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Effect of heat treatment and number of passes on the microstructure and mechanical properties of friction stir processed AZ91C magnesium alloy †

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Abstract

In this paper, the effect of heat treatment and number of passes on microstructure and mechanical properties of friction stir processed AZ91C magnesium alloy samples were investigated. From six samples of as-cast AZ91C magnesium alloy, three plates were pre-heated at temperature of 375°C for 3 hours, and then were treated at temperature of 415°C for 18 hours and finally were cooled down in air. Three plates were relinquished without heat treatment. 8 mm thick as-cast AZ91C magnesium alloy plates were friction stir processed at constant traverse speed of 40 mm/min and tool rotation speed of 1250 rpm. After process, microstructural characterization of samples was analyzed using optical microscopy and tensile and Vickers hardness tests were performed. It was found that heat treated samples had finer grains, higher hardness, improved tensile strength and elongation relative to non-heat treated ones. As the number of passes increased, higher UTS and TE were achieved due to finer grains and more dissolution of β phase ($Mg_{17}Al_{12}$). The micro-hardness characteristics and tensile improvement of the friction stir processed samples depend significantly on grain size, removal of voids and porosities and dissolution of β phase in the stir zone.

Keywords: AZ91C magnesium alloy; Heat treated; Microstructural characterization; Mechanical properties

1. Introduction

As the lightest materials among constructional alloys, Magnesium alloy are expected to be widely used in transportation and aerospace industries [1-3]. FSW/FSP which was invented by the welding institute (TWI) of the UK in 1991 is a welding process in which a non-consumable welding tool is used to generate both the frictional heat and mechanical deformation simultaneously in order to make a solid state joint [4]. Recently, advantages such as low residual stresses, high tensile strength, low distortion due to lower welding temperature and also elimination of porosities and cracks has made Friction stir welding (FSW) as a promising choice for joining particulate reinforced aluminum matrix composites, pure copper and magnesium alloys [5, 6]. Because of no melting occurrence, FSW is considered as a solid state welding process, with a rotating non-consumable tool and a pin extending from a larger shoulder [7]. Based on the principles of the friction stir welding and closely with similar features to Modified friction stir channeling (MFSC), Friction stir processing (FSP) was invented by Charit and Mishra [8-10] for microstructural modification of metal materials. In FSP, a rotating tool is inserted into a material and high plastic deformation is produced. Unlike its pioneers, FSP is used to enhance ductility, induce superplasticity and improve corrosion. Dynamic recrystallization of the deformed zone forms an ultrafine-grained structure [11, 12]. FSP has been successfully applied to various cast aluminum and magnesium alloys to eliminate casting defects and there by improve their mechanical properties [13]. One of these materials is AZ91C alloy. AZ91C alloy contains approximately 9 and 1 wt.% of aluminum and zinc, respectively, as alloying elements. Under equilibrium solidification conditions, microstructure of AZ91C alloy contains α solid solution with β precipitates ($Mg_{17}Al_{12}$) in the matrix. However, under non-equilibrium solidification conditions, microstructure of the alloy has a dendritic form in which brittle β phase exists between dendrite arms [14]. Dendritic structure along with other non-homogeneities is observed in casting specimen and cause the alloy to have brittle structure with low UTS and formability [15]. Therefore, to improve the formability and tensile properties and then to prepare the alloy for subsequent processes such as rolling and extrusion, heat treatment is inevitable. The most important parameters during heat treatment are holding temperature, holding time and cooling rate of the specimen to the room temperature [16]. Plenty of investiga-

