Microstructural Effects of Zn/Mg Ratio and Post Heat Treatment on Tensile Properties of Friction Stirred Process (FSP) Al- x Zn- y Mg Alloys

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The workability and ductility of Al-Zn-Mg alloy are lower. In this study, the Zn content (4.44–6.34 mass%) was reduced and the Mg content (1.11–1.28 mass%) was raised to investigate the relationship between microstructures and tensile mechanical properties of Al- x Zn- y Mg aluminum alloys after friction stir process (FSP). The ZM35 alloy (Zn/Mg ratio is 3.5) and the ZM57 alloy (Zn/Mg ratio is 5.7) had a friction stir process and then three artificial aging treatments were performed to discuss the effects of Zn/Mg ratio on mechanical properties. After FSP and then natural aging for 12 days, the results of the experiment showed that the tensile strengths of ZM35 and ZM57 alloys were lower than the base metal (extruded material with T6, no FSP). Notably, the strength of as-FSP specimens was promoted after aging treatments. Through the mode I (80°C/16 h) treatments and the mode II (80°C/16 h + T6) treatment, the strengths of ZM35 alloy and ZM57 alloy were improved. However, after the mode III (T4 + 80°C/16 h + T6) treatment, the tensile strength and ductility of the ZM57 alloy reduced, and the deterioration rate of ductility reached to 65%; the ZM35 alloy possessed a better ductility, which confirmed the fact that decreasing Zn/Mg ratio improved the workability of friction stir Al-Zn-Mg specimens with an artificial aging treatments. [doi:10.2320/matertrans.M2012003]

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1. Introduction

Al-Zn-Mg series aluminum alloy (7xxx) are the heat treatment alloys which produce precipitates (such as MgZn_2) and widely apply as large structural components in navigation and transportation facilities, due to their good combination of strength, hardness and fracture toughness.^{1–3)} The main addition elements of Al-Zn-Mg series aluminum alloy are Zn and Mg. Different Zn/Mg ratios not only affect the workability but also dominate the mechanical properties.^{4,5)}

Friction Stir Weld (FSW) is a technology of solid-state joining. The temperature of welding process cannot reach the melting point, so there are not some porosities and shrinkage cracks inside the materials. Friction Stir Process (FSP),^{6,7)} using the same methodology as FSW, produces the fine recrystallized grains in the stir zone due to the dynamic recrystallization from severe plastic deformation, and further affects the mechanical properties.

The reports of friction stir welding in 7xxx series aluminum alloys indicated^{8,9)} that both strengths of 7050 and 7075 containing higher Mg content after FSP were lower than that of base metals (7050 and 7075 alloy), and the ductility of 7075-FSP alloy deteriorated obviously. The FSP characteristics of Al-Zn-Mg alloys containing different Zn/Mg ratio has not been investigated and the ductility problems of friction stirred zone should be improved by controlling artificial aging conditions. In this study, after FSP and different artificial aging treatments, Al- x Zn- y Mg aluminum alloy containing lower Mg content (Zn/Mg ratio was 3.5, ZM35; Zn/Mg ratio was 5.7, ZM57) was compared with the effects of Zn/Mg ratio on the microstructures and tensile properties. In addition, the mechanism of artificial aging treatment how to promote the applications of Al- x Zn- y Mg aluminum alloy with FSP was also studied.

Table 1 Chemical composition of ZM35 and ZM57 specimens (mass%).

	Zn	Mg	Mn	Cr	Zr	Fe	Ti	Si	Cu	Al
ZM35	4.44	1.28	0.35	0.09	0.11	0.15	0.03	0.09	0.05	Bal.
ZM57	6.34	1.11	0.21	0.23	0.1	0.09	0.04	0.05	0.05	Bal.

