Microstructure evolution and mechanical properties of friction stir processed TiC/7085Al nanocomposites

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Abstract

In this paper, homogeneously dispersed TiC nanoparticles and fine-grained composite was successfully achieved by friction stir processing (FSP) of in situ 0.5 wt. % TiC/7085Al nanocomposites with different rotational speed. The effects of the rotational speed on the microstructures and tensile properties were investigated. Experimental results showed that the stir zone (SZ) exhibited equiaxed recrystallized grains with fine size and a high fraction of high-angle grain boundaries (HAGBs) caused by dynamic recrystallization (DRX). Moreover, the tensile strength and elongation of the friction stir processed (FSPed) composite were significantly improved compared with the base composite. With the rotational speed of 1000 rpm, the composite has the smallest grain size and the optimum mechanical properties. The average grain size decreased to 1.61 μm, the yield strength (YS), ultimate tensile strength (UTS) and elongation reached to 345 MPa, 429 MPa and 17.8%, respectively.

1. Introduction

Al and its alloys are used in many industry fields, such as aerospace, transportation and structural applications due to their high strength, good ductility and good corrosion resistance [1–3]. In the past three decades, many researches devote to further improve the comprehension properties of the Al alloys. It was found that the Al-based metal matrix nanocomposites (MMNCs) possess high tensile strength, enhanced corrosion resistance, fatigue, creep and wear properties [4–6]. There are many processing methods to manufacture the Al-based MMNCs [7–9]. Among these methods, the Al-based MMNCs fabricated by in situ method exhibit more advantages in microstructure, such as clean particle-matrix interface, strong particle-matrix bonding, fine particles and economic viability [5, 6, 10–12]. However, the reinforced nanoparticles tend to be clustered and agglomerated in the matrix due to their high specific surface energy. These cluster and agglomeration of the nanocomposites in the matrix would decrease the mechanical performance of the nanocomposite [12–15].

Friction stir processing (FSP), a novel solid-state processing technique, develop from the basic principles of friction stir welding (FSW). Recent years, the FSP technology has been successfully used to fabricate the MMNCs [16–18]. Many researches have reported the microstructure evolution and mechanical properties of the Al-based MMNCs fabricated by FSP. Amra produced the CeO2–SiC/Al5083 nanocomposites by incorporating the nanoparticles into the Al5083 alloy matrix using FSP. They found that the grain refinement and uniform distribution of reinforcement particles were achieved inside the nugget zone. Meanwhile, the hardness and wear resistance of the FSPed composite were higher than those of the base metal [18]. Yang fabricated the Al3Zr/6063Al composites by direct melt reaction method and subjected to forging and FSP. After FSP, the grain size of the 5 wt.% Al3Zr/6063Al composite decrease from 378 nm to 153 nm compared with the forged composite. The FSPed composite exhibited superplasticity at the temperature of 400 °C–550 °C with initial strain rate from 1 × 10−3 s−1 to 1 × 10−2 s−1 [19]. Khodabakhshi fabricated 3.5 vol.% SiC/Al–Mg nanocomposite by using multi-step FSP. After five passes FSP, the grain size of the stir zone decreased to 1.4 μm.

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and the SiC nanoparticles homogenously distributed in the Al matrix. Compared with the annealed Al–Mg alloy, the hardness, yield strength and ultimate tensile strength of the FSPed composite improved from 60 HV, 68 MPa and 180 MPa to 123 HV, 120 MPa and 290 MPa, respectively [20]. Bahrami investigated the effect of SiC nanoparticles on the microstructures and mechanical properties of the FSPed SiC/Al composites. They found that the SiC nanoparticles refined the microstructure. At the FSP parameter of 1250 rpm and 40 mm min\(^{-1}\), the addition of SiC nanoparticles lead to 31% and 76.1% improvement of the ultimate tensile strength and elongation, respectively [21]. Therefore, FSP is an effective method to refine the grain size and disperse the nanoparticles in the matrix. Thus, the excellent microstructure could achieve the improvement of the mechanical properties of the nanocomposites.

