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Enhanced mechanical properties of Mg–Al–Zn cast alloy via friction stir processing

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Mg–Al–Zn casting was subjected to friction stir processing (FSP) and subsequent aging. FSP resulted in significant breakup and dissolution of the coarse, network-like eutectic β-$Mg_{17}Al_{12}$ phase distributed at the grain boundaries and remarkable grain refinement, thereby improving significantly the tensile properties of the casting. The FSP Mg–Al–Zn sample exhibited an ultimate tensile strength of 337 MPa and an elongation of 10%. FSP combined with aging is a simple and effective approach to enhance the mechanical properties of Mg–Al–Zn casting.

Keywords: Friction stir processing; Friction stir welding; Magnesium alloy; Microstructural modification; Property

Magnesium alloys are useful lightweight structural materials because of their low density, good die-castability, weldability, recyclability and abundance [1]. Grain refinement is an effective method to enhance the mechanical properties of magnesium alloys. Fine-grained structures have been successfully achieved by applying various techniques, e.g. grain refiners [2], altering, rapid solidification [3], spray co-deposition, recrystallization, and severe plastic deformation such as equal channel angular pressing (ECAP) [4].

AZ91 is a cast magnesium alloy widely used to produce die-cast components. The microstructure of AZ91 alloy is characterized by Al-lean dendrites with a eutectic Al-rich solid solution and intermetallic β-$Mg_{17}Al_{12}$ in between [5]. As-cast AZ91 alloy generally exhibits low strength and ductility due to the network-like eutectic β-$Mg_{17}Al_{12}$ distributed at the grain boundaries. To improve the mechanical performance of the AZ91 alloy, it is necessary to modify the distribution and morphology of the β-$Mg_{17}Al_{12}$ phase. In the past few years, various research efforts have been made to achieve this purpose.

The first category of research is heat-treatment. Conventional T6 heat-treatment, involving solution at ~413 °C for ~16–24 h, followed by aging at ~168 °C for 16 h, results in dissolution of the coarse eutectic $Mg_{17}Al_{12}$ network and reprecipitation of fine $Mg_{17}Al_{12}$ particles, thereby improving the mechanical properties of the AZ91 alloy. However, the conventional T6 procedure is time-consuming, resulting in not only increased material cost but also obvious surface oxidation and grain growth. Recently, an improved heat-treatment procedure, denoted by T_x, was developed for rheo-diecast AZ91 alloy [6]. In this case, a heat treatment around its solvus (~370 °C) for 1–2 h transfers the β network to the isolated particles due to partial dissolution of the β-$Mg_{17}Al_{12}$ with limited grain growth, resulting in an improved combination of strength and ductility.

The second category of research is the plastic deformation route. It was reported that multi-pass ECAP of AZ91, homogenized at 413 °C for 18–19 h, resulted in both significant grain refinement and a change of β-$Mg_{17}Al_{12}$ morphology from a long, rod-like shape to smaller particles [4]. However, it seems that no study has reported processing as-cast AZ91 via ECAP. Furthermore, at least 4–6 passes of ECAP are required to achieve microstructural refinement and homogenization.

Friction stir welding (FSW) is a simple, clean and innovative joining technology invented by The Welding Institute in the UK in 1991 [7,8]. Recently, friction stir processing (FSP), a new solid-state processing technique, has been developed based on the basic principles of FSW [9]. FSP has been proved to be an effective and versatile metal-working technique for producing fine-grained microstructures [9–11] and surface composites [12], and for modifying the microstructures of Al-based

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alloys [13–16] and Ni–Al bronze [17]. For example, FSP on A356 aluminum casting resulted in a significant breakup of coarse acicular Si particles and primary aluminum dendrites, created a homogeneous distribution of Si particles in the aluminum matrix, and eliminated casting porosity, thereby improving significantly the tensile properties of A356 casting, in particular ductility and fatigue strength [14,15].

