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Investigating on microstructural and mechanical properties of Al6061/nano-SiC composites fabricated via friction stir processing

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Abstract

In this investigation, metal matrix composite was fabricated on the surface of Al6061 plates using means of 50 nm SiC particles via friction stir processing (FSP). X-ray diffraction (XRD), electron backscattering diffraction (EBSD) and transmission electron microscopy (TEM) were employed to detect and analyze the morphology and characteristics of precipitated phase and composite strengthening phase as well as the grain microstructure in samples. The microhardness and tensile properties of the samples were also tested. The results show that the ratio of low angle grain boundaries (LAGBs) decreased from 56% to 39% of the base metal owing to severe plastic deformation and the addition of nano-SiC particles, and that of the high angle grain boundaries (HAGBs) increased from 44% to 61%, which significantly refines the grain structure in the stir zone (SZ). The hardness profile of Al/SiC metal matrix composite sample is an inverted ‘U’ curve, which is different from the case that the hardness distribution of FSPed Al6061 alloy is a typical ‘W’ shape. Grain refinement and SiC dispersion strengthening compensate the defects of hardness reduction and uneven distribution caused by the dissolution in stir zone or coarsening in thermo-mechanically affected zone of Mg2Si precipitates due to the increase of temperature. Microhardness in stir zone increased from 78 HV to 100 ± 5 HV, which was improved up to about 28% comparing with base metal Al6061 alloy.

1. Introduction

Aluminium matrix composites are well known for wide utilization in aerospace, automobiles and marine areas owing to their good strength, low density and high specific modulus [1–3]. Dispersion of reinforcement particles on the metal surface and control are more difficult to attain by conventional surface modification techniques or the properties were degraded due to the reaction between the reinforcements and the matrix, which forms detrimental phase [4–6]. Recently, friction stir processing (FSP) have intensively been studied in material connection, microstructure improvement and preparation of metal matrix composites. Srivastava et al [7] concluded that, using the appropriate FSP parameters could obtain the uniform dispersion of reinforcement powder. Sun Kai et al [8] found that in FSP of metal matrix composites fabrication with SiC particles, the counter-clockwise rotation direction generates a more uniform SiC distribution compared with the clockwise rotation direction. Several researchers have discovered that increasing the number of passes leads to refinement of the grains and an improved homogeneity of the reinforced layer, thus in higher hardness [9, 10]. NahedAE-Mahallawy et al [11] fabricated Al/SiC composites using FSP and improved the mechanical and microstructure properties of the base metal.

The majority of published research is primarily directed towards the evaluation of effects of process parameters like speed of rotation, traveling speed, types of reinforcement particles etc on the surface and mechanical properties of fabricated surface composites. However, to the best of author’s knowledge, the effect of the addition of nanoparticles on the transformation for low angle to high angle grain boundaries, and the influence of the transformation on the grain structure are not investigated up. This study aims to fabricate...
aluminum matrix composite with nano SiC particles, and preliminarily investigates the effect of nano SiC particles on the evolution of grain structure with electron backscattering diffraction (EBSD) and transmission electron microscopy (TEM). Likewise it was investigated that the mechanism of hardness change in FSPed area.

2. Material and experimental procedures

2.1. Sample preparation
In this study rolled 6061 aluminum plates were used. Plates were cut into final dimensions of 250 × 75 × 4 (mm). Table 1 displays chemical composition of aluminum plates which were used. SiC particles (particle size about 50 nm) were used as reinforcements. Figure 1 shows transmission electron micrograph of the as-received SiC nano-particles. The friction stir welding tool was machined out of H13 hot working steel. The tool with a shoulder diameter of 18 mm, the screwed cylindrical pin diameter of 6 mm and height of 3 mm.

