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Tailoring grain refinement through thickness in magnesium alloy via stationary shoulder friction stir processing and copper backing plate

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Abstract

To develop ultrafine grains (UFG) in 6.35 mm thick magnesium alloy, stationary shoulder friction stir processing (SSFSP) with steel and copper backing plates was conducted. Steel backing plate produced uniform fine grains (FG) size of 4.98, 4.75, 4.12 µm in top, middle, bottom of the stir zone (SZ), respectively. In contrast, copper backing plate tailored microstructure from FG (4.1µm) in the top to UFG (0.96µm) in the bottom of SZ. SSFSP produced uniform and gradient microstructures, altering temperature gradient by placing steel and copper backing plates, respectively. It is worth to note that UFG microstructure achieved without usage of external cooling, owning to the copper backing plate. Most of the grains found under ~2 µm size in UFG microstructure. FG and UFG microstructures contributed to the enhancement in the ductility and strength, respectively. UFG resulted in significant improvement in hardness and tensile strength by ~80% and 24% of the base material, respectively. The intensity of strong basal texture throughout the thickness found independent of the backing plate type. Microstructure evolutions across the SZ thickness for both processing conditions are discussed using electron back scattered diffraction (EBSD).

Keywords: Stationary shoulder; Ultrafine grained microstructure; Friction stir processing; Grain refinement; Copper backing plate.

Introduction

Among the non-ferrous alloy community (Al, Ti, Mg), magnesium and its alloys are the lightest structural material as a possible replacement of the steel and aluminium in automotive industry [1]. Despite of the rare combination of high specific strength and stiffness, magnesium alloys suffer from the low ductility and poor formability at room temperature [2]. To enhance the ductility in magnesium alloy, alloying have shown significant improvement in ductility by specific dilute solute additions [3-5]. Pourbahari et al. [6, 7] demonstrated another approach of hot extrusion to improve ductility in magnesium alloy through grain refinement. Moreover, equal channel angular pressing (ECAP)[8] and friction stir processing (FSP)[9] refine the grain structure via severe plastic deformation.

In recent years, FSP as a thermomechanical and solid-state technique has shown great potential in grain refinement and properties enhancement [9-13]. Apart from the aluminum and magnesium alloys, FSP has started to show potential for the grain refinement in hard alloys [14, 15]. FSP uses non-consumable rotating tool to generate severe plastic and friction deformation into the base material (BM), and thereby results into fine grained microstructure in the stir zone (SZ). FSP of magnesium alloys, especially commercial AZ31 grade, has successfully demonstrated for grain refinement from fine grain (FG) to ultrafine grain (UFG) or nano-grain with strong basal texture [16-19]. Recently, FSP of cast alloy AZ61[20] and ZKX50[21] demonstrated significant grain refinement. However, the magnesium alloys are quite sensitive to the heat in comparison to aluminium alloys, which makes them difficult to process. Therefore, FSP of magnesium alloys has been conducted with the use of external cooling medium for grain refinement as presented the literature in Table 1.

Table 1 Summary of the grain refinement by external cooling FSP in magnesium alloys.

Base material (BM)	Tool rotation and traverse speeds	Average grain size, µm		Ref.
		BM	SZ	
AZ31 rolled	1100-1500 rpm, 60 mm/min	100	2.8-3.8	Chai, Zhang and Li [22]
AZ31 rolled	1000 rpm, 58 mm/min	18	3.35-7.52	Alavi Nia, Omidvar and Nourbakhsh [23]
AZ31B rolled	3200 rpm, 550 mm/min	100	13.3	Darras and Kishta [24]
AZ31 rolled	1000 rpm, 58 mm/min	18	3.4-7.4	Alavi Nia, Omidvar and Nourbakhsh [25]

