Using Multiple FSP Passes to Cure Onion Splitting of Mg Alloys Deformed at Elevated Temperatures

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The investigation of high temperature mechanical properties of the friction stir processed AZ61 alloy with different passes shows that the one-pass processed AZ61 Mg alloy exhibits inferior superplastic deformation ability along the FSP forward traveling direction due to the onion-ring pre-mature splitting. The homogenous microstructure in the multi-pass friction stir processed Mg alloy can avoid the onion-ring splitting and eliminate the anisotropy behavior of high temperature mechanical properties. [doi:10.2320/matertrans.48.780]

(Received January 9, 2007; Accepted February 7, 2007; Published March 25, 2007)

Keywords: magnesium alloys, friction stir processing, elevated temperature deformation, onion splitting

1. Introduction

Friction stir processing (FSP), a development based on friction stir welding (FSW),¹ is a new solid state processing technique for microstructural modification in metallic materials, such as the generation of fine-grained microstructure and homogenization of the microstructure of metallic matrix composites.²,³ The microstructural characteristics of FSP light metals (such as Al and Mg alloys) have been widely investigated and found that the concentric “onion rings” are the most prominent features in the stirred zone.⁴,⁵ Although a lot of works on the formation of onion rings have been done, the effects of the onion rings on properties are not clearly understood. Only some of authors have reported the influence of onion ring on the mechanical properties at room temperature.⁶-⁹ As for the mechanical properties at elevated temperatures, the influence of onion rings has not been addressed.

The superplastic deformation behavior of FSP aluminum alloys has been discovered and some recognized conclusions have been established so far.²,¹⁰-¹³ The tensile specimens for superplastic tensile tests were usually machined along the transverse direction, where the loading direction is perpendicular to the welding forward direction and the specimen gage section is centered in the fine-grained FSP nugget. Based on the fine and equiaxed microstructures in the FSP alloys, the superplastic deformation behavior in these specimens should be basically isotropic. But the tensile results of the FSP Mg alloys at elevated temperatures reveal different behavior. It is interesting to note that the AZ61 Mg alloy subject to one-pass FSP exhibits relatively poorer ductility along the forward advancing direction as compared with that along the transverse direction. Meanwhile, the tensile specimens along the advancing direction appear the onion splitting at elevated temperatures. Here the onion splitting is referred to the phenomenon that the tensile specimens exhibit a local cracking along the onion rings at elevated temperatures, as a result of the local inhomogeneous grain structures associated with onion rings.

In this study, the influence of onion rings on the fracture behavior of the FSP AZ61 Mg alloys on the tensile deformation at elevated temperatures is examined. Moreover, a simple method, i.e., multi-passes FSP, is demonstrated to effectively improve the onion splitting problem.

2. Experimental Methods

The material used in this study is the AZ61A Mg billets, a kind of solution hardening magnesium alloy with the chemical composition of Mg-6.02%Al-1.01%Zn-0.30%Mn (mass percent). The billet possessed nearly equiaxed grains around 75 µm (based on the linear line intercept method from three cross-sectional planes). The billet was cut into rectangular samples 60 mm in width, 130 mm in length and 10 mm in thickness.

The simplified FSP machine used in this experiment was a modified form of a horizontal-type milling machine, with a 5 HP spindle. The fixed pin tool was 6 mm in diameter and 6 mm in length. The shoulder diameter was 18 mm, and a 1.5° tilt angle of the fixed pin tool was applied. The pin was machined with screws, and the screw pitch distance was 1 mm. The advancing speed of the rotating pin was selected to be 45 or 90 mm/min, with a fixed pin rotation of 800 rpm. The plates were fixed by fixture and ambient air cooling was applied. In order to maintain the entire fixture at the initial room temperature after each pass, the back plate of the fixture was designed to contain three cooling channels with cooling water passing through them. Using the methods described in our previous papers,¹⁴,¹⁵ the strain rate and the maximum temperature experienced during FSP are around 10³ s⁻¹ and 400°C, respectively.

