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Friction stir processing: a novel approach for microstructure refinement of magnesium alloys

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Abstract. In this article, recent investigations on magnesium alloys by friction stir processing (FSP) are addressed. It indicates that remarkable grain refinement and breakup/dissolution of second-phase particles could be achieved simultaneously by FSP. High values of superplastic elongation were achieved in the FSP magnesium alloys at a wide range of strain rates and temperatures. The pinning of heat resistant particles on the grain boundaries in Mg-RE alloys stabilized the fine microstructure, leading to the occurrence of superplasticity at higher temperature and higher strain rate.

Introduction

Magnesium alloy with low density, high thermal conductivity, good damping characteristics, and high specific strength, is one of the lightest metals used for structural applications [1]. However, low ductility resulting from the limited number of independent slip systems in hexagonal close-packed (HCP) structure limits the use of the magnesium alloys. Grain refinement is an effective method to enhance the mechanical properties and formability of the magnesium alloys.

Mg-Al-Zn (AZ) and Mg-Zn-RE (rare earth) alloys were developed for room-temperature property and improved elevated-temperature performance, respectively. For the AZ magnesium alloys, no reliable grain refining additive exists due to the interaction between impurity elements (such as zirconium) and aluminum, thereby resulting in poor grain refinement [2,3]. For the Mg-Zn-RE alloys, bulky second-phase particles in the castings reduce the strength and ductility.

It is reported that plastic working procedure such as thermo-mechanical processing (TMP), can refine both the grains and second-phase particles, thereby improving the ductility and strength of the cast magnesium alloys significantly [4]. ECAP is one of the most important SPD techniques. It is widely reported that ECAP could refine the grain size of the magnesium alloys dramatically.

Friction stir processing (FSP), developed based on the basic principles of friction stir welding (FSW), is a relatively new SPD technique for microstructural modification [5,6]. In this case, a rotating tool with pin and shoulder is inserted in a single piece of material, for localized microstructural modification for specific property enhancement. Attempts to FSP magnesium based alloys have demonstrated that effective microstructural homogenization and refinement can be achieved as a result of severe plastic deformation and dynamic recrystallization.

In this article, our recent investigations on FSP magnesium alloys were overviewed. It indicates that FSP is a very effective technique for modifying the microstructures and achieving good superplasticity in several types of magnesium alloy castings and wrought billets.

Grain refinement

Figure 1 shows micrographs of rolled and FSP AZ31 samples. A fine-grained microstructure of 4.2 μm (Figs. 1(b)) was produced at a tool rotation rate of 1000 rpm and a traverse speed of 100 mm/min (designated as 1000/100), whereas the average grain size of the parent materials (PM) is 15.1 μm (Fig. 1(a)). It is generally believed that the grain refinement by FSP is due to dynamic recrystallization (DRX), although the detailed mechanism is not well known. TEM observations and electronic backscatter diffraction (EBSD) analyses indicated that twin-induced DRX is main mechanism for the coarse grained Mg-Al-Zn alloy [7]. However, the as-rolled AZ31 plate had an average grain size of 15.1 μm with many fine grains. Therefore, the DRX might not be associated with the twining because the twining usually occurred in grains larger than 10 μm [8]. Thus, the DRX behavior may relate to subgrain boundary evolution or nuclei growth.

