Correlation between Microstructural Stability and Tensile Properties of FSWed Al-Mg-Mn Cast Plate during Subsequent Thermal Exposure

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Al-Mg-Mn cast plate was friction stir welded at different rotational speeds for studying the thermal stability of FSWed specimen. As-FSWed specimens possessed a typical fine grain structure, 5.5 μm at 450 rpm, 9.5 μm at 650 rpm and 12.8 μm at 850 rpm could be acquired. But for the specimens as raised up the rotational speed to 850–1650 rpm exerts little effect to the coarsening of grain size. On the other hand, a subsequent thermal exposure from 350 to 500 °C for FSWed specimens will cause significant growth of fine dynamic recrystallization grains, especially as the rotational speed decreased to 450 rpm for the fixed traverse speed, 0.55 mm/s. Tensile properties revealed that extreme grain growth after thermal exposure at 500 °C deteriorated the workability of the weld zone. It should be noted that a stored energy release by 300 °C pre-annealing heat treatment was beneficial for avoiding extra grain coarsening. In this study, the optimum rotational speed for improving tensile properties pertaining to microstructural thermal stability could be recognized as 850 rpm. [doi:10.2320/matertrans.M2011050]

(Received February 15, 2011; Accepted May 13, 2011; Published July 25, 2011)

Keywords: aluminum-magnesium-manganese, friction stir welding (FSW), microstructural stability, thermal exposure, workability

1. Introduction

Fully annealed Al-Mg-Mn cast plate possessing equiaxial grains has been used for vapor deposition equipment, where the thermal exposure temperature often reaches to 500 °C; therefore, a stable thermal property is required during repeated used. For friction stir processed samples, our previous report1) had investigated the correlation between microstructural features and tensile properties. Although little difference of tensile yield strength can be found between base metal (BM) and friction stir processed sample, a significant improvement in tensile ductility can be acquired. For high-temperature application, a fully annealed cast sample, which possesses a comparatively isotropic and equiaxial grain structure, is beneficial for the application of repeated thermal exposure condition.

Furthermore, Al-Mg-Mn alloy possessing a high Mg content is difficult to join by conventional fusion welding because this will seriously weaken its mechanical properties. Friction stir welding has emerged as a promising solid-state joining process for many difficult-welded metals. 2) Although this joining method eliminates the solidification defects caused by fusion welding, a significant microstructural stability problem pertaining to grain growth has been recognized during the subsequent heating process. The sample proceeding friction stir process/welding (FSP/FSW) commonly possesses very fine dynamic recrystallization (DRX) grain structure and many previous reports3-6) have focused on the thermal instability of heat-treatable aluminum alloys, abnormal grain growth (AGG) will occur and can be referred to the reduction of pinning force due to precipitates dissolution during the solution treatment. Only a few studies7,8) are concerned with the thermal instability of non-heat treatable Al-Mg-Mn alloy; therefore, the object of this study was to examine the factors that lead to instability of grain structure pertaining to the variation of tensile properties during subsequent thermal exposure of up to 500 °C.

2. Experimental Procedure

The chemical composition of the sample used in this study is listed in Table 1. This Al-Mg-Mn cast alloy sample was received as a hot-top solidified casting slab and sliced into 10-mm-thick experimental sheets. The Al sheet was sustained by a cast iron back plate, and FSW was carried out with a SKH-51 steel tool. The tool dimension was 15 mm in shoulder diameter, 5.5 mm in pin diameter, and 3 mm in pin length. The FSWed parameters were 0.55 mm/s in process speed, 1° in tool tilt angle and 70 MPa in loading pressure, and tool rotational speeds ranged from 450 to 1650 rpm. Subsequent thermal exposure temperature based on practical application was chosen up to 500 °C. The macro- and micro structural features of as-FSWed and thermally exposed specimens were examined in cross-sectional plane (perpendicular to the FSW path) with a stereo- and optically metallurgical microscope. The surface for metallographic analysis was bath polished with Al2O3 slurry and etched in 10% warm phosphoric acid. Grain size d is defined as the equivalent circle diameter, which is calculated by setting the projected areas of each grain (A) equal to the equivalent area of a circle (∆ = πd4/4).

Dog-bone shaped tensile specimens were machined from both perpendicular and parallel to the welding direction. In particular, the perpendicular specimens were machined to possess the weld zone (WZ) positioned in the middle of the specimen gauge, as shown in Fig. 1. All the tensile specimens were ground out 1 mm in thickness from the rough surface of FSWed specimens. Room temperature tensile test was carried out under an initial strain rate of 1.67 × 10−3 s−1.

