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Micro-nano structure and characterization in the nugget zone of Al-Cu-Mg alloy under friction stir processing

Siyu Sun[1], Peng Liu[1]*, Lanwen Chen[1], Meiqing Cao[2], Jiaying Hu[1], Siyu Liu[1], Xue Qiao[1], Junhao Sun[1]

[1]*School of Materials Science and Engineering, Shandong Jianzhu University, Jinan 250101, P. R. China*
[2]*School of Materials Science and Engineering, Shandong University of Science and Technology, Qingdao 266590, P. R. China*

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Abstract

In this paper, the microstructure in the nugget zone of a friction-stir-processed Al-Cu--Mg alloy was analysed. There are the irregular bulk precipitates and micro-voids in the Al-Cu-Mg alloys. These precipitates were the compounds of Al-Cu system, and the size is less than 1 μm. In a local area, the homogeneous amorphous structure of Al-Cu system in some area of the nugget zone was also detected. The amorphous structure with a halo pattern has a structure characteristic of the long-range disorder. The formation of micro-nano and amorphous structure in the nugget zone could be attributed to the severe plastic deformation, breaking effect of stir tool, higher temperature gradient, hydrostatic pressure and shear stress and strain.

Key words: friction stir processing, aluminium alloys, microstructure, micro-nano structure, electron microscopy

1. Introduction

Friction stir welding (FSW) is an important method of joining materials, patented by the Welding Institute (TWI) in 1991 [1, 2]. However, friction stir processing (FSP), which was derived from FSW, was applied to fabricate composites, process metallic materials and refine the structure of materials [3, 4]. However, most of the researches mainly focused on composites fabricated by adding some reinforcement phase including SiC, TiC, nanoparticles, etc. [5–7]. In these researches, nanostructure produced in-situ aluminium alloy by FSW, or processing has been reported rarely. Although Cai et al. [8] reported that the various strengthening precipitates (θ' and S$'$ phases) were dissolved in the nugget zone where dense dislocations and nano-sized co-clusters were found in the joints of Al-Cu-Li alloys by FSW. Kumar et al. [9] also reported that the typical nano-sized grains in a magnesium alloy were achieved for the study of magnesium alloys using an FSP technique. However, these reports on nanostructures formed in situ aluminium alloys are still little until now. Therefore, the possible micro--nano structure and its characterization are important to know the formation mechanism of nanoscale structures.

In this paper, some fine precipitates and possible nanoscale structure were observed and analysed. The microstructure in stir zone of a friction-stir-processed Al-Cu-Mg alloy was analysed by scanning electron microscope (SEM), scanning transmission electron microscopy (STEM), X-ray diffraction technology (XRD), transition electron microscopy (TEM) and high-resolution transition electron microscopy (HRTEM).

2. Experimental

The friction-stir-processed sheets of 2024-T4 aluminium alloys had dimensions of 150 mm × 60 mm × 3 mm. The nominal chemical composition of aluminium alloys in weight percentage is 3.8–4.9 Cu, 1.2–1.8 Mg, 0.5 Si, 0.5 Fe, 0.3–0.9 Mn, 0.1 Cr, 0.25 Zn, 0.15 Ti and balance Al. The processing direction was parallel to the rolling direction of the sheets. The

*Corresponding author: e-mail address: liupeng1286@163.com

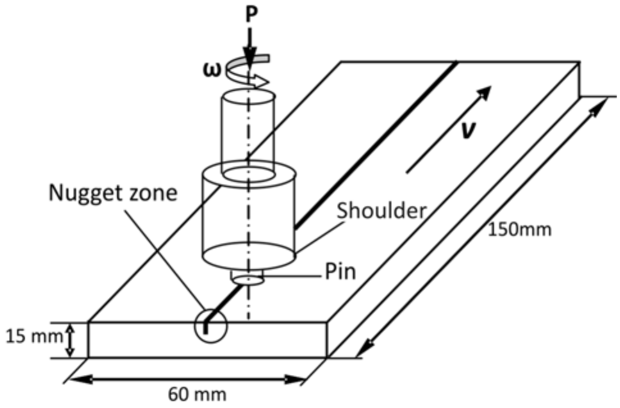

Fig. 1. The schematic of friction stir processing [10].