Multiple passes of FSP are performed on the AZ61 plate, with the pin travel direction being opposite with respect to the previous pass. FSP experiments using one pass (1P) up to four passes (4P) were tried in this study. In order to observe the microstructure in three-dimension of the samples, the horizontal flat plane (called the “H plane”), the longitudinal side plane near the central position of the stirred zone (called the “L plane”) and the transverse cross-sectional plane

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(called the “T plane”) were grinded, polished and etched. The location of the H plane was chosen to be 2 mm below the surface in order to prevent from the surface oxide effect. The location of the L plane was cut along the central line of the stirring zone. The etching solution was comprised of 2.1 g picric acid, 35 ml methanol, 5 ml acetic acid and 5 ml water. These specimens were examined by optical microscopy (OM) or scanning electron microscopy (SEM). The texture was examined by X-ray diffraction (XRD) using the Cu-Kα radiation for the T and L planes. Tensile tests were conducted on the FSP specimens with a gage length of 3–4 mm; with the loading direction parallel or perpendicular to the FSP forward traveling direction and termed as the WD and TD specimens, respectively. The tensile tests were performed at 300 and 400 °C, with an initial strain rate ranging from $10^{-4}$ s$^{-1}$ to $10^{-2}$ s$^{-1}$.

3. Results and Discussions

3.1 Microstructures

Figure 1 shows the optical micrographs at different locations for the 1P45 (one pass at 45 mm/min) and 4P45 (four passes at 45 mm/min) modified alloys, taken from the H planes. The 1P45 alloy reveals the inhomogeneous microstructures from the advancing to retreating side, but the 4P45 alloy exhibits more homogeneous microstructures. For the 1P45 modified alloy, the grain structure shows different contrasts for the relatively darker and lighter ring layers. The
darker ring layer is the fully recrystallized region, exhibiting the well-defined grains measuring around 5–10 μm. In contrast, the lighter ring layer region contains relatively ill-defined grains and tends to be slightly larger measuring greater than 10 μm, suggesting that the grains within this region were incompletely recrystallized after one pass. For the L and T planes, the mixed grain structures containing well-defined and ill-defined grains could also be observed in the 1P45 specimens, as shown in Fig. 2. After four passes, the microstructures become remarkably improved, as viewed from all of the H, L and T planes. The average grain size in the 4P45 specimen is 7.8 μm.

The situations appears to be similar for the 1P90 and 4P90 (at 90 mm/min) specimens, but the average grain size is lower measuring 3.4 μm. The higher advancing speed of 90 mm/min at the same rotational speed of 800 rpm would lead to a lower heat input and even more severe inhomogeneous layer structures. Lower the advancing speed (e.g., to 25 mm/min) or increasing the rotation per minute (e.g., to 1400 rpm) would promote a higher degree of recrystallization and lessen the inhomogeneity, but would induce the larger recrystallized grain size.15) The latter would in-turn result in the poorer ductility at elevated temperatures.16) In comparison, the invoking of multiple FSP passes appears to be effective and feasible scheme in improving this inhomogeneous microstructure.

On the basis of the grain structures seen from the H, L and T planes, a simple schematic illustration of the three-dimensional grain structures in the stirred zone of the modified alloy after 1 pass FSP, The gray-grain layers are referred to the partially recrystallized larger grains, and the lighter layers are referred to the fully recrystallized smaller grains.

3.2 XRD results

The XRD patterns of the 1P45 and 4P45 specimens are shown in Fig. 4. In order to quantify the XRD results, the intensity for any particular diffracted peak is firstly normalized by the intensity sum of the three major diffraction peaks, namely, the (10i0), (0002) and (10i1), and then compared with those for the completely random Mg powders. Thus, the relative intensity is written as

\[
\text{Intensity}_{(h\ kl \ l)} = \frac{I_{(h\ kl \ l)} / I_{(1010)} + I_{(0002)} + I_{(1011)}}{I_{(h\ kl \ l)} / I_{(1010)} + I_{(0002)} + I_{(1011)}}\text{random Mg powders}
\]

![Figure 3](image-url) Schematic illustration of the grain structure in the stirred zone of the modified alloy after 1 pass FSP. The gray-grain layers are referred to the partially recrystallized larger grains, and the lighter layers are referred to the fully recrystallized smaller grains.

![Figure 4](image-url) X-ray diffraction patterns obtained from the (a) H plane, (b) L plane, and (c) T plane for the 1P45 and 4P45 modified alloys.

<table>
<thead>
<tr>
<th>Relative</th>
<th>1P45</th>
<th>4P45</th>
</tr>
</thead>
<tbody>
<tr>
<td>intensity</td>
<td>(0002)</td>
<td>(1010)</td>
</tr>
<tr>
<td>H plane</td>
<td>0.11</td>
<td>2.91</td>
</tr>
<tr>
<td>L plane</td>
<td>0.24</td>
<td>1.57</td>
</tr>
<tr>
<td>T plane</td>
<td>2.18</td>
<td>0.28</td>
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Table 1 lists the summary for these normalized XRD results. As seen from the T plane of the FSP specimen, the (0002) planes exhibit the relative intensity $C_{24}^2 = 5$ while the (10) planes show the much weaker relative intensity $C_{24}^0 = 25$, indicating the strong planar texture in both the 1P and 4P specimens. In the L and H plane, the (10) planes have the stronger relative intensity greater than 1.4, and the (0002) planes hardly have any intensity. These XRD results show that one-pass and multi-passes FSP have the similar texture and are consistent with the previous texture research on the FSP Mg alloys.\textsuperscript{17–19} It is confirmed that the specific FSP texture in the stirred zone, where the (0002) basal planes tend to lie surrounding the stirring pin, is persistently maintained even with increasing FSP pass number.

