

## Achieving an ultra-high strength in a low alloyed Al alloy via a special structural design



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### ABSTRACT

An ultrafine-grained (UFGed) low alloyed aluminum alloy (6061 Al alloy) with a special nano-sized spherical precipitate structure was achieved by friction stir processing (FSP) and post-FSP aging. The evolution of the grains and precipitates during post-FSP aging and their contribution to mechanical properties were investigated. Under a low rotation rate (200 rpm) with water cooling, the average grain size of the FSP sample was only 200 nm. The ultimate tensile strength of the UFG 6061Al alloy aged at 100 °C for 20 min achieved 573 MP with a high elongation of 17%. This strength was nearly 90% higher than that of 6061 Al-T6 alloy (~300 MPa), and was equivalent to that of commercial 7075Al-T651 alloy (~572 MPa, the typical high alloyed Al alloy). Such a good mechanical property was attributed to the good thermal stability and the accelerated precipitation of the FSP UFGed structure under the low-temperature and short-time aging conditions. Further, the contribution of various strengthening mechanisms to the total strength in post-FSP aged Al alloys was quantitatively evaluated. This work provides an effective strategy for obtaining low alloyed high-strength Al alloys, which is beneficial for reducing environmental burdens and improving long-term sustainability.

### 1. Introduction

Global climatic warming resulting from greenhouse gas emissions, such as CO<sub>2</sub>, is an acute environmental issue [1]. Vehicular exhaust is one of the most important sources of greenhouse gases [2]. Recently, lightweight design has been proven to be an effective strategy of decreasing the volume of vehicular exhaust [2]. Thus, lightweight design is the main trend in the automobile industry for reducing greenhouse gases.

It is generally known that using light-weight and high-strength metals is a key technology in lightweight vehicle manufacturing [3,4]. Light metals, such as Al and Mg alloys, are used to achieve the light-weight goal, and alloying elements are usually added to achieve the high-strength [5]. However, alloying makes material development more resource intensive [6]. Meanwhile, alloyed materials with complicated compositions might be also more difficult to recycle. Thus, adding alloying elements might have a negative impact on the long-term sustainability of high-performance metals [6].

To make materials plain, which has been proposed in 2017 [6,7], means that novel properties and performance can be achieved in the “plain” materials with low alloying or even non-alloying via

introducing architectural defects across different length-scales. Various defects, such as vacancy, dislocation, grain boundary and phase boundary, widely exist in crystalline solids. These defects directly influence materials' physical, chemical, and mechanical properties [8]. Thus, to introduce architectural defects is an effective strategy of enhancing the mechanical properties of metals without changing chemical compositions.

6xxx series Al alloys, which are the so-called Al-Mg-Si alloys, are known as typical precipitation-hardened alloys [9]. Compared with other precipitation-hardened Al alloys, such as 2xxx series and 7xxx series Al alloys, alloying elemental contents are lower in 6xxx series Al alloys [10]. More importantly, the cost of adding Mg and Si are cheaper than adding other elements. Thus, 6xxx series Al alloy is a good choice for making materials plain, although the strength of this Al alloy is relatively low. For example, a commercial 6061Al alloy obtained by peak aging only exhibits an ultimate tensile strength (UTS) of ~300 MPa.

Metals and alloys can be strengthened by introducing more grain boundaries via grain refinement [11]. Grain refinement induced by plastic strain in metals and alloys is a well-known phenomenon. Thus, various severe plastic deformation (SPD) methods, such as

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**Table 1**  
Strain, strain rate, and minimum grain size of SPD 6xxx series Al alloys or pure Al.

Fabrication method	Strain	Strain rate ( $s^{-1}$ )	Minimum grain size (nm)	Ref.
ARB	4	< 10	300	[17]
ECAP	8	–	200	[12]
HPT	100	< 25	100	[13]
FSP	35	75	80	[15,18]
SMAT (pure Al)	20–45	$\sim 10^3$	30	[14,16]

accumulative roll bonding (ARB), equal-channel angular pressing (ECAP), and high-pressure torsion (HPT), friction stir processing (FSP), and surface mechanical attrition treatment (SMAT) [12–19], were developed as effective methods for designing grain size in metals and alloys.

Table 1 shows the strain, strain rate, and minimum grain size in 6xxx series Al alloys or pure Al processed by various SPD technologies [12–18]. It can be found that ultrafined or nanostructured grains can be prepared by these SPD methods [13–16,18]. However, among all these SPD methods, FSP is the only method to fabricate bulk metals with UFGs or nanostructured grains [19,20]. For example, Nakata et al. [19] obtained large processed area with a size of  $250 \times 56 \times 4$  mm by multi-pass FSP in an Al-Si-Cu alloy. Therefore, FSP exhibits an incomparable advantage over other SPD methods to produce large-area UFG or nanostructured metals.

As a kind of typical precipitation-hardened Al alloy, precipitation results in the strength increase in 6xxx series Al alloys [21]. However, because of the concomitant grain growth of the ultrafine-grained (UFGed) Al alloys during post-SPD aging [21–23], controlling the grain size in UFG Al alloys during aging is a big challenge. For 6xxx series Al alloys, HPT [13,21], ARB [17], and ECAP [24,25] processes with post-SPD aging only resulted in slight strength enhancement, or even caused a reduction in strength [26]. However, Xue et al. [27] found that unlike other SPD Cu with a big grain coarsening, FSP Cu exhibited a stable UFG structure without grain coarsening after high cycle fatigue tests, which indicated that UFG metals prepared by FSP might be a relatively stable structure. Thus, it is expected to control the grain size of FSP UFG Al alloys during post-FSP aging by selecting proper aging conditions.

In this study, in order to obtain low alloyed high-strength Al alloys, grains were designed at via FSP at the sub-micro scale, while the precipitates were designed via FSP and subsequent post-FSP aging at the nano scale. The microstructural evolution of FSP Al alloys during post-FSP aging was carefully examined. Finally, the contribution of various strengthening mechanisms to the total strength in post-FSP aged Al alloys was quantitatively evaluated.