Table 1 Chemical compositions of base metal (mass%).

<table>
<thead>
<tr>
<th></th>
<th>Mg</th>
<th>Mn</th>
<th>Cr</th>
<th>Fe</th>
<th>Si</th>
<th>Zn</th>
<th>Cu</th>
<th>Ti</th>
<th>V</th>
<th>Al</th>
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<tbody>
<tr>
<td></td>
<td>4.86</td>
<td>0.58</td>
<td>0.06</td>
<td>0.27</td>
<td>0.07</td>
<td>0.04</td>
<td>0.04</td>
<td>0.01</td>
<td>0.01</td>
<td>bal</td>
</tr>
</tbody>
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3. Results and Discussion

3.1 Effect of tool rotational speed on grain size of as-FSWed specimen

Figure 2(a) shows significant grain size refinement observed from the transverse cross section of the FSWed specimens in comparison with BM, which commonly possessed a sharp interface between BM and WZ. From the microstructural features indicated in Fig. 2(b), fully annealed BM specimen is characterized by coarse structure with an average grain size of 80 µm. On the other hand FSW led to a significant grain refinement and spherical broken Al₆(Mn,Fe) particles with an average size of 10 µm, as indicated in Fig. 2(d), which are too large and ineffective in pinning of grain boundaries migration. It is reasonable to propose that Al₆(Mn,Fe) exerted little important effect on AGG in this study. Figure 3(a) exhibits that the grain size of as-FSWed specimens was affected by changing the tool rotational speed and back-plate material. The average grain size increased proportionally to rotational speed as 5.5 µm at 450 rpm, 9.5 µm at 650 rpm and 12.8 µm at 850 rpm. Similar result that the grain size tended to increase with increasing rotational speed was also observed in many previous studies and commonly with respect to the frictional heat input. Frigaard et al. reported that the average heat input per unit time for FSW could be described as \( Q = \frac{4}{3} \pi^2 PR \omega \mu \), where \( P \) was the vertical pressure on the tool, \( R \) was the surface radius of the shoulder, \( \mu \) was the friction coefficient, and \( \omega \) was the
rotational speed. According to the equation, nearly double frictional heat could be achieved in the WZ as raised up rotational speed from 850 to 1650 rpm. From Fig. 3 it should be noted that more heat input did not exert significant effect on further coarsening of grain structure. Detailed statistical data is shown in Fig. 3(b). The grain size distribution curves broadened and shifted to the coarser side as increased the rotational speed up to 850 rpm but demonstrate little effect as further raised the rotational speed even up to 1650 rpm. It is suitable to suggest that the saturation of heat capacity of the cast iron back plate was responsible for suppressing working temperature even stirred at 1650 rpm. For clarification purpose, a stainless steel back plate was substituted and conducted the FSW at an identical condition of 1650 rpm, and the experimental evidence showed that the grain size actually could be enlarged, as indicated in Fig. 3(a). This confirmed that the effect of back plate will play an important role on the grain size of as-FSWed sample and resulted from introduced higher working temperature as well as provides more driving force for grain growth.

3.2 Grain growth feature in thermal exposure

3.2.1 Effect of tool rotational speed

During FSP/FSW, many previous investigations mentioned that the maximum working temperature will be varied within the WZ region. This implies that different high-temperature deformation behavior could be introduced into the stir zone. This factor is indeed closely related to the microstructural stability problem during subsequent thermal exposure application. For quantitatively understanding, an experimental result as shown in Fig. 4, the tensile absorbed energy data demonstrated a significant difference as the BM undergoes tensile test at different elevated temperature. However, the absorbed energy tended to increase with decreasing tensile temperature as well as lower frictional heat input condition of FSW with decreasing rotational speed. Figure 5 is a typical evidence for 450 rpm specimen under a subsequent thermal exposure at 300–500 °C for at least 1 h. It shows that the DRX fine grains in WZ region could maintain the initial condition while thermally exposed at 300 °C even prolonging the duration to 6 h. The onset of grain growth could be recognized at 350 °C with a certain amount of grains growing preferentially from the upper surface and consuming the surrounding fine DRX grains. At the thermal exposure temperatures higher than 400 °C, even the bottom part of WZ became unstable. The grain structure of WZ performed an overall transformation to coarse while the heating temperature arising to 500 °C. However, the grain structure of BM showed a great thermal stability as initial.
state maintain with average grain size of 80 μm. Figure 6 indicates that the structural stability could be improved by increasing the rotational speed; especially, the extent of abnormal grown coarse grains could be suppressed. At 850–1650 rpm the AGG almost did not occur unless the heating temperature arising to 500 °C.

