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Friction Stir Processing of Thixoformed AZ91D Magnesium Alloy and Fabrication of Surface Composite Reinforced by SiC$_p$s

CHEN Tijun, ZHU Zhanming, MA Ying, LI Yuandong, HAO Yuan

(State Key Laboratory of Gansu Advanced Nonferrous Materials, Lanzhou University of Technology, Lanzhou 730050, China)

Abstract: The microstructural evolution characteristics of the thermomechanically affected zone (TMAZ) alloy during friction stir processing (FSP) of thixoformed (TF) AZ91D alloy were investigated. Simultaneously, a surface composite layer reinforced by SiC particles (SiC$_p$s) was prepared on the alloy by FSP and the corresponding tribological properties were examined. The experimental results indicate that dynamic recrystallization and mechanical separation (including splitting and fracture of the primary grains) are the main mechanisms of grain refinement for the TMAZ. A composite surface reinforced by uniformly distributed SiC$_p$s was prepared on the alloy. Compared with the corresponding permanent mould casting alloy and the TF alloy without composite surface, the TF alloy with composite surface has the highest wear resistance and lowest friction coefficient.

Key words: friction stir processing; thixoforming; AZ91D alloy; microstructure evolution; composite surface; tribological properties

1 Introduction

Magnesium alloys are attractive for lightweight structural applications in transportation industry because of their low density and high specific strength and stiffness[1]. However, AZ91D alloy, one of the most commonly used magnesium alloys, suffers from the challenge in meeting the requirements of strength and ductility. In order to improve these properties, thixoforming is a promising way by decreasing grain size and shrinkage porosity[2]. Unfortunately, magnesium alloys prepared by either traditional casting or thixoforming always have poor specific elongation due to their hexagonal close-packed structure[3]. This can be overcome by grain refinement[4].

Friction stir processing (FSP) is a relatively new technique for modifying microstructures of metal materials[5]. This technology has been invented by Mishra *et al* based on the principles of friction stir welding and has been used to prepare traditional cast Al and Mg alloys with ultrafine grains[5]. The results indicates that for the FSPed magnesium alloys, the grain size can be decreased to a few micron meters and the mechanical properties are significantly improved[5-9]. Furthermore, FSP has also been used to fabricate surface composites reinforced by intermetallic particles, SiC particles (SiCps), SiO$_2$ particles, C$_{60}$ molecules or fullerene on traditional cast Al or Mg alloy substrates[10-20]. Mg alloys can not be taken as wear resistant material, but as structural material, it is expected that a sliding motion between components is inevitable in certain applications. Therefore, the wear behavior of Mg alloys is an important scientific topic that should be paid attention. The surface modification through preparing composite by FSP is a promising way to improve Mg alloys' wear resistance.

However, the grain refinement by FSP has been focused on some traditional casting Mg or Al alloys and no investigation has involved the thixoformed alloys. The microstructures of the alloys prepared by these two techniques are significantly different and thus the microstructural evolutions during FSP have their respective characteristics. In addition, only two papers about fabricating surface composites reinforced by SiC$_p$s by using FSP have been found[15, 16]. Mishra *et al* examined the grain size and the hardness of the surface composite on 5083 Al alloy substrate while Morisada *et al* studied the stability of the composites on AZ31 Mg alloy besides the grain size and hardness[15, 16]. However, some important properties, such as wear properties of this kind of composite layer have not been investigated. Therefore, in this work the microstructural evolution characteristics of the thermomechanically affected zone was discussed during

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CHEN Tijun (陈体军): Prof.; Ph D; E-mail: chentj@lut.cn

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FSP of thixoformed AZ91D Mg alloy and a SiC$_p$ reinforced surface composite was prepared by FSP. Correspondingly, the wear resistance was also examined.

2 Experimental

A quantity of commercial AZ91D alloy melt was firstly poured into permanent mould to form some rods with 45 mm diameter. Then they were cut into short rods with 85 mm length as the starting ingots for thixoforming. Each short rod was heated for 90 min at a semisolid temperature of 585 ℃ and then pressured in a 40-ton pressure machine. Consequently, a thixoformed ZA91D plate with dimensions of 100 mm×40 mm×15 mm was obtained. Repeating this process, some thixoformed plates were prepared. For comparison, several traditional permanent mould casting (PMC) plates with dimensions of 200 mm×200 mm×15 mm were also prepared (AZ91D melt at 720 ℃ was poured into a steel mould with ambient temperature).

