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Amorphization of Al-Cu-Mg alloys by friction stir welding

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Abstract

The microstructure and phase constituents in stir zone of friction stir weld for 2024-T4 aluminium alloys were observed and analysed by transmission electron microscopy (TEM), high-resolution transmission electron microscopy (HRTEM) and X-ray diffraction analysis. Homogeneous amorphous structure in some area of the weld nugget zone was detected. The amorphous structure with halo pattern has a structure characteristic of the long-range disorder. X-ray diffraction analysis indicated that a diffuse scattering peak in the shape of steamed bun with amorphous characteristic exists in the degree scope of 15°–29°. Some influencing factors, such as higher temperature gradient, hydrostatic pressure and shear stress and strain, may induce the formation of amorphous structure in weld nugget zone of friction stir weld.

Key words: friction stir welding, amorphous structure, transmission electron microscopy, X-ray diffraction, microstructure

1. Introduction

At present, several processes such as rapid solidification (RS), mechanical alloying (MA), mechanical milling (MM), and a solid-state reaction (SSR) process can be enumerated as methods to produce the amorphous structure. Some amorphous structures of Al-based alloys, for example, Al-Cu-Ti, Al-Cu-Nb, and Al-Cu-Fe were prepared by these processes [1–3]. It is clear that the FSW processing was different from all the above methods in producing the amorphous structure. Friction stir welding (FSW) is a solid-state joining process patented by the Welding Institute (TWI) in 1991 [4]. FSW process has some advantages over conventional fusion welding processes, including nearly defect-free welds with no hot cracking, fine grain structure and minimized distortion. FSW uses a rotating tool consisting of a pin and tool shoulder to induce severe plastic deformation and frictional heating (below melting point temperature of materials) to the base metal to produce a strong metallurgical joining [5, 6].

Recently, microstructure and phase constitutions in the weld of FSW joint for various aluminium alloys were studied [7–10]. Moreover, the study relating to an amorphous structure that formed during the welding was also reported gradually. Firstly, the amorphous structure was detected at the weld interface by friction welding (FW) for aluminium alloys and stainless steel [11, 12]. The test results indicated that the amorphous structure was formed by mechanical alloying (MA) due to the friction process. Moreover, Sato et al. [13] reported that a high density of fine Al_2O_3 particles with an amorphous structure was found at the root part of FSW joint of Al alloy 5052. During the preparation of transmission electron microscopy (TEM) samples, those samples were thinned by focused ion beam (FIB). However, the FIB process was also used to prepare the thin amorphous layer [14–16]. Therefore, the formation mechanism of Al_2O_3 particles with amorphous structure found in above specimen might be not very clear. Tanaka et al. [17] reported that the thin amorphous layer of several nm in thickness including oxygen was found at the weld

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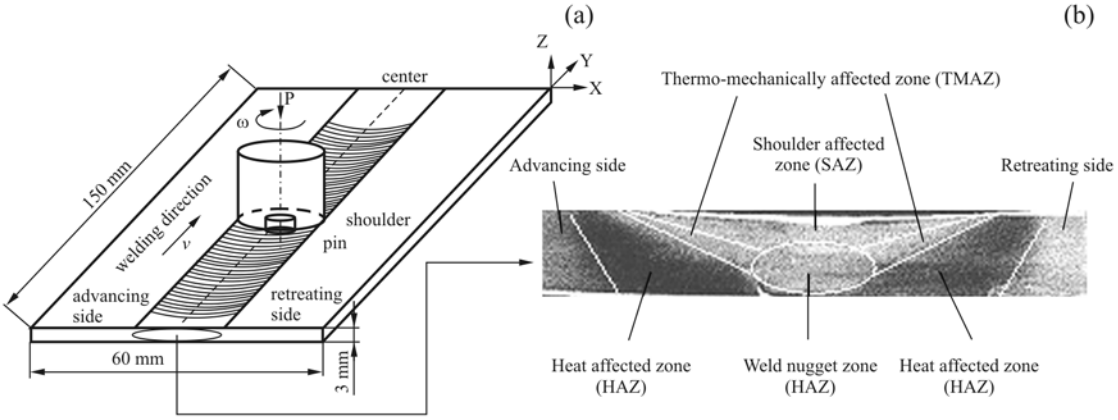

Fig. 1. The schematic of the friction stir welding (FSW) and cross section of FS weld: (a) the schematic of FSW; (b) metallographic image of cross section of FS weld.

interface of friction stir spot welding (FSSW) for aluminium alloy and steel sheets. However, unfortunately, the samples experienced FIB, too. Till now, the study on an amorphous structure formed during FSW which sample has not been processed by FIB has not been reported yet.