2. Experimental Procedure

Two Al-Zn-Mg extrusion alloys containing different Zn/Mg ratios (Zn/Mg ratio is 3.5 as ZM35; Zn/Mg ratio is 5.7 as ZM57) were used in this study. The chemical compositions of ZM35 and ZM57 specimens were listed in Table 1. The specimens under the solution treatment, 80°C/16 h and T6 treatment to obtain ZM35-T6 alloy and ZM57-T6 alloy that were used as a base metal (BM). The processing direction of the FSP was parallel to the extruding direction. Figure 1 shows the schematic illustration of the friction stir process (FSP) and the friction stir tool. In the experiment, advancing side (AS) was defined as the direction of tool rotation with the same direction as the processing direction; retreating side (RS) was defined as the direction of tool rotation with the reverse direction of processing direction. According to plastic deformation degree and the heat-input in the stir zone, it can be distinguished stir zone (SZ), thermomechanically affected zone (TMAZ) and heat-affected zone (HAZ).¹⁰⁾

Tool rotation speed was 1670 rpm and the tool moving speed was fixed at 0.46 mm s^{-1} with 1.5° of tool angle. The downward push force was controlled at 30 MPa. The specimens of base metal would be designated as “ZM35-BM” and “ZM57-BM”. To investigate the improvement on the strength of stir zone (SZ) under different artificial aging conditions after FSP, three different artificial aging conditions were performed, including mode I (80°C/16 h), mode II (80°C/16 h + T6) and mode III (T4 + 80°C/16 h + T6).

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Fig. 1 The schematic illustration of FSP and friction stir tool.

All matrices were analyzed to understand the effects of Zn/Mg ratio on microstructures and micro-hardness distribution. Vickers-Hardness (Hv) was tested with a load 0.98 N for 10 s. In addition, the tensile mechanical properties of FSP specimens and the different artificial aging specimens were performed. The direction of tensile specimen was parallel to the extruding direction (friction stir direction) and the location was controlled in the stir zone. The strain rate was fixed at $8.33 \times 10^{-4} \text{ s}^{-1}$. It could be confirmed the effects of artificial aging treatment condition to improve the brittleness of FSP specimens. Notably, the grain size and distribution of precipitates always varied with heat treatment conditions to affect tensile mechanical properties. Hence, the microstructural characteristics were analyzed by transmission electron microscope (TEM: FEI E.O Tecnai F20 G2) with energy dispersive spectroscopy (EDS) to study the relation between the aging matrices and the tensile fracture mechanism of FSP specimens. TEM samples of the stir zone and base metal were a 3 mm diameter disc with 100 μm -thick. The specimens were prepared by an electro-polishing in a solution of 25 vol% HNO_3 and 75 vol% CH_3OH at -30°C to produce electron-transparent thin sections.

3. Results

Both the matrices of ZM35-BM and ZM57-BM aluminum alloys were extrude structure as shown in Figs. 2(a) and 2(b) respectively. Figures 3(a) and 3(b) showed the microstructures for process direction (PD) plane in the SZ after FSP. From the microstructure of ZM35 and ZM57 specimens, there was an obvious refinement in the equiaxed grains of SZ. The average grain size of ZM35 specimen was 24.6 μm and that of ZM57 specimen was 17.4 μm (Fig. 4). For ZM35 alloy, there were obvious friction stir metal flows extending to the bottom of SZ in the AS of TMAZ, but it was not observed in ZM57 alloy.

Figures 5(a) and 5(b) showed the micro-hardness (Hv) of PD plane with FSP after natural aging for 12 days (the test location was 1 mm under the upper surface to avoid the effect of surface traces). The results revealed the hardness of the

Fig. 2 Microstructure of base metals: (a) ZM35 alloy (b) ZM57 alloy.

Fig. 3 Cross-section of PD plane by friction stirred process: (a) ZM35 alloy (b) ZM57 alloy.

ZM35 and ZM57 alloy no obvious drop in the friction stir zone, but they were still lower than that of BM. Compared with the yield strength (YS) and ultimate tensile strength (UTS) of BM, those of ZM35 and ZM57 alloy were deteriorated (Table 2). This phenomenon was consistent with 7050 or 7075 containing high Mg content after FSP.^{8,9)} It can be explained that the tensile strength of 7xxx serial aluminum alloy through FSP was deteriorated. It is the main reason that the grain size and distribution of precipitates of FSP affected the tensile results. Notably, whether it is BM or

Fig. 4 Microstructures of FSP specimens in the SZ (a) ZM35 alloy (b) ZM57 alloy.

