finer grains.

Magnesium alloys are the lightest metallic structure materials and are very attractive in such applications as automotive, railway and aerospace industries[6]. Several researchers[7−9] have reported that rare earth (RE) additions can improve casting characteristics, high-temperature tensile strength and ambient tensile yield strength of magnesium alloys. Moreover, RE additions can result in the improvement of as-cast microstructure of magnesium alloy in the semi-solid state. LI et al[10] suggested that with the addition of 0.5% RE, the transformation from the primary phase to spheroid particle was accelerated, and the fine and symmetrical spheroid grains were gained during semi-solid isothermal treatment of magnesium alloy AZ91D.

Up to now, magnesium alloys currently used in semi-solid processing are restricted to a few commercial alloys such as AZ91D[11], AM50[12], and ZA84[13]. There is few report concerning the microstructure evolution and thixoforging of ZK60+RE magnesium alloys produced by the SIMA process. In the present work, the microstructure evolution of a ZK60+RE magnesium alloy produced by the SIMA process is studied. Furthermore, the thixoforging of the ZK60+RE magnesium alloy is also investigated.

2 Experimental

The material used was an as-cast ZK60+RE

Corresponding author: CHEN Qiang; Tel: +86-23-68792284; E-mail: 2009chenqiang@163.com
DOI: 10.1016/S1003-6326(09)60117-5

magnesium alloy with composition of Mg-5.3Zn-0.76Zr-1.24Nd-0.62La-0.32Pr (mass fraction, %). As shown in Fig.1, the solidus and the liquidus temperatures for the material were obtained to be 442 ℃ and 636 ℃ from a differential scanning calorimetry(DSC) experiment at heating rate of 2 ℃/min.

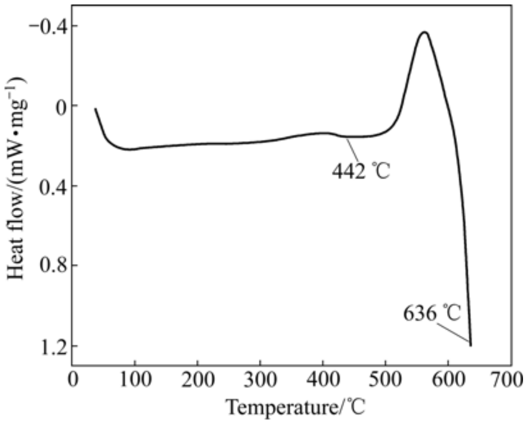

Fig.1 DSC heating curves of as-cast ZK60+RE magnesium alloy

The ZK60+RE magnesium alloy with 59 mm in diameter and 120 mm in length was processed at 300 ℃ by two ECAE passes according to route B_c (90° rotation about the workpiece axis between the passes). For the ECAE die, both the included angle Φ and the angle of curvature Ψ of the outside corner were 90°, which gave an equivalent strain of 0.907 per pass[14]. Molybdenum disulphide (MoS_2) was used as a lubricant. Samples cut from the ECAE-formed bar were heated and partially remelted in a steel pipe of a furnace at 589 ℃ and the temperature was monitored by means of a K-type thermocouple inserted about 2 mm into reference sample. According to the Scheil equation, the expected solid fraction was 0.8. During partial remelting, Ar was used as a protective atmosphere to prevent oxidization. On removal from the furnace, samples were rapidly quenched in cold water. Subsequently, they were polished and etched with 4% HNO_3 aqueous solution.

In the present work, thixoforging was carried out on a hydraulic press. Some cylindrical (d58 mm×40 mm) ingots were cut from the ECAE-formed bars of 59 mm in diameter for thixoforging. The reheating of ingot was carried out in an electrical resistance furnace. During reheating, Ar was used as a protective atmosphere to prevent oxidization. The ingot was rapidly heated to 589 ℃ and isothermally held at this temperature for 10 min. The temperature of the ingot was monitored by a K-type thermocouple embedded in the ingot. After the required temperature was reached, the thermocouple was removed and the heated ingot was transferred to the die. During thixoforging, the speed of the ram was 1 mm/s. The preheating temperature of the die was 400 ℃. The pressure exerted by the ram on the ingot was rapidly increased to 150 MPa, and kept at this level for 100 s.