In the past few years, several studies have been conducted to understand the effect of FSP on the microstructure and properties of magnesium alloys [18,19]. However, no attempt has been made to evaluate the effect of FSP on the microstructure and properties of cast AZ91 alloy. This paper aims to establish a simple procedure to enhance the mechanical properties of AZ91 casting.

Cast AZ91D alloy billets with a composition of 8.35Al–0.49Zn–0.20Mn–0.045Si–0.0015Cu–0.0013Ni–0.0011Fe (wt.%) was used for this study. Plates 8 mm thick were machined from the billets and subjected to FSP at a tool rotation rate of 400 rpm and a traverse speed of 100 mm min^{-1}. To enhance material mixing and breakage of the eutectic β-$Mg_{17}Al_{12}$ phase, two-pass FSP was adopted with a 100% overlap and the same forward directions. A tool with a shoulder diameter of 24 mm and a threaded cylindrical pin of 8 mm in diameter and 6 mm in length was used. After FSP, some of the FSP samples were subjected to aging (168 °C/16 h).

Microstructural characterization and analysis was carried out by X-ray diffraction (XRD), differential scanning calorimetry (DSC) and scanning electron microscopy (SEM), complemented by energy-dispersive spectroscopy (EDS). The SEM specimens were prepared by mechanical polishing and etching using a solution of 3 g picric acid, 20 ml acetic acid, 50 ml ethanol, 20 ml water. Tensile specimens with a gauge length of 25 mm, a width of 4 mm and a thickness of 2.5 mm were machined parallel to the FSP direction with the gauge being completely within the nugget zone. Tensile tests were conducted at a strain rate of 1×10^{-3} s^{-1}.

XRD patterns indicate the presence of α-Mg, β-$Mg_{17}Al_{12}$ and Al_8Mn_5 phases in both as-cast and as-FSP AZ91D samples (Fig. 1). However, both the number and the intensity of β-$Mg_{17}Al_{12}$ diffraction peaks decreased considerably after the FSP, indicating the significant dissolution of the β-$Mg_{17}Al_{12}$ phase into the magnesium matrix.

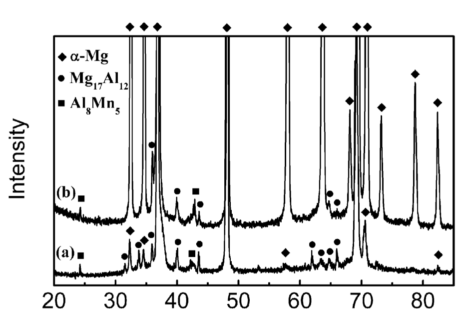

Figure 1. XRD patterns of AZ91D magnesium alloy: (a) as-cast, (b) as-FSP.

The as-received AZ91D was characterized by a coarse eutectic β-$Mg_{17}Al_{12}$ network distributed at the grain boundaries, which is a typical microstructure for as-cast AZ91D (Fig. 2(a)). The maximum solid solubility of aluminum in magnesium is as high as 12.9 wt.% at the eutectic temperature of 437 °C [20]. However, the equilibrium concentration of aluminum in magnesium at room-temperature is about 1.5 wt.%. When aluminum-supersaturated magnesium solid solution is cooled, β-$Mg_{17}Al_{12}$ with a stoichiometric composition of 41.4 at.% Al (44 wt.% Al) will precipitate. The EDS analysis results are summarized in Table 1. The magnesium matrix in the as-received AZ91D casting contains 2.5 wt.% Al (point A in Fig. 2(a)). This value is higher than the room-temperature equilibrium concentration of aluminum in magnesium, indicating that the as-received AZ91D is a little supersaturated. No zinc was detected in the magnesium matrix. On the other hand, point B was composed of 31.5 wt.% Al, 5.3 wt.% Zn and 63.2 wt.% Mg, suggesting that the gray phase is β-$Mg_{17}Al_{12}$ with a certain amount of solid solutionized Zn. It was reported that Zn was mainly distributed in the β-$Mg_{17}Al_{12}$ phase by substituting for a part of aluminum, with a form of $Mg_{17}(Al,Zn)_{12}$ or $Mg_{17}Al_{11.5}Zn_{0.5}$ at temperatures below 437 °C [21]. The fine white particles (point C in Fig. 2(a)) were a compound of aluminum and manganese (Table 1), i.e. Al_8Mn_5 (Fig. 1).