FSP was carried out on one of surfaces of the above plates by using FSW-3LM-015 friction stir welder. A tool pin with height of 5 mm and diameter of 7 mm was used. The shoulder diameter was 20 mm, and a 3° tilt angle of the fixed pin tool was applied. A constant tool rotating rate of 450 r/min was employed, and the rotating tool was traversed at a speed of 60 mm/min along the long axial direction of the plates. To verify the effect of FSP pass on the microstructures and deduce the microstructural evolution characteristics, multiple FSP passes were applied along the same line. Microstructural features were characterized by optical microscope (OM) on the cross-section perpendicular to the welding direction.

For preparing surface composite reinforced by SiC$_p$s, commercial green α-SiC$_p$s (mean diameter: 10 μm) were used. Three grooves with 4.2 mm in depth and 1.25 mm in width were machined on a thixoformed plate along the long axial direction. The distance between the grooves was 6 mm. The SiC$_p$s were filled into the grooves. The tool used in this section has a shoulder with diameter of 18 mm and a pin with diameter of 6 mm and length of 4 mm. The pin was inserted into the groove filled with SiC$_p$s. A constant tool rotating rate of 350 r/min was adopted, and the rotating tool was traversed at a speed of 45 mm/min along the grooves. The tool tilt angle of 3° was used. After the three grooves were FSPed, the microstructure was characterized by OM and scanning electron microscope (SEM) on the cross-section perpendicular to the welding direction. In order to verify the effect of combination of SiC$_p$s on wear resistance, a UMT-2MT reciprocating sliding wear apparatus (ball-on-disk) was used under dry condition. The counterpart is a ball with 3 mm in diameter made by GCr15 steel. An average sliding speed of 50 mm/s, load of 2N and testing time of 10 min were employed. After testing, the volume loss was examined according to the dimensions of worn surface grooves. The ratio of volume loss to sliding distance was taken as the wear rate. For comparison, the wear tests of the permanent mould casting and as-received thixoformed AZ91D alloys were also carried out under the same conditions. The worn surfaces were characterized by SEM.

3 Results and Discussion

3.1 Microstructural evolution characteristics of the thermomechanically affected zone during FSP

Fig.1 shows the microstructures of the thixoformed AZ91D alloy. It can be seen that its microstructure is consisted of spherical primary α phase particles and intergranular secondarily solidified structure (solidified from the liquid phase in semisolid ingot prior to thixoforming) (Fig.1(a)), which is completely different from that of the traditional casting alloy. The microstructure of the casting alloy is composed of primary dendrites and interdendritic eutectics[21]. The primary particle size in the thixoformed alloy is relatively large and up to an average size of about 120 μm while the secondarily solidified structure is very fine due to rapid solidification during thixoforming. Some black particles can be found within the primary particles and they formed from solidification of the liquid pools entrapped within the primary particles at semisolid state, *i e*, they also belong to the secondarily solidified structure [21]. The secondarily solidified structure includes secondarily primary phase (to differentiate from the primary particles, the primary phase solidified from the liquid phase in the semisolid state is named secondarily primary phase) and eutectic structure (Fig.1(b)).

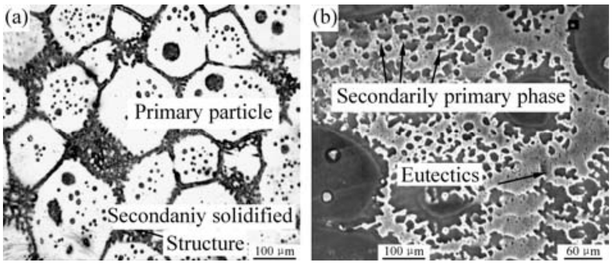

Fig.1 (a) OM and (b) SEM images of the thixoformed AZ91D alloy

The grain refinement mechanism of FSP belongs to sever plastic deformation induced grain refinement[5-9]. In order to verify the details of this kind of grain refinement mechanism, a method that has been frequently accepted is to study the microstructural evolution with the increase of strain during processing. For the friction stir processed (FSPed) alloy, as shown in Fig.2, four distinct zones, nugget zone (nugget), thermomechanically affected zone (TMAZ), heat-affected zone (HAZ) and parent material (PM) can be found. Although the strain in the nugget increases as the increase of stir pass, the strain generated in one pass is so large that the deformation process is difficult to be clearly observed. But for the HAZ, as the description of its name, it is only affected by the heat generated during processing. However, in the TMAZ, both the strain and heat all continuously increase as the stir pass increases. The microstructural evolution in the TMAZ with stir pass, thereby, can be easily verified. In view of the deformation and heat, among the HAZ, TMAZ and PM, the conditions for the TMAZ are the most similar to those for the nugget. Thus, some important information about the microstructural evolution of the nugget can be indirectly obtained through studying the characteristics of the TMAZ. Therefore, the microstructural evolution of the TMAZ was studied during FSP of the thixoformed AZ91D alloy.

