Materials Characterization 121 (2016) 187–194

Contents lists available at ScienceDirect

Materials Characterization

journal homepage: www.elsevier.com/locate/matchar

Microstructure and mechanical properties of friction stir processed Cu with an ideal ultrafine-grained structure

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ARTICLE INFO

Article history:
Received 1 September 2016
Received in revised form 7 October 2016
Accepted 8 October 2016
Available online 11 October 2016

Keywords:
Friction stir processing
Ultrafine-grained structure
Pure copper
Microstructure
Strength
Ductility

ABSTRACT

Ultrafine-grained (UFG) Cu with uniform microstructure was successfully prepared by friction stir processing (FSP) under additional water cooling. FSP Cu exhibited equiaxed grains with low dislocation density, weak texture, and high fraction of high-angle grain boundaries. Isotropy and tension-compression symmetry were achieved in FSP Cu sample, which provided an ideal model material to investigate the intrinsic mechanical behavior of the UFG material. Enhanced strain hardening was achieved in FSP Cu due to the special microstructure which was effective in accumulating dislocations, resulting in good strength-ductility synergy. This study provides a feasible strategy of preparing bulk UFG materials with good mechanical properties.

1. Introduction

Ultrafine-grained (UFG) materials have attracted extensive concern due to their significantly enhanced hardness and strength [1]. However, most of these materials have low tensile ductility at ambient temperature, which limits their practical applications. The elongation to failure is far less than that of their coarse-grained (CG) counterpart [2,3]. Usually, the disappointingly low ductility can be attributed to the artifacts from processing, the plastic instability (necking or shear localization) with little or no strain hardening (dislocation storage) capacity, and low resistance to crack initiation and propagation [4].

One-step severe plastic deformation (SPD) methods, such as equal-channel angular pressing (ECAP), high-pressure torsion (HPT) and dynamic plastic deformation (DPD), provide practical approaches to producing bulk UFG materials that can exhibit mechanical properties controlled by their intrinsic deformation mechanisms [1,3]. Usually, unstable microstructure with high density of dislocations and strong texture is obtained in SPD UFG materials, which results in insufficient strain hardening capacity [4,5]. Plastic instabilities in such non-equilibrium systems of heavily deformed metals are very common and have been proven to occur, and low tensile ductility is still achieved in these UFG materials [6]. Moreover, grain coarsening and large-scale shear bands are easily formed during cyclic deformation, which are very harmful to the fatigue properties of the UFG materials [7]. Therefore, new methods are expected to be produced in order to improving the mechanical properties of the UFG materials.

Based on the principle of friction stir welding (FSW), Mishra et al. [8] developed a new SPD method of friction stir processing (FSP) for microstructural modification of materials. FSP has been shown as an effective technique in many applications, such as refining the microstructure of cast alloys, fabricating surface composites, and producing fine-grained structure, which exhibits good superplasticity [9,10]. Recently, FSP has been proven to be an effective method to prepare bulk UFG materials, such as Al, Mg, Cu, Ni and their alloys [11–18].

The grain refinement mechanism of FSP is dynamic recrystallization (DRX), so fine and equiaxed grains with quite uniform sizes are produced in the processed zone (PZ) [9–19]. Usually, SPD UFG materials exhibit high density of dislocations and most grain boundaries (GBs) are wavy, diffuse, and ill-defined non-equilibrium GBs [4,20]. Differently, large fraction of equilibrium high-angle grain boundaries (HAGBs, misorientation angle ≥15°) are obtained in the FSP UFG materials, and low density of dislocations exist in the ultrafine grains. Therefore, good strength and ductility can be obtained in the FSP UFG materials compared to that of other SPD materials, resulting from the enhanced strain hardening [11–19]. In this study, UFG materials were successfully prepared by FSP with additional water cooling in pure Cu samples, and the microstructure and mechanical properties of the FSP Cu were investigated and the strength-ductility relationships of various UFG Cu samples were discussed.

