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Development of a fine-grained microstructure and the properties of a nugget zone in friction stir welded pure copper

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Defect-free pure copper welds were achieved under low heat input conditions of 400–800 rpm for a traverse speed of 50 mm min^{-1}. The grain size of the nugget zones decreased from 9 to 3.5 μm with decreasing rotation rate from 800 to 400 rpm. "Onion rings", consisting of different-sized grain bands, were observed at 400 rpm, but disappeared at 600–800 rpm. Variations of both microhardness and yield strength of the nugget zones with grain size followed the Hall–Petch relationship.

Keywords: Friction stir welding; Copper; Grain refining; Hardness; Strength

Friction stir welding (FSW), invented by The Welding Institute of the UK in 1991 [1], is an energy-efficient, environment-friendly and versatile joining technique that has proved to be one of the most significant achievements in the field of joining aluminum alloys [2]. While the research and applications of FSW have mainly focused on the aluminum alloys, investigations into the FSW of copper and copper alloys is quite limited [3–7]. This is attributed to the high melting point and good thermal conductibility of copper, which requires a higher heat input during FSW to achieve a defect-free copper weld, thereby resulting in more stringent requirements for tool materials and geometry design. Therefore, although copper has a face-centered cubic structure and good ductility, it is more difficult to obtain a sound FSW joint of copper than of aluminum and magnesium alloys [2]. It has been indicated that the tool material and geometry exert a significant effect on the feasibility of FSW of thick copper plates [7].

The higher heat input requirement for the FSW of copper means that the FSW must be conducted at lower welding speeds and/or higher rotation rates. For example, Okamoto et al. [5] fabricated a copper backplate for cooling by FSW at a tool rotation rate of 1300 rpm and a welding speed of 170 mm min^{-1}. Similarly, Lee and Jung [6] reported that 4-mm-thick copper plate was successfully welded at a rotation rate of 1250 rpm and a traverse speed of 61 mm min^{-1}. It should also be pointed out that the higher heat input during FSW of copper resulted in the generation of coarse recrystallized grains in the nugget zone. A grain size of 70–100 μm was observed in the nugget zone of FSW copper by Okamoto et al. [5] and Lee and Jung [6], a value significantly larger than that obtained in FSW aluminum alloys. It has been reported that a decrease in heat input can reduce the size of the recrystallized grains in FSW aluminum alloys significantly [8,9]. Therefore, it is expected that the recrystallized grain size of FSW copper can be refined by reducing the FSW heat input. A comprehensive investigation by Hautala and Tiainen [7] indicated that it is feasible to join copper by FSW under relative low heat input conditions. They did not measure the grain size in the nugget zone. However, the recrystallized grains appeared to be much smaller than those reported in Refs. [5,6].

It is noted that the studies mentioned above focused mainly on the FSW parameters and transverse tensile properties of the welds. An investigation into the correlation between the microstructure and properties of the nugget zone is lacking. In this study, FSW of commercial pure copper was conducted at various tool rotation rates for a constant welding speed. The purposes of this work are (i) to obtain fine recrystallized grain (<10 μm) in FSW copper by reducing the heat input; and (ii) to

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establish the relationship between the microstructure and properties of the nugget zone.

Commercial pure copper plate, 5 mm thick, under the 1/2H condition was used in this study. The chemical compositions were 0.1O–0.01S–0.01As–0.01Pb (in wt.%). Plates 300 mm in length and 70 mm in width were welded along the rolling direction using a gantry FSW machine (China FSW Center). FSW was conducted at a constant traverse speed of 50 mm min^{-1} with tool rotation rates of 400, 600 and 800 rpm. A tool with a shoulder of 20 mm diameter and a cylindrical threaded pin of 6 mm diameter and 4.7 mm length was used. The tilt angle for all welds was maintained at 2.5° and the plunge depth was controlled at ~0.2 mm. The FSW samples were cross-sectioned perpendicular to the welding direction, polished and then etched with a solution of 100 ml distilled water, 15 ml hydrochloric acid and 2.5 g iron(III) chloride. Microstructural features were characterized by optical microscopy (OM). Grain sizes were measured by the mean linear intercept method. The microhardness of the nugget zone was measured with a 50 g load for 10 s. The microhardness measurement near the "onion rings" was performed under a 10 g load for 10 s. Tensile specimens with a gauge length of 23 mm, a width of 4 mm and a thickness of 2 mm were machined parallel to the FSW direction with the gauge length being completely within the nugget zone. The tensile test was carried out using a Zwick–Roell-type testing machine at a strain rate of 7.2×10^{-4} s^{-1}.