Three grooves which width and depth both are 1 mm were machined on the plate with a spacing of 1 mm. Then, slurry of SiC particles premixed with C$_2$H$_5$OH was filled into these grooves. After drying in vertical vacuum oven. The FSP was conducted on a numerically controlled friction stir welding setup (FSW–LM–AL–16–2D). The rotational and traveling speeds were 1400 r min$^{-1}$ and 50 mm min$^{-1}$, respectively. Tool onward tilt angle of 2.5° and counter-clockwise tool rotation directions were applied. Figure 2 schematically depicts the FSP process.

| Mg | Si | Cu | Zn | Ti | Mn | Cr | Al |
|----|----|----|----|----|----|----|----|
| 0.85 | 0.41 | 0.22 | 0.07 | 0.05 | 0.32 | 0.06 | Bal |

Table 1. Chemical compositions of the Al 6061 alloy (mass fraction)%.
2.2. Sample detection

2.2.1. Phase composition analysis of samples
To identify the phase constitutes in the Al/nano–SiC composites, x-ray diffraction analysis (XRD: Rigaku D/max 2500) was employed. The XRD specimens were scanned using Cu Ka radiation in the range of 30°–70° at a rate of 2° min⁻¹.

2.2.2. Observation of grain size and orientation of samples
Samples for macrostructural study were machined at the cross section in a direction perpendicular to the processing route. These samples were polished following standard metallographic procedures and then etched with Keller’s reagent (1 ml HF, 1.5 ml HCl, 2.5 ml HNO₃ and 95 ml H₂O) for 15 s. Macrographic images were taken by optical microscope (OM: LEIKA DMI3000 M). The samples at the cross section were polished by mechanical polishing and then electrolytic polishing was carried out in HClO₄:C₂H₅OH = 1:4 (vol) solution at a voltage of 15–20 V for 15 s at about −30 °C. The the evolution of grain morphology and grain boundary misorientation angle distribution were examined by electron backscattering diffraction (EBSD) technique.

2.2.3. Morphology and distribution of precipitated phase and composite strengthening phase in Al matrix
Samples processed by FSP were cut into 1 mm thin slices, polished by metallographic sandpaper and thinned by a Gantan-691 ion beam thinner. The distribution of nano-SiC with composite strengthening phase and the changes of precipitates of Al substrate in different regions affected by FSP were analyzed using a JEM-2000EX transmission electron microscope (TEM).

2.2.4. Mechanical properties of samples
Hardness testing was performed in different areas affected by FSP using a FM700 microhardness tester. A load of 100 g was applied for 15 s dwell time, and an interval between two points was 0.1 mm. In order to determine the mechanical properties of Al/ nano-SiC composites, tensile specimens were processed from Al/ nano-SiC composites and base metal, the dimensions of tensile specimens are shown in figure 3. Tensile tests were carried out using a WDW-200 universal testing machine with an initial strain rate of 1 mm min⁻¹ at room temperature.

3. Results and discussion

3.1. Macrostructure and XRD results of FSP samples
The samples FSPed were etched with Keller’s solution, and then observed under optical microscope, usually consisting of four different regions. They are: (a) base metal (BM), (b) heat affected zone (HAZ), (c) thermo-mechanically affected zone (TMAZ), and (d) stir zone(SZ) [12] as shown in figure 4. The spread of nano-SiC particles is more towards the advancing side (AS) in comparison to retreating side (RS). The reason behind this asymmetry may be understood as follows: as tool traverses, there are two sides of the processed zone, namely AS and RS having varied powder distribution and temperature [13]. Althought the relative particle velocity at AS is less as compared to RS, the temperature at the AS is higher as compared to RS [14]. The material on the AS is squeezed to the back of the tool along the counterclockwise direction owing to the action produced by the rotating tool. However, the plastic material is extruded to the back of the tool according to the clockwise direction on the RS. Therefore, the deformed material is scattered more towards the AS with the forward tool movement [15]. Figure 5 Shows the XRD results of the Al/nano-SiC composites FSPed and Al matrix samples.
The x-ray diffraction pattern of 6061 aluminum alloy is shown in figure 5(a). It was found that besides the diffraction peaks of Al, there are also the diffraction peaks of Mg$_2$Si aging precipitates. Diffraction peaks of Al and SiC are detected in the curves of FSPed Al/nano-SiC composites and no Mg$_2$Si aging precipitates are detected as shown in figure 5(b). This maybe attribute to the Mg$_2$Si dissolution affected by the temperature produced during the processing.