## 2. Experimental procedure

Commercial 6061Al alloy plates, 2 mm in thickness and in a T6 aging state, were used in this work as the parent metal (PM). Here, the T6 aging state was chosen as the initial state just because this state of 6061 Al alloy is commonly used in the practical engineering applications. The nominal chemical compositions of PM are listed in Table 2 and the content of the total alloying elements is 2.43 wt%.

The original plates were subjected to FSP along the rolling direction and the tool with a shoulder 10 mm in diameter and a pin 3 mm in root diameter and 1.82 mm in length (taper thread pin) was used in this

**Table 2**  
Chemical compositions of 6061Al-T6 sheets (wt.%).

Mg	Si	Fe	Cu	Zn	Al	Total alloying elements
1.08	0.59	0.3	0.21	0.25	bal.	2.43

study. Additional rapid water cooling was used to obtain a low heat input during the FSP process. FSP was conducted at rotation rates of 400 and 200 rpm with a constant traverse speed of 100 mm/min, which were defined as 400-W and 200-W, respectively. Meanwhile, conventional FSP process was also completed in air at 400 rpm with the same traverse speed, and was defined as 400-A for the samples. In order to further improve the mechanical properties, the 200-W sample was artificial aged after FSP. The aging temperatures were from 80 °C to 175 °C.

The cross-sectional FSP samples were machined perpendicular to the processing direction. The microstructural characterization of FSP samples was investigated using optical microscopy (OM, HC-300Z/OL, Olympus Corporation) and the detailed microstructure of the FSP and post-FSP aged samples were carried out by transmission electron microscopy (TEM, FEI TECNAI G<sup>2</sup> 20) combined with energy dispersive spectroscopy (EDS, OXFORD) and scanning transmission electron microscopy (STEM). The grain size was measured by TEM with the linear intercept method. The volume fraction of precipitates was determined by the area of the whole area of the TEM image divided by the area of the precipitates, and the relative volume fraction of precipitates for the post-FSP aged sample was determined by the volume fraction of precipitates for the T6 aging state divided by that for the post-FSP aged samples. 6061 Al alloy in a solid solution state and 200-W sample were analyzed by means of differential scanning calorimetry (DSC). The DSC experiments were carried out under an argon atmosphere using a METTLER-1100LF system between room temperature and 500 °C with a heating rate of 10 °C/min.

Vickers microhardness measurements were performed on the processed zones (PZ) under a load of 50 g for 15 s. Tensile specimens with a gauge length of 2.5 mm were machined in the PZ parallel to the processing direction and the geometry of the tensile specimen is shown in Fig. 1. Tensile tests were carried out at room temperature at an initial strain rate of  $1 \times 10^{-3} s^{-1}$ .

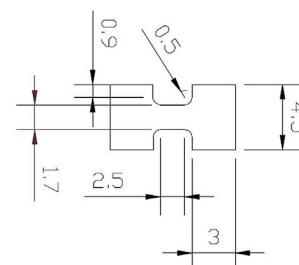
## 3. Results

### 3.1. Microstructural characterization and mechanical properties of FSP samples

Fig. 2 shows the cross-sectional macrostructures of FSP samples under various processing conditions. Under the investigated processing parameters, there were no defects, such as cavities and cracks in the FSP samples. Besides, the PZs produced by FSP exhibited basin shapes with wide top regions.

The TEM micrographs and statistical distributions of the grain sizes in the PZs of FSP samples are shown in Fig. 3. UFGs were obtained in all FSP samples (Fig. 3(a), (c), and (e)) and the average grain sizes of the 400-A, 400-W, and 200-W samples were 700, 400, and 200 nm (Fig. 3(b), (d), and (f)), respectively. It is obvious that finer grain sizes were attributed to the decrease of the heat input via the decrease of the rotation rate or additional water cooling methods.

The magnified TEM micrographs of the precipitates in the 400-W and 200-W samples are shown in Fig. 4. Many spherical precipitates



**Fig. 1.** Geometry of tensile specimens.

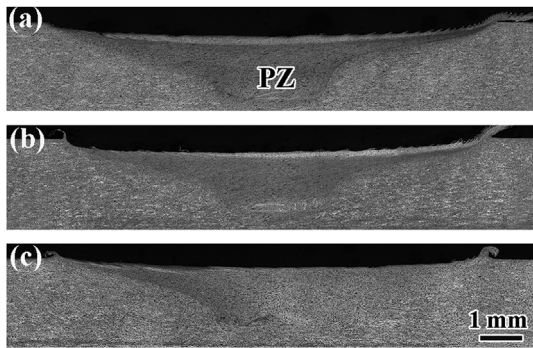


Fig. 2. Macrostructural characterization of FSP 6061 Al alloys: (a) 400-A, (b) 400-W, and (c) 200-W.

with the size of 10–70 nm were found in the 400-W sample (arrow in Fig. 4(a)). However, for the 200-W sample, only few small spherical precipitates were detected, and the average particle size was less than 10 nm (Fig. 4(b)). Thus, the 200-W sample can be approximately considered as an UFG Al alloy in a solid solution state.

The STEM micrographs and EDS mapping of the precipitates in the 400-A sample are shown in Fig. 5. Some large spherical precipitates rich in Fe, Cr, and Mn elements (Points 1 and 2) can be observed in this sample. These spherical precipitates were also obtained in FSP and HPT 6061 Al alloys by Feng et al. [28] and Mohamed et al. [13]. In addition, many light rod-like precipitates only rich in Mg and Si elements were also observed in the 400-A sample (Points 3 and 4). Sauvage et al. [21] reported that these light precipitates in SPD 6xxx series Al alloy were determined to be  $\beta$  phase, depending on the results of Mg/Si ratio and diffraction patterns.

Table 3 shows the microhardness values of the PM and various FSP samples. The PM showed a typical hardness value of 6061 Al alloy in a T6 state, which was 102 HV. The microhardness value of the 400-A sample was only 72 HV, which were smaller than the PM. In comparison, obvious hardening was observed in the 400-W and 200-W samples, and the microhardness values were 113 and 147 HV, respectively.