Figure 7 shows the quantitative data of average grain size in WZ region with 1 mm below friction-stirred surface. FSWed specimens stirred with lower rotational speed (850–1650 rpm) introduced higher frictional heat input in the WZ region, which resulted in little difference in the average grain size even when thermally exposed at 500 °C. However, experimental results confirmed that the FSWed fine grains demonstrated different growth behavior, and it should be noted that the specimens with higher rotational speed (850–1650 rpm) the AGG almost did not occur unless the heating temperature arising to 500 °C.

Table 2 Correlation between grain size ratio, $d_{\text{max}}/d_{\text{ave}}$ and AGG.

<table>
<thead>
<tr>
<th>specimens</th>
<th>$d_{\text{ave}}$</th>
<th>$d_{\text{max}}$</th>
<th>$d_{\text{max}}/d_{\text{ave}}$</th>
<th>350 °C</th>
<th>400 °C</th>
<th>500 °C</th>
</tr>
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<tbody>
<tr>
<td>450FSW</td>
<td>5.5 μm</td>
<td>17.1 μm</td>
<td>3.1</td>
<td>AGG</td>
<td>AGG</td>
<td>AGG</td>
</tr>
<tr>
<td>650FSW</td>
<td>9.5 μm</td>
<td>18.4 μm</td>
<td>1.9</td>
<td>Non</td>
<td>AGG</td>
<td>AGG</td>
</tr>
<tr>
<td>850FSW</td>
<td>12.8 μm</td>
<td>37.6 μm</td>
<td>2.9</td>
<td>Non</td>
<td>Non</td>
<td>AGG</td>
</tr>
<tr>
<td>1250FSW</td>
<td>13.7 μm</td>
<td>27.4 μm</td>
<td>2.0</td>
<td>Non</td>
<td>Non</td>
<td>AGG</td>
</tr>
<tr>
<td>1650FSW</td>
<td>14.1 μm</td>
<td>23.5 μm</td>
<td>1.7</td>
<td>Non</td>
<td>Non</td>
<td>AGG</td>
</tr>
</tbody>
</table>

(1) Boarder initial grain size distribution is expected to contribute a larger thermodynamic driving force for AGG. However, in this study the profile of grain size distribution (Fig. 3) and grain size ratio, $d_{\text{max}}/d_{\text{ave}}$ (Table 2) show little compelling correlation between grain size heterogeneity and AGG.

(2) Lower boundary misorientation possesses lower energy and mobility within the higher textured grain assembly. If some grains of another texture component are present, higher boundary energy and mobility promote abnormal grains to grow preferentially. Higher strain and lower working temperature favor both smaller grains and higher proportion of low angle grain boundary. Similar result was also reported in the literature of FSWed AA5083 H-18, in which larger fraction of low angle grain boundary are found in the region of higher strain and lower heat input. In this study the specimen FS450 of lowest rotational speed is supposed to possess a highest proportion of lower angle grain boundary, and which is referred to easier occurrence of AGG.

(3) The heterogeneous dissolution of unstable particles leads to a non-uniform reduction of pinning force, which acts preferential growth of abnormal grains. Several types of particles reported in Al-Mg series alloys included Al₆(Mn,Fe), Al₅Fe, Mg₂Si and Mg₅Al₈. Al₆(Mn,Fe) particles with average size of 10 μm were observed in this study and which had been reported to be less important to grain boundary migration. Al₅Fe is quite stable at 500 °C because of very slight solid solubility in Al matrix (~0.001 mass%). Since sub-micron Mg₅Al₈ and Mg₅Si tend to precipitate on grain boundary and play important roles on boundary pinning, the reduction of pinning force due to thermal instability is considered as the main factor of AGG. A preheating temperature higher than 350 °C can completely eliminate Mg₅Al₈. Mg₅Si starts to partially dissolve in the heat treatment of 500 °C.

