Hall–Petch Tensile Yield Stress and Grain Size Relation of Al–5Mg–0.5Mn Alloy in Friction-Stir-Processed and Post-Thermal-Exposed Conditions

Chun-Yi Lin*a, Truan-Sheng Lui*a, Li-Hui Chen and Fei-Yi Hung

Department of Materials Science and Engineering, National Cheng Kung University, Tainan 70101, Taiwan, ROC

Friction stir process (FSP) and post thermal exposure were carried out to a microstructure-thermostable cast Al–5Mg–0.5Mn alloy to examine the grain size dependence of tensile yield stress by using Hall–Petch relation, \( \sigma_y = \sigma_0 + k_yd^{-1/2} \). The measurement of preferred crystalline orientations and grain boundaries characteristic is used to explain the difference of the Hall–Petch parameters. FSP produces fine grain structure and, for the obtained average grain size from 3.7 to 12.8 \( \mu m \), near-random crystalline orientations and certain proportion of rigid coincidence site lattice boundaries (CSLs) is detected. Two-step post thermal exposure leads to a quick development of rotated (001) type crystalline orientations and the obtained coarse grains with average size from 229 to 508 \( \mu m \) are also commonly surrounded by general high angle boundaries. The calculation of Taylor factor and CSLs proportion reveals that stronger texture and grain boundary hardening effects exist in FSPed specimens than in thermal exposed specimens. These items are the major factors leading to larger angle boundaries. The calculation of Taylor factor and CSLs proportion reveals that stronger texture and grain boundary hardening effects exist in FSPed specimens than in thermal exposed specimens. [doi:10.2320/matertrans.M2013334]

1. Introduction

Friction stir welding (FSW) emerged as a solid-state joining process produces defect-free joints with better mechanical properties than fusion welding processes. Friction stir processing (FSP), which is essentially similar to FSW, can be applied to surface modification. Fine and equiaxed grain structure generated from combination of frictional heat and intense plastic deformation during FSW/FSP. Previous studies on 5083 Al alloy, one of the widely investigated non-heat-treatable alloys, has shown that the level of grain refinement can be controlled by process parameters and Hall–Petch equation can be used to describe the grain-size dependence of tensile strength or hardness. Hall–Petch equation correlates tensile yield stress (\( \sigma_y \)) and average grain size (\( d \)) as

\[
\sigma_y = \sigma_0 + k_yd^{-1/2}
\]

where \( \sigma_0 \) is friction stress and \( k_y \) is stress intensity coefficient associated with the stress required to extend dislocation activity into adjacent unyielded grains. Hall–Petch relation has been investigated in 5083 Al alloy in several previous studies. For the conditions of FSW/FSP, microhardness data showed that \( k_y \) slope varies from 14 to 53 Hv·\( \mu m^{-1/2} \) with a variation of average grain size from 0.2 to 25 \( \mu m \). These values can be converted to \( k_y \) slopes as 46 to 173 MPa·\( \mu m^{-1/2} \) by taking \( Hv = 3\sigma_y \). On the other hand, the super-high rolled and consequently annealed specimens with the grain size between 3.1 to 12 \( \mu m \) had a \( k_y \) slope of about 188 MPa·\( \mu m^{-1/2} \). Above-mentioned reviews show that different intense straining processes or annealing situations can lead to a wide range of \( k_y \) values in 5083 Al alloy. However, the influential factors on Hall–Petch parameters in 5083 Al alloy are still not clearly clarified yet. According to several associated reports, FSP produc-

*1Graduate Student, National Cheng Kung University
*2 Corresponding author, E-mail: z7408020@email.ncku.edu.tw

2. Experimental Procedure

2.1 FSP, thermal exposure and tensile test

Non-heat-treatable Al–5Mg–0.5Mn cast slabs were composed of 4.86% Mg, 0.58% Mn, 0.27% Fe, 0.07% Si, 0.06% Cr, 0.04% Cu (in mass%), and balancing Al. The cast slabs were machined to 4 mm in thickness and friction-stir-processed by using 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 fixed parameters were 0.55 mm/s in process speed, 1° in tool tilt angle and 70 MPa in loading pressure. Three tool rotational speeds, namely 450, 650 and 850 rpm (labeled as 450F, 650F and 850F) were chosen to produce three kinds of fine-grain specimens.