The engineering stress-strain curves of the PM and all FSP samples are shown in Fig. 6. The ultimate tensile strength (UTS) of 328 MPa were obtained in the PM. Under the condition of 400 rpm and air cooling (the 400-A sample), the UTS were only 232 MPa. Under water cooling, the UTS of the 400-W sample reached up to 376 MPa. The elongation to failure of the 400-W sample was as high as 18.5%, which

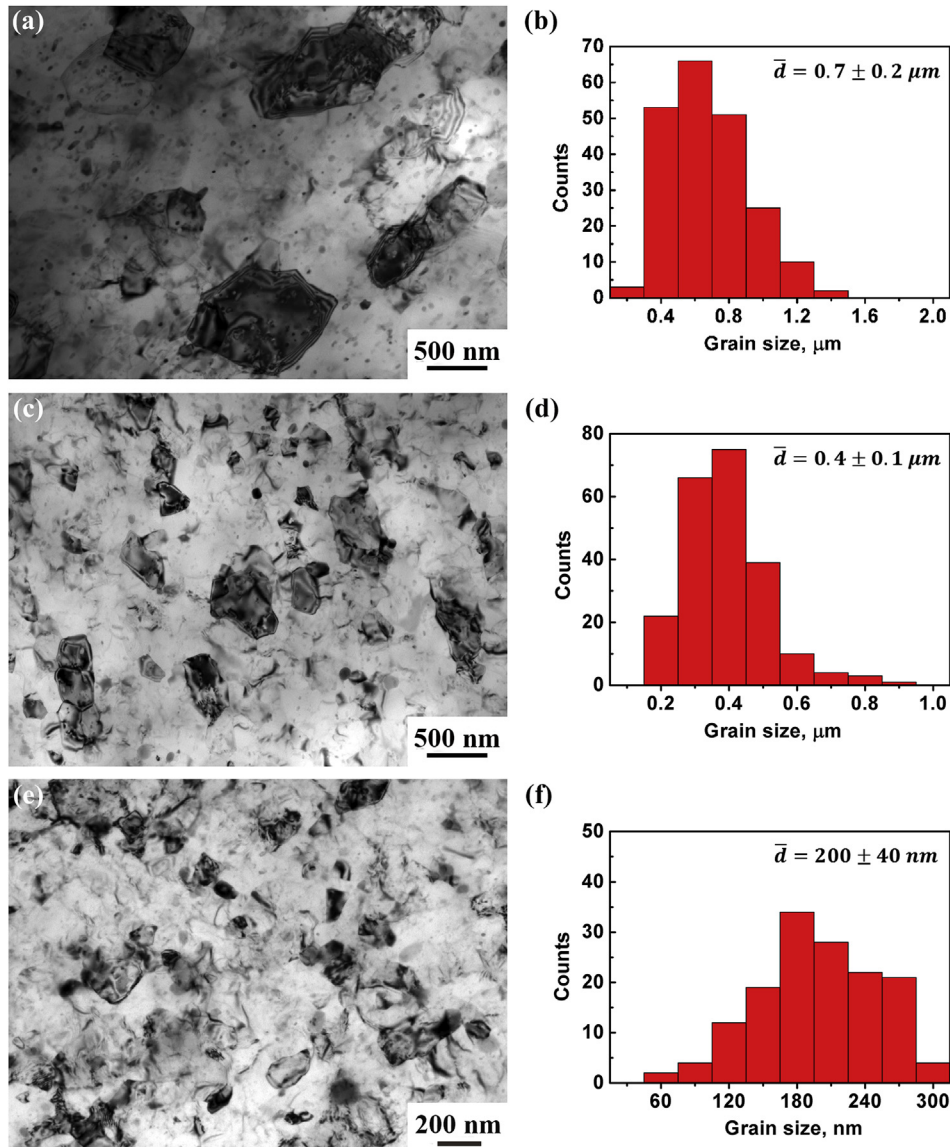


Fig. 3. Grain structures and grain size statistical distributions in PZs of FSP samples: (a) and (b) 400-A, (c) and (d) 400-W, and (e) and (f) 200-W.

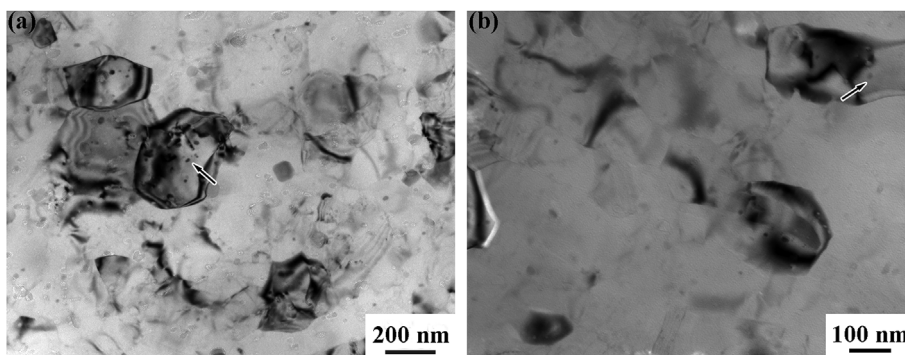


Fig. 4. TEM micrographs of precipitates in (a) 400-W and (b) 200-W samples.

