Microstructures and hardness of stir zone for friction stir processed and post-processed heat treatment 7B04-O aluminium alloy

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Abstract. Friction stir processing (FSP) with different rotation speeds was conducted on 7B04-O aluminium alloy thin-sheet, then the post-processed heat treatment (PPHT) was carried out. The microstructures and hardness of the stir zone (SZ) under as-processed (AP) and PPHT states were investigated. The result showed that the hardness of the SZ was enhanced under the AP state, attributing to the grain refinement. The effect of PPHT on the hardness of the material was different, compared with the AP state, the hardness of the SZ with a low rotation speed under PPHT state was deteriorate while it was enhanced when high rotation speed was adopted. When FSP was conducted on the O-temper base metal (BM), the initial precipitates in the matrix were coarse and stable, reducing the dissolution rate of the precipitates. High heat input was required in order to induce sufficient dissolution of the precipitates, which was beneficial to elevating the impact of PPHT on hardness. The amount and morphology of the precipitates played important roles in the SZ of improvement in hardness.

1. Introduction
Friction stir processing (FSP) was developed as a generic tool for microstructure modification based on the basic principles of friction stir welding. This new solid-state processing technique has been developed by Mishra and coworkers [1]. FSP causes intense plastic deformation and elevates temperatures in stir zone (SZ) [2]. SZ, consisting of recrystallized and fine-grained microstructure, is called dynamic recrystallized zone [3]. And FSP has been proved to be an efficient, cheap and reliable method to produce fine-grained materials [4].

In order to improve the mechanical properties of the SZ produced by FSP, many different methods including optimizing FSP parameters, applying post-processed heat treatment (PPHT), expediting cooling speed, and using submerged FSP, have been explored to minimize the dissolution or coarsening of the strengthening precipitates [5]. Due to the convenience and efficiency, more and more studies have been concentrated on PPHT.

High-strength, precipitation-hardening 7xxx-series aluminum alloy are used extensively in aircraft primary structures. And FSP has been proved to be a successful method in producing fine grains in
7xxx aluminum alloy [6]. Normally FSP and PPHT are often applied in aluminum alloys which are in the hardened condition. However, conducting the FSP on the base metal (BM) in this condition reduces tool life because of the high resistance to deformation, increasing the cost of production. By contrast, there are some advantages in carrying out the FSP with BM in the soft condition (O-temper) as the FSP tool forces are found to be low, which means the FSP system need not to be very expensive [7]. Thus it is necessary to research the effect of FSP and PPHT on the microstructures and mechanical properties in this alloy system in the soft condition. In the present work, FSP was conducted on 7B04-O aluminum alloy with different rotation speeds, and PPHT was applied in order to investigate the effect of PPHT on the microstructure and hardness of 7xxx-series aluminum alloy at O temper.

2. Experimental
Rolled 7B04 aluminum alloy sheets of 2 mm in thickness were used. Annealing heat treatment comprised of initial heating to 400 °C, soaking for 1 hr, followed by furnace cooling to 150 °C and air cooling down to ambient temperature for obtaining the O-temper. The sheets were then processed by FSP with different rotation speeds, which were 1600 rpm and 800 rpm. The traverse speed was 200 mm/min. A tool with a concave shoulder 10 mm in diameter and a M4 cylindrical pin was employed. The tool was made of H13 tool steel with a hardness of HRC 45. After FSP, the artificial aging was carried out at 165 °C for a soaking period of 6 hrs in an electric oven. Microstructural characterization was performed on the cross-section of plates using optical microscopy (OM), electron back-scattered diffraction (EBSD) and transmission electron microscopy (TEM). Differential scanning calorimeter (DSC) test samples were cut from the stir zone under AP state. Samples were heated at a constant heating rate of 10 K/min from room temperature to 550 °C. The Vickers micro-hardness was carried out along the centerlines of the cross-section with a distance between neighboring measured points of 0.5 mm under a load of 50 g for 10 s.