3.3 Tensile behavior at elevated temperatures

Tensile loading was conducted at 300 and 400°C. However, independent of the 1P or 4P specimens stirred at 45 or 90 mm/min, the tensile elongations at 400°C are consistently below 100% due to the rapid grain growth during the heating and deformation at elevated temperatures.\textsuperscript{20} The focus was placed on the behavior at 300°C. The representative stress-strain curves of the WD and TD specimens for the 1P45 and 4P45 modified alloys are presented in Fig. 5. It is noted that the flow stress of the WD specimens at $1 \times 10^2$–$1 \times 10^3$ s$^{-1}$ were higher than that of the TD specimens for the 1P45 FSP modified alloys, suggesting that the material flow or the grain boundary sliding did not proceed smoothly in the WD specimens. The latter might be a result of the inhomogeneous microstructures in the 1P specimens.

Figure 6(a) shows the tensile elongation variation of the WD specimens loaded at 300°C as a function of loading strain rate. It can be seen that the ductility of the 1P45 specimen did not exceed 200% throughout. It will be demonstrated below that the lack of satisfactory superplasticity is due to the premature failure as a result of onion splitting. In our previous research results for the similar Mg
alloys\textsuperscript{21–24}) which have been subjected to severe extrusion (with reduction ratio over 100 : 1) or equal channel angular pressing (6 or 8 passes) to result in a grain size less than \(10 \mu m\), the superplasticity elongations were much higher, and even exhibiting the low temperature superplasticity (LTSP) or high strain rate superplasticity (HSRSP) behavior. In contrast, the current one-pass FSP modified Mg alloys with a grain size of 3–8 \(\mu m\) could not perform the comparable superplasticity. The situation of the 4P45 specimens appears to be superior, exhibiting tensile elongations greater than 200\% at a lower strain rate of \(1 \times 10^{-3} \text{s}^{-1}\).

Figure 6(b) shows the elongation variation of the TD specimens loaded at \(300^\circ \text{C}\) and \(1 \times 10^{-3} \text{s}^{-1}\): (a) the 1P45 WD specimen, (b) the 1P45 TD specimen, (c) the 4P45 WD specimen, (d) the 4P45 TD specimen, and (e) the higher magnification of the 1P45 WD specimen.

3.4 Fractured surface examinations

On examination of the failed tensile specimens, the 1P specimens along the WD direction always exhibit the onion-like splitting phenomenon after deformation at elevated temperatures, as shown in Fig. 7(a). The splitting arcs are nearly perpendicular to the loading direction. The same 1P specimens extracted from the TD direction did not possess onion splitting, as shown in Fig. 7(b). In addition, the multi-passes 4P45 modified alloys, independent of the WD and TD specimens, also did not exhibit onion-like splitting, as one example shown in Figs. 7(c) and 7(d).
3.5 Influence of FSP passes on deformation at elevated temperatures

The apparent strain rate sensitivity coefficient \( (m_a) \) for the FSP modified alloys can be calculated employing the following equation,

\[
m_a = \frac{\partial \log \sigma}{\partial \log \dot{\varepsilon}_p}.
\]

on the base of the linear fit for logarithmic stresses over the strain rate range of \( 1 \times 10^{-4} \)–\( 1 \times 10^{-2} \) s\(^{-1} \) at 300°C and a true strain of 0.1. The values of \( m_a \) of all of the 1P and 4P alloys along the WD and TD are all around 0.4. With the consideration of the threshold stress, the true strain rate sensitivity was calculated to be all around 0.5, suggesting that grain boundary sliding might be the dominant deformation mechanism for all of the FSP modified AZ61 alloy.

There should not be severe deformation mechanism differences to result in the anisotropic tensile behavior.

The texture would result in the anisotropic mechanical behavior for the Mg alloys. From the XRD results of 1P and 4P modified alloys, as listed in Table 1 and shown in Fig. 4, the texture appears to be similar for the 1P and 4P FSP modified alloys. Therefore, the texture factor in influencing the mechanical behavior at elevated temperatures might be eliminated.