Table 2 Tensile properties of ZM35 and ZM57 before and after FSP.

ZM35	UTS (MPa)	YS (MPa)	UE (%)	TE (%)
BM (T6)	398.2	358.2	7.7	15.2
FSP + natural aging 12 days	353.5	223.9	17.1	20.7
ZM57	UTS (MPa)	YS (MPa)	UE (%)	TE (%)
BM (T6)	464.6	394.6	9.3	13.4
FSP + natural aging 12 days	391.8	243.9	16.1	19.6

FSP specimens, the micro-hardness and tensile strength of ZM57 alloy were higher than that of ZM35 alloy. In other words, raising Zn/Mg ratio had better tensile mechanical properties.

The FSP specimens were performed by artificial aging treatment to improve the deterioration of the tensile mechanical properties. Figure 6, Fig. 7 and Table 3 showed the tensile mechanical properties and the deterioration rates of FSP-ZM35 and FSP-ZM57 alloys through three artificial aging treatments respectively. The results revealed the tensile strength of these alloys after mode I (80°C/16h) treatment was similar to that after natural aging for 12 days, and the deterioration rate of YS was 39 and 31% respectively. For mode II (80°C/16h + T6) or mode III (T4 + 80°C/16h + T6) treatments, the tensile strength of these alloys was significantly improved. In addition, compared with mode I

Fig. 5 Micro-hardness of PD plane with FSP after natural aging for 12 days. The test location was a 1 mm under the upper surface: (a) ZM35 alloy (b) ZM57 alloy.

and mode II, when FSP specimens were carried out by T6 treatment, their YS was promoted 44 and 38% respectively. Compared mode II with mode III in the degree of solid solution after FSP by T4 treatment, the tensile strength of ZM35 alloy raised slightly, but that of ZM57 alloy reduced. Notably, both ultimate elongation (UE) and total elongation (TE) of ZM57 alloy reduced to 65%.

4. Discussion

According to previous reports,^{11–14} the heat of FSP caused the precipitates to dissolve into the matrix, which the strength in the friction stir zones decreased. Hence, the precipitates of the base metal specimen and the FSP specimens were analyzed. Figure 8(a) showed the microstructure of the precipitates in the ZM57-BM after solid solution treatment

Fig. 6 Tensile strength of FSP specimens with different artificial aging treatments: (a) ZM35 alloy (b) ZM57 alloy.

Fig. 7 Elongation of FSP specimens with different artificial aging treatments: (a) ZM35 alloy (b) ZM57 alloy.

Table 3 The deterioration of tensile properties after different artificial aging. It can be calculated as (FSP specimens – T6 specimens)/T6 specimens. (–) stands for deterioration.

ZM35	UTS	YS	UE (%)	TE (%)
Mode I (FSP + 80°C/16h)	–11.3%	–38.9%	148%	56.6%
Mode II (FSP + 80°C/16h + T6)	–8.7%	–12%	7.8%	–3.3%
Mode III (FSP + T4 + 80°C/16h + T6)	–6%	–7.3%	–10.4%	–9.9%
ZM57	UTS	YS	UE (%)	TE (%)
Mode I (FSP + 80°C/16h)	–15.6%	–31.6%	93.9%	61.4%
Mode II (FSP + 80°C/16h + T6)	–10%	–5.6%	–25.8%	–9.7%
Mode III (FSP + T4 + 80°C/16h + T6)	–20.9%	–16%	–65.6%	–62.7%

by TEM and many spherical particles (Al_3Zr) distributed in the matrix (Fig. 8(b)). In contrast to the previous studies,^{15–18)} the Al_3Zr particle was coherent with the matrix after solid solution treatment and aged. Notably, the Al_3Zr

particles of Fig. 8 were incoherent with the matrix in the FSP-ZM57 specimen through artificial aging treatment. From previous studies,^{16–18)} the characteristic is related to dynamic recrystallization (DRX) during the FSP because the precipitates in the grain boundaries of DRX do not retain their initial orientation. The precipitates pass through the grain boundaries of DRX that will change their initial orientation, causing incoherent between precipitate phases and matrix.