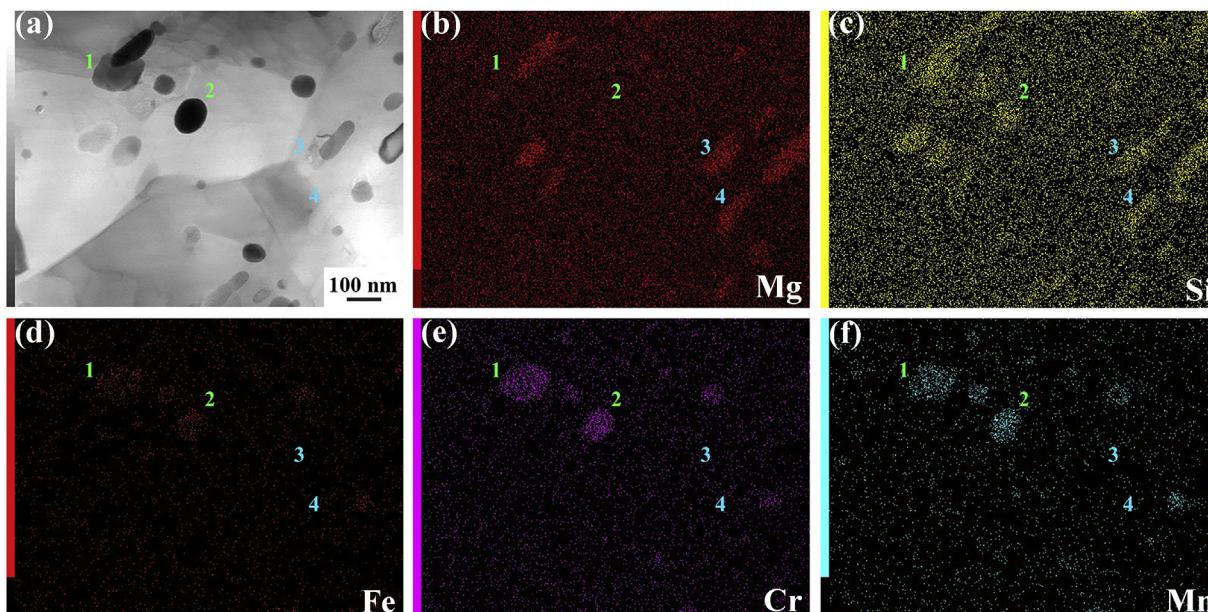


Fig. 5. (a) STEM image and corresponding EDS mappings with (b) Mg, (c) Si, (d) Fe, (e) Cr, and (f) Mn for 400-A sample.

Table 3  
The microhardness of PM and various FSP samples.

	PM	400-A	400-W	200-W
Microhardness (HV)	102 ± 3	72 ± 1	113 ± 2	147 ± 4

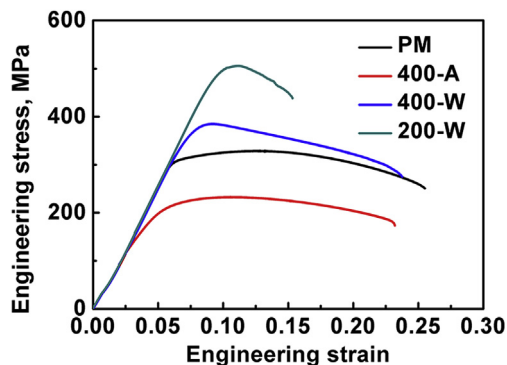


Fig. 6. The engineering stress-strain curves of PM and various FSP samples.

exhibited a similar value of the PM (21.0%). For the 200-W sample, the UTS of 506 MPa was achieved, which was significantly higher than that of the PM.

### 3.2. Microstructural evolution and mechanical properties during post-FSP aging

From the above results, it was observed that only few precipitates existed in the 200-W sample (Fig. 4(b)), so the strength of this sample can be improved by post-FSP aging. Fig. 7(a) shows the microhardness profiles of 200-W samples after post-FSP aging at different temperatures and time. At the aging temperatures of 80 °C–140 °C, the microhardness rapidly increased to a peak value. Thereafter, the microhardness value decreased with the extension of the aging time. Under the aging conditions of 100 °C and 20 min, the microhardness value was the highest, which reached to a high microhardness level of 161 HV. At a conventional aging temperature of 175 °C, the microhardness value decreased all the time during post-FSP aging.

The engineering stress-strain curves of 200-W samples after post-FSP aged at 100 °C for different durations are shown in Fig. 7(b). Clearly, the 200-W sample after aging 20 min exhibited the highest strength and elongation, with UTS reaching up to 573 MPa, and a surprising high elongation of 17% was obtained. This result was very exciting because this strength was nearly 90% higher than that of 6061 Al-T6 alloy (~300 MPa), and was equivalent to that of commercial 7075Al-T651 alloy (~572 MPa, the typical high alloyed Al alloy).

The grain evolution of the 200-W samples aging at 100 °C with different aging time is shown in Fig. 8. The grain sizes of post-FSP aged samples at different aging time were measured, as depicted in Fig. 8(a). With aging for 20 min, the average grain size just reached to 240 nm

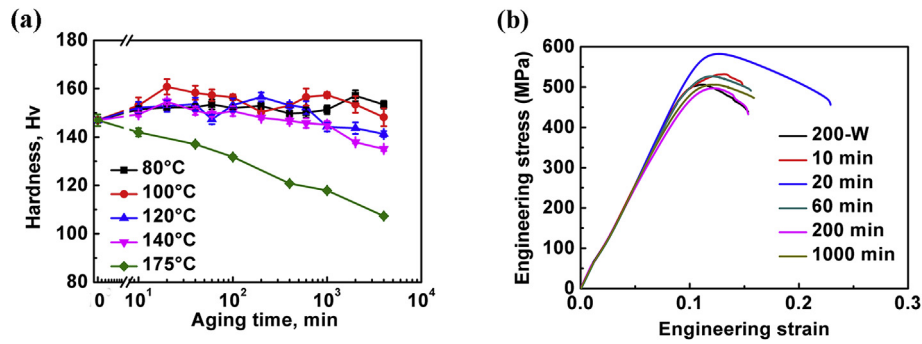


Fig. 7. (a) Variation of microhardness of 200-W samples with post-FSP aging time at different temperatures and (b) engineering stress-strain curves of 200-W samples by post-FSP aging at 100 °C.

(Fig. 8(b)), which was slightly larger than that of the 200-W sample (200 nm). It indicated that the FSP Al-Mg-Si UFG structure exhibited good thermal stability during short-time aging at 100 °C. When the aging time was longer than 200 min, grain growth was observed in the aged samples. It is noticed that although the grain size increased with the aging time increasing during post-FSP aging, the average grain size of all samples were less than 400 nm, which were also in the range of UFG scale.