It is known that grain boundary sliding needs to be accommodated by other mechanisms, for example, grain-boundary migration, diffusion flow, or dislocation slip. The 1P modified alloy possesses the periodical arrangement with the semicircle-like shape in the three-dimensional space for the fully and partially recrystallized grains, as shown in Fig. 3. The partially recrystallized larger grains in the WD specimens might lead to difficulties in functionizing smooth grain boundary sliding and accommodation, and in-turn act as the preferred sites for cavity formation. The interface region between the fully and partially recrystallized layers is subjected to normal and shear stresses during grain boundary sliding deformation. The effect of combined normal stress \( (\sigma_n) \) and shear stress \( (\sigma_s) \) near the inhomogeneity creates strain concentration at the periphery of the induced cavity that is dominated by lateral (circumferential) tensile strain. As shown in Ref. 25, 26), the magnitude of the local tensile strain increases with particle size because the constrained zone of plasticity at the interface becomes larger. Growth of an interface defect by plasticity is a preferred concept for cavity formation because it is energetically more favorable than breaking atomic bonds under high hydrostatic tension.

Once cavity is formed, debonding of this particular interface will occur preferentially until fracture. In the mean time, the effect of hydrostatic tension on other interfaces will be weakened. Consequently, the constant deformation along WD would steadily form the cavities along the interface between the well-defined and ill-defined grain boundary. Finally, the cavities would influence the fracture behavior forming the semicircle-like cracks, as shown in Fig. 7.

Figure 8 shows the schematic illustration of the onion splitting course for the one-pass FSP modified alloy as tensile-loaded parallel to WD. Direction (also the welding direction for the WD specimens). As the tensile direction is applied along TD of the 1P modified alloy, the layers with partially recrystallized larger grains would behave as a long fibrous structure along the loading direction which would not induce onion splitting. Therefore, the TD specimens of the 1P45 modified alloy would perform better ductility than the WD specimens at elevated temperatures. The 4P specimens with a homogeneous grain microstructure could smoothly deform at elevated temperatures, no matter along WD or TD. It is demonstrated that multi-passes FSP could cure the onion splitting through the improvement of the inhomogeneous microstructure.

The analogous situation also occurs during ECAP which needs multiple passes to accumulate the strain for the Mg alloys to fully recrystallize and refine the grain structures. One-pass ECAP at 200–300°C could generate the strain of 0.5–1 for various \( \Phi \) angles; however, the fine grain volume fraction was around 50~60% and these grains were located at the periphery of original and large grains. For ECAP over four passes, the accumulated strain of 2–4 could refine entirely the matrix grains to homogeneous and fine grains via complete dynamic recrystallization. FSW/FSP has been considered as an extrusion-like process from the retreating side to the back of the pin and deposited along the semi-circle arc at the stirred zone. Accordingly, the extrusion ratio is an important variable. Based on the model proposed by Arbogast, the extrusion ration (R) is calculated to be \( \sim 7 \); therefore, the average strain, \( \ln(R) \), is approximated as 1.9. The working temperature was estimated to be around 400°C (0.73 \( T_m \)). One-pass FSP could refine grains from 75 \( \mu m \) to 3–8 \( \mu m \) but not 100% completely recrystallized and refined. After 4-passes FSP, the accumulated strain up to 7.6 (1.9 \times 4) could completely refine all grains and render the microstructure more homogeneous. The current results provide a concept that multi-passes FSP may be a superior and feasible choice to modify and refine the grains more homogeneously.

4. Conclusions

The AZ61 Mg based alloys processed by friction stir processing for one pass exhibits the onion pre-mature
splitting phenomenon as loading along advancing WD direction at elevated temperatures due to the inhomogeneous microstructure consisting of fully recrystallized smaller and partially recrystallized larger grains. Such inhomogeneity of the current FSP alloy would induce onion splitting premature failure and in-turn result in anisotropy of tensile ductility at elevated temperatures. The multi-passes FSP could effectively cure the onion premature splitting by accumulating a higher degree of strain to fully recrystallize the initial grains and to improve the inhomogeneous microstructure. It is suggested that multi-passes FSP may be a necessary practice for magnesium based alloys as the processed alloys are intended to be loaded at elevated temperatures.

Acknowledgements

The authors would like to gratefully acknowledge the sponsorship from National Science Council of ROC under the project NSC 94-2216-E-110-010. The author X. H. Du is grateful to the post-doc sponsorship from NSC under the contrast NSC 95-2816-E-110-001.

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