3.2. Grain microstructure distribution of samples in friction stir processing area

Figures 6(a) and (b) shows the grain morphology and size distribution affected by deformation and recrystallization during rolling of the Al6061 alloy. It can be seen that the base Al6061 alloy comprises of elongated grains parallel to the rolling direction. The average grain size of Al6061 alloy is 23.6 μm. After friction stir processing, the grain microstructure will change affected by drastic rise of temperature and severe plastic deformation in this region. Figure 6 reveals the microstructural characteristics of FSPed samples observed by EBSD in different regions. Compared with the base metal, material undergoes severe plastic deformation at elevated temperature which results in the formation of fine grains in the SZ. Comparing figures 6(a) with (c), we realize that FSP had decreased grain size from 23.6 μm to 4.7 μm, part of which are less than 1.8 μm, and transformed the rolled microstructure with coarse elongated grains to a structure consisting of fine equiaxed grains. This phenomenon is known as a distinct dynamic recrystallization (DRX) [16–18]. In this study, the grain size distributions of TMAZ on the AS were not uniform, as shown in figure 6(e). The grain size of individual grains in the bottom is similar to that of base metal due to insufficient deformation strain, while the grain in the middle-upper is the smallest. Figure 6(g) is an inverse pole diagram whose color designate the crystallographic orientation with respect to the sample normal. It clearly shows that there is no obvious texture and the grain orientation is random distribution in FSPed samples, which maybe lead to the 'isotropic' mechanical properties.

Figure 7 shows the image quality map superimposed with the grain boundary map and grain boundary misorientation map of FSPed samples in different areas obtained from EBSD. It was highlighted that two types of low angle grain boundaries (LAGB) (red 1°–5°, green 5°–15°) and a high angle grain boundary (HAGB) (beyond 15°) that is indicated in blue. Table 2 shows the summarised results of grain size, LAGBs, HAGBs for various regions of FSPed samples. There are many mechanisms for the evolution of grain structure, such as dynamic recovery (DRV), geometric dynamic recrystallization (GDRX), continuous dynamic recrystallization (CDRX) and discontinuous dynamic recrystallization (DDRX) [19]. The materials in stir zone (SZ) undergo severe plastic deformation at high temperature lower than its melting point during FSP. The flow stress increases due to
the high stacking fault energy (SFE) of Al, and then dislocation grows and annihilate, which leads to DRV occurs quickly [20–22]. Increasing dislocation density leads to an increase in recovery rate, and dislocations begin to rearrange to form subgrains boundaries (1°–5°), as shown the white arrow in figure 7(c). Upon dynamic equilibrium, subgrain boundaries begin to merge by absorbing increased dislocations continuously and transform into low angle grain boundaries (5°–15°) are shown the green arrow in figure 7(c). Therefore, when the subgrains reach the critical grain size, CDRX begins and the subgrains rotate slowly, resulting in increasing their misorientation and transforming them from LAGBs to HAGBS (>15°) are showed in blue arrow in

Figure 6. EBSD images of Grain morphology and grain size distribution chart in different areas of FSPed samples, (a) and (b) BM, (c) and (d) SZ, (e) and (f) TMAZ, (g) Inverse pole figure.
However, the presence of nano-SiC particles in SZ increases the influence factors on the grain evolution in FSP. On one hand, higher temperature produced by FSP promote grain boundary migration results in grain growth. On the other hand, the existence of nano-SiC particles restrict the movement of grain boundaries and impede grain growth by pinning effect. Furthermore, the sheared SiC particles could break the initial grains. According to Barmouz et al [23] it is found that the grains are broken and a large number of LAGBs are created during plastic deformation, which leads to increase the nucleating sites. Besides, presence of nano-SiC particles also increases nucleation sites for recrystallization [24]. Above factors lead to the formation of fine equiaxed grains in SZ, and the ratio of LAGBs decreases from 0.56 to 0.39 of the base metal.