3. Result
3.1 Microstructures in the stir zone
Optical and transmission electron microscopy images of the BM are shown in figure 1. It can be seen that the elongated grains of the BM resulting from the rolling process has an aspect ratio of 3.0-4.0 (figure 1a). The result of TEM (figure 1b) indicates that the particles in the matrix are coarse (larger than 50 nm). In the SZ, the microstructure is characterized by the fine and equiaxed recrystallized grains caused by the severe plastic deformation and thermal exposure during FSP (figure 2). The result of EBSD shows that the average grain size of the SZ in the sample processed at 800 rpm and 200 mm/min (800/200 sample) is 1.15 μm, and 1.07 μm in the sample processed at 1600 rpm and 200 mm/min (1600/200 sample). There is not much difference in grain size when different rotation speeds were adopted in the present case.

Figure 1. Optical micrograph of BM (a) and TEM micrograph of precipitates in the BM (b).
3.2 DSC analysis of strengthening precipitates

The results of DSC analysis of the SZ with different rotation speeds under AP state are shown in figure 4. An exothermic peak was found below 300 °C during DSC heating for 1600/200 and 800/200 samples. The appearance of this exothermic peak indicates the formation of η(η′)-MgZn₂. It is well known that the area under the peaks is related to the volume fraction of the precipitates. From the
calculation about area $\eta$ ($\eta'$) peak, there is less $\eta$ ($\eta'$)-MgZn$_2$ formed in 800/200 sample compared with that in 1600/200 sample.

![Figure 4](image)

**Figure 4.** DSC analysis of the precipitates in the SZ and the BM under AP state.

3.3 Hardness

The hardness profiles on the cross-section under AP and PPHT states are shown in figure 5, exhibiting a reverse “U” shape. The hardness in the SZ of 1600/200 and 800/200 samples under AP state are higher than that of the BM. Compared with the un-processed BM, which has an average hardness of 65 HV, the average hardness in the SZ is 115 HV and 124 HV, respectively, corresponding to the 800/200 and 1600/200 samples. The increment in hardness of FSP samples under AP state is mainly caused by the difference in precipitation and grain refinement strengthening effects, which will be quantitatively calculated in this paper. Under PPHT state, the hardness in the SZ of both 1600/200 and 800/200 samples also increase compared with the hardness of the BM. Figure 6 shows the average hardness in SZ with different rotation speeds. It can be seen that the effects of PPHT on hardness are different between 1600/200 sample and 800/200 sample. Compared with AP state, the hardness of 1600/200 sample increases signally, while the hardness of 800/200 decreases under PPHT state.

![Figure 5](image)

**Figure 5.** Hardness distribution across the cross-section of plates under different states (a) 800/200 and (b) 1600/200 samples.

![Figure 6](image)

**Figure 6.** The average hardness in the SZ with different rotation speeds at different heat treatment conditions.

3.4 Effect of the BM condition on the dissolution of the precipitates during FSP

The PPHT did not enhance the hardness of 800/200 sample in the present study. Under PPHT states, the strengthening precipitates reform and its quantity is in proportion to the amount of the dissolved precipitates during FSP. Sufficient reformed precipitates contribute to the elevation in the hardness. Under the soft (O-temper) condition, the initial precipitates in the BM are the coarse stable phases which are difficult for dissolution because of their high dissolution points and larger sizes. And it suggests that the dissolution rate of the precipitates is reduced due to the existence of the coarse stable
precipitates. As mentioned above, for 800/200 sample, the low dissolution rate restrains the dissolution of the precipitates, leading to a negative effect of PPHT on the hardness. More heat input has been provided in 1600/200 sample than in 800/200 sample, which is beneficial to induce sufficient dissolution of the precipitates and elevates the hardness of the SZ during PPHT.