AZ31 cast	1000 rpm, 37 mm/min	75	0.085	Chang, Du and Huang [26]
AZ61 cast	800 rpm, 240 mm/min	NR	4.6	Luo, Cao, Zhang, Qiu and Zhang [27]
AZ91D cast	600 rpm, 400 mm/min	31	1.3	Xu, Bao and Shen [28]
AZ91 cast	700 rpm, 120 mm/min	NR	0.4	del Valle, Rey, Gesto, Verdera, Jiménez and Ruano [29]
AZ91 cast	600 rpm, 60 mm/min	72 ± 3	2.8 ± 0.8	Chai, Zhang and Li [30]
AE42 cast	700 rpm, 60 mm/min	20	1.5	Arora, Grewal, Singh, Dhindaw and Mukherjee [31]
Mg–Nd–Y cast	800 rpm, 40 mm/min	33	1.9	Cao, Zhang, Zhang and Qiu [32]

NR: Not Reported

Woo et al. [33] reported texture-dependant tensile properties of the SZ in FSPed AZ31B Mg alloy. Yuan et al. [34] exhibited a texture-dependant Hall-Petch relationship between yield stress and fine grain size in the SZ of FSPed AZ31alloy. Recently, Peng at al. [35] demonstrated the separate effect of the grain size and texture on the hardness and tensile behavior of FSPed AZ31 alloy at different tool rotational speeds. To produce nano-grain or UFG via FSP, rapid cooling by different means is mandatory during FSP because of the thermo mechanical nature of the process. Chang et al. [26, 36] used liquid nitrogen cooled FSP to produce UFG microstructure in AZ31 alloy. While Yuan et al. [37] used copper backing plate to increase the cooling rate during FSP of AZ31 alloy, exhibiting the UFG with strong basal texture and their effect on mechanical behavior.

So far, UFG AZ31 Mg alloy sheets in small thickness (2-3 mm) are produced by FSP at rapid cooling. It is obvious that temperature gradient increases as processing thickness increases in heat sensitive Mg alloy, especially in the case of rapid cooling during FSP. Recently, stationary shoulder FSP (SSFSP) demonstrated a probe-dominated SZ with uniform grain refinement throughout the SZ thickness in AZ31 [38] and AA7075 [39] alloys due to the absence of shoulder action. Moreover, stationary shoulder helps to achieve smooth surface appearance with little or no flashes [40, 41]. Therefore, the aim of the present study is to tailor the through-thickness microstructure in thick AZ31 Mg alloy using steel and copper backing plates during SSFSP.

Materials and Method

6.35mm thick commercial grade AZ31B magnesium alloy with starting grain size of ~25µm was subjected to the SSFSP. The stationary shoulder tool with 18mm shoulder diameter and threaded conical probe of 6 mm length was used for FSP. The details of the stationary shoulder can be found in the previous studies [38, 42]. Prior to the selection of process parameters, series of experiments at different rotational speeds (500-1200 rpm) and traverse speeds (100-200 mm/min) were conducted to see the effect of different heat input conditions on resulting SZ. Then after, SSFSP was investigated at the best combination of process parameters i.e., a probe rotational speed of 700 rpm and traverse speed of 150 mm/min using steel and copper backing plates. For the sake of simplicity in discussion, the samples produced by steel and copper backing plates are designated by sample *S* and *C* respectively. In order to understand the effect of backing plate on the microstructure gradient developed in the SZ, the grain refinement has been investigated through the SZ thickness i.e., top, middle and bottom locations (as marked in graphical abstract) using electron back scattered diffraction (EBSD) with a step size of 0.1 µm. EBSD specimens were prepared mechanical polishing followed by electro polishing (10 vol% perchloric acid and 90 vol% ethanol) using a voltage 5 V for 20 s to generate strain free surface. The obtained results were analyzed using CHANNEL 5, Oxford Instruments HKL software. Grain size measurements were conducted using the linear intercept method. Low and high angle grain boundaries were separated by using 15° criterion. A recrystallized grain can be identified in a set of plastically deformed grains by the grain orientation spread (GOS) method. In crystal orientation mapping, a grain is defined to encompass a set of pixels having a misorientation less than the user-specified threshold angle. The grains are defined with the grain tolerance angle above 5° in present GOS method. It was measured by CHANNEL 5 software, with inbuilt function of ‘recrystallized fraction component’. Recrystallized grain criterion of GOS <= 2, GOS >2° <=5°, GOS >5° for recrystallized, substructured, and deformed were used to measure recrystallized fraction, respectively. The Vickers microhardness across the centre of SZ thickness under the loading conditions of 200 g load and 10 s dwell time. The dog bone shape tensile specimens with gage dimensions of 20 mm length, 2 mm width and 1.5 mm thickness were extracted in processing direction (PD) from the SZ. The room temperature tensile testing was conducted at the strain rate of 1×10^{-3} s^{-1}.

