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Study on distribution of long-period stacking ordered phase in Mg–Gd–Y–Zn–Zr alloy using friction stir processing

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ABSTRACT

Friction stir processing (FSP) was applied to Mg–Gd–Y–Zn–Zr casting at various process parameters to study the influence of heat and deformation conditions on the distribution of long-period stacking ordered (LPSO) phase. FSP resulted in grain refinement, fundamental elimination of β-Mg_3RE eutectics and formation of LPSO phase. Various distributions of LPSO phase, including no LPSO phase, LPSO phase both at the grain boundaries and within the grains and LPSO phase only within the grains could be obtained after FSP at the investigated process parameters. Based on the microstructure analyses and formation mechanism of LPSO phase, this varied distribution is believed to be caused by the combined influence of temperature, plastic strain and cooling rate. Detailed influence of these factors on the distribution of LPSO phase was discussed.

1. Introduction

Mg–Gd–Zn based alloys have received considerable attention in recent years [1–5], because of their superior mechanical properties provided by the special microstructure, especially the long-period stacking ordered (LPSO) phase. Recent studies revealed that the distribution of LPSO phase influenced mechanical properties greatly [6,7]. While the grain boundary distributed LPSO phase could improve ductility, the LPSO lamellae within the grains could enhance yield strength. This indicates that, besides the grain size, texture, and the size of LPSO phase, the distribution of LPSO phase may play an important role in the mechanical property optimization of Mg–Gd–Zn based alloys.

In Mg–Gd–Zn based alloys, the LPSO phase forms mainly in two ways: (1) transformation from β phase and (2) precipitation from the supersaturated matrix. The transformation occurs at above 425 °C [8], with the transformed LPSO phase being mainly distributed at the grain boundaries. The precipitation occurs at above 350 °C, with the LPSO lamellae developing on the bases of stacking faults (SFs), and growing and coarsening with increasing annealing time and temperature [1,9,10]. The precipitated LPSO phase can be distributed both at the grain boundaries and within the grains [11].

During solution treatment of Mg–Gd–Zn based cast alloys, the solute segregation in grain boundary particles favors the transformation [12]. As a result, the LPSO phase is distributed mainly at the grain boundaries [1]. Small amounts of LPSO phase can extend into the grains by prolonging the annealing time or decreasing cooling rate [4,6,13]. During subsequent plastic deformation, the already formed LPSO phase can only be refined, thus its grain boundary distribution is seldom modified [14,15]. In our recent study, as a result of high temperature severe plastic deformation, friction stir processing (FSP) can eliminate solute segregation in Mg–Gd–Y–Zn–Zr casting and enhance precipitation of LPSO phase, leading to the distribution of LPSO phase only within the grains [16].

From the above statements, it is clear that the distribution of LPSO phase is closely related to temperature, plastic deformation and cooling rate. Therefore, investigating the relationship between these factors and the formation of LPSO phase is essential to control the distribution of LPSO phase and further enhance the mechanical properties of the Mg–Gd–Zn based alloys.

FSP is a short-route technique for microstructural modification [17–20]. The high temperature severe plastic deformation generated by FSP can dissolve the eutectics, refine particles, and alleviate severe solute segregation in cast magnesium alloys. Besides, the temperature, deformation and cooling rate during FSP can be varied by controlling the process parameter, e.g. the rotation rate and traverse speed. Moreover, the heat and deformation conditions also vary in local regions of the stir zone (SZ) [21–24]. Consequently, various microstructures can be obtained in the SZ by FSP [25–28]. It is therefore expected that FSP is an ideal method to study the relationship between the processing conditions (temperature, deformation and cooling rate) and the distribution of LPSO phase.

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In this study, FSP was applied to a cast Mg–Gd–Y–Zn–Zr alloy and the microstructure at different process parameters were systematically investigated. The aim is to elucidate the relationship between heat and deformation conditions and the formation of LPSO phase and establish the processing methods for controlling the distribution of LPSO phase.

2. Experimental

6 mm thick Mg–9.4Gd–4.1Y–1.2Zn–0.4Zr (wt%) cast plates were subjected to FSP. A conventional tool with a shoulder 20 mm in diameter, a threaded conical pin 8 mm in root diameter and 4.3 mm in length was used. FSP was carried out at various rotation rates and traverse speeds, as summarized in Table 1. Temperature histories were measured below the SZ using K-type thermocouples, which were embedded 4.3 mm below the plate surface in the centerline of the SZ.

Specimens for microscopy were machined from the cross-section of the SZ perpendicular to the FSP direction. Microstructural characterization was conducted by optical and transmission electron microscopy (OM and TEM, FEI Tecnai G^2 20) in the centerline of the SZ at 0.2, 2.2 and 4.2 mm below the plate surface, i.e. the top, center and bottom regions, respectively. Specimens for OM were prepared by mechanical polishing and etching using a solution of 4.2 g picric acid+10 mL acetic acid+70 mL ethanol+10 mL water. Thin foils for TEM were prepared by low-energy ion milling. The volume fractions of second-phase particles were analyzed by an image analysis software (Image-Pro Plus 6.0).