In this paper, the microstructure in stir zone of friction stir welded Al-Cu-Mg alloys (2024-T4 aluminium alloys) was analysed using TEM and HRTEM. During the preparation of TEM samples, some techniques such as ion beam thinner that possibly could induce amorphous structure were avoided. Therefore, the double jet electro-polishing was applied to make the samples to obtain the effective thinning area in this study. Then, the foils are further treated by ion-cleaning at low temperature 40 ℃ to avoid somewhat pollution. The X-ray diffraction technology was also used to analyse the existence of amorphous structure.

2. Experimental

The examined joined sheets of 2024-T4 aluminium alloys had dimensions of 150 mm × 60 mm × 3 mm, as shown in Fig. 1a. The nominal chemical composition 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, and balance Al. The two 3 mm thick sheets were butt welded, using the friction stir welding technique. The welding direction was parallel to the rolling direction of the sheets. The diameter of tool shoulder was 20 mm, and that for the pin was 6 mm. The tool rotational speed and travel speed were 475 rev min^{-1} and 5 mm s^{-1}, respectively.

A series of specimens was cut after friction stir welding, and then these specimens were made into metallographic samples. The samples were etched using mixed solution 1.0 % HF + 1.5 % HCl + 2.5 % HNO_3 + 95 % H_2O. The microstructure of a cross section of FS weld is shown in Fig. 1b. The weld of FSW joint is composed of weld nugget (WN), thermo-mechanically affected zone (TMAZ) and heat affected zone (HAZ). The WN was an important region that was directly influenced by the tool pin. Then, the thin foils were cut from the central stir zone along the cross section and were polished to 50 μm thickness. Moreover, the thin foils were mechanically cut to make 3 mm diameter discs. At last, 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 operating at 200 kV. The operation modes were selected-area diffraction and high-resolution transition electron microscopy (HRTEM). For XRD analysis, Rigaku-2500 equipment using copper target (Kα) radiation was used. The test conditions were 40 kV voltage and 200 mA current.

3. Results and discussion

3.1. TEM and HRTEM analysis

A compound different from the substrate structure among the crystal grain was found by TEM (see Fig. 2a, region A and B). Figure 2b shows the local TEM image of region A (size of ∼ 1 μm). The HRTEM images in region A are shown in Fig. 3a. Figure 3a shows the selected area diffraction pattern of this region. According to Fig. 3a, the compound has distinct structure characteristic of the long-range disorder, and the halo pattern was also detected in this region. The TEM imaging over a wider area (see Fig. 2), HRTEM imaging (see Fig. 3a), and diffraction pattern (see Fig. 3a) can confirm the compound as a homogeneous amorphous structure. The homogeneous amorphous structure does not exist as a large amount

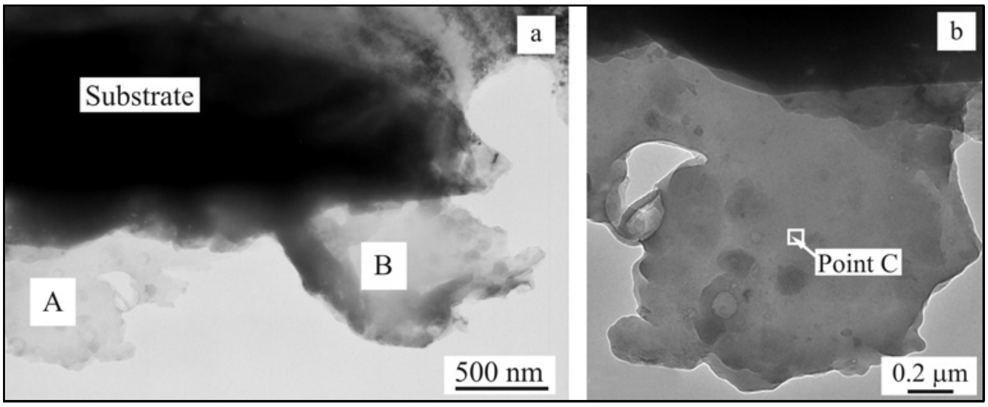

Fig. 2. TEM image in weld nugget zone of FS weld: (a) TEM image of new compound and substrate in weld nugget zone of FS weld; (b) the sectional TEM image of compound A.