For Al-based MMNCs, TiC is an ideal reinforced particle because it possesses high melting point, high hardness and chemical stability, especially for its low lattice mismatch with Al matrix [22, 23]. Moreover, we have successfully fabricated the TiC/Al nanocomposites from Al, Ti and carbon nanotubes (CNTs) system [24]. It was observed that some TiC nanoparticles agglomerated in the Al matrix, thus the mechanical properties were decreased. Therefore, it is necessary to disperse the TiC nanoparticles in the Al matrix by FSP method. However, the research focused on the TiC/Al nanocomposites fabricated by FSP is rare. In this study, the FSP was applied to in situ TiC/7085Al nanocomposites to promote the TiC nanoparticles distribution in the 7085Al matrix. The purpose of this research is to reveal the microstructure evolution and mechanical properties of the FSPed nanocomposites fabricated by different PSP parameters. Through microstructure observation and tensile properties test, the strengthening mechanism and microstructure-property relationship were also discussed.

2. Materials and methods

In this paper, the 0.5 wt.% TiC/7085Al nanocomposite was fabricated through in situ casting method. The related details can refer to [25]. The ingots were cut into the plates with size of 140 mm × 80 mm × 8 mm by wire cut electrical discharge machining (WEDM) and then subjected to FSP. A H13 steel stirring tool having a flat shoulder of Φ18 mm and a righthand threaded pin of Φ6 mm root and 5 mm length was tilted by 2.5° with a 0.2 mm tool shoulder plunge depth. The FSP was conducted with rotational speed of 800, 1000, 1200 rpm, respectively, at a constant traveling speed of 60 mm min\(^{-1}\). Then the samples for tensile tests and microstructure observations were cut along the direction of FSP by WEDM. Schematic representation of samples preparation for various microstructure observation and mechanical property test is displayed in figure 1.

Microstructural observation was characterized by optical microscopy (OM), field emission scanning electron microscopy (FE-SEM) equipped with an electron backscatter diffraction (EBSD) detector and analysis system, transmission electron microscopy (TEM). EBSD specimens are electropolished in a solution of 5 ml HClO\(_4\) and 95 ml pure C\(_2\)H\(_6\)O at 20 V for 15 s at room temperature. TEM simples were polished to a thickness of approximately 50 um and then thinned by twinjet electropolishing.

As shown in figure 1, the tensile test samples were machined from the middle of SZ along the FSP direction with a gauge length of 8 mm, a width of 3 mm and a thickness of 2 mm. Tensile tests were conducted on a screw-drive universal machine at a tensile rate of 1 × 10\(^{-3}\) s\(^{-1}\). Meanwhile, the tensile tests of composite without FSP were also carried out for comparison. At least three tests were repeated for each composite using different
processing parameter and average values are reported. After tensile tests, fracture surfaces were studied using FE-SEM observation.

3. Results and discussion

3.1. Materials flow pattern and grain
Figure 2 shows the optical micrographs from the thickness cross-section and the stir zone of the FSPed nanocomposites. It can be seen that the onion ring pattern structure emerged in the FSPed composites. This onion ring distribution profile can be divided into the stir zone (SZ), thermo-mechanical affect zone (TMAZ), base material (BM), advancing side (AS) and retreating side (RS) which have been marked out. Figure 2(a)–(f) shows the microstructures of the SZ processed by different FSP parameters. As shown in figure 2(a)–(f), the grains size in the SZ is greatly reduced compared with that in BM, the coarse dendrites were broken up into fine equiaxed grains. The sizes of the equiaxed grains under 800 rpm, 1000 rpm and 1200 rpm are 1.9 μm, 1.6 μm and 2.2 μm, respectively. This indicated that with the increase of the rational speed, the grain size of the composites in SZ decrease first and then increase.