Morphological and microchemical characterizations of samples were conducted with scanning electron microscope (SEM) equipped with an energy dispersive X-ray spectrometer (EDS). The shape factor was calculated from the following equation: $F=(4\pi A)/P^2$, where P is the perimeter and A is the area (F=1, sphere; $F\rightarrow 0$, needle). Grain sizes were measured using a mean linear intercept method. The mechanical properties of thixoforged ingots were measured using an Instron 5569 testing machine at a cross head speed of 1 mm/min.

3 Results and discussion

3.1 Microstructure of as-cast and ECAE-formed ZK60+RE alloys

Fig.2 shows SEM image and energy dispersive

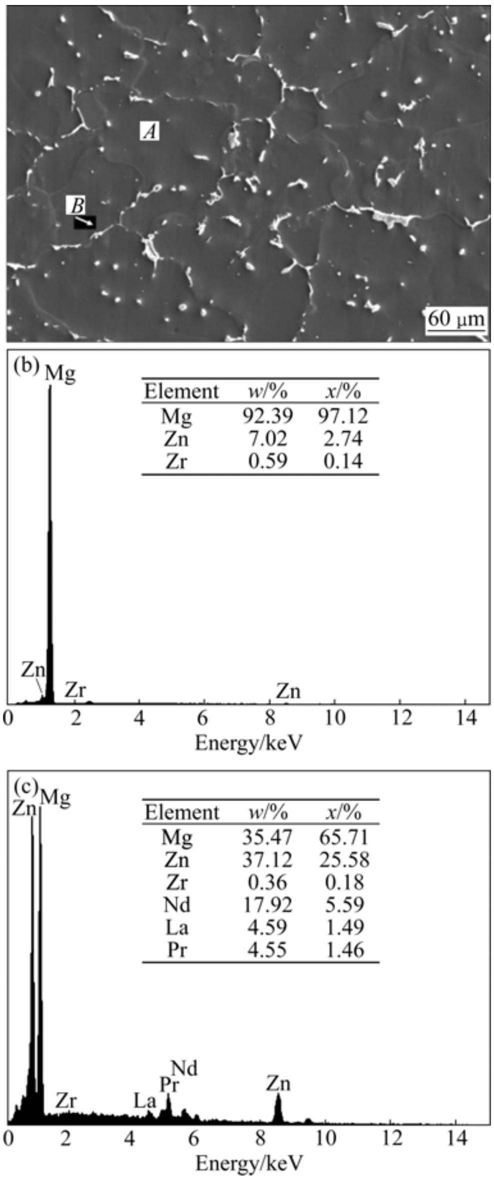

Fig.2 SEM image (a) of as-cast ZK60+RE alloy and EDS spectra of point A (b) and point B (c) in Fig.2(a)

X-ray spectra (EDS) of the as-cast ZK60+RE alloy. As shown in Fig.2, because of non-equilibrium solidification, low-melting point phase (bright area) is precipitated as discontinuous network primarily at grain boundaries. The EDS analysis results indicate that the as-cast ZK60+RE alloy consists of the α-Mg matrix and eutectic compounds. Mg-Zn-RE eutectic compound distributes at grain boundaries and Zn solid solution in the α-Mg matrix. However, it cannot yet be detected the existence of RE in the matrix of ZK60+RE alloy. Fig.3 shows the longitudinal and transverse morphologies of the as-cast ZK60+RE alloy after ECAE at 300 ℃ for two passes. After ECAE, grains are elongated in the extrusion direction as a consequence of dislocation. The eutectic compound also orientates itself in the extrusion direction.