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http://dx.doi.org/10.1016/j.matchar.2016.10.009
1044-5803/

2. Experimental Procedures

In order to compare with other findings of UFG Cu more accurately, we used two base materials (BM) with different purities: one is the common commercially pure Cu T3 (99.9%) and the other is high purity oxygen free Cu TU1 (99.99%). The schematic illustration of FSP process is shown in Fig. 1. Before FSP, the initial Cu plates were annealed at 700 °C for 2 h, and the grain size was about 200 μm. To yield low heat-input during FSP, the Cu plates were first fixed in water and additional cooling by flowing water was adopted. Detailed parameters about the water cooling have been stated in the previous study [21]. FSP process was performed on the Cu plates using a rotating tool with the shoulder 20 mm, 12 mm and 10 mm in diameter at relatively low rotation rates of 400 rpm, 600 rpm and 400 rpm, respectively. The FSP samples prepared by the above parameters were designated as FSP-T3-a, FSP-T3-b, FSP-T3-c and FSP-TU1-a, FSP-TU1-b, FSP-TU1-c for the T3 Cu and TU1 Cu samples, respectively.

Microstructural examination was completed with optical microscopy (OM), scanning electron microscope (SEM) equipped with electron backscatter diffraction (EBSD) system, and transmission electron microscopy (TEM). EBSD experiments were performed using an Oxford HKL Channel 5 system on a LEO Supra 35 FEG SEM with step size of 70 nm. In order to investigate the microstructural uniformity in the PZ, EBSD scans were performed in various areas of FSP T3 Cu samples. TEM observation was carried out on a FEI Tecnai G^2 20 microscope operating at 200 kV. Thin foils for TEM were twin-jet electropolished by a solution of 25% alcohol, 25% phosphorus acid and 50% deionized water at 263 K.

Micohardness tests were performed on the cross-section (*x*-*z* plane in Fig. 1) of FSP Cu samples, five points were tested on each specimen (the test positions were shown as the squares in Fig. 2), and the relationship between the grain size and hardness value was discussed. Moreover, in order to investigate the homogeneity of the mechanical properties in FSP UFG Cu, the hardness distribution in the PZ was tested in FSP-TU1-b sample. During the hardness test, a load of 50 g with a holding time of 15 s was used. For the tensile test, the dog-bone-shaped specimens with a gauge length of 5 mm and a width of 1.4 mm were machined along the processing direction from the PZ, and polished to a thickness of 0.7 mm. Meanwhile, we investigated the tensile and compression behaviors in different directions in the PZ of FSP-T3-a sample, i.e., perpendicular and parallel to the processing direction, respectively (*x* (transverse) and *y* (longitudinal) directions in Fig. 1). Uniaxial tensile tests were conducted at room temperature at an initial strain rate of $1 \times 10^{-3}\ s^{-1}$. For the compression test, the gauge length of the specimen is 4 mm and the size of cross section is 2×2 mm.

3. Results and Discussion

Fig. 2 shows the typical cross-sectional macrograph of the FSP Cu for FSP-TU1-b sample. A typical basin-shaped PZ which widened near the upper surface was observed on the transverse section of the FSP Cu,

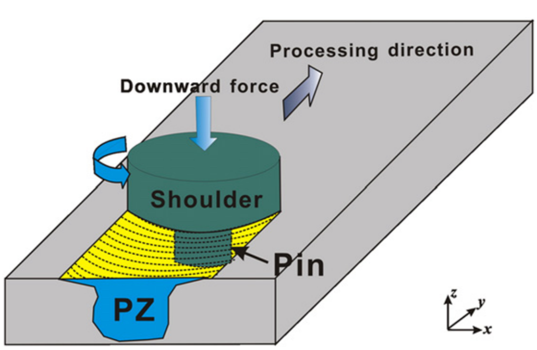

Fig. 1. Schematic illustration of FSP process.