schematic of friction stir processing is shown in Fig. 1. A cylindrical tool made of tool steel was applied. The diameter of the tool shoulder is 20 mm, and that for the pin is 6 mm. The tool rotational speed and travel speed were 475 rpm and 150–375 mm min^{-1}, respectively.

A series of specimens were cut from the stir zone after FSP, and then these specimens were made into metallographic samples. The NZ is an important region that was directly influenced by the tool pin. Some samples of TEM analysis were cut from the nugget zone. Then, thin foils were also cut from this region along the cross-section and were polished to 50 μm thickness. The thin foils were mechanically cut to make 3 mm diameter discs. At last, the discs were prepared into the thin foils for STEM investigations. Ion beam thinner with a cooling stage was used during the sample preparation to avoid the influence of oxygen. The microstructure in nugget zone of a friction-stir-processed Al-Cu-Mg alloy was observed and analysed by NEOPHOT32 metallographic microscope and SEM (FEI Quanta 200 FEG). The fine microstructure investigation was carried out on STEM instrument of S5500 type. Also, the discs were prepared into the thin foils for TEM investigations using double jet electro-polishing, using a solution containing HNO_3 and CH_3OH (1 : 3) at 18 V and –20 ℃. Microstructure investigation was carried out on a JEOL 2011 FX instrument with a W-filament operated at 200 kV. For XRD analysis, Rigaku-2500 equipment using copper target (Kα) radiation was used. The test conditions are 40 kV voltage and 200 mA current.