Generally, the relative volume fraction of the precipitates can be defined as:

$$V_R = \frac{V_S}{V_{T6}} \quad (1)$$

where  $V_S$  is the volume fraction of the precipitates in the post-FSP aged sample and  $V_{T6}$  is the volume fraction of the precipitates in 6061 Al alloy at a peak aging (T6) state. The relative volume fraction of the precipitates in 200-W samples aging at 100 °C under different aging time is showed in Fig. 9(a). With aging for 20 min, the relative volume fractions of the precipitates increased rapidly and the sizes of most precipitates were less than 10 nm (Fig. 9(b)). With increasing the aging time, both the relative volume fractions and the size of the precipitates continued to increase while the increasing trend decreased (Fig. 9(c), (d), and (e)).

## 4. Discussion

### 4.1. Grains and precipitates in FSP and post-FSP aged samples

Various SPD technologies, with high strain and strain rate as well as low temperature have been shown to be beneficial and effective methods of obtaining fine grains [29–31]. Due to the ultra-high strain and strain rate [15,18], FSP is able to realize the grain refining at a low processing temperature. It is well known that decreasing heat input is a key method to reduce the temperature during FSP of Al alloys. Decreasing rotation rate, increasing traverse speed and using additional cooling condition are conventional methods for reducing the heat input during FSP [32–34]. In this study, the average grain size of the 200-W sample (200 nm) was the finest, which was mainly attributed to a very low heat input resulting from the extremely low rotation rate and the additional water cooling condition.

It is well known that the concomitant growth of the UFG occurs in the SPD samples during the aging process, which results in a large decrease of the strength [21]. Thus, selecting an appropriate temperature for controlling grain growth was a critical issue for the UFGs during the aging process. During a conventional peak aging process of 175 °C for 17 h, the microhardness value of post-FSP aged sample (typically with 113 Hv) was much lower than that of 200-W sample (147 Hv). Sauvage et al. [21] also reported that during aging at 170 °C, the

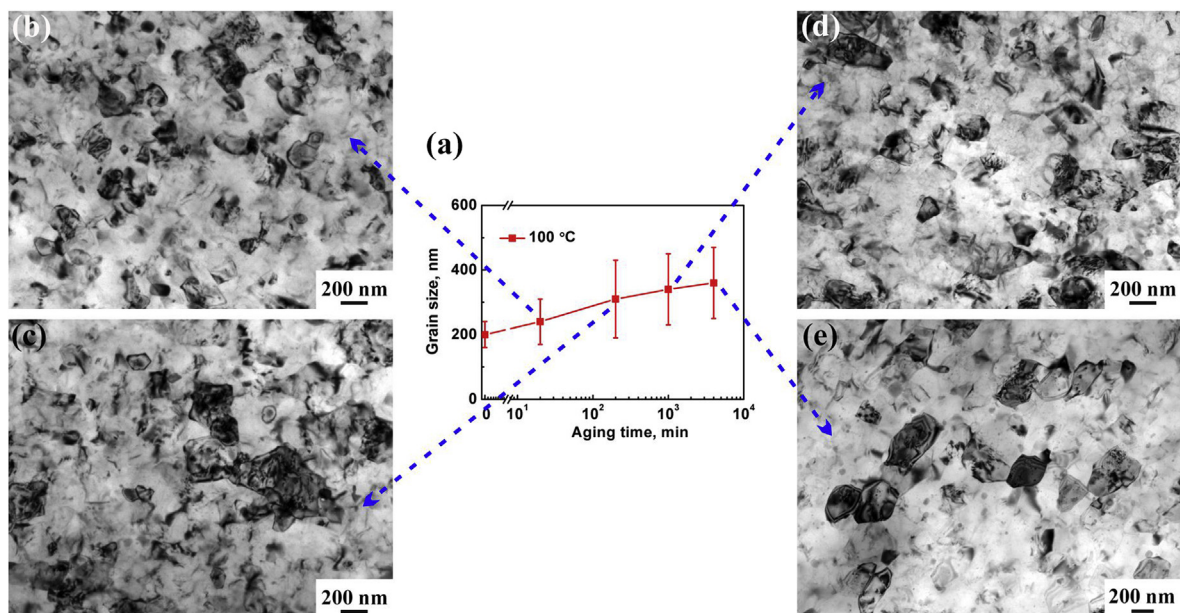


Fig. 8. (a) Variation of grain sizes of 100 °C aged 200-W samples with different aging time. TEM images showing grain structures for aging time of (b) 20, (c) 200, (d) 1000, and (e) 4000 min.