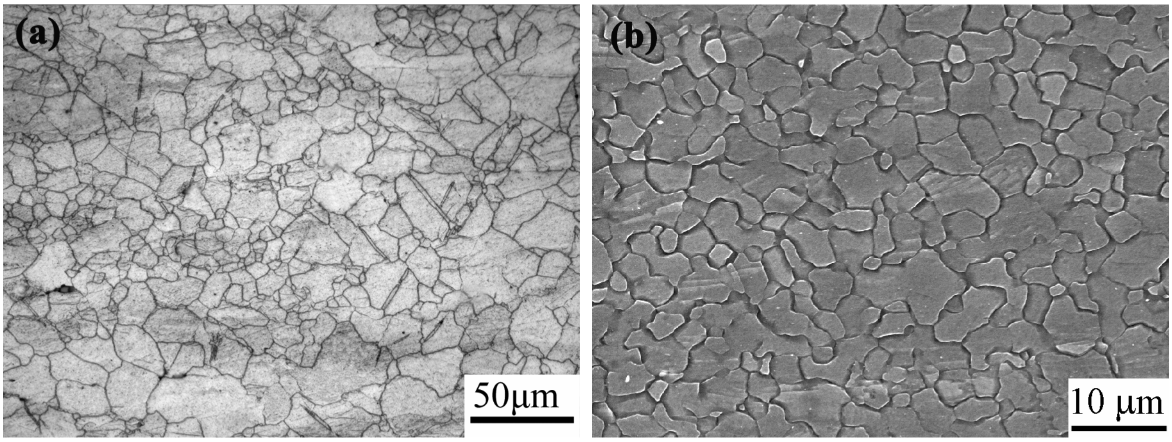

Figure 1. Optical micrographs of (a) rolled and (b) FSP AZ31 samples

An interesting phenomenon is that the average grain size of the FSP AZ31 samples did not exhibit a continuous increase with the rotation rates (Fig.2). The grain refinement in the FSP samples is due to the complex effects of temperature rising and strain rate caused by the friction and stir of the tool. In the previous studies, the grain size was larger under high heat input such as high rotation rate. Chang et al [9] calculated the Zener-Holloman parameters of different FSP parameters and rationalized the grain size by the Zener-Holloman parameters. However, their study was performed at rotation rates lower than 1800 rpm for a traverse speed of 90 mm/min. For the present study, the case is altered for the samples prepared at extremely high rotation rate of 3800 rpm (the micrograph is not shown). It can be inferred that the temperature did not increase linearly with increasing the rotation rate. At extremely high rotation rate, both the strain rate and the strain could be very high. In this case, the deformation behavior and DRX mechanisms might be different from those at lower rotation rates. The Zener-Holloman parameters need to be recalculated carefully by combining temperatures, strain rates and strain values at extremely high rotation rates in the future work.

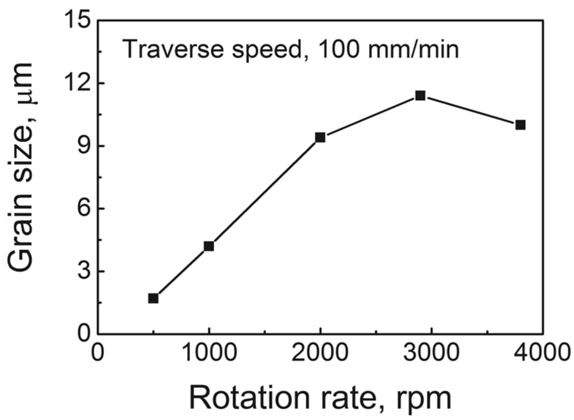

Fig.2 Variation of grain sizes with rotation rates for FSP AZ31 samples

Figure 3 shows the microhardness profiles measured on the transverse cross-sections of the FSP AZ31 samples prepared at a constant traverse speed of 100 mm/min for various rotation rates of 500-3800 rpm. The obvious high hardness in the stir zone (SZ) of the FSP sample at 500/100 was attributed to the finer grain size (1.7 μm). Except for 500/100, the refinement in the grains in the FSP samples, compared with the PM, did not change the hardness obviously. This agrees with the recent studies where a weak influence of grain size on the mechanical properties was reported in FSP AZ31B Mg alloy [8,9]. The texture intensity, dislocation density and subgrain size also exert influence on the hardness. The effect of the grain refinement on hardness could be undermined by these factors.

