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Improvement of yield stress of friction-stirred Mg–Al–Zn alloys by subsequent compression

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The AZ61 magnesium alloy shows a lower yielding strength when subjected to friction stir processing (FSP) due to the unique (0002) basal texture that roughly surrounds the stir pin column surface. It demonstrates that a subsequent compression along the normal direction could improve the unfavorable texture by inducing deformation twins, thus raising the yielding stress significantly. A post-FSP compressive strain of ~6% could raise the yielding stress of the AZ61 alloy from 140 to 260 MPa.

Keywords: Magnesium alloy; Friction stir processing; Yielding strength; Compression; Twinning

Friction stir welding (FSW) [1] is a solid-state welding process that has a specific extrusion-like material flow from the retreating to the advancing side at elevated temperatures [2,3]. Friction stir processing (FSP) is similar to FSW, but it concerns the modification of the materials, such as the uniform distribution of the precipitate of cast alloys or grain refinement, by special plastic flow at elevated temperatures. Therefore, FSP has great potential for producing a homogenized microstructure. The application of FSP for the modification of Al alloys has been widely reported [4,5].

Light Mg alloys have also attracted FSP-related research [6–11]. The hardness of the FSP Mg alloys has been reported to be upgraded due to grain refinement [6–8], but the tensile strength, particularly the yielding strength (YS), does not appear to have been improved, and is sometimes even lower than that of the base materials [8,10,11]. In contrast, after extrusion, the same Mg alloys, with the same refined grain size, could possess a higher YS or ultimate tensile strength (UTS) [11]. To explain this phenomenon, Wang et al. [11] rationalized that the extrusion process would result in the (0002) basal planes of Mg alloys lying parallel to the extrusion direction, thus making the operation of the basal slip system more difficult as the applied stress axis is parallel to the extrusion direction. With FSP, the simple shear and rotating deformation around the pin tool would cause the intense (0002) basal planes to roughly surround the pin column surface of the pin tool [8–11]. Such a significant FSP texture in Mg-based alloys would lead to the easy operation of the basal slip system. In other words, the FSP Mg alloys will possess a lower YS than extruded Mg alloys. This drawback would not occur in face-centered cubic Al alloys.

The low yield stress of the Mg alloys after FSP or FSW is a drawback in their application as structure materials. Any method that can improve this disadvantage is therefore of importance. In this study, a simple concept is demonstrated that can greatly improve the YS of the FSP Mg alloys by 85%.

The AZ61A billets used in this study have a chemical composition (in mass%) of Mg–6.02Al–1.01Zn–0.30Mn. This alloy is a solution-hardened alloy with minimal precipitation. The billet, fabricated through semi-continuous casting, possesses nearly equiaxed grains of around 75 μm (measured by the linear line intercept method). FSP of the as-received billet was performed on a modified horizontal-type miller using an FSP tool with a shoulder diameter of 18 mm, a pin diameter of 6 mm and a pin length of 6 mm. The advancing speed of the rotating pin was kept at 45 mm min^{-1}, with a rotation speed of 800 rpm. Four FSP passes were applied, with the subsequent pass being opposite to and 100% overlapped with the previous pass, in order to

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effectively homogenize the grain structures in the entire stirred zone. The sample used for FSP is rectangular in shape, measuring 130 mm in length, 60 mm in width and 10 mm in thickness. The detailed experimental methods have been presented elsewhere [6,7].

The FSP specimens along the stirred zone were cut into rectangular samples measuring 15 mm in width, 30 mm in length and 10 mm in thickness. The FSP specimens were subjected to a subsequent compression process at room temperature, with the compression axis parallel to the normal direction (ND) of the FSP specimens, as shown in Figure 1. These FSP specimens were compressed to two different levels of strain, one to ~3% (termed as FSP-cp3) and the other to ~6% (termed as FSP-cp6).