Results and Discussion

The probe dominated SZ in both the samples (*S* and *C*) developed due to the nonrotating action of the shoulder at the top of the SZ, as shown in Figure 1(a) and Figure 2(b). As it can be seen from these macrostructures that shape and size of the SZ is independent of the type of backing plate used in processing. The microstructure evaluation the SZ thickness is shown in Figure 1. For sample *S*, the average grain sizes of 4.98 ± 3.29, 4.75 ± 3.26, 4.12 ± 2.11 μm were obtained at the top, middle, bottom of the SZ, respectively. It shows that sample *S* produced almost uniformly distributed FG microstructure throughout the thickness, attributing to the small temperature gradient developed by the stationary shoulder tool and steel backing plate. The processing temperature across the thickness governs the grain growth of the recrystallized grains and hence the grain size. It can be inferred that stationary shoulder acts as a moving "backing" plate, generating shoulder-free deformation zone (refer macrograph of the SZ given in graphical abstract) at the top of the SZ. Therefore, plastic and frictional deformation takes place only by the probe, resulting smaller temperature gradient between a moving backing plate (stationary shoulder) at the top and a rest backing plate at the bottom of BM. Because of the same material of these backing plates, the difference in heat transfer via to the respective backing plate conduction becomes negligible.

For sample *C*, average grain sizes of 4.1 ± 2.2, 3.19 ± 1.77, 0.96 ± 0.4 μm were achieved at the top, middle, bottom of SZ (see Figure 2). Such a dramatic reduction in the grain size attributed to the rapid cooling by copper backing plate underneath the BM. Moreover, similar crystal orientations among the grains in the middle and bottom of the SZ can be seen in sample *C*. Copper backing plate remarkably increases rate of heat conduction at the bottom in such a way that unidirectional heat flow takes place from top to bottom, and thereby rate of grain growth decreases rapidly in the direction of heat transfer. Therefore, copper backing plate developed eye-catching microstructure gradient across the thickness, unlike steel backing plate.

The grain refinement in sample *C* exhibits the tailored microstructure ranging from FG to UFG while moving from top to bottom in the SZ, unlike the sample *S*. The UFG microstructure at the bottom of the SZ achieved due to the availability of the rapid sink by copper backing plate. Similarly, Yuan et al. [37] reported UFG microstructure with an average grain size varying from 0.7 to 0.6 μm from the top of SZ to bottom in FSPed AZ31 thin sheet (2 mm thick). Chang et al. [26, 36] used liquid nitrogen cooling (very fast cooling rate) to achieve UFG microstructure in FSPed AZ31 thin sheet (3 mm thick). The grain boundary maps across the SZ thickness for sample *S* and *C* are presented in Figure 3 and Figure 4, respectively. The large fraction of high angular grain boundaries (HAGBs) observed through thickness in both the samples. The grain size across the thickness found almost uniform in sample *S* as compared to sample *C*, as shown in Figure 5 (a). Moreover, effect of backing plate on grain size distributions across the thickness in sample *S* and *C* can be well understood from Figure 5 (b, c). For sample *C*, significant difference in grain size distribution at bottom was found in comparison to the top and middle region. The limited fraction of the grains near copper backing plate (i.e. bottom region) obtained < 1 µm grain size (termed as ultrafine-grained), while sample *S* found without any significant difference in the grain size distribution across the thickness. Therefore, it can be inferred that backing plate also influences the grain size distribution in addition to the grain refinement. The grain refinement in SZ results from the dynamic recrystallization during the process. Hence, the fractions of recrystallizations in the FG and UFG structures are to be considered for an effective grain refinement. Sample *S* reported slight increasing trend of fraction of recrystallized grains as the SZ thickness increases, as shown in the EBSD recrystallization maps in Figure 6. The UFG microstructure grains exhibited the highest fraction of the recrystallized grains ~80% with almost null fraction of the deformed grains, as depicted in Figure 7. It can be inferred that fraction of recrystallized grains increases while moving from FG to UFG.