450F specimens were two-step thermally exposed to obtain different levels of coarse-grained specimens. Strain-energy-release treatment took place at 300°C for 0, 1, and 6 h in the first step and subsequent thermal exposure was carried out at 500°C for 1 h in the second step. Which were labeled as TTEx, where \( x = 0, 1 \) and 6 means the holding hour of strain-energy-release treatment. Following FSP and post thermal exposure, tensile test was applied at room temperature with an initial strain rate, \( 1.67 \times 10^{-3} \) s\(^{-1} \). Flat tensile specimens in I-shape were machined with gage longitudinal axis parallel to the processing direction. The gage length of tensile specimens was 10 mm and cross-section was 2 mm in thickness and 2.2 mm in width, which was covered within the marked region of stir zone (SZ) as shown in Fig. 1.
2.2 Metallographic observation

The microstructural observation focused on the marked region of SZ in the cross-sectional plane (perpendicular to the friction-stir processing path). Electron back-scattered diffraction (EBSD) analysis was used to identify the misorientation angle and grain size. The total scanning areas of as-FSP and as-TTE specimens are approximate $8 \times 10^4$ and $1.2 \times 10^5 \mu m^2$, respectively. The step size of scans was 0.4 $\mu m$, the binning of Kikuchi pattern image was 8 x 8, and the further information was processed by OIM Data analysis software. According to the same viewpoint with previous studies,14,15) only the conventional grains surrounded by high angle grain boundaries (HAGBs) were accounted for the grain size calculation. HAGBs were defined as misorientation angle $>15^\circ$ and grain size was calculated as the equivalent circle diameter of the grain area.

2.3 Texture analysis

Texture data on the marked region was collected by X-ray diffractometer and the orientation distribution functions (ODFs) were calculated from the incomplete pole figures [111], [200] and [220]. The ODFs were calculated by Bunge’s series method16) and represented in Euler’s space at constant $\phi_2$ sections. The texture component is labeled as $(hkl)_{uvw}$, where $(hkl)$ is the crystallographic plane perpendicular to processing direction, and $[uvw]$ is the crystallographic direction parallel to normal direction.

In this study, Taylor orientation factor ($M$) was used to understand the textural effect on the Hall–Petch parameters. Since the tensile direction was parallel to the processing direction, the stress pole of specific texture component (hkli)uvw was [hkli]. $M$ value was calculated with Chin’s method,17,18) which assumed tensile deformation as axisymmetric flow strain state and $M$ value of each stress pole could be approached based on [111](110) slip.

3. Experimental Results

3.1 Grain structure

EBSD-OIM maps in Figs. 2(a)–2(c) show the grain orientation and boundary characteristics in the marked regions of 450F, 650F and 850F. Equiaxed grains possess near-random crystalline orientations and mainly neighbor upon each other with high angle grain boundaries. The average grain size is acquired as 3.7 $\mu m$ at 450F, 7.8 $\mu m$ at 650F and 12.8 $\mu m$ at 850F. The grain-to-grain misorientation angle distribution histograms (0 to 65 deg) in Fig. 2(d) reveal that all FSPed specimens own excess 90% HAGBs. The statistic data of coincident site lattice boundaries (CSLs) identification in Fig. 2(e) show that there are 10 to 13% of CSLs within FSPed specimens and low $\Sigma$ boundaries (i.e., $\Sigma < 15$) occupy about 2/3 in CSLs population.