The nugget zone of the FSP AZ91D is characterized by fine grains (∼15 μm) and fine particles distributed at the grain boundaries (Fig. 2(b)). No fine particles were found within the grain interior by SEM examinations even under a very high magnification. The coarse eutectic β-$Mg_{17}Al_{12}$ network in the as-received AZ91D disappeared after FSP. EDS analyses indicated that the grain interior contains as much as 7.5 wt.% Al (point D), i.e. 89% of the aluminum concentration in this AZ91D alloy. This value is much higher than that (2.5 wt.% Al) in the as-received alloy, indicating significant dissolution

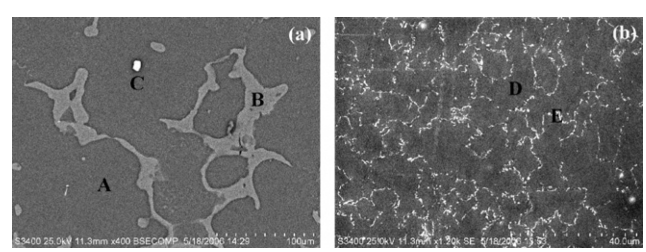

Figure 2. SEM images of FSP AZ91D magnesium alloy: (a) secondary electron image (SEI) of the base metal and (b) backscattered electron image (BEI) of the nugget zone.

Table 1. The results of EDS analysis of Figure 2 (wt.%)

Location	Al	Zn	Mn	Mg
A	2.5	–	–	97.5
B	31.5	5.3	–	63.2
C	29.0	–	68.7	2.3
D	7.5	–	–	92.5
E	10.0	–	–	90.0

of the eutectic β-$Mg_{17}Al_{12}$ phase into magnesium matrix during FSP. At the grain boundaries (point E), a higher aluminum concentration of 10.0 wt.% is attributed to the presence of fine β-$Mg_{17}Al_{12}$ particles. From Figure 2 and Table 1 it is clear that FSP resulted in significant breakup and dissolution of the coarse eutectic β-$Mg_{17}Al_{12}$ that was previously distributed at the grain boundaries. In the FSP sample, no zinc was detected at either the grain interior or the boundaries. This indicates that zinc was dissolved into the α-Mg with the breakup and dissolution of the β-$Mg_{17}Al_{12}$ phase and it is difficult to detect the presence of zinc due to the relatively low zinc content (0.49 wt.%).

Based on the Mg–Al phase diagram, a heating temperature of 437 °C causes the dissolution of the eutectic β-$Mg_{17}Al_{12}$ phase into the magnesium matrix. DSC analyses indicated the presence of two endothermic peaks in the as-cast AZ91D sample (Fig. 3). The first peak, with an incipient melting temperature of 425 °C, corresponds to the dissolution of the eutectic β-$Mg_{17}Al_{12}$ phase, whereas the second peak at higher temperature is due to the melting of the magnesium. However, for the as-FSP sample, the endothermic peak corresponding to the dissolution of the eutectic β-$Mg_{17}Al_{12}$ phase disappeared, indicating that the eutectic β-$Mg_{17}Al_{12}$ phase has been basically dissolved into the magnesium matrix during the FSP (Fig. 3). The DSC results are in good agreement with the EDS analyses (Table 1). However, there is a broad exothermic peak around 250 °C and a broad endothermic peak around 370 °C in the as-FSP sample. In order to reveal the nature of the exothermic and endothermic peaks in the as-FSP sample, a DSC run to ~475 °C was conducted on the aged FSP sample. The exothermic peak around 250 °C disappeared and the endothermic peak moved somewhat to a lower temperature in the aged FSP sample. Thus, the exothermic peak around 250 °C in the as-FSP sample is due to the precipitation of fine β-$Mg_{17}Al_{12}$ particles, considering the fact that the magnesium matrix of the as-FSP sample is highly supersaturated (Table 1). On the other hand, the endothermic peak in both as-FSP and aged FSP samples might be associated with the dissolution of fine β-$Mg_{17}Al_{12}$ particles because the solvus of the AZ91 alloy is ~370 °C.