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tions have been done to investigate microstructural and mechanical properties of AZ91 alloy. Rouhi et al. [17] studied the effects of welding environment on microstructure and mechanical properties of friction stir welded AZ91C magnesium alloy, their results indicated that the tensile strength of the joint can be improved from 210 to 410 MPa using external water cooling. Ni et al. [18] studied the low cycle fatigue properties of friction stir welded joints of a semi-solid processed AZ91D magnesium alloy. They reported that a recrystallized fine grained microstructure was generated in the stir zone after FSW and a fatigue life fairly close to that of the base metal. Chai et al. [19] studies the high strain rate superplasticity of a fine-grained AZ91 magnesium alloy prepared by submerged friction stir processing. They reported that SFSP results in remarkable grain refinement due to the enhanced cooling rate compared with normal FSP and The SFSP AZ91 specimen exhibits considerably enhanced superplastic ductility with reduced flow stress and higher optimum strain rate, as compared to the normal FSP specimen. The excellent HSRS of the SFSP material is attributed to its finer grain structures, which contain a larger fraction of grain boundary and grain growth and cavities coalescence is the main failure mechanism for the normal FSP and SFSP alloys during superplastic deformation. Chai et al. [20] investigated heat treatment on microstructure and mechanical properties of AZ91 magnesium alloy prepared by FSP. They found that the possibility of heat treatment to achieve higher mechanical properties. Zhang et al. [21] investigated superplastic tensile behavior of the fine grained AZ91 by means of hot tensile tests at different temperature and strain rate ranges. They found that the friction stir processed AZ91 alloy exhibits excellent superplasticity and a maximum elongation of 1604% was achieved. Chai et al. [22] subjected the as-cast AZ91 plate to normal friction stir processing (processed in air) and submerged friction stir processing (processed in water, SFSP) and investigated microstructure and superplastic tensile behavior of the experimental alloys. Remarkable grain refinement was observed due to the enhanced cooling rate compared with normal FSP, with an average grain size of 1.2 μm and 7.8 μm. They attributed excellent high strain rate superplasticity of the SFSP alloy to its finer grain structure and higher fraction of grain boundary. Although, comprehensive study on the effects of heat treatment on microstructure and mechanical properties of FS processed as-cast AZ91 alloy is lacking, several papers have investigated the selected properties of AZ91 magnesium alloy. Čížek et al. [15] investigated the microstructure and mechanical properties of AZ91 magnesium after heat treatment and hot forming. Their results showed the possibility of heat treatment to achieve higher mechanical properties. In this study, the FSP technique was applied to produce as-cast AZ91C plates with 8 mm thickness. Two groups of plates with heat treatment and without heat treatment were prepared at the constant process conditions. Microstructural properties were investigated by means of optical microscopy. Tensile strengths of the specimen with different annealing condition and tool rotation speeds were investigated by standard tensile test. The hardness behavior was discussed by dislocation density and grain size variation during FSP.

Table 1. Chemical composition of as cast AZ91C magnesium alloy.

Element	Mg	Al	Zn	Mn	Si	Fe	Cu	Ni
Weight (%)	Bal.	9.1	0.6	0.21	0.085	0.002	0.009	0.001

(a) (b)

Fig. 1. (a) FSP process and applied machine; (b) a high carbon steel cylindrical tool.

2. Materials and experimental procedures

As-cast AZ91C magnesium alloy plate (160 mm × 50 mm × 8 mm) was selected for the present study. The measured chemical composition of as-cast AZ91C magnesium alloy is given in Table 1.

Six samples of this alloy were used to produce FSP samples. Three plates were pre-heating at temperature of 375°C for 3 hours and then were treated at temperature of 415°C for 18 hours (solid solution heat treatment-T4 after ASTM) and finally were cooled down in the air. Other 3 plates were relinquished without heat treatment to study the heat treatment effect. The FSP machine used in this experiment was a modified form of a vertical-type CNC milling machine. The FSP process and applied machine are shown in Fig. 1(a). A high carbon steel cylindrical tool, with square pin profile, 16 mm shoulder diameter, 8 mm pin diameter and pin length of 4.5 mm was used, as shown in Fig. 1(b).

The optimum processing condition for FSP were established at a tool transverse speed of 40 mm/min and a tool rotation speed of 1250 rpm with a tilt angle (angle between spindle and work piece normal) of 3° [17]. Since the process forces are intense during FSP, plates were fixed by a fixture. Different passes were used. The detailed parameters are summarized in Table 2. Defect-free sample of FSP is shown in Fig. 1(a). The processed plates were cut in transverse direction and etched with Keller's reagent (5 ml acetic acid, 7 g picric acid, 15 ml water, 120 ml ethanol, 5 ml HCl and 8 ml nitric acid) for 2 s. Vickers microhardness tests were conducted using a 100 g load for 10 s. The grain structures and the particle distribution of etched specimens were examined by Optical microscopy (OM). Grain size and particle size measurements

Table 2. Different experimental process conditions.

Sample		TE (%)	UTS (MPa)	Hardness on the nugget zone (HV)	Grain size (μm)
Non-Heat treated	Base metal	140	86	108	9
	1 pass	103	92	118	16
	2 pass	94	95	125	17
	3 pass	88	106	140	19
Heat treated	Base metal	13	113	90	114
	1 pass	19.5	123	97	93
	2 pass	21	144	111	86
	3 pass	23	165	119	78

were carried out using an image analyzer. The tensile tests were carried out using an Instron-type testing machine with a crosshead speed of 1 mm/min according to the American Society for Testing of Materials (ASTM E8M-04) standards.