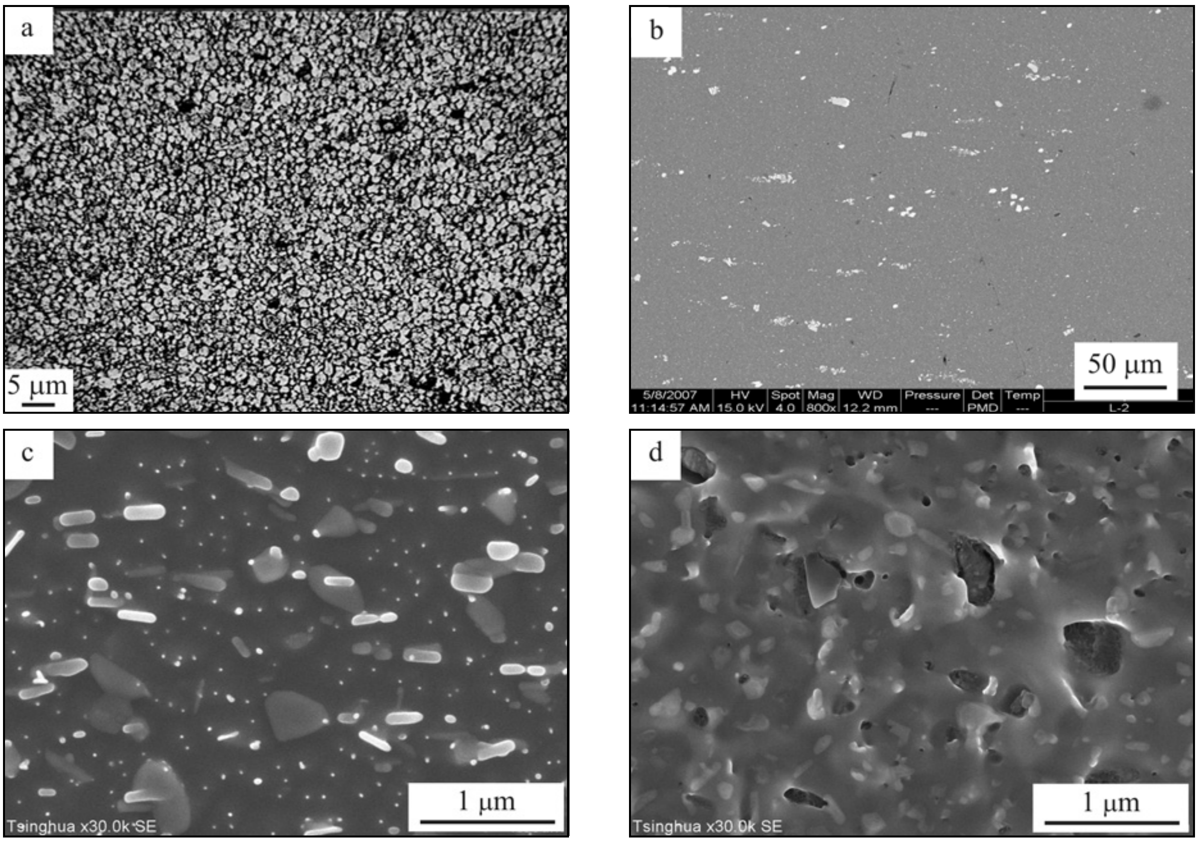

Fig. 2. The microstructure of base metal and nugget zone of a friction-stir-processed Al-Cu-Mg alloy: (a) optical image in nugget zone, (b) SEM image in nugget zone, (c) STEM image in base metal, and (d) STEM image in nugget zone.