For Mg–Al alloys, it takes up to ~40 h to achieve the complete dissolution of the eutectic β-$Mg_{17}Al_{12}$ phase due to low diffusion rate of aluminum in magnesium matrix [22]. For the FSW/FSP thermal cycles, both heating and cooling rates are quite high. For example, the duration at above 200 °C during FSP of A356 aluminum alloy is only 25 s [23]. It seems impossible to achieve the dissolution of most of the eutectic β-$Mg_{17}Al_{12}$ phase in such a short period for a conventional thermal cycle. However, for FSW/FSP, severe plastic deformation in the nugget zone, with a strain rate of 10^0–10^2 s^{-1} and a strain of up to ~40 [18,24], facilitates significantly the dissolution of the β-$Mg_{17}Al_{12}$ phase, thereby generating an aluminum-supersaturated solid solution.

For AZ91 supersaturated solid solution, aging at 168 °C for 16–24 h resulted in the precipitation of the β-$Mg_{17}Al_{12}$ phase in continuous or discontinuous form [20]. For the present as-cast AZ91D alloy, aging resulted in the discontinuous precipitation of lamellar β-$Mg_{17}Al_{12}$ phase around the original coarse eutectic β-$Mg_{17}Al_{12}$ network (Fig. 4(a)). This is due to the fact that the magnesium matrix of the as-received AZ91 was a little supersaturated. On the other hand, extensive continuous precipitation was observed within the grains in the aged FSP AZ91D sample (Fig. 4(b)). This is attributed to the high supersaturation of the AZ91D solid solution resulting from the FSP thermal cycles as discussed above.

Figure 5 summarizes the room-temperature tensile properties of cast and FSP AZ91D samples. The as-received AZ91D casting exhibited lower yield and ultimate tensile strengths (73 and 111 MPa) and elongation (2.5%). This is attributed to the presence of the coarse eutectic β-$Mg_{17}Al_{12}$ network at the grain boundaries that tends to crack or debond from the magnesium matrix early under lower stress during tensile deformation (Fig. 6(a) and (b)). Similar fracture characteristics have been previously observed in a cast AZ91 alloy

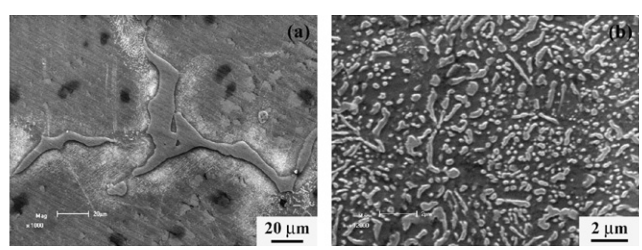

Figure 4. SEM secondary electron image of aged AZ91D magnesium alloy: (a) the base metal, (b) the nugget zone of the FSP sample.

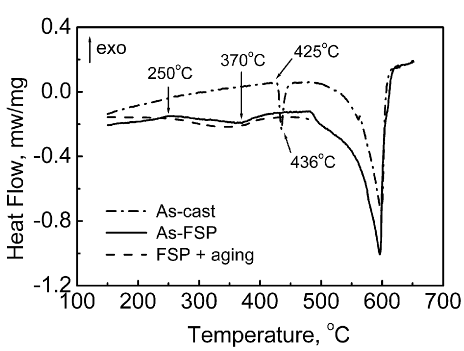

Figure 3. DSC curve of AZ91D magnesium alloy.

