Modification of mechanical properties of friction stir welded Cu joint by additional liquid CO₂ cooling

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Abstract

A notable effect of the retained heat after the friction stir welding (FSW) of Cu was studied by the application of additional liquid CO₂ cooling. The adjustment of the welding parameters made it possible to conduct the FSW processes, but the cooling rate of the joint wasn't sufficiently changed due to the existence of the post-annealing effect. However, the accelerated cooling can reduce the post-annealing effect and provide an ultrafine grain structure with a high dislocation density in the stir zone, and a joint with mechanical properties better than the base metal level was achieved.

Keywords: Friction stir welding, Copper, Rapid cooling, Microstructure, Mechanical properties

1 Introduction

Cu has a wide range of applications in many industrial fields due to its excellent properties, such as high electrical and thermal conductivities, the favorable

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combinations of strength and ductility, and excellent corrosion resistance [1-7]. In contrast to its advantages, the conventional fusion welding of Cu requires a relatively high heat input due to its high thermal conductivity, resulting in a remarkable grain coarsening in the fusion zone, a wide heat-affected zone (HAZ) and a serious distortion with a high residual stress. One possible method to overcome these problems is friction stir welding (FSW), which is a solid-state joining process invented by The Welding Institute (TWI) of the UK in 1991 in an attempt to join aluminum alloys. The FSW has been significantly expanded to the welding of high melting point materials, such as Cu, steels and Mo [8-11]. During the FSW, a rotating tool is plunged into the clamped and butted plates to produce an intense local frictional heating which does not melt the plates and traverses along the weld center. Because the FSW is operated at a temperature lower than the melting point of the materials, a high-quality joint with a narrow HAZ and less distortion can be obtained. For example, Khodaverdizadeh et al. [2] successfully fabricated 5-mm-thick Cu joints using FSW at the tool rotation rates of 600 rpm and 900 rpm, and welding speeds of 25 mm/min and 75 mm/min. However, both the ultimate tensile strength and the yield strength of the FSW joints were lower than those of the base metal (BM) due to the grain coarsening and the recovery of dislocations in the stir zone (SZ). In order to enhance the mechanical properties of the joints, many researchers have recently operated the FSW of Cu under relatively low heat input conditions, and defect-free joints can then be achieved [12,13]. Liu et al. [12] obtained a sound 3-mm-thick FSWed Cu (1/2H condition) joint under a low heat input condition of 400 rpm and

100 mm/min, which produced a fine-grained structure (4 μm) in the SZ. Moreover, Xie et al. [13] successfully achieved a fine-grained microstructure (3.5 μm) in the SZ of copper (5-mm-thick with 1/2H condition) joints under the FSW condition of 400 rpm and 50 mm/min. According to their results, although the tensile strength of the SZ approached the BM level, the yield stress was still lower than that of the BM. This means that further improvement is needed with the microstructure control.

In addition, another characteristic of the FSW is the ultra-grain refinement. The mechanism of this feature has been occasionally referred to as dynamic recrystallization (DRX) [14]. During the early stage of the FSW research, only the continuous DRX was regarded as the dominant mechanism since the microstructure formed in the SZ looks like that in the hot deformation with a continuous DRX [15]. Later, the careful observations to clarify the inhomogeneity of the dislocation density in the SZ [10,16] suggested that the grain refinement was due to the multistep evolution consisting of not only the DRX, but also the dynamic recovery as well as the post-annealing effect after the stirring. Also, Fujii et al. reported that the post-recovery can be considered to provide some meaningful effect on the microstructure in the SZ based on the dislocation distribution observed by transmission electronic microscopy (TEM) [17]. Therefore, in this study, in order to reduce the post-effect on the FSWed Cu joint, we developed an additional liquid CO₂ cooling method to increase the cooling rate of the joint during the FSW in order to obtain a joint with mechanical properties better than the BM level.

2 Experimental details

Commercially pure Cu plates (99.97 %, 1/2H condition) with its size of $140^L \times 70^W \times 2^T \text{ mm}^3$ were used for the FSW. The rotating tool was made of a WC-based material and equipped with a columnar probe with no thread. To prevent oxidation of the samples, the FSW processes were carried out with Ar gas flowing around the rotation tool. The rapid cooling equipment was placed behind the rotating tool with movement speed the same as the welding speed [18]. Rapid cooling media is liquid CO_2 (-78°C) [18] with its cooling capacity is much better than that of other cooling media, such as water. A K-type thermocouple was placed under the work pieces and set in the groove of the back plate to measure the temperature history of the SZ. The designed welding parameters and the rotating tool size are shown in **Table 1**. The tool was tilted backwards at a 3 deg. from the plate normal direction and the axial load was 1.5 t. The welded joint obtained using conventional FSW without rapid cooling was denoted RT550. By using rapid cooling, in order to receive a similar peak temperature as the RT550 joint, the welding parameter should be changed to offset the thermal dissipation caused by the liquid CO_2 , and then the obtained sample was denoted LCO₂800. For comparison, the conventional FSW with a relatively lower heat input than RT550 was operated as well, and denoted RT400.