3.5 Strengthening effect of grain refinement and precipitation

FSP drastically changed the microstructure in the SZ. And the main differences in microstructure between the BM and the SZ in this study are the grain size, the amount and morphology of the precipitates. Consequently, the hardness is mainly affected by the grain refinement and precipitation strengthening. Based on the report of Feng [8], the hardness of a material is roughly proportional to the yield strength, therefore, the yield strength model is also suitable for predicting the variation in hardness.

Table 1. The volume fraction and the mean radius of the precipitates.

| Samples      | BM     | 800/200 | 1600/200 |
|--------------|--------|---------|----------|
| f            | 5.86   | 2.93    | 2.40     |
| r            | 75     | 60      | 55       |

Grain refinement strengthening $\sigma_{\text{GB}}$ is described well by the Hall-Petch relationship as

$$\sigma_{\text{GB}} = \sigma_0 + kd^{-1/2}$$

where $\sigma_{\text{GB}}$ is the increase in yield strength due to grain boundary strengthening, $\sigma_0$ is the yield strength for pure aluminum is about 16 MPa, $k$ is the Hall-Petch constant about 0.065 MPa m$^{-1/2}$, $d$ is the average grain size [9].

The precipitation strengthening $\sigma_p$ can be given as

$$\sigma_p = \frac{M}{b} \sqrt{\frac{3f}{2\pi}} \cdot (2\beta G b^3) \cdot \frac{1}{r}$$

where $r$ is the mean precipitates radius, $G$ is the shear modulus (27 GPa for aluminum), $b$ is Burgers vector (2.84 Å for aluminum), and $f$ is the volume fraction of the precipitates, $\beta$ is a constant (0.36 by Ref [10]). The initial precipitates in the BM are coarse, and in the SZ, there is insufficient time for dissolved precipitates to reform under AP state. The detailed information of the precipitates is listed in table 1. The summary of the grain refinement and the precipitation effects, and the hardness in the SZ and BM is shown in table 2. For the O-temper BM, the precipitation strengthening of the BM is small due to the existence of initial coarse precipitates, and the decrease of the precipitation strengthening caused by the FSP is limited. Since the hardness of the SZ is enhanced by the grain refinement. Besides, more precipitates dissolve in 1600/200 samples during FSP, leading to a higher solid solution strengthening, and increase the hardness further compared with that of 800/200 sample.

Table 2. Summary of the grain refinement and precipitation strengthening effects.

| Samples      | $\sigma_{\text{GB}}$ (MPa) | $\sigma_p$ (MPa) | Hardness (HV) |
|--------------|--------------------------|-----------------|--------------|
| BM           | 21                       | 37              | 65           |
| 800/200 AP   | 76                       | 32              | 115          |
| 1600/200 AP  | 79                       | 33              | 124          |

Under PPHT state, the precipitation strengthening rises and the solid solution strengthening drops as the $\eta$ ($\eta'$)-MgZn$_2$ reforms. Sufficient reformed fine $\eta$ ($\eta'$)-MgZn$_2$ are required in order to offset the loss of the solid solution strengthening. As to 1600/200 samples, there are enough fine $\eta$ ($\eta'$)-MgZn$_2$ reforming, and the precipitation strengthening elevates the hardness sharply. On the contrary, the reformed fine precipitates in 800/200 sample are insufficient on account of its small amount of the dissolved precipitates during FSP.
4. Conclusion

(1) FSP was demonstrated to be very effective in producing fine grains in the SZ, and a same level grain size in the SZ was obtained with rotation speeds of 800 rpm and 1600 rpm. The hardness of the SZ under the AP state was enhanced, which was higher than that of the BM. After PPHT, the hardness of SZ under high rotation speed condition had been enhanced, while the hardness was deteriorated when the low rotation speed was adopted.

(2) For FSPed 7B04-O Al, the strengthening effect of grain refinement overwhelmed the softening effect of precipitate dissolving, and the elevation in the hardness of the SZ under the AP state was mainly caused by the grain refinement. Under the PPHT states, the amount and morphology of the precipitates played important roles in the enhancement in the hardness, sufficient fine reformed precipitates contributed to elevating the hardness.

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