Typical cross-sectional macrographs of the FSW copper weld are shown in Figure 1. Under the investigated welding parameters, no welding defect was detected in the welds. It is noted that the heat input in this study is much lower than that in the previous reports [5,6]. This indicates that sound welds can be achieved under relatively low heat input conditions. The macroscopical morphologies of the nugget zones at various parameters were significantly different. While the nugget zone at 600 and 800 rpm did not exhibit a clear outline, an elliptical-shaped nugget zone was observed at 400 rpm with obvious onion rings, and there was a clear boundary between the nugget zone and the thermomechanically affected zone.

The discrepancy reflected significantly different microstructures in the nugget zones produced at various rotation rates. As shown in Figure 2, the grain size of the nugget zone decreased with decreasing rotation rate. The grain size determined by the linear intercept method is summarized in Table 1. The average grain size was 12, 9 and 3.5 μm for tool rotation rates of 800, 600 and 400 rpm, respectively. Compared with the parent metal, FSW resulted in a significant decrease in the grain size of the nugget zone due to the occurrence of the dynamic recrystallization. The reduction in the grain size with decreasing rotation rate is attributed to the reduced heat input. At a constant traverse speed, the decrease in the rotation rate reduced the heat input of the FSW, thereby decreasing the size of the recrystallized grains. Similar observations have been made in FSW aluminum alloys [8,9]. In the previous studies, an average grain size of 70–100 μm in the nugget zone of the FSW copper was reported by Okamoto et al. [5] and Lee and Jung [6]. That was associated with the higher heat input used in their studies. This investigation clearly indicates that by reducing the heat input of the FSW the grains of copper can be significantly refined.

Onion rings – typical structures found on the nugget zone of the FSW aluminum alloys – have been reported by various investigators. The formation of the onion rings has been explained by the geometrical effect [10], the variations in grain size [11] and the particle-rich bands [12,13]. It has been suggested that FSW is similar to a severe extrusion process, so in the process of tool rotation, a thin-layer plasticized material is pushed to the back of the tool, causing the accumulation of multi-layer plasticized material [13]. Therefore, the cross-section of the nugget zone shows the onion rings. Krishnan found that the spacing of the onion rings was equal to the forward movement of the tool in one rotation [10]. On the other hand, Sutton et al. [13] reported that the onion rings were characterized by a segregated banded microstructure consisting of alternating hard

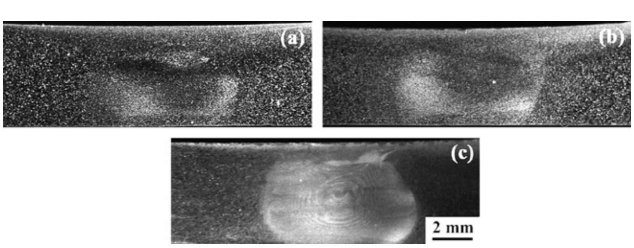

Figure 1. Cross-section macrographs of FSW copper joints: (a) 800 rpm, (b) 600 rpm, (c) 400 rpm (the advancing side is on the right).

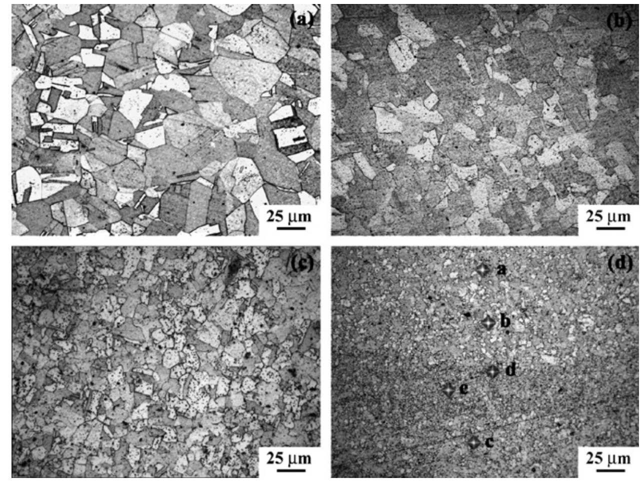

Figure 2. Optical micrographs showing the grain microstructure: (a) parent copper, (b) FSW, 800 rpm, (c) FSW, 600 rpm, (d) FSW, 400 rpm.