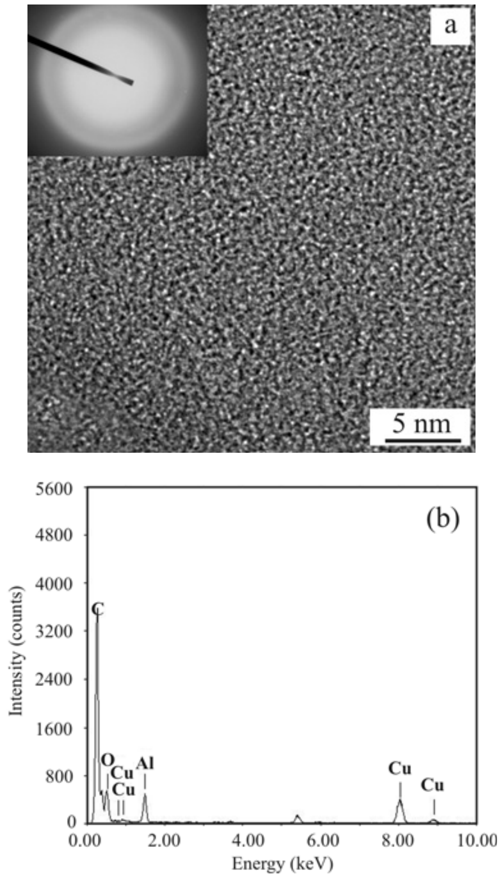

Fig. 3. HRTEM image, selected area electron diffraction pattern and EDS results: (a) HRTEM image and selected area electron diffraction pattern of Fig. 2b; (b) EDS results of point C in Fig. 2b.

through TEM observation, but shows scattered distribution among the crystal grains. The chemistry elements of amorphous structure can be determined by energy dispersive spectroscopy (EDS). According to Fig. 3b, the amorphous structure is composed of aluminium (Al) and copper (Cu). Moreover, a significant number of carbon elements (C) exist in test results. This reason may be that test foils were polluted and deposited on the surface of test foils, such as conductive adhesive, carbonization of organic compound in the sample cleaning, etc. However, the existence of oxygen element may be because the foil was oxidized in the air.

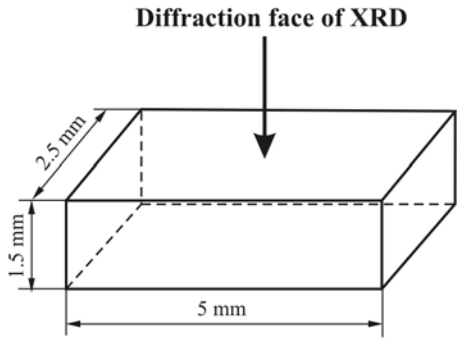

Fig. 4. The schematic of samples for X-ray diffraction analysis.

3.2. X-ray diffraction analysis

To further determine the existence of an amorphous structure, the weld nugget zone of FS weld was analysed using X-ray diffraction technology. The XRD samples were cut along the central weld. During the preparation of samples, the diffraction face was ensured in the weld nugget zone. The schematic of XRD sample was shown in Fig. 4. The base material of 2024 aluminium alloy was also cut and prepared into the same XRD sample comparing with FSW sample. The XRD analysis results are shown in Fig. 5.

According to Fig. 5, there is a lower diffuse scattering peak in the range 15° to 29°. This proved the existence of amorphous structure in the nugget zone.