3.2 Microstructure evolution of ECAE-formed ZK60+RE alloy during isothermal holding

Fig.4 shows SEM images of ECAE-formed ZK60+RE alloy when holding at 589 ℃ (φ_s=0.8) for different times. As shown in Fig.4, when the holding time is 20 min, elongated grains are entirely replaced by recrystallized grains, which indicates that the recrystallisation has occurred during reheating. With further prolonging the holding time, coalescence and spheroidization are activated. Following partial remelting in each case, the microstructure consists of globular grains with grain boundary liquid films. Fig.5 shows the effect of holding time on grain size and shape factor. As shown in Fig.5, increasing holding time from 20 min to 90 min, mean grain size and shape factor increase, which indicates that prolonging holding time can cause grain coarsening and improve the degree of spheroidization.

The previous investigation showed that non-dendritic microstructure evolution of deformed alloy had three stages during isothermal holding, that is, the fragmentation of contacted solid particles caused by liquid films, spheroidization and coarsening[15]. The

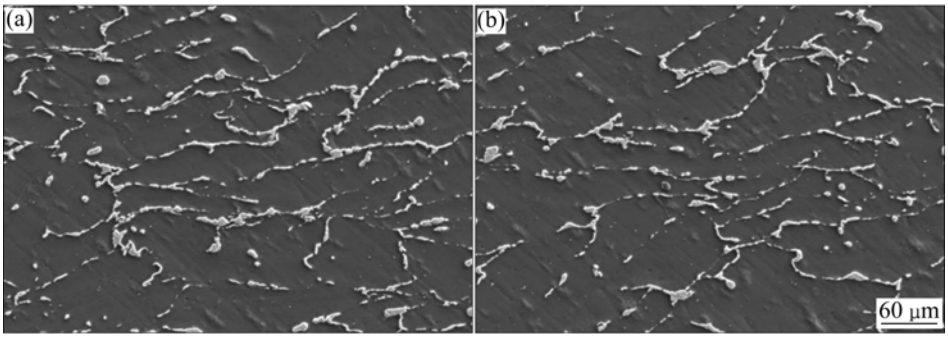

Fig.3 SEM images of two-pass ECAE-formed ZK60+RE alloy: (a) Longitudinal section; (b) Transverse section

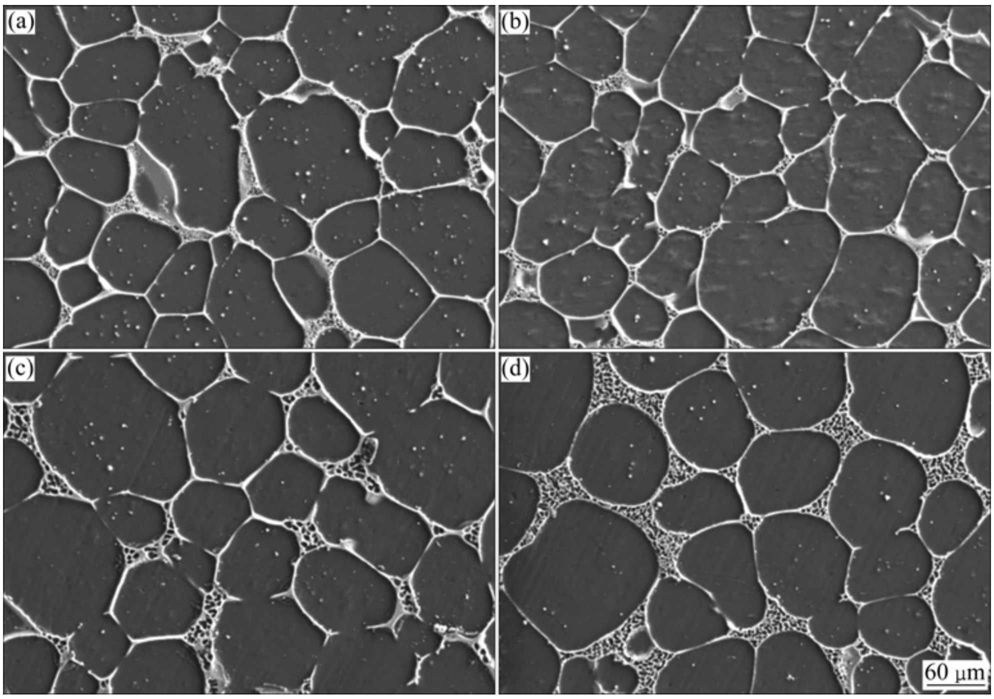

Fig.4 SEM images of ECAE-formed ZK60+RE magnesium alloy partially remelted at 589 ℃ (φ_s=0.8) for different time: (a) 20 min; (b) 40 min; (c) 60 min; (d) 90 min