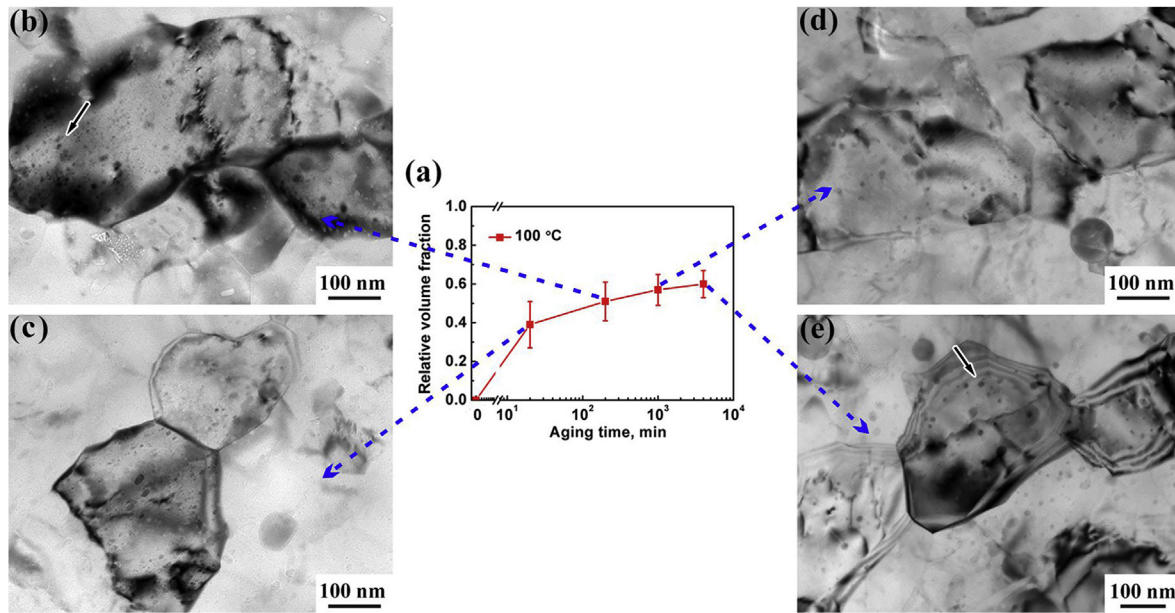


Fig. 9. (a) Relative volume fraction of precipitates in post-FSP aged 200-W samples after different aging times, TEM micrographs of precipitates in samples at (b) 20, (c) 200, (d) 1000, and (e) 4000 min.

grains of HPT UFG sample (the average grain size was about 200 nm) grew to 1000 nm. Meanwhile, for the HPT UFG structure, during aging at a relatively low-temperature (130 °C and 100 °C), the grain size also grew to 600 nm and there was a sharp drop for the hardness during aging [21]. However, in this study, during aging at 100 °C, the average grain size of all FSP samples were less than 400 nm, which indicated that UFG did not experience obvious coarsening during the low-temperature aging process. This might be because that the dynamic recrystallization (DRX) occurs in the FSP of Al alloys, which can release stored energy [18]. Thus, the FSP UFG structure exhibited good thermal stability during short-time aging at 100 °C and the microhardness of FSP sample rapidly increased to a peak (161 Hv) after a short-time.

The DSC curve could analyze the thermal information associated with precipitation of alloys. The DSC curves of the 6061 Al alloy in a solid solution state and the 200-W sample are given in Fig. 10. Usually, there are four exothermic peaks in the DSC curve of Al base alloy: peak A (~80 °C, GP zones), peak B (~260 °C,  $\beta''$  precipitates), peak C (~310 °C,  $\beta'$  precipitates), and peak D (~420 °C,  $\beta$  precipitates) [35,36]. However, there was no exothermic peak B in the DSC curve of 200-W sample, which indicated that  $\beta''$  precipitates were not precipitate during post-FSP aging. Meanwhile, Sauvage et al. [21] reported that according to the results of Mg/Si ratio and diffraction patterns, the precipitates in post-HPT aged 6xxx series Al alloy were only  $\beta$  and  $\beta'$ . Compared with 6061 Al alloy in a solid solution state, 200-W sample

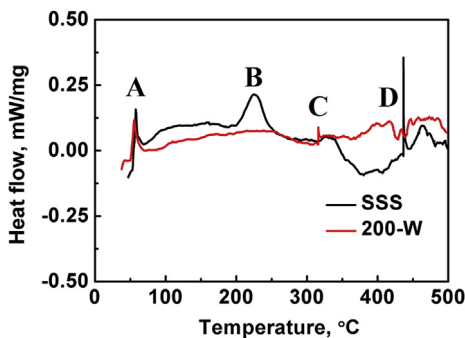


Fig. 10. The DSC curves of 6061 Al alloy in a solid solution state and the 200-W sample.

had lower GP zones and  $\beta'$  precipitates temperatures, implying an accelerated precipitation rate. The smaller peak area of exothermic peak C also reflected the accelerated precipitation kinetics of 200-W sample.

A mass of defects including vacancies and grain boundaries were generated by plastic deformation during SPD [37,38]. A high density of vacancies were quickly trapped by solute atoms to form clusters after SPD [35], which led to lower GP zones and precipitates temperatures and an accelerated precipitation rate. Moreover, grain boundary was a fast channel for element diffusion [33]. The high volume fraction of grain boundaries resulted in the increase of precipitation kinetics. Thus, under the low-temperature and short-time aging conditions, the precipitation of UFG 6061 Al alloy is rapid. Therefore, the high hardness and strength for the aged 200-W FSP sample at 100 °C for 20 min were attributed to the good stability and the accelerated precipitation kinetics of the UFGed structure.

#### 4.2. Relationship between microstructures and properties in post-FSP aged samples

It has been generally accepted that the hardness contribution in a precipitate strengthened alloy of each microstructural characteristics were calculated as follows [13,21]:

$$HV = HV^{Al} + \Delta HV^{GB} + \Delta HV^{precip} + \sum_i \Delta HV_i^{sol} + \Delta HV^{dislo} \quad (2)$$

where  $HV^{Al}$  is the pure Al hardness, which is about 18 HV [39],  $\Delta HV^{GB}$  is the hardening of grain boundary,  $\Delta HV^{precip}$  is the hardening of precipitate, and  $\Delta HV_i^{sol}$  is the solution hardening from the solute  $i$ ,  $\Delta HV^{dislo}$  is the strain hardening by dislocations.

Due to the low dislocation density of FSW/FSP grains and poor storage capacity of dislocations in UFGs [40,41], the hardening of dislocation effect can be redlined in this study.

Hall-Petch equation can be used to describe the relationship between the hardness (or yield stress) and grain boundary of metals, which can be extended to the SPD Al alloys [13,21]. According to the results of Saito et al. [42], the hardness of a FSP 1050 Al alloy can be written as

$$HV = 18 + 35d^{-\frac{1}{2}} \quad (3)$$

where  $d$  is the grain size. Thus, the grain size contribution is determined to be  $\Delta HV^{GB} = 35d^{-\frac{1}{2}}$ .