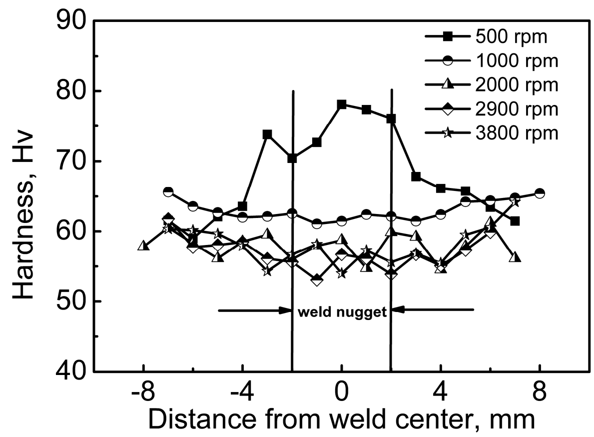

Figure 3. Microhardness profiles of FSP AZ31 alloy

Dissolution of second-phase particles

Mg alloys with higher alloy element contents are widely used as die-cast or wrought alloys, e.g., AZ91 and Mg-RE. The as-cast structure is characterized by coarse α-Mg dendrites and network-like eutectic or coarse intermetallic phases along the grain boundaries. The Mg castings need a long time solution to improve mechanical properties before serving or plastic forming. However, the solution process results in coarsening of Mg grains and severe oxidizing. Figure 3 shows micrographs of as-cast, solutionized, and two pass FSP AZ80 alloy. While it is easy to identify the grain coarsening after solution treatment (Fig. 3(b)), the grains of as-cast AZ80 were significantly refined through a two pass FSP procedure with most of β-$Mg_{17}Al_{12}$ phase being broken-up and dissolved (Fig. 3(c)). The high dislocation density by FSP allows the occurrence of pipe diffusion [10]. The pipe diffusion rate is at least 1000 times higher than the bulk one for magnesium [11]. Thus, FSP could result in the refinement of the Mg grains and the dissolution of the β phase simultaneously, producing a supersaturated solution with the fine grains. A post-FSP aging precipitated out fine β-$Mg_{17}Al_{12}$ phase particles, thereby providing an excellent combination of strength and ductility [12].

For the cast AZ91 alloy, it is reported that two pass FSP at a low rotation rate of 400 rpm refined grains and erased the onion rings consisting of fine β phase particles by reducing heat input and increasing the strain value [13]. Our recent work performed on as-cast Mg-Y alloys indicated that one pass FSP at a high rotation rate of 800 rpm and a low traverse speed of 50 mm/min could refine the grains (~12 μm) and break up the coarse Mg_5Y. Compared with the same FSP Mg-Y alloys at 800 rpm and 100 mm/min, the grain sizes were almost constant, but no visible Mg_5Y remained. The thermal stability of Mg-RE alloys is remarkably higher than that of Mg-Al-Zn alloys. Therefore, it is necessary to investigate DRX behavior of FSP Mg-RE alloys for a better understanding of grain size controlling mechanisms.

Superplasicity for FSP alloys

In the previous works [14-16], it was reported that good superplasticity was achieved via FSP with an optimum strain rate of 10^{-4} to 10^{-3} s^{-1} in AZ91, AM60 and Mg-Zn-Y-Zr alloys. The strain rates were higher than those for conventional coarse-grained magnesium alloys. Ref. [15] reported enhanced superplasticity in Mg-6Zn-0.5Zr-1Y (ZK60-1Y) alloy with grain size of 5.6 μm via FSP at a traverse speed of 100 mm/min and a rotation rate of 800 rpm. A maximum elongation of 635% was achieved at a temperature of 450 ^{o}C and a strain rate of 3×10^{-3} s^{-1}. The superplastic deformation kinetics of the FSP Mg alloy was significantly faster than that of ECAP ones because the FSP alloys contain a higher ratio of high-angle grains boundaries [17,18].