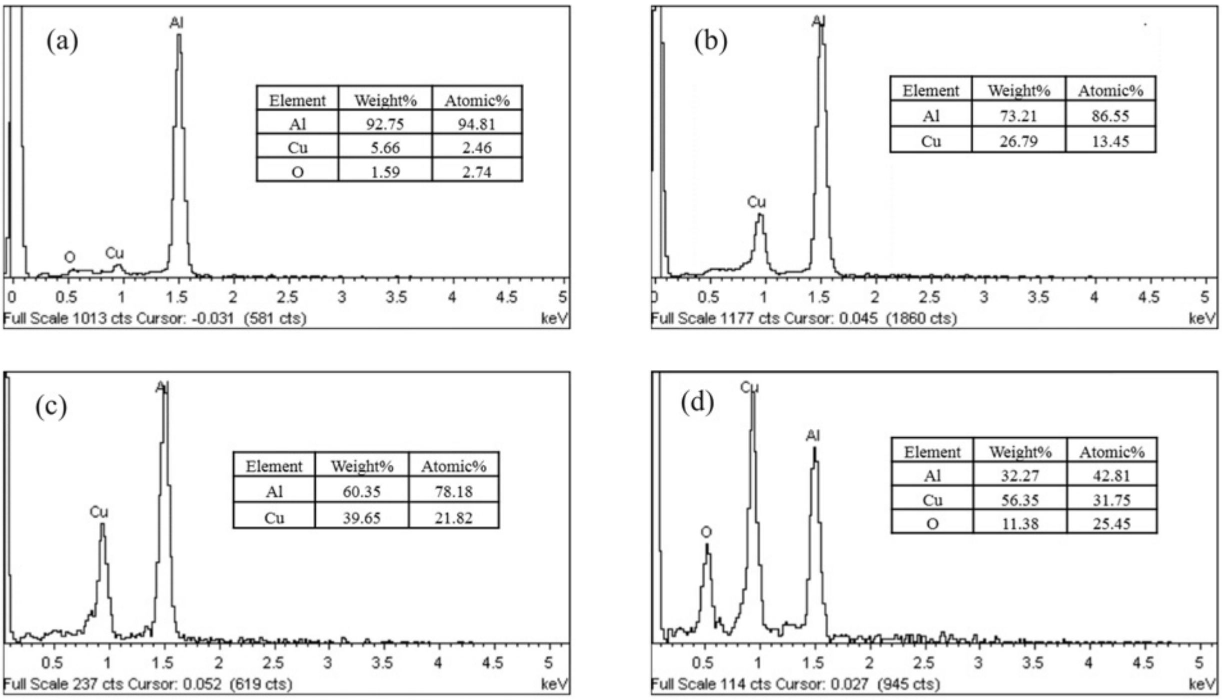

Fig. 3. The EDS results of base metal and nugget zone of a friction-stir-processed Al-Cu-Mg alloy: (a) EDS result for S1 in base metal, (b) EDS result for S2 in base metal, (c) EDS result for S4 in nugget zone, and (d) EDS result for S5 in nugget zone.

3. Results and analysis

3.1. Microstructure characterization

The NZ experienced the dynamic recovery and dynamic recrystallization by a tool pin [11], and a fine equiaxed grain structure shown in this zone (see Fig. 2a). Moreover, according to the SEM result, there are some visible precipitates in the nugget zone (see Fig. 2b). This is favourable to enhance the mechanical properties of the friction stir welded joints. A novel STEM technology was used to study the microstructural change of NZ metal experienced an elevated temperature, large plastic deformation and stress. The test results are shown in Fig. 2c, d.

According to Fig. 2c, besides Al substrate (S3) there are two kinds of precipitates observed in the base metal. These precipitates show the strip structure (S1) and bulk structure (S2). There are some fine punctuate precipitates dispersed among some big precipitates. However, there are the irregular bulk structures (S4), micro-voids and particles of the second phase (S5) in the Al-Cu-Mg alloys experienced the FSP including the Al substrate (S6) in Fig. 2d. According to Fig. 3, these precipitates or particles of second phases were the compounds of Al-Cu system, and the composition and structure of these precipitates or particles of second phases should also be different.

Moreover, after the FSP the size of these precipitates observed becomes smaller, and the size of some precipitates is less than 1 μm. Therefore, the Al-Cu-Mg alloys have experienced the obvious grain refinement. Also, there are a large number of micro-voids in the nugget zone; the diameter is about 1 μm. Barcellona [12] reported that a large number of micro-voids were observed in the nugget zone of AA2024-T4 aluminium alloys under FSW.

3.2. X-ray diffraction

According to XRD analysis, in the nugget zone of friction stir processed Al-Cu-Mg alloys the main phases were Al_2Cu, α-Al, Cu_2Mg and $Mg_{17}Al_{12}$ (see Fig. 4). That is to say, the fine precipitates close to nano-scale observed in nugget zone might be an Al_2Cu phase. However, according to the EDS analysis by STEM, there should be several different phase constitutions for micro-nano structure.

These structures formed may be the reason that some large precipitation strengthening phases in the nugget zone were possibly broken up and changed into the micro-nano structure by the severe stirring effect. The EDS analysis indicated that some structures in the nugget zone were composed of Al-Cu structure. Moreover, in base metal, there were a large number of large-scale precipitates of Al-Cu system. When the FSP was finished, some large-scale precipitates were not observed in the nugget zone. The reason is possi-