Table 1. Grain size and microhardness of pure copper under different conditions

Conditions	Grain size (μm)	Hardness (Hv)
Parent metal	18	82.2
FSW, 800 rpm	12	63.1
FSW, 600 rpm	9	72.8
FSW, 400 rpm	3.5	99.6

particle-rich and hard particle-poor regions. Furthermore, Biallas et al. [12] found that the onion rings vanished when the traverse speed decreased or the rotation rate increased. The result in this study is consistent with that in Ref. [12], i.e. the onion rings disappeared at the higher rotation rates of 600 and 800 rpm.

There were almost no secondary phase particles in the commercial pure copper, therefore the onion rings are not associated with the particle-rich and particle-poor regions. OM observations indicated that the onion rings consisted of alternating coarse grain bands (CGB) and fine grain bands (FGB), as shown in Figure 2d. The average grain size in the FGB was 1.2 μm. The formation of CGB and FGB might be associated with insufficient stirring and lower heat input at lower rotation rate. With increasing rotation rate, the enhanced stirring and heat input generated a relatively uniform grain structure in the nugget zone, therefore the onion rings tend to disappear.

Table 1 shows the Vickers microhardness of the parent metal and the nugget zones of the FSW copper under various rotation rates. The parent metal had a hardness of 82, which is consistent with the 1/2H condition. The FSW produced two competitive factors influencing the hardness of the nugget zone. On one hand, the FSW resulted in remarkable annealing softening, thereby reducing the hardness of the nugget zone. On the other hand, the significant grain refinement resulting from the FSW increased the hardness of the nugget zone. At the higher rotation rates of 600 and 800 rpm, although the grain size of the nugget zone was refined substantially, the hardness of the nugget zone was still obviously lower than that of the parent metal because the annealing softening effect was dominant. Similarly, Okamoto et al. [5], Lee and Jung [6] and Hautala and Tiainen [7] noted that the hardness of the nugget zone was lower than that of the parent copper after the FSW. Decreasing the tool rotation rate from 800 to 400 rpm caused the hardness of the nugget zone to increase continuously. At the lower rotation rate of 400 rpm, with further refinement of the grains (Fig. 2d), the fine grain strengthening effect enhanced significantly and exceeded the annealing softening effect. Therefore, the hardness of the nugget zone at 400 rpm was significantly higher than that of the parent metal. In order to measure the microhardness of the onion rings, a smaller loading (10 g) was used. Points a, b and c in Figure 2d were located in the CGB of the onion rings, while points d and e were located in the FGB of the onion rings. It was noted that the microhardness (105.4 Hv) in the FGB was higher than that (97.2 Hv) in the CGB. This result again showed that the microhardness of the nugget zone increased with decreasing recrystallized grain size.

Figure 3 shows the mechanical properties of the nugget zone and parent metal. The following important observations were made. First, the FSW resulted in reduced yield strength and improved ductility in the nugget zone due to significant annealing softening during the FSW. This is consistent with the hardness results (Table 1). However, the nugget zone in various FSW samples exhibited similar ultimate tensile strength, which was close to that of the parent metal. Second,

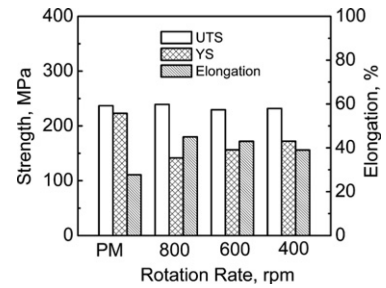

Figure 3. Mechanical properties of the parent metal and FSW copper samples.

for the FSW samples, the decrease in the tool rotation rate led to an increase in the yield strength and a decrease in the ductility of the nugget zone due to the reduced grain size (Table 1).