The microstructures of the FSP, FSP-cp3 and FSP-cp6 specimens were characterized by optical microscopy (OM) on the transverse cross-sectional plane (the "T plane") and the flat horizontal plane (the "H plane"), as shown in Figure 1. The texture was examined by X-ray diffraction (XRD) using Cu Kα radiation for the T and H planes. Vickers hardness tests were conducted on the T plane, using a Vickers indenter with 200 gf load for 10 s. The FSP, FSP-cp3 and FSP-cp6 specimens were machined into tensile specimens along the welding direction (WD) from the center area of the stirred zone, which is located 2 mm below the top surface. The tensile specimens had a gage dimension of 4 mm in length, 3 mm in width and 2 mm in thickness. Tensile tests were conducted with an initial strain rate of $1 \times 10^{-3}\ s^{-1}$ at room temperature.

Figure 2 shows the fully recrystallized grain structure in the stirred zone of the FSP AZ61 specimen. The grain size is measured to be 7.8 μm on average, compared with the 75 μm in the as-received billet. The microstructure of the FSP-cp3 specimen shows a high number of twins inside the grains, as shown in Figure 2. After further compressive strain to ~6%, the density of twins inside the grains was higher than that of the FSP-cp3 specimen. It is well known that deformation twinning plays an importance role on the plastic deformation for the hexagonal close-packed (hcp) metals. The c/a ratio of the hcp structure would influence the favorable operation of deformation twinning under tension or compression deformation. The magnesium alloys with a c/a ratio of ~1.632 would readily favor the deformation twinning when the imposed stress state is compression perpendicular to the c-axis [12]. From previous research results [8–11], the (0002) basal planes are roughly surrounding the pin column surface, meaning that the c-axis for most grains in the stirred zone would be perpendicular to the ND. It follows that a large number of deformation twins would be induced in the FSP-cp3 and FSP-cp6 specimens during post-FSP compression.

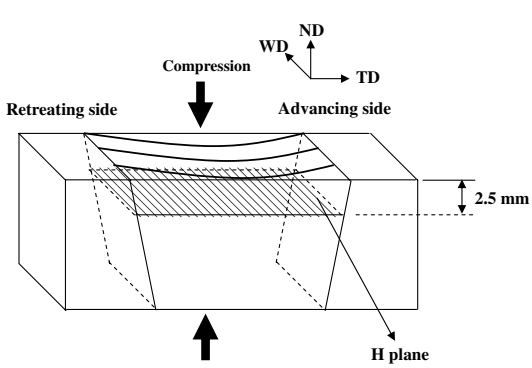

Figure 1. Schematic illustration of the H plane in the stirred zone and the subsequent compression process.