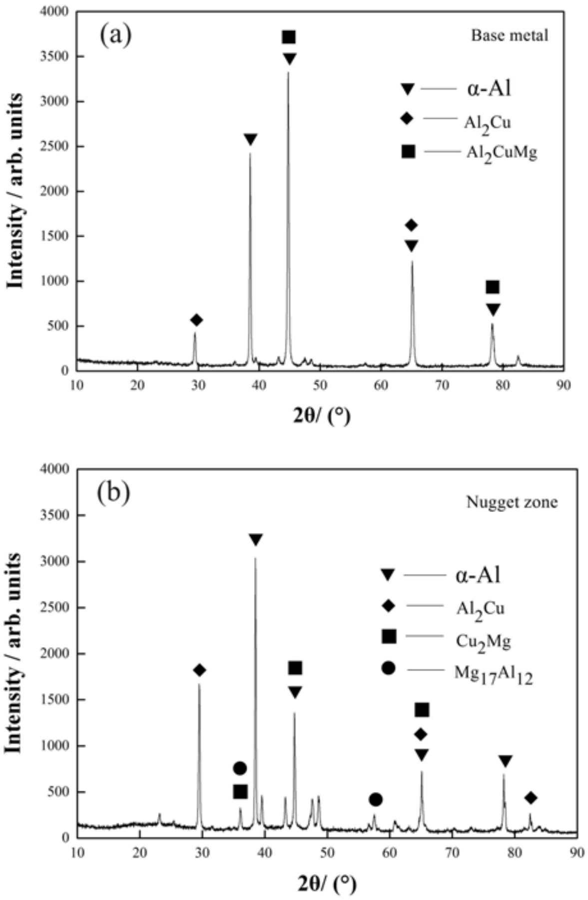

Fig. 4. XRD results of the base metal (a) and nugget zone (b).

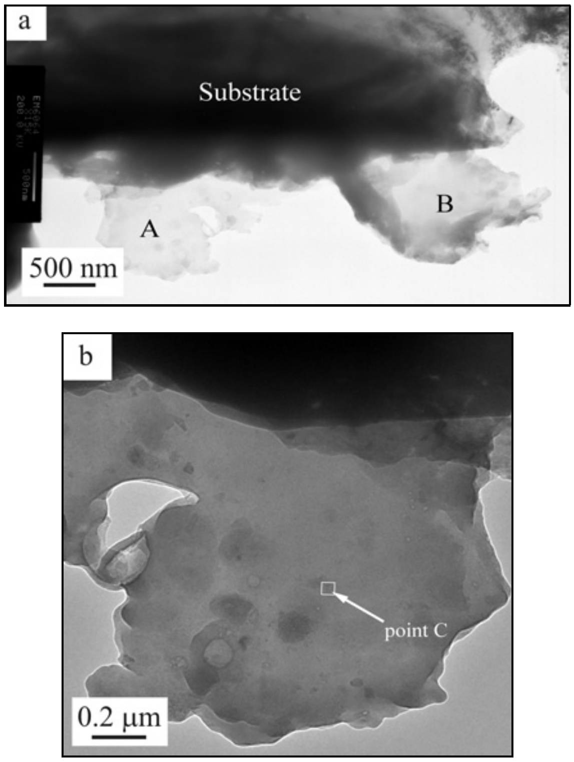

Fig. 5. TEM image of the compound and substrate in nugget zone: (a) TEM images and (b) the local TEM image of compound A.

bly according to the dislocations movement or breaking effect of stir tool. It is well known that the dislocations movement could enlarge the phase interface of large-scale precipitates, which form more fine grain with the increase of deformation. Some large-scale or bulk precipitates near the grain boundary, Si and β-$Mg_{17}Al_{12}$ broke up into fine structure dispersed on grain boundary or in grain during the FSP [13]. Therefore, the micro-nano structure formed was the effect of the dislocations movement and breaking effect of the stir tool.

3.3. TEM and HRTEM analysis

A compound different from the substrate structure in nugget zone of friction-stir-processed Al-Cu-Mg alloy was found by TEM (see Fig. 5a, region A and B). Figure 4b shows the local TEM image of region A (size of ~ 1 μm). The HRTEM images of point C in region A are shown in Fig. 6. Figure 6 shows the selected-area diffraction pattern of this region. According to Fig. 6, the compound has obvious structure characteristic of the long-range disorder, and the halo pattern was also detected in this region. The TEM imaging over wider area (see Fig. 5), HRTEM imaging (see Fig. 6), and diffraction pattern (see Fig. 6) can confirm the compound as a homogeneous amorphous structure. The homogeneous amorphous structure does not exist as a large amount through TEM observation, but shows scattered distribution among the crystal grain. According to Fig. 6b, the amorphous structure is composed of aluminium and copper.