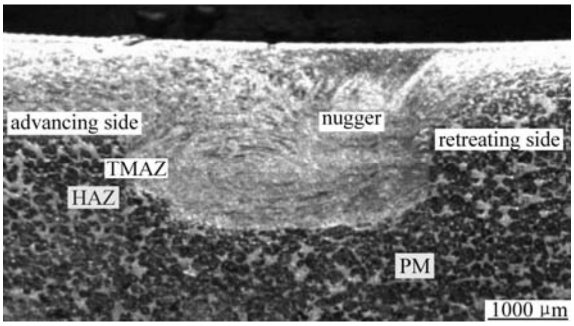

Fig.2 OM images of the cross-section of the thixoformed AZ91D alloy FSPed for two passes

Fig.3 shows the images of the TMAZs of the alloys FSPed for different stir passes. Comparing Figs.3(a) and 1(a), it can be seen that the primary particles were elongated after being processed for 2 passes. As the stir pass further increased, the primary particles became longer and longer and gradually became into thin strips (Fig.3(b)). Simultaneously, it can be found that the number of the strips significantly increased compared with that of the primary particles in a given visual field (comparing Figs.1(a) or 3(a) with 3(b)). One reason is that the primary particles in the other field was elongated and extended to this field. The other is that there was multiplication phenomenon for the particles during being elongated. As shown by arrows A in Fig.3(a), it can be seen that the secondarily solidified structure made the primary particles branched and mechanically split them into several parts during the subsequent elongation. Similarly, as shown by arrow B, the spherical black particles within the primary particles could also play a role of splitting. In addition, it can be found that the strips are not continuous and integrated, but are composed of lots of small-size columns (Fig.3(d)). This implies that the long primary strips dynamically recrystallized during processing, and of course, it is possible that they were mechanically separated into short columns when the strain exceeded their fracture utmost. When the pass further increased, due to the dynamic recrystallization and mechanical separation (including splitting and fracture of the primary grains), the small-size columns gradually evolved into very fine and uniform particles (Fig.3(c)), a microstructure similar to that of the nugget (Fig.4). This implies that both the deformation and heat in the TMAZ and nugget are of certain similarities although the detailed material flow (deformation) and the heat amount in these two zones are very different[6,8,22], and some important information about the microstructure evolution in the nugget can be obtained through studying that in the TMAZ.

Fig.5 quantitatively presents the grain size in the nuggets of the alloys FSPed for different passes. It showsc that the grain size significantly decreased as the stir pass increased. After being stirred for 6 passes, the grain size decreased from the original 120 μm to 3.1 μm. According to the previous investigations, the Mg alloys with such microstructure should have excellent ductility[5-9].

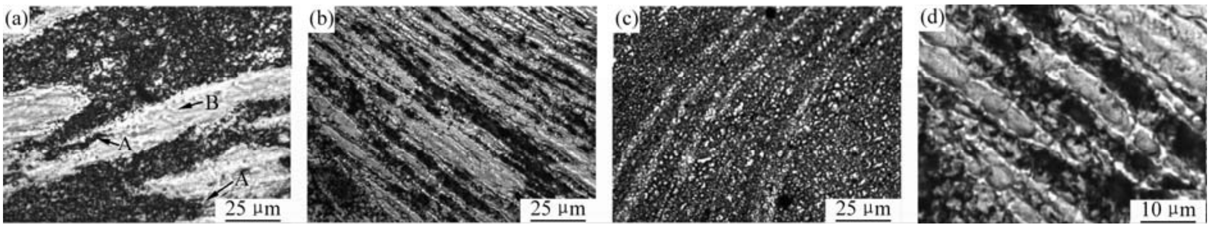

Fig.3 OM images of the TMAZs of the thixoformed AZ91D alloys FSPed by (a) 2, (b) 4, (c) 6 passes and (d) large magnification of micrograph (b)