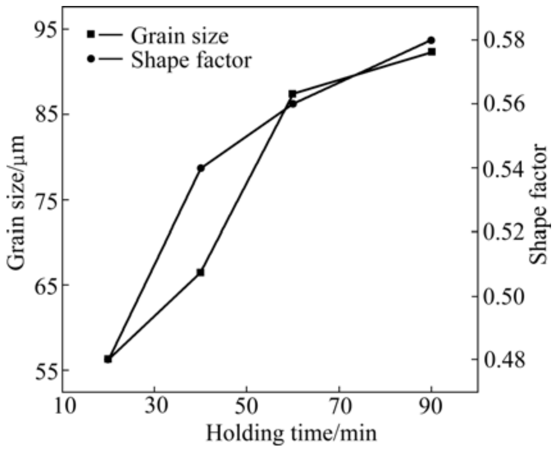

Fig.5 Effect of holding time on mean grain size and shape factor of primary solid particles in semi-solid state

initial sizes of recrystallized grain in the semi-solid state depend mainly on the degree of deformation. The greater the degree of deformation, the smaller the recrystallized grain size in the early stage of isothermal holding. However, it is seen that increasing plastic deformation only in a small strain range could result in the refinement of solid particles in the semi-solid state. LIANG et al[16] found that no significant differences in the microstructure of AZ91D alloy with increasing ECAE passes from two to eight. According to the present results, prolonging time also promotes the degree of spheroidization. As shown in Fig.5, the shape factor of solid particles in the semi-solid state increases from 0.48 to 0.58 with prolonging holding time from 20 min to 90 min. This agrees well with the work of YANG et al[13]. However, the difference between the present study and the work of YANG et al lies in the fact that in the present results, the grain size increases with prolonging holding time. When the material is deformed by two-pass ECAE, a large amount of distortion energy is stored in the material, which provides the driving force for recrystallization during reheating. Compared with the as-cast ZA84 magnesium alloy, the fragmentation of contacted solid particles of ZK60+RE alloy can be achieved in shorter time. As shown in Fig.4(a), with 20 min isothermal holding, recrystallized grains surrounded by liquid films are achieved. However, for as-cast ZA84 magnesium alloy, the fragmentation of contacted solid particles is achieved after holding for 120 min. Although prolonging holding time promotes the degree of spheroidization and improves the filling ability of feedstocks in thixoforging, it results in grain coarsening and undesirable mechanical properties of final components. Therefore, holding time must be controlled properly.

The coarsening behavior of semi-solid alloys with high solid fraction still remains a controversial issue. MANSON-WHITTON et al[17] suggested that the coarsening rate increased with increasing φ_s for φ_s less than approximately 0.75, and then decreased again with further increasing φ_s for φ_s greater than approximately 0.75. Therefore, they developed a modified liquid film migration model to take into account of the necks between the contacting solid particles based on the experiments with a number of spray-formed alloys. The modified liquid film migration model proposed by MANSON- WHITTON et al was given by

$$\bar{r}^3-\bar{r}_0^3=\frac{3D_L C_L(\infty)\sigma V_m(1-\varphi_s)}{2RT(C_L(\infty)-C_s)(1-\varphi_{s,0})(\varphi_{s,0}^{-1/3}-1)}t \quad (1)$$