The main precipitations (Al_3Zr ; $\eta(\text{MgZn}_2)$) of 7xxx series aluminum alloy have different interface energy that can improve the tensile characterization by orientation and the post heat treatments. When the precipitate phases (Al_3Zr) became incoherent phases and their interface energy is higher than that of coherent phases, the stable phases $\eta(\text{MgZn}_2)$ easily precipitated in the phase boundaries.^{17,18)} Figure 9 showed the precipitates of FSP-ZM57 alloy with mode I in the matrix by TEM with EDS. The results revealed the $\eta(\text{MgZn}_2)$ phases precipitated around the Al_3Zr particles to reduce the oversaturated degree of solute atoms in the matrix. Hence, the effect of precipitate strengthening was not obvious and the strength of FSP specimen was lower than that of BM specimen.

Fig. 8 TEM analysis of ZM57-BM alloy after T4 + 80°C/16 h: (a) bright-field image (b) EDS.

Fig. 9 Observations of FSP-ZM57 alloy with mode I in the precipitates and matrix.

The strength (YS) of FSP-ZM35 and FSP-ZM57 alloy specimens after mode I treatment were not improved obviously, the FSP-ZM57 alloy specimen after mode I treatment was examined by TEM. Figure 10(a) showed many fine precipitates distributed uniformly in the matrix, and Fig. 10(b) had the SAD pattern of GP zones in $[001]_{Al}$. According to the pattern, its morphology was similar to

the figure indicated by X. J. Jiang *et al.*^{19–22} as shown in Fig. 10(c), and the structure was belong to GPI. This explained why the FSP specimen after mode I treatment had a limited improvement in the tensile strength. The contribution of GPI to the tensile strength is less than that of GPII.²³ According to the previous reports^{24–26} indicated, after the solid solution treatment, the Al–Zn–Mg

Fig. 10 The precipitates of FSP-ZM57 alloy with mode I by TEM: (a) bright-field image of GP zones in the matrix, [001]-projection, (b) and (c) selection area diffraction (SAD) schematic pattern in [001]_{Al} zone axis.

supersaturated solid solution decomposed in the following sequence:

Supersaturated solid solution

→ GP zones → η' → $\eta(\text{MgZn}_2)$

The metastable GP zones is coherent and the metastable η' phase is semicoherent. After mode II treatment, the major strengthening phase (GP zones) transferred to η' phase and

raised the strength of FSP-ZM35 and FSP-ZM57 specimens. The tensile properties of FSP-ZM35 and FSP-ZM57 alloy specimens were different through mode III treatment. In addition, the strength of FSP-ZM35 alloy through mode II and mode III was similar. It can be explained that the solid solubility of the second phase particles (such as G.P. Zone; η' and Al_3Zr) during FSP and that of T4 specimens were almost the same. So, the effect of precipitation strengthening of FSP specimens was not remarkable after T4 treatment. However, the strength or ductility of FSP-ZM57 alloy mode II and mode III were different. It can be explained that the second phase particles (G.P. Zone; η' and Al_3Zr) of matrix during FSP were not solid solution completely. The tensile properties of FSP-ZM57 after mode III were severely deteriorated, which was related to the solid solution effects. After post heat treatment, the tensile strength of FSP specimens with larger Zn/Mg ratio (ZM57 alloy) was higher; the ductility of FSP specimens with smaller Zn/Mg ratio (ZM35 alloy) was better. Judging from the above, decreasing Zn/Mg ratio improved the workability of friction stir Al–Zn–Mg specimens.

5. Conclusion

After FSP, the tensile strength of Al–Zn–Mg aluminum alloys was promoted by different artificial aging treatments. The mechanical properties of FSP-ZM57 alloy were better than that of ZM-35 alloy after mode II (80°C/16 h + T6) treatment because the grain refinement degree of FSP-ZM57 was better. After mode III (T4 + 80°C/16 h + T6) treatment, the tensile properties of FSP-ZM35 alloy were slightly promoted and that of FSP-ZM57 alloy were deteriorated because of the solid solution effects.

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