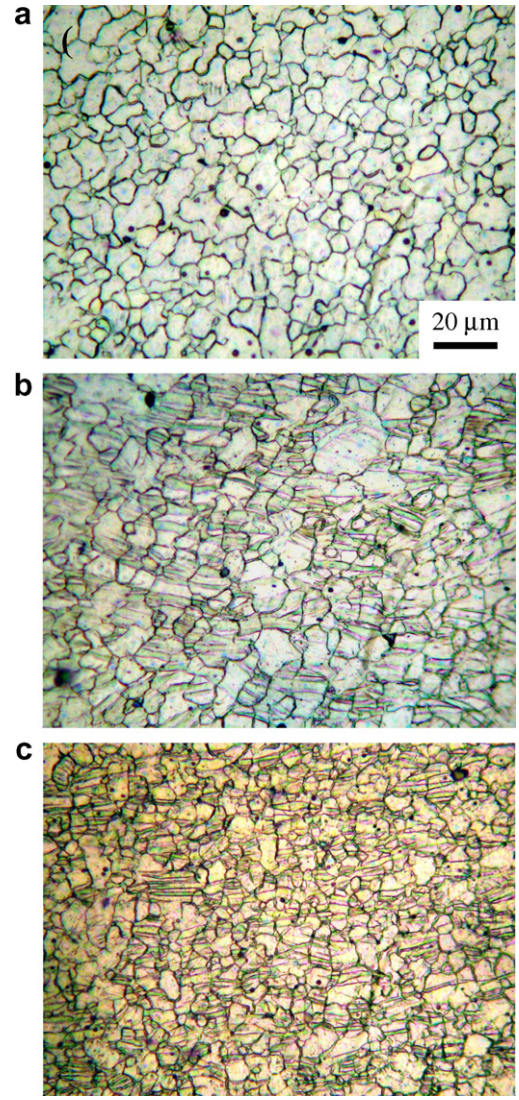

Figure 2. Optical microscopy showing the grain structures of the (a) FSP, (b) FSP-cp3 and (c) FSP-cp6 specimens.

The XRD patterns of the FSP, FSP-cp3 and FSP-cp6 specimens are shown in Figure 3. In order to quantify the XRD results, the intensity for any particular diffracted peak is first normalized by the intensity sum of the three major diffraction peaks, namely the $(10\bar{1}0)$, (0002) and $(10\bar{1}1)$, and then compared with those for the completely random Mg powders. Thus, the relative intensity is written as $\text{Intensity}_{(hkil)} = [I_{(hkil)}/I_{(10\bar{1}0)} + I_{(0002)} + I_{(10\bar{1}1)}]_{\text{sample}}/[I_{(hkil)}/I_{(10\bar{1}_0)} + I_{(0002)} + I_{(10\bar{1}1)}]_{\text{random Mg powders}}$. Table 1 lists the summary for these quantified XRD results. As seen from the T plane of the FSP specimen, the (0002) plane exhibits the relative intensity of ~2.61, while the $(10\bar{1}0)$ plane shows the

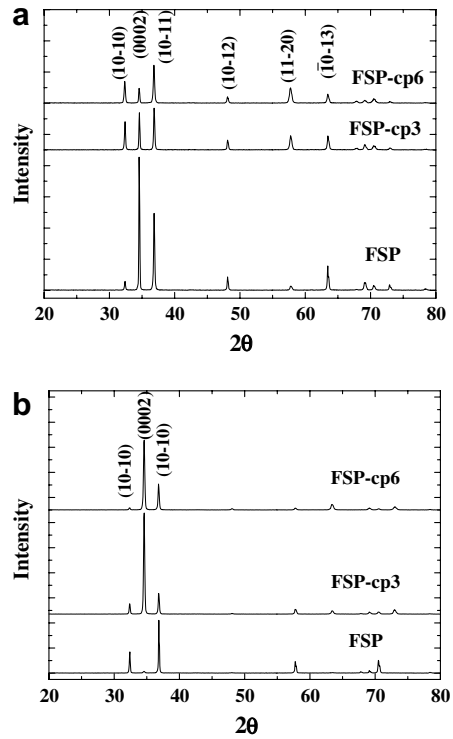

Figure 3. XRD measured from the (a) T plane and (b) H plane for the FSP, FSP-cp3 and FSP-cp6 specimens.

Table 1. Relative XRD intensity measured from the T and H planes

Relative intensity	T plane		H plane	
	(0002)	$(10\bar{1}0)$	(0002)	$(10\bar{1}0)$
FSP	2.61	0.21	0.10	1.41
FSP-cp3	1.52	1.30	3.30	0.39
FSP-cp6	0.87	1.51	3.10	0.11

weaker relative intensity of ~0.21. In the H plane, the $(10\bar{1}0)$ plane has the stronger relative intensity of ~1.41, and the (0002) plane has hardly any intensity. These XRD results of the current FSP AZ61 specimens are consistent with the previous texture research on the FSP Mg alloys [8–11].