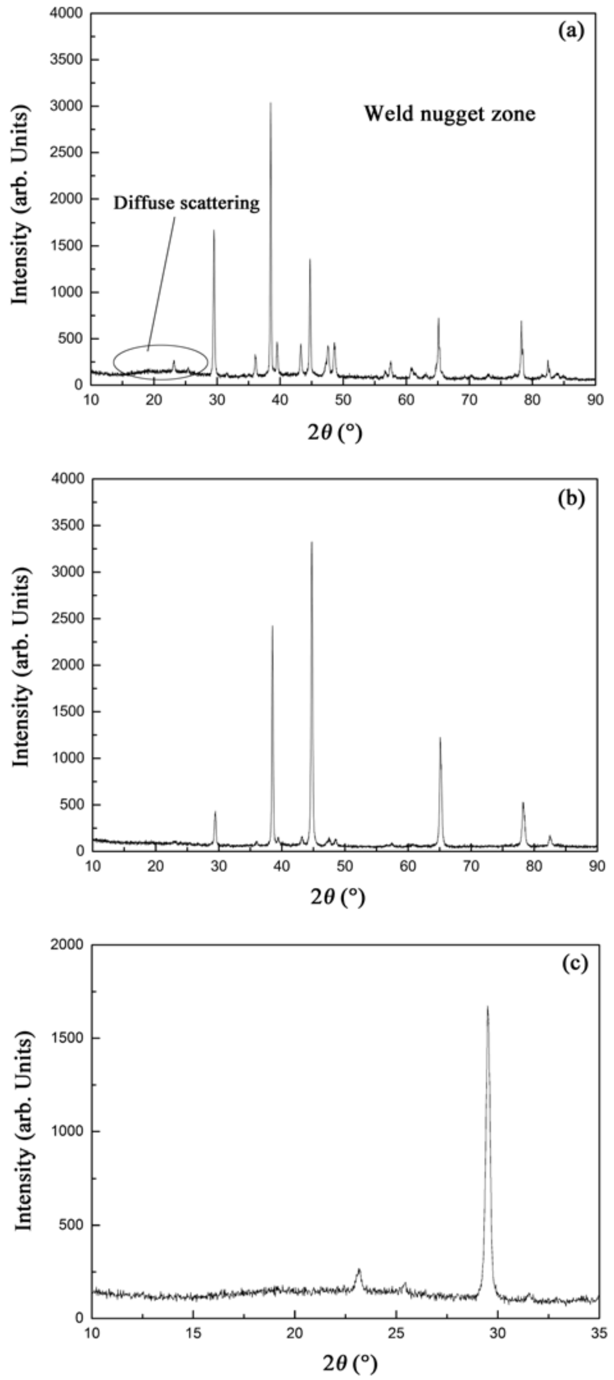

Fig. 5. X-ray diffraction pattern of weld nugget zone and base materials: (a) XRD results of weld nugget zone; (b) XRD results of base metal; (c) large local image of diffuse scattering region in Fig. 5a.

However, the intensity of diffuse scattering peak is very low, leading to the low content of amorphous structure. Moreover, there are two weak crystal peaks located at 23° and 25°, respectively. According to the report of Daniels et al. [18], Al-Cu-Fe-Cr film manufactured by physical vapour deposition (PVD) had an amorphous structure that gives a broad diffuse scattering region at low angles (16°–20°). However, the diffraction intensity is also very low. Moreover, Wu et al. [19, 20] reported that there was an obvious pre-peak in a smaller angle region of X-ray diffraction curve for Al-based amorphous structure. The intensive chemical short-range order in Al substrate might result in the pre-peak [21]. Therefore, Fig. 2c provides the indirect evidence for the formation of amorphous structure.

3.3. Discussion

The amorphous structure was detected in weld nugget zone (WNZ, Fig. 1b) of FS weld of 2024 aluminium alloys. This indicated that FSW technology could provide an outstanding physical condition for the formation of amorphous structure. During FSW, the weld metal was plastically deformed by the rotating tool and friction (see Fig. 1a). At the same time, the forging force of tool shoulder and stir action of tool pin induce shear stress and strain. This further accelerates grain refinement. Therefore, the amorphous structure is likely to be formed in the special physical condition.

The physical condition of FSW processing for 2024-T3 aluminium alloys similar to 2024-T4 aluminium alloys in this letter has been reported [22, 23]. The temperature, force, and torque have been measured during friction stir welding. According to this report, the temperature gradient along the welding direction in the weld nugget zone was about 2 ℃ mm^{-1}. The hydrostatic pressure of tool shoulder imposed on the weld nugget 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 higher strain. The original grain and subgrain boundaries in weld nugget zone mainly appear to be replaced with fine, equiaxed recrystallized grains [24]. However, the structure ordering of some atoms in weld nugget zone might be restrained by higher shear stress and strain in the special physical condition. Therefore, these influencing factors may induce the formation of amorphous structure in weld nugget zone of FS weld for 2024 aluminium alloys finally. Unfortunately, the area with an amorphous structure is very small. However, multi areas were found in the studied sample, noting that since the FSW is a quasi-steady state process, so there should be lots of small amorphous structures scattered in the bulk weld. Further studies on the amorphous structure in friction stir welds are being carried out, and more details might be reported in the future.

4. Conclusions

We have studied the microstructure in the FS weld for 2024 aluminium alloys by TEM, HRTEM, and XRD. A homogeneous amorphous structure with structure characteristic of the long-range disorder has been found. The halo pattern was also detected in this area. The homogeneous amorphous structure shows scattered distribution among the crystal grains. A diffuse scattering peak in the shape of steamed bun with amorphous characteristic exists in the degree scope of 15°–29°. Compared with TEM analysis results indicated that some amorphous structure was formed in weld nugget zone of FS weld, but the amorphous structure was very small. Some influencing factors, such as higher temperature gradient, hydrostatic pressure, shear stress and strain, may induce the formation of amorphous structure in stir zone of FS weld.

Acknowledgements

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