Figure 8 and Figure 9 show strong basal (0001) texture developed throughout the thickness in sample *S* and *C*, respectively.. For sample *S*, the intensity of the basal poles is the lowest near the top of SZ, inferring the lack of plastic flow occurred near the top surface of SZ due to the non-rotating action of the shoulder. In contrast, Vargas et al. [21] reported the highest intensity of texture near the top SZ in FSPed cast ZKX50 alloy, because of the intense plastic flow by rotating shoulder tool processed cast alloy. The middle and bottom of SZ in sample *S* developed texture with same intensity as well as orientation (rotation of basal pole about ND ~45°), revealing the consistency in corresponding shear deformation by probe.

On the other hand, sample *C* showed the small variation in the intensity of texture through thickness in comparison to sample *S*. The orientation of the basal pole in sample *C* can be observed parallel to the ND, and it moves towards PD as thickness increases, becoming almost parallel to PD at the bottom of SZ. Sample C texture orientation through thickness is in the good agreement with the previous study of FSPed 1.5 mm thick AZ31 alloy [37]. It is well known that texture evolution in Mg alloy depends on material flow around the rotating tool, and it is governed by the FSP parameters and tool geometry [37, 43, 44]. The results of the present study clearly indicate that stationary shoulder minimizes the intensity of the texture at the top of SZ in comparison to the middle and bottom of SZ in case of both the samples. Despite of the same SSFSP parameters and tool geometry used in both samples, the cooling influences distribution of texture through thickness, similar observation reported by Huang et al. [45].

Enhancement of mechanical properties such as hardness and tensile properties in the FSPed alloys may be achieved by different strengthening mechanisms. These strengthening mechanisms include grain size strengthening, solid solution strengthening, precipitation strengthening, and dislocation strengthening. At least three tensile specimens for each condition were tested. The statistical value of local mechanical properties, i.e., tensile strength (UTS) and elongation (El) throughout the SZ thickness for both conditions are summarized in Figure 10(b). The resultant properties of SSFSPed sample closely relate to the corresponding microstructure and texture evolution. Hardness distribution through thickness in sample *S* showed slight enhancement but homogenously distributed within the tiny range of 5-10 HV (see Figure 10a). Such a uniform hardness distribution is attributed to the uniform FG microstructure throughout the thickness in sample *S*. The copper backing plate exhibited increasing trend of hardness with the depth, prevailing to the FG to UGF microstructure across the thickness. Because of UFG microstructure at the bottom of SZ, exceptional increase in the hardness about 75-80% of the BM was achieved. This hardness distribution confirms the influence of grain refinements in the SZ and hence grain size strengthening is believed to be key strengthening mechanism for the enhancement in SZ hardness. In addition to that, strong basal texture also contributed to the improvement in SZ hardness.