3. Results and discussion

3.1 Microstructural characterization

The as-received AZ91C is characterized by the classical cast structure in which different floating crystals can be recognized. In all specimens distributed zones characterized by large porosity were observed. Microstructure of base metal consisted of primary α grains (light) embedded in the dark eutectic β phase ($Mg_{17}Al_{12}$) and is shown in Fig. 2(a). Due to severe plastic deformation in the Nugget zone (NZ) and high strain rate during the FSP process, dissolution of eutectic β phase is accelerated significantly and $Mg_{17}Al_{12}$ precipitates started to disappear after the FSP process as shown in Fig. 2(b). At stir zone of friction stir processed specimens, grain size had been significantly reduced and the β precipitations were solved due to the high heat input of the process. Performing FSP led to fabrication of ultra-fine-grained structure. The average grain size in the base metal of AZ91C magnesium was 140 μm. Microstructural characterization of heat treated and friction stir processed samples showed following privileges relative to non-heat treated samples:

- Heat treated samples had finer and more uniform microstructure relative to non-heat treated ones. Improvement of structure is attributed to finer grains of base metal in heat treated samples which causes finer microstructure of the NZ during dynamic recrystallization, as shown in Fig. 2(c).
- More precipitates of $Mg_{17}Al_{12}$ in the grain boundaries were dissolved by FSP in heat treated samples.
- In heat treated samples porosities and voids of the AZ91C alloy disappeared during the FSP (Fig. 2(d)) while, some voids were still observed in non-heat treated ones, as shown in Fig. 2(e).

The effect of single, second and third passes on the grain size of the stir zone was also investigated. As shown in Table 2, the average grain size in the nugget zone decreased by increasing the number of passes. It was found that by increasing the number of passes, dissolution of β phase increased and the grain size decreased in the NZ.

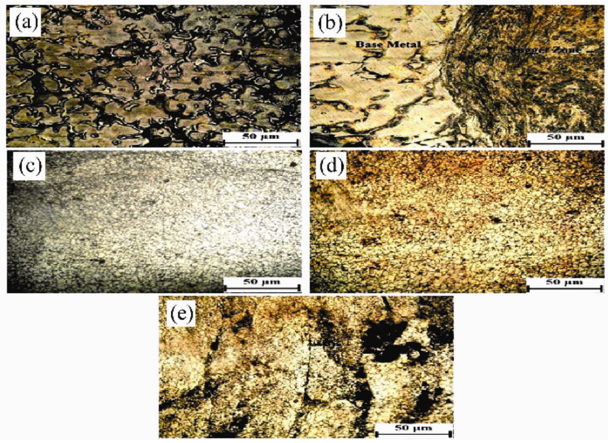

Fig. 2. Microstructure of (a) base metal; (b) base metal and the NZ with dissolution of eutectic β phase; (c) heat treated samples with during dynamic recrystallization; (d) heat treated after removal of porosities and voids; (e) non-heat treated samples with voids.

3.2 Microhardness characterization

Fig. 3 illustrates the variations of the hardness along the center line on a cross-section of the samples at different passes and heat treatment conditions. It has been observed that, in most cases, the NZ has slightly higher hardness values than the base metal. The increase in the hardness value of NZ is related to the extra-fine grained structure generated by severe grain refinement due to the dynamic recrystallization. In the early stages of plastic deformation on FSP process, grain boundaries are important obstacles to slip, so fine-grained stir zone of friction stir processed AZ91C alloy is stronger than coarse-grain base metal and has higher hardness [22, 24, 26].

The dependence of initial yield strength on grain size is often expressed by the Hall-Petch relationship:

$$\sigma = \sigma_0 + K_Y d^{-1/2} . \quad (1)$$

Similar equation can be written for the dependence of hardness, H, on grain size:

$$H = H_0 + K_H d^{-1/2} . \quad (2)$$

Using Hall-Petch relation, it is proved that by increasing the number of passes, grain size of NZ decreased and led to an increase in hardness value of NZ. The maximum amount of hardness was 119 HV and was observed in the specimen produced in three passes. Khayyamin et al. [24] also found that by increasing the number of passes the average hardness of fabricated Mg based nanocomposite improves up to 124 HV.

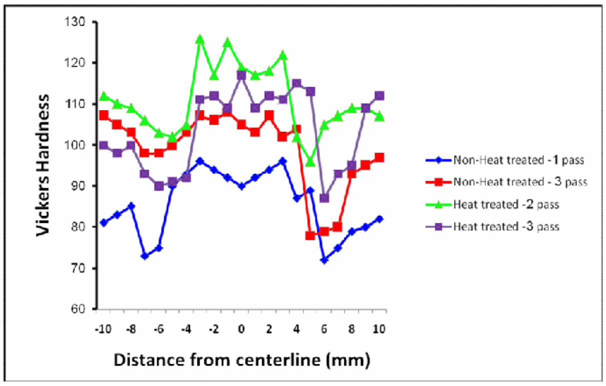

Fig. 3. Vickers microhardness profile of the samples.