After the welding, the joints were cross-sectioned perpendicular to the welding direction for optical microscopy. Electron back scattering diffraction (EBSD) measurements were carried out using a scanning electron microscope (SEM) equipped with a field emission gun. The cut-off angle is 2° when the misorientation angle was

evaluated in the EBSD data. The microstructures in the SZ were also characterized by TEM. For the TEM sample preparation, thin polished samples were double-jet electro-polished using a solution of HPO_4 : CH_3OH : H_2O =1: 1: 2 in volume. The Vickers hardness of the joints was measured along the mid-thickness of the plates with a 50 g load for 15 s. The transverse tensile specimens covering various regions with a gauge size of $28^{\text{L}} \times 5^{\text{W}} \times 1.8^{\text{T}} \text{ mm}^3$ were machined with the tensile axis perpendicular to the welding direction. The tensile tests were carried out using an Instron-type testing machine with a crosshead speed of 1 mm/min. For the specimens fractured in the BM, in order to evaluate the tensile strength of the SZ, the specimens containing only the SZ were also machined with a gauge size of $12^{\text{L}} \times 3^{\text{W}} \times 1.8^{\text{T}} \text{ mm}^3$ with the tensile axis parallel to the welding direction.

3 Results and discussion

3.1 Temperature history

Figure 1 shows the temperature histories of the central SZ for the different welding conditions. All of the three underwent similar changes; the temperature rapidly increased as the rotation tool approached the measurement point and then decreased after the rotation tool passed through and away from the point. Without rapid cooling, although the SZ of the RT550 and RT400 joints showed different peak temperatures of 430 °C and 402 °C, respectively, they experienced a similar cooling rate of about 15 °C/s around the peak temperatures. Cooling rates of the SZs were calculated from peak temperatures to room temperature (25 °C), respectively. That is to say,

decreasing the heat input can reduce the peak temperature of the SZ, but the cooling rate of the SZ was still very slow. When the rapid cooling was operated, the rotating tool and the back plate as well as the specimen were significantly cooled by the liquid CO₂. Thus, it was necessary to increase the heat input by increasing the rotation rate or decreasing the welding speed to offset the thermal dissipation. For comparison, we tried to obtain a similar peak temperature as that of the RT550 joint. After many attempts, it was successfully found that under the condition of 800 rpm and 150 mm/min, the SZ showed a peak temperature of 428 °C, which was very similar as that of the RT550 joint, but the cooling rate around the peak temperature was increased to 38 °C/s. The times above 100 °C of the RT550, RT400 and LCO₂800 joints were 25.5 s, 24.0 s and 10.6 s, respectively. Clearly, the high temperature duration which can lead to recovery and grain growth during the post-welding period was significantly reduced by the rapid cooling.

3.2 Microstructure evolution

The cross-sectional macrographs of the joints under different welding conditions are shown in **Fig. 2**. No obvious welding defects, such as tunnel and root defects [19], were detected in all the joints. The macroscopical morphologies of the joints for the different welding conditions were similar, and all the joints showed a bowl-like SZ [10]. In the LCO₂800 joint, the size of the SZ became slightly smaller probably due to the thermal dissipation caused by the rapid cooling.

According to previous studies [2,20], the microstructure around the joint center

exhibits several distinct zones, i.e., the SZ at the weld central, the HAZ surrounding the SZ, and the thermo-mechanically affected zone (TMAZ) between the SZ and the HAZ. These zones can be also found in all the joints in this study. **Figure 3** shows the optical microstructures of the HAZ, TMAZ and SZ in the transition zone on the advancing side of the joints obtained under the different welding conditions. The mean grain sizes of the HAZs of the RT550 and RT400 joints are 31 μm and 28 μm , respectively, slightly larger than that of the BM (25 μm); whereas the HAZ of the LCO₂800 joint had a grain size (25 μm) same to that of the BM due to the additional rapid cooling. The TMAZ and SZ of all three joints have finer grain sizes, and the mean grain size of TMAZ is somewhat similar to that of the SZ as reported in previous studies due to the recrystallization was occurred in the TMAZ as well [12,21,22]. Therefore, the difference between the TMAZ and the SZ was not obvious. The same unclear results for the TMAZ were also reported by Lee et al. [23]. It should be noted that the TMAZ and the SZ in the LCO₂800 joint showed ultrafine structures whose mean grain size is less than 2 μm .