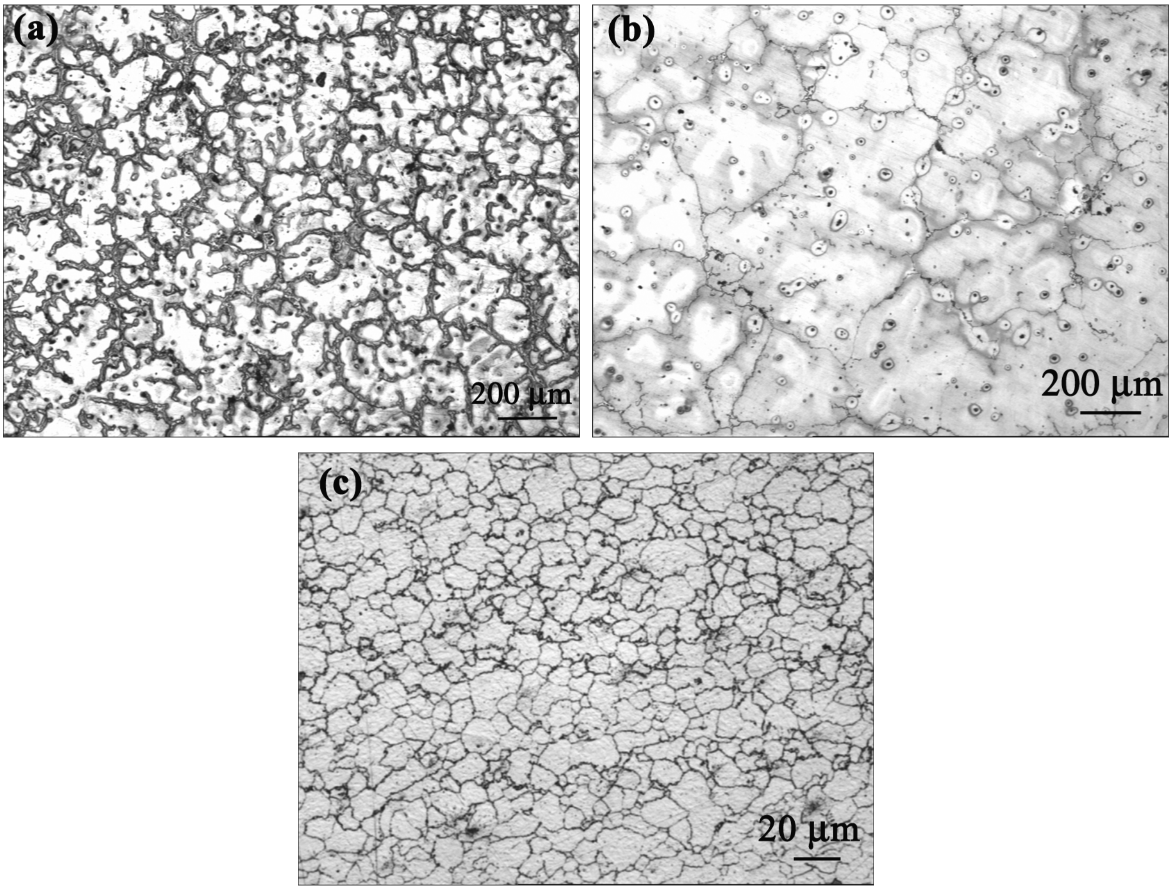

Figure 4. Optical micrograph of AZ80 samples under (a) as-cast, (b) solutionized, (c) two pass FSP

Figure 5 shows our recent superplasticity investigation of the same ZK60-1Y alloy as one in Ref [15] via FSP at a parameter combination of 700 rpm-100 mm/min with a copper back plate. The average grain sizes were 4.2 μm (Fig. 5(a)), which were finer than that in Ref. [15] due to the higher cooling rate and lower heat input. The maximum elongation of 682% was achieved at 375^{o}C and 10^{-3} s^{-1} (Fig. 5(b)). While similar superplastic elongations were achieved in two FSP samples, the optimum temperature was reduced by 75^{o}C with decreasing the grain size from 5.6 to 4.2 μm.

When ZK60 alloy was subjected to FSP at a rotation rate of 1000 rpm and a traverse speed of 100 mm/min, fine grains with an average size of 2.9 μm were obtained (Fig.5c). A maximum elongation of 1180% was achieved at 275 ^{o}C and 3×10^{-4} s^{-1} (Fig. 5(d)). The better superplasticity in the FSP ZK60 alloy than that in the FSP ZK60-1Y alloy is mainly attributed to finer grain size in the FSP ZK60. This indicates that the microstructure of the magnesium alloys could be easily tailored by adjusting the FSP parameters for different purposes. However, it is worthy of note that the elongation of FSP ZK60 alloy decreased significantly above 300^{o}C, thus the optimum strain rate for maximum elongation can hardly be increased. On the contrary, the ZK60-1Y alloy contains quasicrystal I phase (Mg_3Zn_6Y) with a eutectic temperatures of 450^{o}C. Therefore, the FSP ZK60-1Y alloy exhibited a good thermal stability at high temperature due to the pining of the fine second-phase particles on the grain boundaries. In this case, good superplastic elongation could be achieved even at above 350 ^{o}C. According to the constitutive relationship for superplasticity, the increase in the testing temperature shifts the optimum strain rate to high value. Therefore, it is very likely to achieve high strain rate superplasticity in the magnesium alloys containing the rare earth elements by FSP, which produced fine-grained magnesium alloys with dispersively-distributed thermally stable second-phase particles.