The remarkable improvement in ductility achieved as an effect of not only rotation of basal pole tilt, but also FG structure, large fraction of HAGBs and recrystallized grains, as shown in Figure 10(b). As it can be seen form the stress-strain curves (Figure 11), the specimens fracture without any pronounced post-uniform elongation. For sample *S*, there is no significant variation in tensile curves of top and middle region, where as the curve of bottom region shows increased UTS due to grain boundary strengthening. However, the elongation of bottom region reduces in comparison to top and middle regions. It is also worth to note that anisotropy in tensile behavior across the SZ thickness for sample *S* do not show any huge difference, owning to uniform grain refinement and special basal pole tilt texture evolution. In contrast to sample *S*, very large gradient in microstructure and the properties found across the SZ thickness in sample *C*. Moreover, this ductility improvement could also be related to the corresponding basal pole rotation in favourable direction to the deformation direction. The significant reduction in ductility from FG to UFG is due to stress localization. It is interesting to see the local tensile properties across the SZ, where FG of sample *C* exhibited minimum anisotropy in comparison to those of sample *S* because of the similar basal pole tilt developed for FG structures of sample *C*. Hence, the local tensile anisotropy is investable in Mg alloys due to the texture variation, same has been reported in the previous studies of FSPed AZ31 alloy [46, 47]. The grain refinement effect was found dominant over strong basal texture effect for ductility improvement. Meanwhile, FG structure could accommodate more plastic deformation and resulted in higher elongation [17]. FG grains with inclined texture obtained higher elongation in comparison to the strong basal texture of UGF grains. So, it can be inferred as UFG structure contributes to the high strength, whereas FG structure results into good ductility [35, 48, 49]. The texture evolved in sample *C* was dominated by the strong basal texture, with the basal plane oriented nearly parallel to the normal direction. This means that the tension along the processing direction should result in nearly-zero Schmid factor for the basal slip and thus the ductility should be very low. Hence, the ductility of the UFG structure reduced by 15% of that of the BM, believing the combined deformation mechanism of grain size strengthening and very strong basal texture. Moreover, this reduction in the ductility can further be accelerated as the grain refinement increases beyond the present UFG size, as reported in the previous study [50] that UFG structures of 0.7 µm and 0.4 µm obtained 9.5 % and 3 % ductility, respectively. Overall, Sample *C* could be expressed as tailored Mg alloy that offers FG as well as UFG structures across the thickness without using any external source of cooling. On the other side, sample *S* offers uniform FG structures and properties in Mg alloy across the thickness using stationary shoulder tooling system. Figure 12 shows the SEM images of the fractured FG and UFG microstructure mini-tensile specimens across the thickness. The deformation behavior of bottom of SZ in sample *C* exhibited dimple structure with tearing edges. Also, few dimples are found to be deeper in nature in bottom of SZ in comparison to the middle of SZ for sample *C*, owning to the UFG microstructure at the bottom of SZ. The significant reduction in the ductility from FG to UFG microstructure is believed due to stress localization. For sample *S*, the deformation behavior (mixture of flat-shaped cleavages and dimples) across the thickness was more or less similar due to the uniform grain refinement. The local tensile properties in both the samples are attributed to the grain refinement and the texture. The summary of the through thickness grain refinement and corresponding mechanical properties is given in Table 2.

Table 2 Summary of grain refinement and local mechanical properties though SZ thickness

	BM	Steel backing plate: SZ			Copper backing plate: SZ		
		Top	Middle	Bottom	Top	Middle	Bottom
Avg. grain size (μm)	25±2.5	4.98±3.29	4.75±3.26	4.12±2.11	4.1±2.2	3.19±1.77	0.96±0.4
HAGBs (%)	-	70	63	60	62	57	70
Hardness (HV0.2)	45-55	57.7	62.7	63.4	68.3	78.2	88.4
UTS (MPa)	220	165	176	214	188	193	272
El (%)	15.6	27.4	24.3	20.4	22.3	22.9	13.2

Conclusions

Present study demonstrated SSFSP of Mg alloy by using stationary shoulder tool to achieve the uniform grain refinement throughout the thickness. Moreover, the grain refinement throughout the thickness was tailored by using copper backing plate. In summary, stationary shoulder acted as a moving backing plate at the top of BM. SSFSP produced probe-dominated SZ with uniform FG structure through thickness in case of steel baking plate. The microstructure gradient throughout the thickness can be modified by varying the material of backing plate. Grain refinement was tailored from FG to UFG across the thickness via copper blacking plate. UFG benefited in significant enhancement of hardness and UTS by ~80% and 24% of the BM, respectively. While FG delivered noteworthy enhancement of the ductility at the minimum cost of UTS. The strong basal texture developed throughout the thickness, irrespective of type of backing plate used.

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