The comparison between the microstructures of the SZs of the joints with or without rapid cooling clarified the significant change. **Figure 4** shows the grain orientation color maps of the SZs as well as the BM. The BM showed an average grain size of 25.0 μm , whereas the SZs have much smaller equiaxed grains. The average grain sizes of the RT550, RT400 and LCO₂800 were 17.0, 14.0 and 1.8 μm , respectively. Without rapid cooling, by decreasing the heat input from the FSW at 550 rpm and 200 mm/min to that at 400 rpm and 400 mm/min, the grain size of the SZ

was slightly refined. This result just follows the previous study that the recrystallized grain size can be reduced by decreasing the ratio of the tool rotation rate/traverse speed [24]. As the rapid cooling proceeded, the grain size of the SZ was significantly reduced to 1.8 μm . The black and white lines in **Fig. 4** indicate the high angle boundary (16 to 62.8 deg. [25]) and low angle boundary (2 to 15 deg.), respectively. Many low angle boundaries were found in the BM because the BM is a work hardened condition (1/2 H). For the RT550 and RT400 joints, the low angle boundaries almost disappeared after the FSW, whereas, for the LCO₂800 joint, the low angle boundaries of the SZ significantly increased.

Figure 5 shows the number fraction of the misorientation angles of the central SZs as well as the BM. The BM has two major peaks (**Fig. 5(a)**). One peak was located at 60 deg., which is mainly contributed by the annealing twin boundaries [26]. The other peak was located at 2 to 5 deg., which indicates a dislocation tangle since the base metal was a work-hardened condition. In contrast, for the SZs, only one large peak appeared. Although the heat input was reduced from the FSW at 550 rpm and 200 mm/min (**Fig. 5(b)**) to that at 400 rpm and 400 mm/min (**Fig. 5(c)**), the peak locations at 60 deg. were not changed. This indicated that the number fraction of the twin boundaries was still very large. That is to say, the post-annealing effect cannot be significantly reduced by the conventional FSW without any additional cooling method. On the other hand, for the LCO₂800 joint, although the peak temperature of the SZ was similar as that of the RT550 joint, the number fraction of the twin boundaries significantly decreased. Meanwhile, the dislocations introduced by the severe plastic

deformation in the FSW and fine grain structure were maintained in the SZ due to the reduction of the post-annealing effect by rapid cooling. Therefore, the large peak was appeared in the low angle region (**Fig. 5(d)**).

Figure 6 shows the TEM bright-field images of the BM and the microstructures of the SZs obtained under different welding conditions. The BM shows a tangled dislocation structure with some twin boundaries which are typical for the deformation structure of the FCC metals with a relatively low stacking fault energy [10,27]. As for the RT400 joint, the grains containing a slight dislocation can be easily found in the SZ as shown in **Fig. 6(b)**, and the twins with their width much smaller than that of the BM can also be observed as is in **Fig. 6(c)**. On the other hand, the microstructure in the SZ of the LCO₂800 joint (**Fig. 6(d)**) shows two obvious differences from the other specimens. One is the number of dislocation cells. This indicated that the slip deformation mode occurred in the SZ, and the increasing stacking fault energy due to the temperature increase [28] allows the formation of the dislocation cell structure. The other difference is the disappearance of the twins which is also related to the temperature dependence of the stacking fault energy. It is noted that the TEM results well match the EBSD measurement that the SZ of the RT400 joint shows large quantity of high angle boundaries and twin boundaries, while the SZ of the LCO₂800 joint shows a higher number of low angle boundaries. Although Galvão et al. [29] received smaller grain sizes (1-2 μm) in the SZ of FSWed copper joint by using complex tool shoulder, the influence of post effect on the substructures in the SZ were not been well investigated. In this study, however, not only the grain size in the SZ

was remarkable refined, but the dislocation density was also increased (**Fig. 5(d)** and **Fig. 6(d)**) for the reduction of the post-annealing effect by using the liquid CO₂ cooling.

3.3 Mechanical properties

Figure 7 shows the Vickers hardness of the joints obtained under the different welding conditions. The BM showed an average hardness profile of 96 HV. For the welded joints without rapid cooling, although the grain size in the SZ was refined, the hardness profiles were much lower than that of the BM, and with decreasing the heat input, the hardness of the SZ slightly increased, but was still obviously lower than that of the BM due to disappearance of the substructures caused by the post-annealing effect. Similar results were reported by Hwang et al. [30] and Lee et al. [23], who also found that the hardness of the SZ of the FSW joints were lower than that of the BM. However, for the LCO₂800 joint, the hardness of the SZ was much higher than that of the BM due to the ultrafine grained structure and the large number fraction of low angle boundaries which were mainly composed of the dislocation cells.