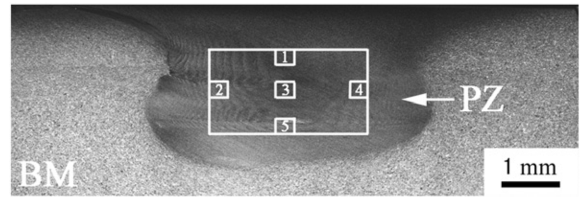

Fig. 2. Cross-sectional macrostructure and schematic positions for hardness test of processed zone (PZ).

and obvious onion rings could be observed which was similar to other observations in various studies [9,22,23].

Fig. 3 shows the EBSD microstructure at different positions of every 1.2 mm along the centerline of transverse sectioned PZ for FSP-T3-a sample. As can be seen from Fig. 3, all the EBSD micrographs in the PZ exhibited equiaxed grains, and the same average grain size and distribution characteristic were achieved in each area. Simultaneously, the microstructure of FSP-T3-b sample of every 120 μm along the vertical centerline from top to bottom in the PZ was analyzed by EBSD, as shown in Fig. 4. Similarly, the microstructure of each area from top to bottom in the PZ was almost the same, which also exhibited equiaxed grains with smaller grain size than that of the FSP-T3-a sample. Clearly, the microstructure was almost the same in every position of the whole PZ. Therefore, FSP is an effective method to prepare bulk UFG materials with uniform microstructure.

The typical TEM microstructure of FSP-T3-a, FSP-T3-b, FSP-TU1-b, and FSP-TU1-c samples is shown in Fig. 5. As can be seen from the TEM microstructure, the grains of FSP Cu exhibited equiaxed recrystallized state, and the dislocation density was low. Thus, the ultrafine grains obtained in FSP Cu under forced cooling were similar to those of the recrystallized grains in the nugget zone/PZ of conventional FSW/FSP processes, and DRX should be the main grain refinement mechanism in the present FSP Cu [9].

Moreover, as we can see from the TEM microstructure of the FSP Cu, under a same parameter (such as the b parameter), grain refinement was more notable in T3 Cu than that of TU1 Cu, as shown in Fig. 5b and c. The average grain sizes (containing sub-grains) of FSP-T3-b and FSP-TU1-b samples were about 500 nm and 800 nm, respectively (Table 1). It means that the presence of a small amount of impurity particles in T3 Cu provide more nucleation sites during recrystallization, which can effectively prevent the grain growth, resulting in the smaller grain size than that of the TU1 Cu.

However, the microstructure of FSP-TU1-b sample was similar to that of FSP-T3-a sample, and the average grain size was about 800 nm. Further, the microstructure of FSP-TU1-c sample was similar to that of FSP-T3-b sample, and the average grain size was about 500 nm. It is clear that approximately same microstructure was achieved in T3 Cu and TU1 Cu samples under different FSP parameters, and it is significant to investigate the mechanical behaviors of various UFG Cu samples.

Plenty of individual straight boundaries that traversed the whole grains were frequently observed in the ultrafine grains, as illustrated by the arrows in Fig. 5. According to the typical selected-area electron diffraction pattern shown in the upper right corner of Fig. 6a, most of these straight boundaries were Σ3 coincident-site lattice (CSL) twin boundaries (TBs) and exhibited the typical twin relationship of {111}/[112] type in Cu. These TBs should be annealing TBs which formed during the recrystallization process, according to the previous studies [15,24,25]. Moreover, from the typical high-resolution TEM image in Fig. 6b, it can be seen that these annealing TBs were perfectly coherent and no lattice dislocation was detected. These annealing TBs in FSP Cu were different from the deformation TBs in SPD Cu where excessive dislocations existed [4,20].

The TEM microstructural characteristics were further confirmed by EBSD studies in FSP-T3-a sample, as shown in Fig. 7. Similar to the TEM observations, the microstructure of FSP Cu was characterized by