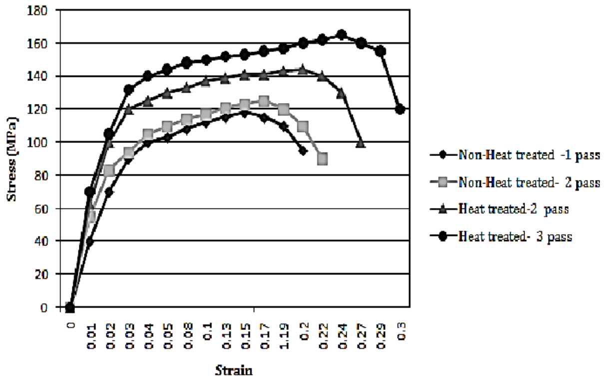

Fig. 4. Stress-strain curves for friction stir processed samples.

As shown in Fig. 3, in samples with same number of passes, heat treated ones had higher hardness. Heat treated samples had finer grains due to limitation of grain growth and according to Hall-Petch relationship, higher hardness value was achieved [25, 27].

3.3 Tensile properties

Fig. 4 indicates the stress-strain curves for samples fabricated in different passes and heat treatment conditions. UTS and elongation of base metal were lower than friction stir processed specimens due to the presence of coarse grains and hard $Mg_{17}Al_{12}$ precipitates in the grain boundaries. The fracture mechanism in all specimens was brittle and samples showed low tensile elongations as it was expected from previous paper [23, 27, 28].

As shown in Fig. 4, heat treated specimen has higher UTS and Tensile elongation (TE) relative to non-heat treated ones. As we see, the minimum amount of UTS and TE in nugget zone for samples with heat treatment were about 123 MPa and 19.5% respectively while, mentioned values were 118 MPa and 16% respectively for non-heat treated specimens. In addition, UTS and TE of base metal increased from 108 to 113 MPa and from 9 to 13% respectively by applying heat treatment method. Tensile strength and elongation in the nugget zone arose by increasing the number of passes in both types of heat treatment specimens. While UTS and TE in heat treated sample, fabricated in single pass, were 123 MPa and 19.5% respectively, mentioned values increased to 165 MPa and 23% in third pass.

The UTS and TE of the nugget zone in as-cast AZ91C alloy are determined by several major factors. First, grain size of SZ that depends on heat input and plastic deformation during FSP process. Second, porosities, voids and β phase ($Mg_{17}Al_{12}$) of the cast alloys which were controlled by heat treatment and the number of passes in this investigation. Since heat treated samples had finer and more uniform microstructure and porosities, voids and β phase were removed or at least were reduced during heat treatment, these samples had higher UTS and elongation relative to non-heat treated ones. Increasing the number of passes, the average grain size in the nugget zone decreased and according to the Hall-Petch relation UTS improved.

4. Conclusions

Effect of heat treatment on microstructure, microhardness and tensile strength during friction stir processing of cast magnesium alloy AZ91C has been investigated. Also the effect of additional FSP pass numbers on the microstructure, hardness and mechanical behavior of the AZ91C specimens was discussed. The conclusions are summarized as follows:

(1) By performing friction stir processing on the AZ91C base metal, UTS and elongation of the parent metal increased and by performing FSP in more than one pass, distribution of the reinforcement became more uniform and UTS rose consequently.

(2) The base metal microstructure of the as-cast AZ91C applied in this investigation was characterized by a fully divorced eutectic of β-$Mg_{17}Al_{12}$ in α-Mg matrix.

(3) Heat treated samples of friction stir processed as-cast AZ91C consisted of finer grains in stir zone, less precipitates of $Mg_{17}Al_{12}$ in the grain boundaries and fewer porosities and voids relative to non-heat treated specimens. As the number of passes increased, dissolution of β phase increased and the grain size decreased in SZ.

(4) Severe grain refinement caused by the dynamic recrystallization generally lead to an increase in the hardness value of SZ. Higher hardness value was achieved in heat treated samples.

(5) Porosities, voids and β phase ($Mg_{17}Al_{12}$) were controlled by heat treatment and the number of passes. According to Hall-Petch relationship, higher UTS and TE value was achieved in heat treated samples due to finer microstructure in SZ. As the number of passes increased higher UTS and TE was reached due to finer grains and more dissolution of β phase.

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