For TTEx specimens, OIM graphs in Figs. 3(a)–3(c) indicate they possess coarse irregular grains and these grains are typically surrounded by HAGBs. Average grain size is acquired as 508 $\mu m$ at TTE0, 340 $\mu m$ at TTE1 and 229 $\mu m$ at TTE6. Grain boundary distribution histograms in Fig. 3(d) indicate that only less than 3% of low angle grain boundaries (LAGBs) exist in TTEx, which suggests that almost no subgrain structure is identified within the coarse grains. CSLs statistic histograms in Fig. 3(e) shows that there are about 8% CSLs in TTEx specimens and $\Sigma$ distribution is more random than FSPed specimens.

3.2 Texture characteristics

The results of texture analysis are represented with ODFs in Figs. 4 and 5. The statistic data of texture components are synthesized in Tables 1 and 2, where “others” represents those two weak components to be exactly identified and they are reasonable to be considered as random texture. From Fig. 4 and Table 1, 450F, 650F and 850F specimens are characterized with weak texture signals. FSPed specimens possess weak components [110][001] (Goss orientation, labeled as G), (045)[054] and (302)[203]. These specific texture components contribute quite few and random components occupy more than 90% in volume fraction. According to Fig. 5, while 450F specimen is thermal exposed at 500°C, fine grains tend to grow into some particular crystalline orientations. Comparison among all texture components in Table 2 reveals that there are 50 to 75% rotated [001] type crystalline orientations in TTEx specimens. It suggests that grain coarsening under 500°C prefers to develop with [001] plane aligning to processing plane whether stain-energy-released treatment is carried out or not.

<table>
<thead>
<tr>
<th>Component</th>
<th>Vol%</th>
<th>$M$</th>
<th>Component</th>
<th>Vol%</th>
<th>$M$</th>
<th>Component</th>
<th>Vol%</th>
<th>$M$</th>
</tr>
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<tbody>
<tr>
<td>Goss</td>
<td>3.2%</td>
<td>3.67</td>
<td>Goss</td>
<td>2.1%</td>
<td>3.67</td>
<td>Goss</td>
<td>2.3%</td>
<td>3.67</td>
</tr>
<tr>
<td>(045)[054]</td>
<td>1.2%</td>
<td>3.58</td>
<td>(045)[054]</td>
<td>1.3%</td>
<td>3.58</td>
<td>(045)[054]</td>
<td>1.5%</td>
<td>3.58</td>
</tr>
<tr>
<td>(302)[203]</td>
<td>2.8%</td>
<td>3.39</td>
<td>(302)[203]</td>
<td>1.2%</td>
<td>3.39</td>
<td>(302)[203]</td>
<td>1.0%</td>
<td>3.39</td>
</tr>
<tr>
<td>Others</td>
<td>93.8%</td>
<td>3.06</td>
<td>Others</td>
<td>94.2%</td>
<td>3.06</td>
<td>Others</td>
<td>94.1%</td>
<td>3.06</td>
</tr>
</tbody>
</table>

$M = 3.10$ $M = 3.09$ $M = 3.09$
3.3 Tensile properties

Table 3 shows the tensile properties of as-FSP and as-TTEx specimens, in which deterioration of tensile strength and ductility can be recognized in TTEx by comparing to FSP, especially in yield strength. As shown in Fig. 6, the yield points are plotted as yield stress against the reciprocal of square root of average grain size, and the linear relation is so-called Hall–Petch relation in eq. (1). The intercept of the straight line on the stress axis is friction stress, $\sigma_0$, and the line gradient is stress intensity coefficient, $k_y$. The experimental data can be classified into two groups and each of them well fit on respective linear lines. The Hall–Petch parameters for group FSP in Fig. 6(a) are 128.7 MPa in $\sigma_0$ and 128.2 MPa·$\mu$m$^{-1/2}$ in $k_y$. On the other hand, $\sigma_0$ is 111.1 MPa and $k_y$ is 40.9 MPa·$\mu$m$^{-1/2}$ for group TTEx in Fig. 6(b). Both parameters in FSPed specimens are larger than those in TTEx. The influential factors are discussed in the following section.