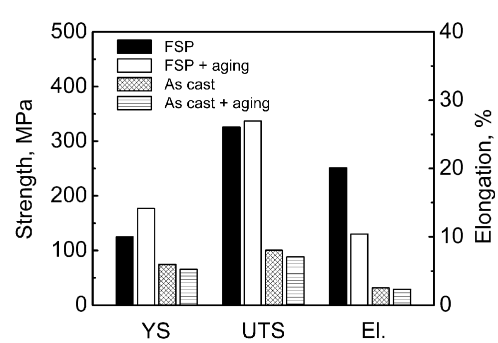

Figure 5. Tensile properties of AZ91D magnesium alloy (YS: yield strength, UTS: ultimate tensile strength, El.: elongation).

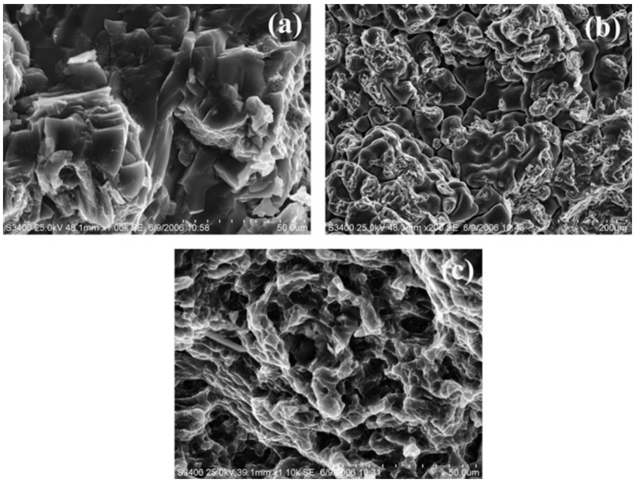

Figure 6. SEM images showing the fracture surfaces of AZ91D samples: (a) and (b) as-cast, and (c) as-FSP.

[25]. FSP resulted in a significant improvement in tensile properties, particularly ductility, with the sample exhibiting a typical ductile dimple-fracture (Fig. 6(c)). This is mainly attributed to significant breakup and dissolution of the coarse eutectic β-$Mg_{17}Al_{12}$ phase and remarkable grain refinement. First, significant breakup and dissolution of the coarse eutectic β-$Mg_{17}Al_{12}$ phase reduced the possibility of crack and debonding of the β-$Mg_{17}Al_{12}$ phase under lower stress, thereby improving considerably the ductility and strength of the FSP sample. Second, the remarkable grain refinement increases significantly the yield strength of the AZ91D alloy because of larger Hall–Petch slopes, k, in magnesium alloy, resulting from the higher Taylor coefficient of magnesium alloy compared to that of aluminum alloy. Third, the solid solution strengthening resulting from the supersaturated aluminum also contributed to the improvement in strength of the FSP sample.

Aging did not improve the tensile properties of the cast sample, but conversely reduced somewhat both strength and ductility. This is due to the fact that aging did not change the coarse-grained structure and eutectic β-$Mg_{17}Al_{12}$ network, and conversely a small amount of precipitation exerted an adverse effect on the mechanical properties. After aging, the FSP sample exhibited greatly enhanced yield and ultimate tensile strengths (177 and 337 MPa), and reduced elongation (10%) due to extensive continuous precipitation of fine β-$Mg_{17}Al_{12}$ particles.

In summary, the following conclusions are reached: (1) FSP on AZ91 casting caused significant breakup and dissolution of the coarse eutectic β-$Mg_{17}Al_{12}$ network, with 89% of aluminum being dissolved into the matrix, and remarkable grain refinement (~15 μm), thereby improving significantly the tensile properties, in particular ductility. (2) Post-FSP aging resulted in continuous precipitation of fine β-$Mg_{17}Al_{12}$ particles, thereby increasing considerably the yield strength and decreasing the ductility. (3) FSP combined with aging is an effective approach to enhance the mechanical properties of cast AZ91 alloy.

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