Precipitation strengthening was originated from the precipitate's ability to obstruct the dislocation movement. Assuming that the precipitates are homogeneously distributed, the corresponding yield stress contribution can be written as [43,44]:

$$\Delta\sigma^{precip} = 2M\beta Gb/L \quad (4)$$

where,  $M$  is the dimensionless mean orientation factor ( $M = 3.06$ ),  $\beta$  is a constant ( $\beta = 0.28$ ),  $G$  is the shear modulus ( $G = 26.9$  GPa),  $b$  is the burger vector corresponding to (110) dislocations ( $b = 0.29$  nm), and  $L$  is the average distance between precipitates. For SPD 6xxx series Al alloy, a ratio  $\sigma_y/HV$  is usually determined to 2 [21]. Thus, precipitation hardening is determined to be  $\Delta HV^{precip} = 6.68/L$ .

Myhr et al. reported that the influence of solute hardening on the yield stress can be written as [44]:

$$\Delta\sigma_i = k_i C_i^{2/3} \quad (5)$$

where  $\Delta\sigma_i$  is the absolute increment in yield stress by solute  $i$ ,  $C_i$  is the concentration of solute  $i$ , and  $k_i$  is the scaling factor for the solute  $i$ . The related values for Si and Mg solutes were proposed as follows:  $k_{Si} = 66.3$  MPa (wt%)<sup>-2/3</sup> and  $k_{Mg} = 29$  MPa (wt%)<sup>-2/3</sup> [44].

Usually, for 6xxx series Al alloy with an artificially peak-aged state (T6 state), all alloying elements (Mg and Si) are considered to be precipitated [9]. Thus, the volume fraction of alloying elements in the Al matrix of all post-FSP aged samples can be obtained, according to the precipitate relative volume fraction of post-FSP aged samples (Fig. 9(a)). Then, the solid solution contribution on the hardness can be estimated as:  $\Delta HV^{sol} = \Delta\sigma_i/2$  [21].

Based on the above analyses, microhardness profiles of each microstructural feature in the 100 °C aged 200-W samples are shown in Fig. 11(a). Due to the grain growth and the precipitation, the grain boundary hardening and solute hardening decreased, while the microhardness profile of the precipitate hardening increased first and then decreased. Because the precipitation trend of precipitates in UFG 6061 Al alloy was strong, precipitate hardening rapidly increased in a short aging time. Then, with increased aging time, precipitate hardening decreased due to the coarsening of the precipitates. The peak of alloying elements hardening (including precipitate and solute hardening) occurred before precipitates coarsening (1000 min), which was a high microhardness level of 83 HV. With aging for 20 min, the grain boundary hardening, solute hardening, and precipitate hardening of aged FSP sample were 72, 24, and 55 HV, which contributed 45%, 15% and 34% to the total hardness, respectively. Thus, the grain boundary hardening and precipitate hardening are the key reinforcing mechanisms of aged FSP Al alloys, which confirmed that the high strength of post-FSP aged 200-W sample was mainly attributed to the good thermal stability and accelerated precipitate kinetics. Meanwhile, alloying elements hardening of this aged FSP sample reached to 79HV, which was slightly lower than that of 6061Al-T6 (~84 HV).

Following this approach, the measured and estimated

microhardness profiles of 100 °C aged 200-W samples are plotted in Fig. 11(b). One can see that the general changing trend for the measured and estimated microhardness profiles show the similar variation trend and the relative difference between the measured and estimated microhardness values did not exceed 5%.

#### 4.3. Low alloyed high-strength Al alloy

The properties of materials are usually determined by their structure. Using the structural and mechanical information presented above, the grain size, microhardness, and microstructure characteristics of the 6061 Al alloy are summarized in Fig. 12. Four regions were differentiated according to structural and mechanical characteristics, as shown in Fig. 12(a). Under the traditional aging process in the commercial 6061 Al alloy plate, the microstructure was characterized by large grains (Fig. 12(d)) and needle-like precipitates (Fig. 12(e)) and the microhardness of this 6061Al-T6 was ~102 HV. When the aging time was too long, the microstructural characteristics were large grains and coarse precipitates and the microhardness was very low (overaging zone in Fig. 12(a)). For the SPD samples, a microstructural characteristic of UFGs exhibited (Fig. 12(c)) and the microhardness could reach up to ~140 HV. By post-SPD aging, the microstructures were characterized by UFGs and spherical precipitates (Fig. 12(b)), with a very high microhardness of 161 HV was achieved in this study. Thus, the combination of SPD and post-SPDed aging was an effective strategy for obtaining a high-strength Al alloy.

The use of materials with low alloying elements is an important and beneficial consideration for reducing environmental burdens and improving long-term sustainability. A comparison of the strengths of Al alloys from different processing technologies and alloying element contents in the present work and previous studies is shown in Fig. 13(a) [13,17,24,26,34,42,45–49]. Most Al alloys exhibit the characteristic of high alloying element content and high strength, with their data points located under a blue curve. Only a data point of Al alloys under the combination process of FSP and post-FSP aging in this study clearly separate from the curve, showing a better comprehensive characteristic including higher strengths and lower element content compared to commercial Al alloys via other processing methods. The UTS of ultra-fine-grained low alloyed aluminum alloy reached up to 573 MPa, which were equivalent to those of commercial high alloyed Al alloy (such as 7075Al-T651 alloy, ~572 MPa).