4. Discussion

4.1 Parameter $\sigma_0$

Friction stress $\sigma_0$, a grain-size-independent parameter, is commonly interpreted as intragranular stress opposing the passage of dislocations along the slip plane. The friction stress is expressed as:

$$\sigma_0 = M \tau_0$$

where $M$ is Taylor orientation factor, $\tau_0$ is critical shear stress corresponding to the tensile stress $\sigma_0$ required to motivate
dislocation slip in an isolated grain. The metallurgical factors on the friction stress are understood in terms of texture hardening, solute locking and precipitate hardening.

The statistic data of texture components in Table 1 show that rotated (001) type components predominates in the specimens from group TTE but the specimens from group
FSP contain exceeding 90% “others” texture, i.e., random components. In Taylor’s calculation, M value of random aggregates was obtained as 3.06 by averaging over all axial orientations. On the other hand, while the tensile stress pole lies at h001 i, M value is 2.45. Average M values are calculated in consideration of volume fraction of each texture component. The value, C22 M FSP = C22 M TTE x, ranges from 3.09 to 3.10, and for TTE x ranges from 2.60 to 2.71. The value of C22 M FSP / C22 M TTE x is 1.16, which is quite similar to the ratio of friction stress, (σ0)FSP / (σ0)TTE x = 1.158. This suggests that the difference of σ0 is mainly contributed from texture hardening effect. The value of critical shear stress, τ0, is similar between both groups which can be referred to the insignificantly influential factors as precipitate hardening and solute locking. Precipitate hardening is generally accepted to be ignorable in non-heat treatable Al–Mg alloy. The working temperature of FSP and exposed temperature of TTE x are commonly higher than 300°C. While the heating temperature arising to 300°C, the solubility of major solute locking element Mg exceeds 5 mass%. Mg atoms are expected to completely dissolve into Al matrix in all experimental conditions. The locking force from Mg solute atoms is insignificantly different for all specimens.

4.2 Hall–Petch slope, k_y

k_y value in Hall–Petch relation expresses the intensity of grain-size effect. Since grain boundaries provide prior positions for stress concentration or relaxation during tensile deformation, k_y value is interpreted as the intensity of localized stress concentrations required for yielding propagation across grain boundaries. Wyrzykowski and Grabski’s research[23] on aluminum alloy indicated CSLs possess a more ordered structure and lower boundary energy than general high angle boundaries; for this reason, CSLs are capable of sustaining more stress concentration. CSLs, especially in low Σ boundaries, have higher boundary rigidity, provide more retarded force for yielding propagation across and cause an increase of k_y value. The entirely distinct distribution profiles of CSLs between two groups are responsible for the different k_y value. In this study, more existence of LAGBs in FSPed specimens (4–10%) contributes more additional retarded force for yielding deformation than TTE x specimens (<3%). That is considered as another factor which leads to higher k_y value of FSPed specimens.
5. Conclusion

(1) Control of rotational speed and holding time of strain-energy-release heat treatment can be used to produce different levels of fine and coarse-grain structure. By using Hall–Petch relation, experimental specimens can be divided into two groups, in which specimens from group FSP possess higher values of $\dot{\gamma}$ and $k_y$ than those from group TTE.

(2) As-FSP specimens are characterized by weak Goss, $(045)[054]$ and $(302)[203]$ texture components and TTE specimens possess much stronger texture components with $(001)$ plane aligning to processing plane. $\dot{M}$ values for group FSP and group TTE are 3.09–3.10 and 2.60–2.71, respectively. The relation $\sigma_0$\textsubscript{FSP}/$\sigma_0$\textsubscript{TTE} $\approx \dot{M}_{\text{FSP}}/\dot{M}_{\text{TTE}}$ suggests difference of $\sigma_0$ between two groups mainly depends on the average value of Taylor’s orientation factor.

(3) As-FSP specimens contain more fraction of low $\Sigma$ boundaries and larger total fraction of CSLs than TTE specimens. More ordered structure in CSLs owns better capability of stress concentration and it strengthens the grain boundary rigidity. Besides special boundaries, more LAGBs within grain structure in specimens from group FSP also contribute to higher $k_y$ value.

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