After a post-FSP subsequent compression, the FSP-cp3 and FSP-cp6 specimens would transform the grain preferred orientation from (0002) to $(10\bar{1}0)$ planes on the T plane, and the $(10\bar{1}0)$ would also transform to (0002) planes on the H plane, consistent with our previous analysis [12]. The $\{10\bar{1}2\}\langle 10\bar{1}\bar{1}\rangle$ has been identified as the main twinning system for the deformation of magnesium metals or alloys at room temperature. Symmetry conditions require that the second undistorted planes $(\bar{1}012)$, be rotated. This will lead to the (0002) planes in the matrix possessing an angle of 86.3° with respect to the (0002) planes in the twinned domain [12]. In other words, the twinned grains will have a reorientation of nearly 90° after twinning deformation. Therefore, the (0002) planes in the stirred zone of the FSP specimens would be rotated to be nearly perpendicular to the ND (by 86.3° or to say nearly 90°) after subsequent compression. This would make the (0002) peak on the H plane much more intense, and that on the T plane appreciably reduced, in the FSP-cp3 and FSP-cp6 specimens. The texture transformation becomes more intense with increasing subsequent compression strain, implying that the twinned domains gradually grow with increasing accumulated compressive strain.

The microhardness readings, H_v, measured from the T plane of the FSP, FSP-cp3 and FSP-cp6 specimens are depicted in Figure 4. The average hardness of the FSP specimen is around 75 H_v. After compression, the hardness of the FSP-cp3 and FSP-cp6 could increase to 81 and 93 H_v. The increment ratio of the FSP-cp3 and FSP-cp6 specimens relative to the FSP specimen is 8 and 24%, respectively. The minor hardness increment is a result of twinning and work hardening. Table 2 summarizes the hardness for the various specimens.

Figure 5 shows the comparison of tensile engineering stress and strain curves of the FSP, FSP-cp3 and FSP-cp6 specimens. The YS of the as-received billet

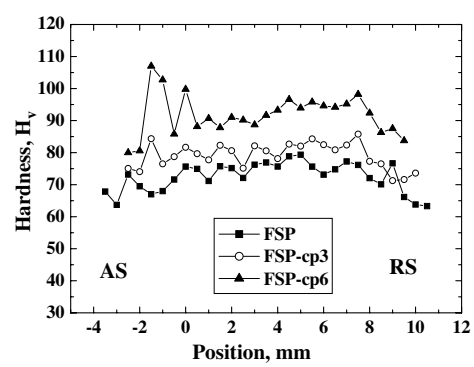

Figure 4. Variation of the H_v microhardness distributions in the FSP, FSP-cp3 and FSP-cp6 specimens; AS and RS indicate the advancing and retreating sides.

Table 2. Comparison of the mechanical properties of the FSP specimens with and without subsequent compression

Material	H_v	YS (MPa)	UTS (MPa)	Elongation (%)
AZ61 billet	60	140	190	13
FSP	75	140	327	18
FSP-cp3	81	178	362	15
FSP-cp6	93	261	366	10

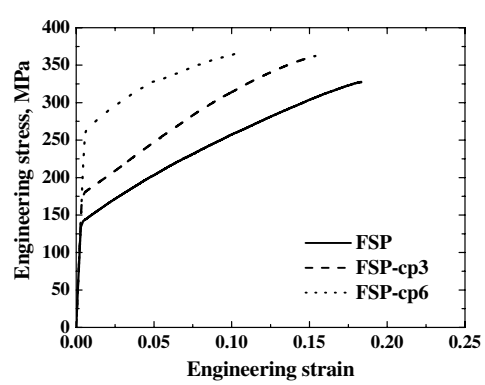

Figure 5. The engineering stress and strain curves for the FSP, FSP-cp3 and FSP-cp6 specimens.

was 140 MPa. After FSP modification, the YS was not improved, staying around 140 MPa, but the grain size was refined from 75 μm to 7–8 μm. The weak grain size dependence of YS for the FSP Mg alloys is due to the specific texture in the stirred zone [11]. However, the YS of the FSP-cp3 and FSP-cp6 specimens could be raised to 178 and 260 MPa, respectively. The increment ratio of the FSP-cp3 and FSP-cp6 specimens relative to the FSP specimen is 27% and 86%, respectively. It is considered that both twinning strengthening and work hardening would contribute to the stress increment.