Besides, the UTS of 6061 Al alloys processed by different methods combined with aging in the present work and previous studies is shown in Fig. 13(b). It is obvious that compared to other processing methods and their aged samples, such as HPT/HPT + aging [13,17,24], higher UTS exhibited in the FSP and post-FSP aged samples, which was mainly attributed to the fact that a UFGed structure was obtained by FSP [34], and a good thermal stability and accelerated precipitation of the FSP UFGed structure exhibited when aging at a low-temperature (100 °C) for a short time (20 min). As a result, the UTS of FSP and post-FSP aged

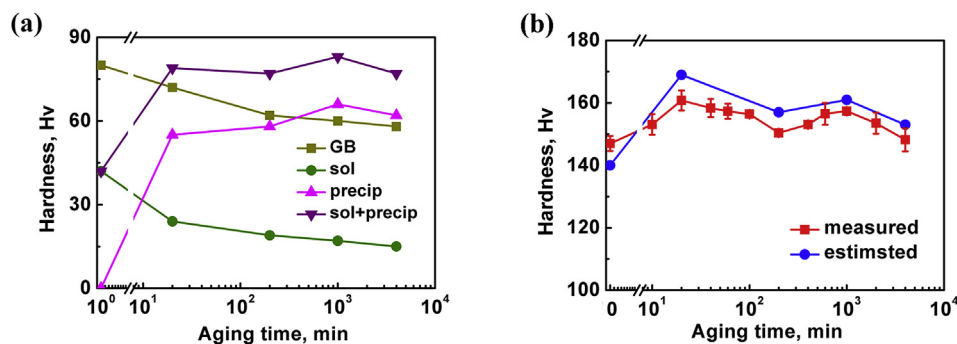


Fig. 11. (a) Microhardness contributions of each microstructural feature in 100 °C aged 200-W samples, (b) measured and estimated microhardness profiles of 100 °C aged 200-W sample.

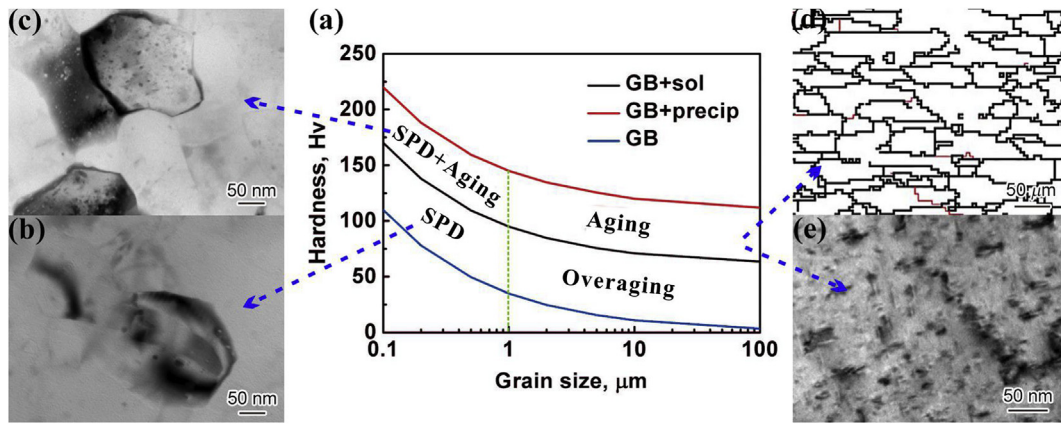


Fig. 12. (a) A map of grain sizes and hardness in 6061 Al alloy; microstructures of (b) post-FSP aging and (c) FSP 6061 Al alloy; and (d) grains and (e) precipitates in 6061 Al alloy in the T6 temper state.

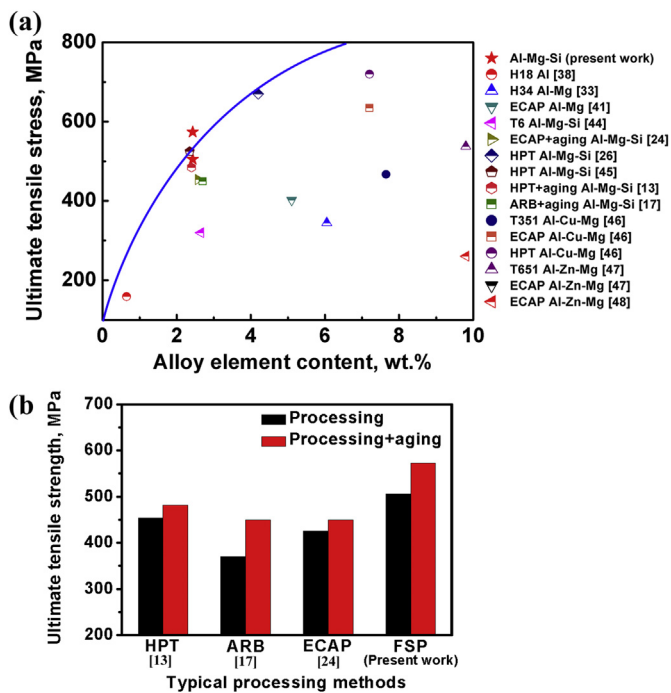


Fig. 13. (a) A comparison of the strength of Al alloys under different processing technologies and alloying element contents, (b) strength of 6061 Al alloy with different processing methods combined with aging.

sample was higher than other processed and post-processing aged 6061 Al alloys [13,17,24]. Therefore, this study provided an effective method for the preparation of low alloyed high-strength Al alloys.

## 5. Conclusions

A low alloyed high-strength Al alloy was achieved via friction stir processing (FSP) combined with post-FSP aging, and the following conclusions were drawn:

1. The ultrafine grains (200 nm) were obtained at 200 rpm with rapid water cooling. The hardness and UTS of FSP 6061 Al alloy increased with decreased rotation rate and increased cooling rate, which were as high as 147 HV and 506 MPa, respectively, for 200 rpm with rapid water cooling conditions.
2. After post-FSP aging at 100 °C for 20 min, the hardness and UTS of UFG 6061 Al alloy reached up to 161 HV and 573 MPa, respectively, which were nearly 90% higher than those of 6061 Al-T6 alloy, and

equivalent to those of the typical commercial high alloyed Al alloy (7075Al-T651 alloy).

3. The high strength for the post-FSP aged UFG 6061 Al alloy was attributed to its good thermal stability and the accelerated precipitation kinetics under the low-temperature and short-time aging conditions.
4. Quantitative evaluation of strengthening mechanisms showed that the grain boundary hardening and precipitate hardening are the key reinforcing mechanisms of aged FSP Al alloys, which contributed 45% and 34% to the total hardness, respectively. The relative difference between the measured and estimated microhardness values did not exceed 5%.

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## Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.msea.2019.03.126>.

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