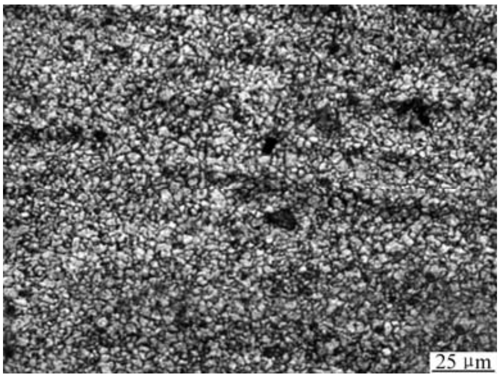

Fig.4 OM image of the nugget of the thixoformed AZ91D alloy FSPed 6 passes

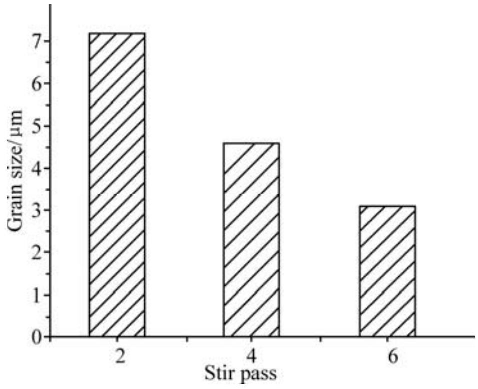

Fig.5 Variation of grain size of the thixoformed AZ91D alloy with stir pass

In addition, from the above results, it can be found that the morphology evolution of the primary grains in the TMAZ can be clearly shown due to the unique microstructure of the thixoformed alloy, relatively large-size spherical primary particles uniformly distributed in the secondarily solidified structure. That is to say that it is a good way to take a thixoformed alloy as the research object in order to verify the microstructural evolution mechanisms of the corresponding traditional casting alloy during FSP.

3.2 Surface composite reinforced by SiC_ps and wear resistance

Fig.6 shows the microstructure of the surface composite prepared by FSP. It indicates that the SiC_ps macroscopically uniformly distributed in the Mg matrix (Fig.6(a)), but they agglomerated at local zones in microscopic scale (Fig.6(b)). It can be expected that this agglomeration can be overcome by multiple FSP passes. This work will be carried out in our future work.

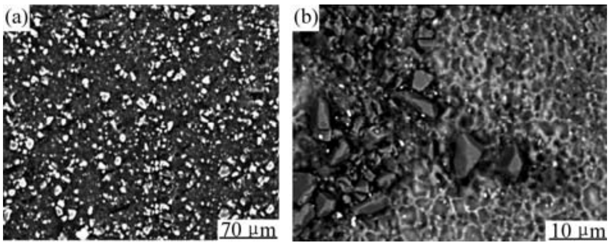

Fig.6 SEM images of the surface composite reinforced by SiC_ps

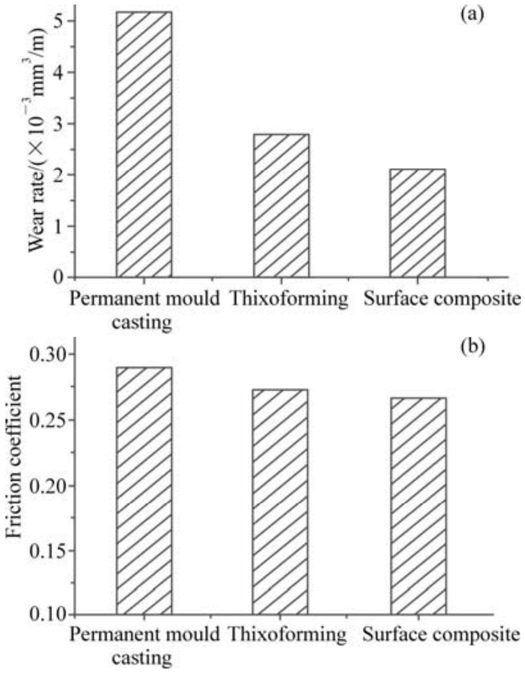

Fig.7 (a) Wear rates and (b) friction coefficients of the AZ91D alloys processed by different routes