3.2.3 Pre-annealing heat treatment

Figure 8 indicated an optical microstructural feature of the FSWed specimen with rotational speed 450 rpm in transverse cross section after strain-energy-release treatment at 300 °C for 3–6 h and subsequent thermal exposure at 500 °C for 1 h. Although a certain amount of coarse-grown grains still can be observed in the center of the WZ, it should be noted that the abnormal-growth grains with sizes larger than 1000 μm could be avoided by this 300 °C pre-annealing heat treatment.
Except heterogeneity of second phase particles, grain size and textural components, the non-uniform dislocation density in WZ also accelerate anomalous coarsening phenomenon in post weld heat treatment. A maximum critical temperature of 300°C was chosen for pre-annealing heat treatment due to the instability of grain structure at temperature above 350°C. The anomalous coarsening phenomenon in the subsequent thermal exposure can be suppressed by pre-annealing heat treatment at 300°C for 6h. In comparison with as-FSWed specimen, pre-annealing specimen is lower and much more homogeneous in microhardness profile, especially in the region with depth between 1.8 to 2.8 mm, as shown in Fig. 9. The region is similar to the central WZ with extremely large grains of the thermal exposed specimen. According to the observation in the previous and present studies, 300°C is insufficient for dissolution of second phase particles, grain growth and texture development. The stored energy release and dislocation elimination by pre-annealing heat treatment are considered as the effective factor to reduce the driving force for the anomalous coarsening phenomenon in the thermal exposure.

### 3.3 Correlation of grain growth and tensile properties after thermal exposure at 500°C

Figure 10(a) showed the ultimate tensile strength and elongation of the specimens with an easy growth rotational speed of 450 rpm. The specimens were machined perpendicular to the welding direction with the WZ region positioned in the middle part of specimen gauge as indicated in Fig. 1. It should be noted that very little deterioration could be recognized in the yield strength and ultimate tensile strength after 500°C thermal exposure for 1h in comparison with BM. From Fig. 10(b), it indicates that the thermally exposed joint fractures in the BM part. WZ region with coarse grains also offered a fair amount of plasticity during tensile deformation. It could be concluded that either the sharp interface between WZ and BM or the extreme grain growth in WZ after thermally exposure exert little deterioration of the strength and ductility of the FSWed workpieces.
In order to clarify the effect of the extreme grain growth on the workability of FSWed specimens, a tensile test was carried out with specimens entirely cut from WZ and parallel to the welding direction as indicated in Fig. 1. Figure 11 shows the map of ultimate tensile strength and uniform elongation as well as the workability of the WZ region. It could be verified from the experimental evidence that FSW could significantly improve the workability and the tensile deformation resistance tended to increase as lowered the rotational speed that pertained to grain refinement. In this study, it was also suitable to suggest that the rotational speed of 850 rpm was an optimum FSWed condition for improving the tensile properties of WZ, which pertained to the better thermal stability.

4. Conclusion

The correlation between microstructural stability and tensile properties of FSWed Al-Mg-Mn cast plate during subsequent thermal exposure was investigated. The main results were as follows.

(1) FSW significantly refined the grain structure, and the grain size decreased as lowered the rotational speed, which was due to the less frictional heat input. While the rotational speed arising from 850 to 1650 rpm, the saturated condition of systematic heat keeping resulted in a similar working temperature, and it exerted very slightly effect on the grain size of the FSWed zone.

(2) FSWed specimens with lower rotational speed possessed higher stored energy were more susceptible to promote the grain growth during subsequent thermal exposure. The initial temperature for AGG tended to increase as increasing rotational speed. The shoulder-

Fig. 8 Macrostructure of FSPed specimens with condition of 450 rpm after stored-energy-release heat treatment at 300°C for (a) 3 h and (b) 6 h, and subsequent thermal exposure at 500°C for 1 h.

Fig. 9 Microhardness profile on the cross-sectioned plane: (a) 450FSW, and (b) pre-annealing heat treated specimen at 300°C for 6 h.

Fig. 10 Tensile test perpendicular to welding direction of FSWed specimen with rotational speed of 450 rpm and the subsequent thermal exposure: (a) tensile property, (b) tensile fracture feature of FSWed specimen and (c) tensile fracture feature of thermally exposed specimen (TME).
contacted region of WZ was the most preferred location for grain growth; on the other hand, the grains at the bottom of WZ grew with smaller size than upper region.

(3) A stored-energy-release heat treatment of friction stir processed specimens at 300°C was beneficial to avoid extreme grain growth and accompanied with homogeneous grain structure within WZ even under subsequent thermal exposure at 500°C.

(4) Tensile ductility of friction stir processed material can be significantly improved, but the microstructural instability problem was easier to occur in the condition of thermal exposure as decreased the rotational speed and resulted in significant deterioration of ultimate stress and uniform elongation.

Acknowledgement

This paper has been supported by the Chinese National Science Council (Contract: NSC-98-2221-E-006-064-MY2), for which the authors are grateful.

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