The EBSD gain structural maps and the misorientation angle distributions from the SZ of the composites are shown in figure 3. In figure 3(a)–(c), different grain misorientation with low angle grain boundary (LAGBs) (3° ≤ θ ≤ 15°) and high angle grain boundary (HAGBs) (θ > 15°) are showed in different colours. LAGBs represented as black line and HAGBs represented as grey line. From the figure 3(a)–(c), the grain size shows a tendency of firstly decrease and then increase with the increase of rotational speed. Statistically, the grain sizes of the SZ at the rotational speed of 800 rpm, 1000 rpm and 1200 rpm are 1.9 μm, 1.6 μm and 2.2 μm, respectively. From the statistical results of orientation distribution, the fraction of HAGBs under the rotational speed of 800 rpm is 63%. When the rotational speed increases to 1000 rpm, the proportion of HAGBs increases to 69.8%. Whereas, when the rotational speed increases further to 1200 rpm, the proportion of HAGBs decreases to 64.1%.

Figure 4 shows the recrystallized microstructure of the composite with different speed after FSP. Recrystallized grains, sub-grains, and deformed grains are shown in blue, yellow and red, respectively. The recrystallized microstructures show that dynamic recrystallization has basically taken place in the composite after FSP. At 800 rpm, there are many sub-crystalline regions and the fraction of recrystallization region reaches 79%. It is observed that the recrystallization region increases significantly to 92% when the rotational speed increases to 1000 rpm. The fraction of recrystallization region decreases to 82% when the rotational speed increases to 1200 rpm.

3.2. Texture
Figure 5 shows the pole figures of the composites with different rotational speed after FSP. The simple shear textures {001} 〈110〉 and {111} 〈211〉 corresponding to C and A* components were obtained, respectively. It can be seen that with the increase of the rotational speed, the texture components remain constant. The persistence of shear texture is mainly due to the high stacked fault energy of Al alloy.
3.3. Distribution of TiC nanoparticles

TEM micrograph of the FSPed composite under the rotational speed of 1000 rpm is illustrated in figure 6. It was found that the TiC nanoparticle homogeneously distributed in the Al matrix. Meanwhile, there still exist some subgrains with partial HAGBs which indicate that dynamic recrystallization (DRX) has not completely finished or was continuous in progress. It is believed that TiC nanoparticles can pin (sub-) grain boundaries which could stabilize the substructure and may affect DRX.

3.4. Tensile properties

Figure 7 shows the typical properties of the FSPed TiC/7085Al nanocomposites, and the detailed data of the yield strength (YS), ultimate tensile strength (UTS) and elongation are listed in table 1. It is observed that the YS, UTS and elongation are all improved after FSP. The YS, UTS and elongation of the composite are 332 MPa, 360 MPa, and 3.5%, respectively, for the sample without FSP. At the rotational speed of 800 rpm during FSP, the YS, UTS and elongation are 323 MPa, 375 MPa and 10.5% respectively. When the rotational speed increases to 1000 rpm, the YS, UTS and elongation are 312 MPa, 429 MPa and 17.8% respectively. With the increase of rotational speed to 1200 rpm, the YS, UTS and elongation are 307 MPa, 407 MPa and 15.3%, respectively.

Figure 8 shows the SEM fracture morphologies of the composites from the fracture surfaces after tensile tests. According to the morphologies, the fracture of the composite samples exhibits the combined ductile-
cleavage fracture. The dimples on the fracture surface indicate that the composites undergo the ductile fracture. For the sample without FSP, the size of dimples on the fracture surface of nanocomposites is large (see figure 8(a)). After FSP, the fracture surfaces are composed of fine and uniform dimples (see figures 8(b)–(d)). This characterization is in agreement with the improved mechanical properties.

3.5. Grain refinement mechanism
There are many literatures reported about the dynamic mechanisms responsible for microstructural refinements during FSP of Al alloys [26–30]. The main mechanisms involved during FSP consist of dynamic
recovery (DRV), dynamic recrystallization (DRX). During these mechanisms, DRV mechanism is considered as the dominant dynamic restoration mechanism during hot-working processes for Al and its alloys [26]. Due to the high stacking fault energy (SFE) of Al, DRV occurs by multiplication and interaction of dislocation at the

Figure 7. The tensile properties of the 7085 Al-base alloy and FSPed TiC/7085Al nanocomposites.