The amorphous structure was detected in the nugget zone of friction-stir-processed Al-Cu-Mg alloy. This indicated that FSP technology could provide important physical condition for the formation of the amorphous structure. During FSP, the weld metal was plastically deformed by the rotating tool and friction. At the same time, the forging force of tool shoulder and stir action of tool pin induces shear stress and strain. This further accelerates grain refinement. Therefore, the amorphous structure is likely to be formed in this special physical condition.

According to our previous researches [10], the temperature gradient along the welding direction in the stir zone of FS processed aluminium alloy was about 2 ℃ mm^{-1}. The hydrostatic pressure of tool shoulder imposed on the weld metal in stir zone was about 20–80 MPa, and the shear stress of tool pin was about 2–10 MPa. The higher shear stress also can induce

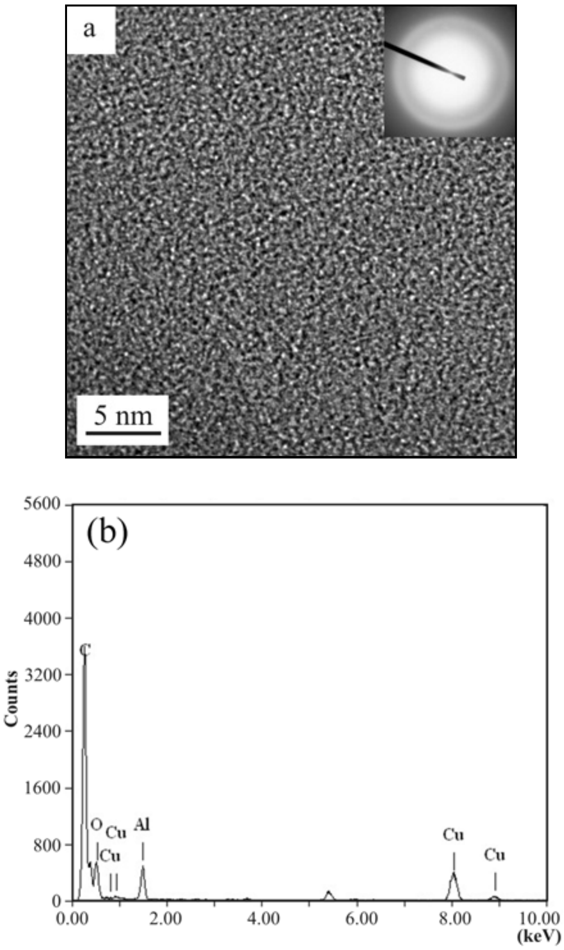

Fig. 6. HRTEM image (a) and EDS analysis (b) of point C in Fig. 5b.

higher strain. The original grain and subgrain boundaries in stir zone mainly appear to be replaced with fine, equiaxed recrystallized grains [14, 15]. However, the structure ordering of some atoms of weld metal in the nugget zone might be restrained by higher shear stress and strain in this special physical condition. Therefore, these influencing factors may induce the formation of amorphous structure in the stir zone of friction-stir-processed Al-Cu-Mg alloy finally.

4. Conclusions

1. The microstructure in the nugget zone of a friction-stir-processed Al-Cu-Mg alloy was analysed using SEM, STEM and XRD. There are two kinds of precipitates with the strip and bulk structure. There are also some micro-voids and particles of the second phase in the nugget zone, which were the compounds of Al-Cu system. These precipitates observed become smaller, and the size of some precipitates is less than 1 μm under FSP.

2. A homogeneous amorphous structure with structure characteristic of the long-range disorder has been found in the nugget zone of Al-Cu-Mg alloys by FSP. The amorphous structure is composed of aluminium (Al) and copper (Cu). Some influencing factors, such as higher shear stress, higher strain and grain refinement, may induce the formation of micro-nano and amorphous structure in the nugget zone.

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