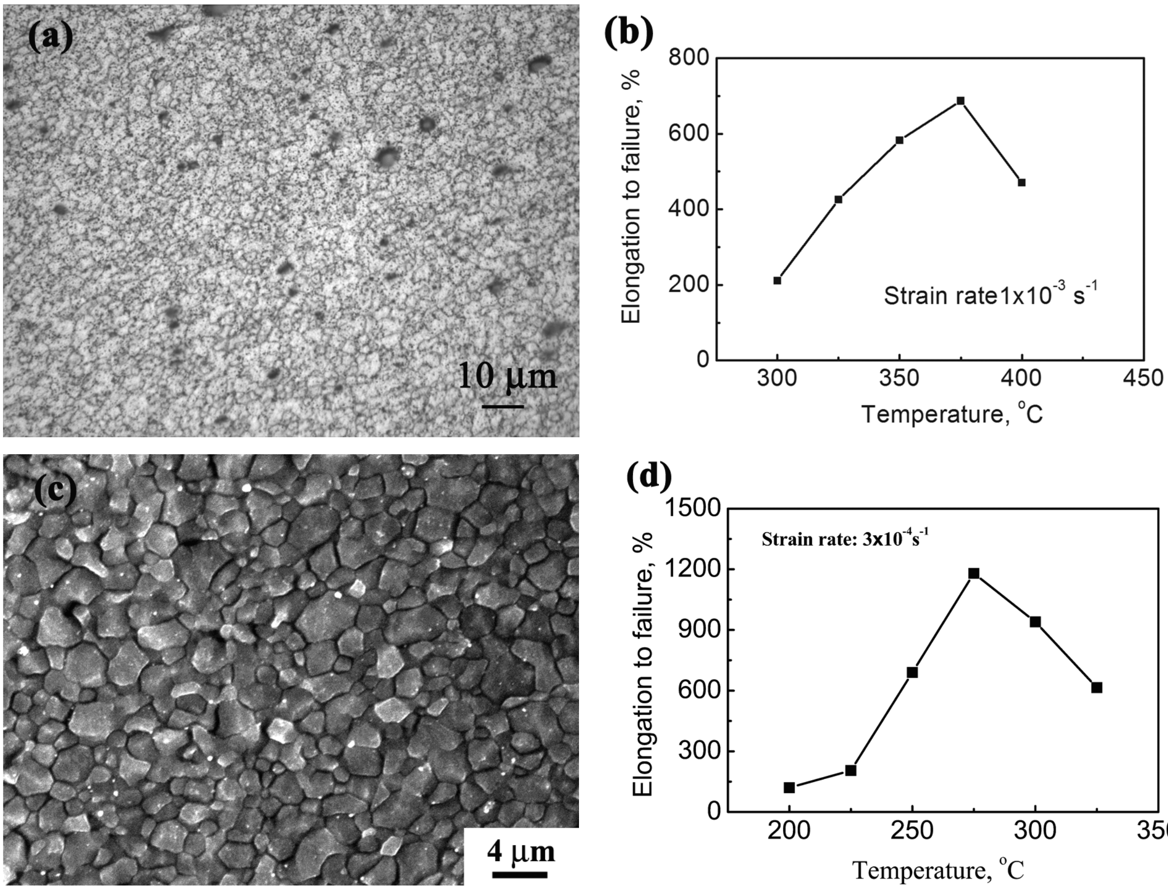

Figure 5. superplasticity of FSP AZ 31 alloys

SUMMARY AND FUTURE OUTLOOK

FSP caused significant refinement of grains and breakup of coarse eutectic or second-phase particles simultaneously. The grain refinement was significant even at high rotation rate with high heat input. The grain size controlling factor and DRX mechanism need to be elucidated to understand the relationship of process parameter and grain size.

FSP resulted in the dissolution of second-phase particles. In this case, FSP produced a fine-grained supersaturated solution. A post-FSP aging resulted in the precipitation of the fine β-$Mg_{17}Al_{12}$ particles. Therefore, it is likely to enhance the mechanical properties of the cast magnesium alloys by a simple FSP procedure.

FSP fine-grained magnesium alloys exhibited good superplasticity. A good thermal stability of RE containing Mg alloys shifted the optimum temperature and strain rate for superplasticity to higher values. The FSP Mg-RE alloys should be further investigated to achieve high strain rate superplasitcity.

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