Recently, Chen et al. [14] summarized the hardness/yield strength vs. grain size relationship in a number of copper samples produced by various processing techniques. They reported that the hardness data follow the Hall–Petch line extrapolated from the coarse-grained copper (hereafter referred to as the CG-copper H–P line) [15] even when grain size is as small as 10 nm. However, for the ultrafine-grained copper samples prepared by severe plastic deformation (SPD), both the hardness and yield strength are obviously higher than those predicted by the CG-copper H–P line due to the existence of dense dislocation walls, tangles, cell walls or subgrain boundaries [14]. For the purpose of comparison, the present data of the FSW copper samples were plotted

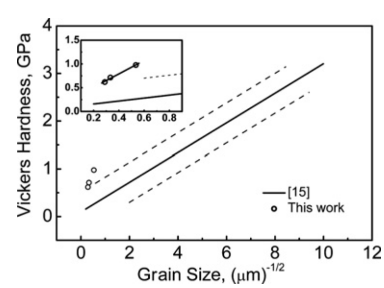

Figure 4. Relationship between microhardness and grain size. The solid line represents the CG-copper H–P line [15] and the dashed lines show the range of hardness data which follow the CG-copper H–P line [14].

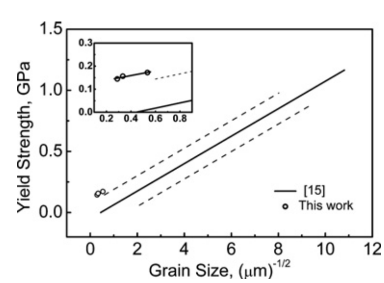

Figure 5. Relationship between yield strength and grain size. The solid line represents the CG-copper H–P line [15] and the dashed lines show the range of yield strength data which follow the CG-copper H–P line [14].

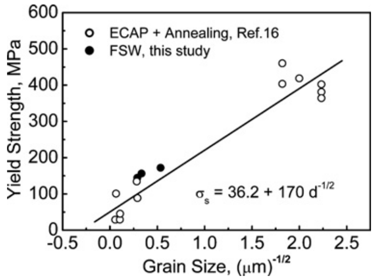

Figure 6. Relationship between yield strength and grain size.

as hardness/yield strength vs. grain size in Figures 4 and 5, with the CG-copper H–P line [15] and the boundary lines showing the range in which the hardness and yield strength data were considered to follow the CG-copper H–P line [14]. Figures 4 and 5 reveal that while the hardness and yield strength data of the FSW copper samples follow the Hall–Petch relationship, they are obviously higher than the CG-copper H–P lines and exceed the boundary lines defined by Chen et al. However, both the hardness and the yield strength data of the FSW copper samples are very close to those of the SPD ultra-fine-grained copper samples [14]. This indicates that, like those of the SPD copper samples, the hardness and yield strength data of the FSW copper do not fit the CG-copper H–P line.

More recently, Estrin et al. [16] summarized the yield strength vs. grain size relationship in a number of pure copper samples, produced by equal channel angle processing (ECAP) and subsequent annealing, from various investigators. It was indicated that for a wide range of the grain sizes, the annealed ECAP copper samples followed the Hall–Petch relationship: $\sigma_s = 36.2 + 170d^{-1/2}$ (Fig. 6). From Figure 6, it is clear that the present yield strength data from the FSW samples is very close to the linear fitting line, i.e. the yield strength data from both the FSW and the annealed ECAP copper samples can be described by the same Hall–Petch equation.

The present study indicates that the grain microstructure of commercial pure copper can be adjusted via the FSW technique to achieve tailor-made properties. It is possible to achieve a micrometric or even submicrometric grain microstructure by reducing the heat input or applying the active cooling. It is expected that the properties of the FSW copper joints can be remarkably enhanced by controlling the FSW conditions. Research into this subject is currently in progress.

In summary, the following conclusions are reached:

1. Defect-free copper welds were achieved under relatively low heat input conditions with a fine-grained microstructure of 3.5–9 μm being produced at a rotation rate of 400–800 rpm for a traverse speed of 50 mm min^{-1}. The grain size in the nugget zone of the FSW copper decreased with reducing tool rotation rate.
2. Onion rings were observed in the nugget zone for a lower rotation rate of 400 rpm. These were identified to consist of alternating coarse grain bands and fine grain bands. At the higher rotation rates of 600 and 800 rpm the onion rings disappeared.
3. With decreasing grain size of the nugget zone, the microhardness and yield strength of the nugget zone increased and the ductility decreased, and the variation trend followed the Hall–Petch relationship but did not fit the Hall–Petch line for coarse-grained copper.

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