The aim of producing SiC_p reinforced surface composite is to improve the tribological properties of the Mg alloy. The present result about the wear resistance is shown in Fig.7(a). It can be seen that the wear resistance of the as-received TF alloy is obviously better than that of the PMC alloy while that of the surface composite (SC) is higher than that of the as-received TF alloy. Correspondingly, the friction coefficient decreased in turn of the PMC, TF and SC (Fig.7(b)). That is to say that the SC can obviously improve the wear resistance and the alloy with such composite surface has the best tribological properties among these three materials. According to the authors' previous investigation, the main reason why the wear resistance of the TF alloy is higher than that of the PMC alloy is that the thixoforming can significantly decrease the amount of porosity in the AZ91D alloy and increase the alloy's hardness[23]. The present result also demonstrates that the hardness of the TF alloy is really obviously higher than that of the PMC alloy (Fig.8). It is also found that the hardness of the alloy with composite surface is higher than that of the TF alloy, which is the reason why the wear resistance of the SC is superior to that of the TF alloy. This implies that the combination of SiCps can strengthen the Mg matrix and then improve its wear resistance.

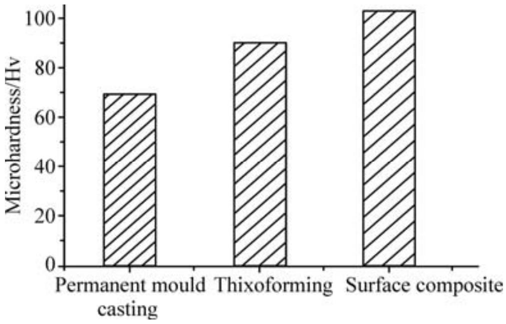

Fig.8 Microhardness of the AZ91D alloys processed by different routes

In order to verify the wear mechanisms, the worn surfaces of these three materials were observed. The results are shown in Fig.9. The worn surface of the PMC alloy is covered by wide and straight grooves (Fig.9(a)), which is the typical characteristic of severe micro-machining wear[23, 24]. In this wear regime, some material transferred on the counterface (ball surface) and was hardened due to plastic deformation, and then ploughed the specimen surfaces, resulting in the formation of the straight grooves. But for the TF alloy, the grooves on the worn surface is narrow, discontinuous and curvous (Fig.9(b)). Under this condition, the small debris particles could take as abrasive to machine the specimen surface driven by the counterface ball. Because these particles did not hard attach on the counterface, the machining was discontinuous and the machining direction also changed at any moment, which resulted in the formation of discontinuous and curvous grooves. This implies that the dominative wear mechanism is the micro-machining and abrasive wear[23]. For the SC alloy, the grooves are more narrow, curvous and denser (Fig.9(c)). It is well known that these are the typical characteristics of abrasive wear. It can be expected that the SiC_ps on the worn surface could detach from the matrix during testing and then took as abrasive. Compared with the TF alloy, the amount and number of the abrasive particles for the SC is significantly larger and their hardness is also higher. Therefore, it is reasonable that the dominative mechanism is abrasive wear for the SC.

From the above discussion, it can be concluded that the wear regime becomes mild in turn of the PMC, TF and SC and the wear resistance increases in this sequence (Fig.7(a)). In addition, the proportion of abrasive wear in the whole wear increases in this sequence and the friction force of abrasive wear is relatively small. Thus the friction coefficient decreases in this sequence (Fig.7(b)).

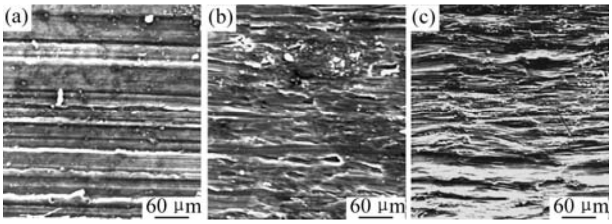

Fig.9 SEM images of the worn surfaces of (a) permanent mould casting AZ91D alloy, (b) as-received thixoforming AZ91D alloy and (c) surface composite on the thixoformed AZ91D alloy

4 Conclusion

The microstructural evolution of the TMAZ can be clearly observed during FSP of the thixoformed AZ91D alloy because of the unique microstructure formed from the thixoforming technology. So it is a good way to take the thixoformed alloy as research object in order to verify the microstructural evolution mechanisms during FSP. The main mechanisms of grain refinement during FSP include thermodynamic recrystallization and mechanical separation.A composite surface with uniform distribution of SiC_ps can be successfully prepared on the thixoformed AZ91D alloy by FSP.Because of the strengthening role of SiC_ps, the thixoformed AZ91D alloy with the composite surface has good tribological properties.

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