Figure 7. The image quality map superimposed with the grain boundary map and grain boundary misorientation map of FSPed samples in different areas, (a) and (b) BM, (c) and (d) SZ, (e) and (f) TMAZ.
TMAZ is formed under the conjoint action of deformation induced by the tool pin and moderate heat generated by the tool. The grains are deformed severely but not dynamically recrystallized absolutely owing to insufficient deformation strain compared with the stirring zone (SZ), and some grains are elongated affected by shear force of the tool. Therefore, the size of individual grains in the bottom is similar to that of base metal, while the grain in the middle–upper is the smallest showed in figure 7(e). The ratio of LAGBs \((1^\circ–15^\circ)\) in TMAZ is higher than that in BM. The severe viscous material flow in SZ intensifies the plastic deformation at TMAZ adjacent to SZ during FSP. As a consequence the microstructure of TMAZ region may be dislocation cell structure and these cells metastably remain as the subgrains inside grains when there is no the succeeding recrystallization \([25]\). Therefore, the LAGBs \((1^\circ–15^\circ)\) increase significantly in TMAZ, as shown in figures 7(e) and (f).

### 3.3. Distribution of precipitated phase and composite strengthening phase nano-SiC in different areas of FSPed sample

The possible strengthening mechanisms which may operate in rolled 6061 aluminium alloy plate are deformation strengthening and precipitation strengthening. Figures 5(a) and 6(a) show the XRD patterns of precipitated phase \(\text{Mg}_2\text{Si}\) and the grain morphology in the base metal, respectively. However, the grain structure and the size of precipitated phase of the base metal will change in the region affected by high temperature and severe plastic deformation after FSP, which leads to that the machanical properties will change. Figure 8 shows the bright field TEM micrographs in different areas of FSPed samples. It was revealed that the morphology of grain and precipitated phase \(\text{Mg}_2\text{Si}\) in figure 8(a). Figure 8(b) shows the refined grains and dispersed nano-SiC particles in SZ, but no \(\text{Mg}_2\text{Si}\) was discovered. Figure 8(c) shows a few nano-SiC particles and a coarsened precipitates \(\text{Mg}_2\text{Si}\) in the TMAZ. In addition, the peak temperature in the SZ is often evaluated by the following formula \([26]\):

\[
T = K \left( \frac{\omega^2}{v \cdot 10^4} \right) \alpha \cdot T_m
\]  

where \(T_m\) is the melting point of material, \(\omega\) is the rotational speed, \(v\) is the traveling speed, and \(\alpha\) and \(K\) are constants varied between \(0.04 \sim 0.06\) and \(0.65 \sim 0.75\), respectively. Based on the formula (1), the peak temperature is about 480 °C in SZ, which is in good agreement with Sato et al.\([27]\) and Yuqing et al.\([19]\) findings in the case of temperature. The precipitates were fully dissolved in this temperature and then the cooling rate was accelerated, which inhibited the precipitation of the precipitates, forming metastable solid solution. Figure 8(b) shows that there are uniform distribution of SiC particles in grain and at the boudary but no \(\text{Mg}_2\text{Si}\) was obersved. Dislocations generated at the interface between SiC particles and Al matrix as a result of unequal thermal expansion coefficient of matrix and SiC particles. Dislocation wall and dislocation cell are formed owing to sliding and annihilating of dislocations during FSP, and then transformed into subgrains as recrystallized nucleus. Whereafter, subgrain boundaries rotate to form HAGBs by absorbing dislocations. Therefore, the Al matrix is significantly refined and most of the nano-SiC particles remain in the grain interior when dislocations near the interface form grain boundaries. Furthermore, the pinning effect associated with nano-SiC particles that exist near grain boundaries restricts the movement of them, which refined the grains, as shown in figure 8(b).

The effect of temperature in TMAZ is less than that in SZ, some grains are refined as well as there are two types phases. One of the phases is precipitated phase \(\text{Mg}_2\text{Si}\) whose size has been coarsened, which is affected by the temperature produced by FSP, and the other is nano-SiC whose size has not been affected by temperature, which may be squeezed into the TMAZ by the pin and shoulder of the tool, as shown in figure 8(c).