Figure 8 shows the fracture location of the FSWed joints obtained under the different welding conditions. The tensile specimens of the RT550 and RT400 joints fractured almost in the geometric center of the SZ, while the tensile specimen of the LCO₂800 joint fractured in the BM. It can be found that all the specimens fracture at the locations with the lowest hardness in the samples, which well matches with the hardness measurement [31]. The fracture surface images of the BM and the SZs

obtained under different welding conditions are shown in **Fig. 9**. All the SZs experienced a ductility fracture because a high number of dimples were observed on the fracture surfaces. For the BM, the dimples were elongated and indicated a slip fracture mechanism. For the SZs, it can be seen that there are many regular dimples on the fracture surface, indicating a micro-void coalescence mechanism which is typical for ductile fracture.

The nominal stress-strain curves of the BM and the SZs based on the tensile tests are shown in **Fig. 10**. Two important observations were obtained. First, the FSW process without rapid cooling resulted in a reduced yield stress and large elongation in the SZ. By decreasing the heat input, the yield point of the RT400 joint slightly increased, but still cannot reach the BM level. In contrast, with rapid cooling, the yield stress of the SZ (220 MPa) increased by about 13 % compared with that of the BM (194 MPa). Second, for the RT550 and RT400 joints, although the yield stress was much lower than that of the BM, their tensile strength approached to the BM level due to the high strain strengthening capacity of Cu [2].

Based on the above description, it is concluded that the post-annealing effect significantly affects the microstructure and mechanical properties of the SZ. If only the heat input is decreased by adjusting the welding parameters, the cooling rate of the SZ cannot be changed. As a result, the yield stress of the SZ also showed a lower profile than that of the BM for the relatively low dislocation density. In contrast, as the rapid cooling occurred during the FSW, not only the grain size of the SZ was ultimately refined, but also the dislocation density of the SZ significantly increased.

Therefore, the SZ showed a significantly enhanced yield strength and the fracture occurred in the BM.

4. Conclusions

In this study, 2-mm thick commercial Cu plates were welded by FSW under different conditions with/without rapid cooling. The grain structure and mechanical properties in the SZ were investigated. Based on the above description, the following conclusions can be drawn:

(1) Defect-free Cu joints were obtained under the well-designed welding conditions.

By decreasing the heat input without any additional rapid cooling, the cooling rate of the SZ could not be changed. With the liquid CO₂ cooling, the cooling rate significantly increased, and the post-annealing effect was effectively reduced.

(2) Based on microstructure observations, without rapid cooling, the SZ showed a fine-grained structure and a low dislocation density in the grains. By using rapid cooling, the SZ showed a large number of sub-structures, which were composed of dislocation cells and tangles due to the reduced recovery and grain coarsening caused by the rapid cooling.

(3) Based on the tensile tests, without rapid cooling, the yield stress of the SZ slightly increased due to the grain refinement, and the tensile strength of the SZ did not reach the BM level. By using rapid cooling, both an ultra-grain refinement and dislocation strengthen mechanism occurred in the SZ, therefore, the yield stress of the SZ increased by about 13 % compared with that of the BM, while keeping an

adequate elongation. Meanwhile, the tensile strength of the SZ exceeded the BM level as well.

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Fig. 10. Nominal stress-strain curves of the BM and the SZs in the joints obtained under different welding conditions.

Table 1. Welding parameters and tool size used in the experiments

Welding parameters			
Sample name	Rotation rate (rpm)	Welding speed (mm/min)	Cooling condition
RT550	550	200	Room temperature
RT400	400	400	Room temperature
LCO ₂ 800	800	150	Liquid CO ₂
Tool size			
Shoulder diameter (mm)	Probe diameter (mm)	Probe length (mm)	
12	4	1.9	

Figure 1

Figure 2

Figure 3

Figure 4

Figure 5

Figure 6

Figure 7

Figure 8

	Fracture location
RT550	A photograph of a RT550 specimen, which is a long, thin, brownish metal bar with a central neck. The left end is labeled 'AS' and the right end is labeled 'RS'. A vertical dashed line indicates the fracture location at the center of the neck.
RT400	A photograph of a RT400 specimen, which is a long, thin, brownish metal bar with a central neck. A vertical dashed line indicates the fracture location at the center of the neck.
LCO ₂ 800	A photograph of a LCO ₂ 800 specimen, which is a long, thin, brownish metal bar with a central neck. The left end has a red 'd' marking and the right end has a red 'd' marking. A vertical dashed line indicates the fracture location at the center of the neck.

Figure 9

Figure 10