Table 1. The tensile strength and elongation of TiC/7085Al nanocomposite after FSP.

| Speed / rpm | YS / MPa | UTS / MPa | Elongation / % |
|-------------|-----------|-----------|----------------|
| 0           | 300       | 360       | 3.5            |
| 800         | 323       | 375       | 10.5           |
| 1000        | 345       | 429       | 17.8           |
| 1200        | 330       | 407       | 15.3           |

Figure 8. The fracture microstructures of the (a) 7085 Al-base alloy and FSPed TiC/7085Al nanocomposites; (b) 800 rpm; (c) 1000 rpm; (d) 1200 rpm.
initial stages of deformation, in which they rearrange and form LAGBs. However, with the plastic stain proceeded during FSP, the DRV mechanism would convert into the DRX mechanism. Thus, the LAGBs annihilated and transformed into HAGBs \cite{27}. And less LAGBs and more recrystallization grains were found with the increase of the rotational speed, as shown in figure 3 and 4. Regarding the DRX mechanisms, the dynamic nucleation initially occurs at the sub-grains formed by DRV, and this is followed by migration of HAGBs. During the subsequent strain through the FSP process, high densities of dislocations form in the presence of the TiC nanoparticles, which increase the nucleation rate during DRX by accommodating the incompatible strain generated during FSP.

As for the composites, there are two refinement mechanisms to be considered during FSP, i.e., particle stimulated nucleation of recrystallization (PSN) and Zener pinning. According to the previous research, because the size of the TiC particle is far smaller than 1 μm, the PSN effect can be excluded in this study \cite{31}. Therefore, Zener pinning mechanism is attributed to the grain refinement and formation of a finer grain structure in the SZ of FSPed nanocomposite \cite{32}. However, when the rotational speed increased to 1000 rpm, more heat input was introduced to the SZ, resulting in the nucleation grains grown further and the LAGBs combined. That is why the SZ of the composite with the rotational speed of 1000 rpm has finer grain and less LAGBs.

3.6. Strengthening mechanism

In the case of the FSPed composites with homogeneously distributed TiC nanoparticles, the major contributions to the improvement of the tensile strength include grain refinement strengthening, Orowan strengthening and coefficient of thermal expansion (CTE) strengthening.

Firstly, according to the well-known Hall-Petch relationship, the YS is improved with the decrease of the grain size. In the FSPed composite, the far smaller grain size in SZ leads to the improvement of the YS.

Secondly, the TiC nanoparticles uniformly dispersed in the Al matrix after the FSP. According to the Orowan strengthening, the TiC nanoparticles would pin up the dislocation to form the Orowan ring when the dislocations passed by. Thus, the strength increased with the interaction between the TiC nanoparticles and the dislocations.

Thirdly, owing to the CTE between TiC nanoparticle and Al matrix during applied deformation, the dislocations generated around the TiC nanoparticles. The increased dislocation density increased the strength.

On the other hand, when the large and clustered nanoparticles and pre-existing porosity exit in the composites, the cracks are easy to nucleate, grow and coalesce resulting in the low ductility. In this study, the base composite exits some agglomerations of TiC nanoparticles and pre-existing porosity which decrease the ductility. During FSP process, the TiC nanoparticles dispersed uniformly, and the pre-existing porosity closed. As seen from figure 8 and table 1, the ductility of the composites increased significantly after FSP.

4. Conclusions

The 7085Al nanocomposite with fine grain size and uniformly distributed TiC nanoparticles was successfully fabricated by FSP of in situ 0.5 wt.% TiC/7085Al nanocomposite. The microstructures were observed by EBSD and TEM analysis techniques. The results show that many fine recrystallized grains exited in the FSPed composite due to the pinning of the homogeneously dispersed TiC nanocomposites. Meanwhile, the YS, UTS and elongation of the composites are all improved after FSP. With the rotational speed of 1000 rpm, the grain size and recrystallization degree were 1.61 μm and 92%, respectively. Compared with the base composite, the YS, UTS and elongation were improved 15%, 19% and 409%, respectively. The improved tensile strength is attributed to the grain refinement, Orowan strengthening and CTE strengthening mechanisms. The superior elongation may also relate to the uniformly dispersed TiC nanoparticles and closed pre-existing porosity during FSP.

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