### Table 2. Summarised results of grain size, LAGBs, HAGBs for various regions of FSPed samples.

| Regions | Average grain size (μm) | Fraction of LAGBs in % \((1^\circ–5^\circ)\) | Fraction of LAGBs in % \((5^\circ–15^\circ)\) | Fraction of HAGBs in % \((15^\circ–60^\circ)\) |
|---------|----------------------|---------------------------------|---------------------------------|-----------------------------------|
| BM      | 23.6                 | 39                              | 17                              | 44                                |
| SZ      | 4.7                  | 31                              | 8                               | 61                                |
| TMAZ    | 22.2                 | 58                              | 5                               | 37                                |
3.4. Mechanical properties

3.4.1. Microhardness

Figures 9 and 10 [28–30] show the hardness profiles along the cross section for Al/nano-SiC metal matrix composite sample and rolled Al6061 alloy plate which are affected by FSP, respectively. It can be seen that the hardness profile of two samples is obviously different. Figure 9 shows an inverted ‘U’ curve, i.e. the hardness value of SZ is the highest (the average hardness value of base metal is 78 Hv, the hardness value of SZ is 100 ± 5 HV). However, it is clearly observed that hardness distribution profile along the cross section of the FSPed Al sample without SiC presents typical ‘W’ shape, as shown in figure 10. Not only the hardness value in SZ is lower...
than that of base metal, but also there are two regions with the minimum hardness value in the processed region. According to the Hall–Petch relationship [31]: $HV = 40 \pm 72d^{-1/2}$, the finer the grain, the greater the hardness value. For the Al/nano−SiC composites sample fabricated by FSP, the hardness value of SZ and TMAZ are improved under the conjoint action of the pinning effect of SiC hard particles on grain boundaries or dislocations and fine grain strengthening. As for the reduction in hardness for the sample of figure 10, which can attribute to the dissolution or coarsening of precipitate Mg$_2$Si during FSP weakened the dispersion strengthening. Since the second phase particles had nearly disappeared in the SZ, the decrease in hardness of the SZ relative to the BM, as shown in figure 10. When the grain is not refined sufficiently, and the precipitate Mg$_2$Si is partially coarsened in TMAZ, there are two regions of the minimum hardness in figure 10 (circle).

### 3.4.2. Tensile properties

Figure 11 depicts the engineering stress-strain curves for Al6061 alloy and Al/nano−SiC metal matrix composite. Table 3 shows the summarised results of tensile strength, yield strength, and elongation of Al6061 alloy and Al/SiC metal matrix composite. It is evident that Al/nano−SiC metal matrix composite exhibited higher tensile strength and yield strength, which increase by 19.1% and 41.5% respectively, while elongation decreases by about 21.6% as compared to base Al6061 alloy. In this study, Al6061 matrix strengthening was obtained by grain refinement and the mechanical properties as well as dispersion distribution of the hard phase nano-SiC particles.

Table 3. Summarised results of tensile strength, yield strength, and elongation of Al6061 alloy and Al/SiC metal matrix composite.

| Material         | Tensile strength (MPa) | Yield strength (MPa) | Elongation (%) |
|------------------|------------------------|---------------------|---------------|
| Al6061 alloy     | 350                    | 159                 | 38.8          |
| Al/SiC composite | 417                    | 225                 | 30.4          |
4. Conclusions

In this study, nano-SiC reinforced Al6061 composites are successfully fabricated using FSP. Based on the analysis of XRD, EBSD, TEM and mechanical properties of the prepared samples, the following conclusions were drawn:

1. The results of EBSD analysis for the affected area of the fabricated Al/nano-SiC composites by FSP show that due to severe plastic deformation and the addition of nano-SiC particles, the ratio of LAGBs decreased from 56% to 39% of the base metal, and that of HAGBs increased from 44% to 61%, which significantly refines the grain structure in SZ. The ratio of LAGBs increased significantly and the grain structure is refined partially in TMAZ. There is no obvious texture in FSPed sample, and the grain orientation is nearly random distribution.

2. Since the action of the pinning effect of SiC hard particles on grain boundaries or dislocations, the hardness profile of Al/nano-SiC metal matrix composite sample is an inverted ‘U’ curve. The hardness distribution of FSPed Al6061 alloy is a typical ‘W’ shape, which is related to the grain is not refined sufficiently and the precipitate MgSi is dissolved or coarsened partially. Microhardness in SZ increased from 78 Hv to 100 ± 5 HV, which was improved up to about 28% comparing with base metal Al6061 alloy.

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