The basal slip system, $\{0002\}\langle 11\bar{2}0\rangle$, played an important role in the tensile deformation of magnesium metals or alloys because it needs a lower critical resolved shear stress (CRSS). However, the other, non-basal, slip systems, $\{10\bar{1}0\}\langle 11\bar{2}0\rangle$ and $\{10\bar{1}1\}\langle 11\bar{2}0\rangle$, need a higher CRSS [13]. The Schmid factor m is defined as $m = \cos\chi \cos\lambda$, where χ is the angle between the normal of the slip plane, e.g. (0002), and the tensile stress axis, and λ is the angle between the slip direction, e.g. $\langle 11\bar{2}0\rangle$, and the tensile stress axis.

The χ angle between the normal of the basal (0002) planes in the twinned grains and the tensile stress axis along WD is only 3.7° (0.021π), from 86.3° to 90°. The optimally oriented slip direction, $\langle 11\bar{2}0\rangle$, is lying on the plane defined by the normal of the basal plane and the stress axis. This corresponds to the smallest possible λ angle for a given χ angle, leading to the highest value for $\cos\lambda$. Therefore, the term of the λ can be expressed as $\cos\lambda = \cos[(\pi/2) - \chi] = \sin\chi$. Taking into account the symmetry of the $\langle 11\bar{2}0\rangle$ slip direction rotated up to $\pm\pi/6$ about the c-axis of the optimally oriented grain, the average cosine function from $-\pi/6$ to $\pi/6$ can be weighted by the factor $3/\pi$ as $\cos\lambda = (3/\pi)\sin\chi$. Therefore, the factor m as a function of χ can be written as $m(\chi) = (3/\pi)\cos\chi\sin\chi$. The average Schmid orientation factor m for the twinned domains can be obtained as follows:

$$\frac{1}{0.021\pi}\int_{86.3^\circ}^{90^\circ}\frac{3}{\pi}\cos\chi\sin\chi\,d\chi \approx 0.03. \quad (1)$$

In addition, based on our previous research results [11], the Schmid factor m for the FSP magnesium alloys (without subsequent compression and thus without extensive twins) has been calculated to be ~0.3. Therefore, the Schmid factor for the FSP-cp3 and FSP-cp6 specimens can be qualitatively expressed as $m(V) = 0.3V_{\text{matix}} + 0.03V_{\text{twin}}$, where V is the volume fraction of the matrix and twinned domains for the FSP + compression specimens. Based on the above XRD results, it is speculated that the volume fraction of twinned domains in FSP-cp6 is higher than that in FSP-cp3, therefore, $m_{\text{FSP-cp6}}$ would be lower than $m_{\text{FSP-cp3}}$. It is well known that the relationship of the tensile stress and CRSS for the basal slip system can qualitatively be expressed as $\sigma_{\text{tension}} \propto \tau_{\text{basal}}/m$. From this equation, the YS for these specimens would exhibit the relationship $\sigma_{\text{FSP-cp6}} > \sigma_{\text{FSP-cp3}} > \sigma_{\text{FSP}}$. However, if the subsequent compressive strain is too large, it will result in microcracks or even fracture of the FSP specimens. Therefore, an appropriate compressive strain of 3–6% could improve the low YS of the FSP Mg alloys at room temperature by inducing deformation twins and transforming the FSP texture.

In summary, the subsequent compression after FSP along ND can transform the (0002) basal plane to lie perpendicularly to the ND direction by deformation twinning. The new (0002) planes orientation in the twinned domains can lower the Schmid factor and raise the YS from the original ~140 MPa of the FSP specimen to ~260 MPa for the FSP-cp6 specimens. It demonstrates that a simple second processing of subsequent compression to 3–6% is an effective way in improving the low YS for FSP magnesium alloys.

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