3. Results

3.1. Microstructure

As shown in Fig. 1a, microstructure of the as-cast Mg–Gd–Y–Zn–Zr alloy is characterized by coarse α-Mg grains and large grain boundary irregular β-Mg_3RE eutectics in a dark contrast and a small number of LPSO particles in a gray contrast, as reported previously [16]. In addition, the fine lamellae observed within the grains were determined to be the SFs by selected area electron diffraction (SAED), as shown in Fig. 1b.

Fig. 2 shows macrographs of the FSP samples. A basin-shaped SZ with a wide top region was observed at all process parameters. According to the contrast difference and the contributions of the shoulder and pin in formation of the SZ, all the SZs can be divided into two sub-zones: the shoulder-driven zone (SDZ) in the upper region and the pin-driven zone (PDZ) in the lower region [29–31]. As shown in Fig. 2a and b, the SDZ in a dark contrast was evidently observed and occupied half the SZ in FSP 1500-25 and 2000-25 samples. Higher magnification images of the two samples revealed voids at the SDZ/PDZ interface, as indicated by arrows in Fig. 2g and h, suggesting insufficient material flow between the SDZ and PDZ [30]. As the rotation rate increased, voids were eliminated, the SDZ expanded to almost the whole SZ, and the PDZ significantly shrank into the narrow bottom region, as shown in Fig. 2c and d. In the FSP 3000-25 sample, a light etching region was even observed to extend from the bottom to the upper region of the SZ (as marked by the dashed line in Fig. 2d), indicating the occurrence of upward material flow. This is coincident with previous study, which showed that vertical material flow can be generated when using a threaded conical pin at a high rotation rate [32,33]. At a constant rotation rate of 2500 rpm, the PDZ expanded as the traverse speed increased (Fig. 2c, e and f).

Fig. 3 shows the variation of grain size in different regions of the SZ with the process parameter. Significant grain refinement occurred after FSP. Gradient grain size was observed in all the SZs with the largest grains and grain size deviation in the top region and the finest in the bottom region. In the FSP 1500/25 sample, the grains were the finest with a size of 2.8 μm in the bottom region. The grain size in the top and center regions increased with increasing the rotation rate and decreasing the traverse speed, which is coincident with the change of heat input with the process parameter. However, grain size in the bottom region changed slightly with the process parameter.

Fig. 4 shows OM images of the top region in the SZ at various parameters. Nearly equiaxed grains with fine lamellae in the grains were observed. The grain sizes in the top regions for the FSP 1500-25, FSP 2000-25, FSP 2500-25, FSP 3000-25, FSP 2500-50 and FSP 2500-100 samples are 11.7 ± 2.4 μm, 22.1 ± 3.8 μm, 27.0 ± 2.4 μm,

Table 1
FSP parameters and sample definition for Mg–Gd–Y–Zn–Zr casting.

Sample definition	1500-25	2000-25	2500-25	3000-25	2500-50	2500-100
Rotation rate (rpm)	1500	2000	2500	3000	2500	2500
Traverse speed (mm/min)	25	25	25	25	50	100

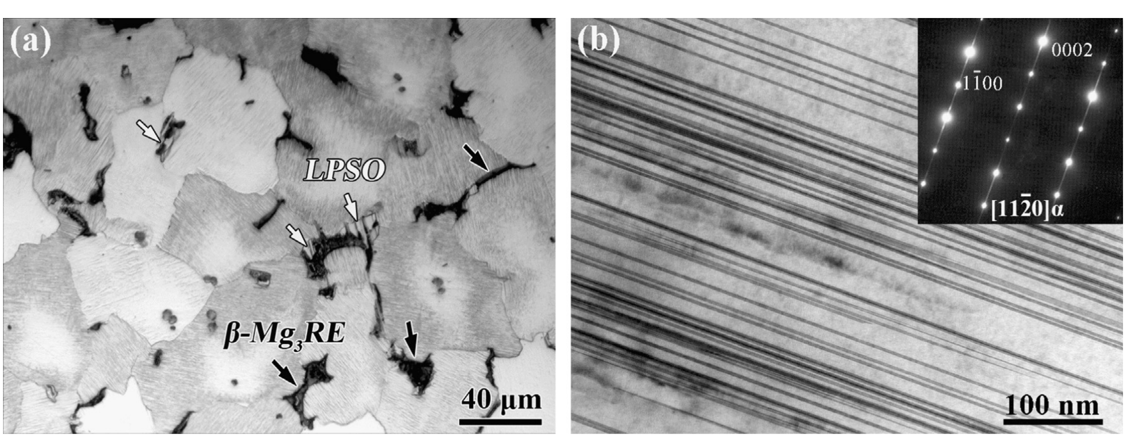

Fig. 1. (a) OM and (b) TEM images of as-cast Mg–Gd–Y–Zn–Zr alloy.