where $\bar{r}$ is the average grain size after reheating for time t; $\bar{r}_0$ is the average initial grain size; D_L is the solute diffusion coefficient in the liquid; $C_L(\infty)$ and C_s are the compositions of the bulk liquid and solid, respectively; σ is the solid/liquid interfacial energy; V_m is the molar volume; R is the universal gas constant; T is the absolute temperature; φ_s is the solid fraction and $\varphi_{s,0}$ is the critical solid fraction (0.75). The liquid film thickness remains constant with increasing solid fraction but the contact area between solid phases increases. Furthermore, coarsening is thought to be controlled by diffusion through the liquid rather than the solid−solid contacts. Under this condition, the reduction of solid area in contact with liquid results in the decrease in coarsening rate. It is interesting to note that in the present study, the reduction of liquid film thickness results in solid−solid contacts with prolonging holding time, which in turn determines the dominant role of coalescence (Fig.4). Therefore, the grain coarsening behavior of ZK60+RE magnesium alloy cannot be explained by the modified liquid film migration model proposed by MANSON-WHITTON et al. The coarsening behavior of magnesium alloys with high solid fraction should be re-examined.

3.3 Tensile mechanical properties of thixoforged ZK60+RE produced by SIMA

Fig.6 shows a photograph of thixoforged components

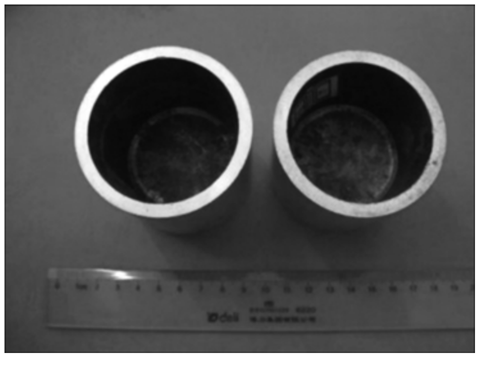

Fig.6 Photograph of SIMA ZK60+RE alloy components

of ZK60+RE magnesium alloy produced by the SIMA route. The trial results indicate that the SIMA components have good surface.

Fig.7 shows the mechanical properties of the thixoforged ZK60+RE components in comparison with those of as-cast and two-pass ECAE-formed ZK60+RE samples. Each tensile value is the average of three measurements. As shown in Fig.7, the tensile properties of the thixoforged ZK60+RE components show a significant improvement over those of as-cast samples and close to those of two-pass ECAE-formed samples with higher yield strength (YS), ultimate tensile strength (UTS) and elongation. The YS of the Mg alloy has a strong dependence on grain size[18]. However, the UTS is also determined by the amount of defects present in the sample. The major defects affecting UTS are gas pores and shrinkage cavity. The more the number of defects is, the lower the UTS and the elongation are[18]. Because ZK60+RE alloys are predeformed by ECAE, these defects can be eliminated to some extent, which leads to higher strength and elongation compared with as-cast samples.

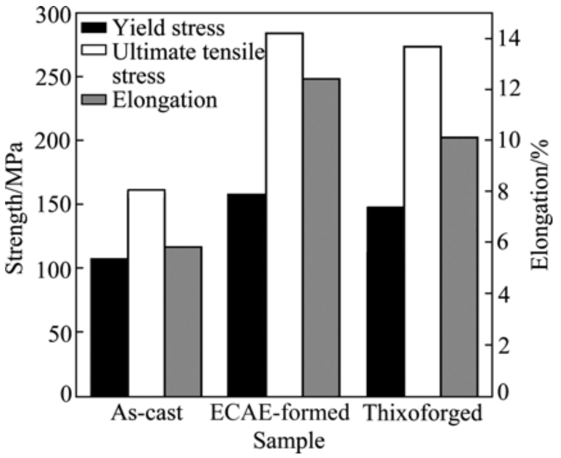

Fig.7 Mechanical properties of thixoforged ZK60+RE components in comparison with those of as-cast and two-pass ECAE-formed ZK60+RE samples

4 Conclusions

1) Semi-solid microstructure of ZK60+RE magnesium alloy, treated by two-pass ECAE, consists of globular grains with grain boundary liquid films. Prolonging isothermal holding time results in grain coarsening and spheroidization.

2) The SIMA ZK60+RE magnesium alloy treated by the two-pass ECAE process can be successfully thixoformed. The tensile properties of the thixoforged ZK60+RE samples show a significant improvement over those of as-cast samples and close to those of two-pass ECAE-formed samples with higher yield strength, ultimate tensile strength and elongation.

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(Edited by YANG Bing)