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Microstructure evolution, hardening and thermal behavior of commercially pure copper subjected to torsion deformation

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ABSTRACT

Commercial pure copper was torsion-deformed under equivalent strains from 1 to 5 at ambient temperature. Microstructure evolution of torsion samples was characterized using metallographic observation on the longitudinal section parallel to the torsion axis and transverse section perpendicular to the torsion axis. Elongated ultrafine grained structure was observed on the edge area of the bar samples. The grain size decreased with increasing shear strain. While no obvious refinement was significantly improved on the central area. Transmission electron microscopy (TEM) observation showed that microstructure refinement correlated with the accumulation and rearrangement of dislocations. The results of Vickers hardness measurement indicated that the inhomogeneous hardness distribution on the transverse section of samples was similar to the counterpart of microstructure. The variations of hardness and microstructure both depended on the value of torsional simple shear depending on the radial positions of the samples. The hardening of torsional samples was mainly attributed to dislocation strengthening. Differential scanning calorimeter (DSC) results revealed that an exothermic peak occurs in the temperature range ~250–300 °C during the DSC scan of torsion samples.

1. Introduction

Severe plastic deformation (SPD) is now becoming increasingly attractive as an effective technique for producing bulk ultrafine grained (UFG) and nanocrystalline materials with grain sizes less than 1 μm [1]. Several SPD techniques are now available for employing high strains into bulk materials, such as equal channel angular pressing (ECAP), high pressure torsion (HPT), multi-directional forging (MDF) and accumulative roll bonding (ARB) [2]. It has been known that certain amounts of strain are necessary to obtain ultrafine grains with large misorientations [3]. Generally, SPD methods consist of combined loads of torsion, compression or/and tension [4,5]. Amongst all SPD techniques, HPT – as an effective combination loading of torsion and compression employing a high compressive torsion deformation into the materials – exerts its exceptional advantages in grain refinement [6]. Torsion deformation has been recognized as an efficient SPD mode for obtaining a higher plasticity strain compared to that in tension or compression deformation [7,8]. Hence, this process has been modified to produce bulk UFG materials, e.g. HPT [9] and twist extrusion (TE) [10]. However, the change in microstructure and mechanical properties of UFG materials fabricated by torsion deformation has been little systematically studied.

Early in the last century, some scholars [11–14] used different methods to study the unique deformation characteristics of torsion deformation. However, related research was rarely carried out in this century. Bassim et al. [11] explained the difference in work hardening for commercial copper deformed in both tension and torsion deformations. Szekely et al. [15] observed hardness distribution variation with distance from the torsional axis in the torsional samples. Kim et al. [16] used finite element analysis to investigate the plastic deformation behavior of cylindrical copper samples during torsion. Khamsuk et al. [17] investigated microstructure evolution in commercial purity aluminum during torsion deformation using electron backscatter diffraction analysis (EBSD). It can be seen that the previous study concentrated only on torsional microstructure observation.

The reduction of grain size can cause the non-equilibrium state of ultra-fine grain boundary accompanying the decrease of deformation storage energy. Studying the thermal stability of the material crystal structure is of great significance because it relates not only to the suitable working temperature of the fine microstructure but also to the preparation method of the UFG material. Particularly noteworthy is that many different analytical techniques have been employed to study SPD-processed materials, including transmission electron microscopy (TEM), scanning

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electron microscopy (SEM), X-ray diffractometry and EBSD. As the thermal analysis technique widely used to measure the thermal stability of processed materials, differential scanning calorimeter (DSC) analysis is now becoming increasingly attractive [18]. The ultrafine microstructures of SPD-processed materials are often inherently thermally unstable. The energy released in the DSC analysis is directly related to the grain boundary change [18]. Recently DSC has been increasingly applied for SPDed materials. Nevertheless, there are only a limited number of reports describing the application of DSC to copper materials processed by HPT [19], ARB [20,21] and ECAP [22–24].

The present work aims to clarify the change in the microstructure and mechanical properties of commercial pure copper during the torsion process. The microstructure stability and deformation storage of UFG copper were also quantitatively studied using DSC measurement.

2. Pure shear in torsion deformation

The significant deformations ranging from pure shear to simple shear have been increasingly recognized in the SPD process [25]. Simple shear is observed in shear bands of many effective SPD processes of HPT, ECAP and TE [26]. The stress state of deformed material is in the torsional shear deformation (Fig. 1). The distribution of shear stress mainly lies in the longitudinal and transverse inside vertical sections. The angle between the direction of principal stress σ_1 or σ_3 and the axial direction is $\sim 45^\circ$ ($\sigma_2=0$). The values of principal stress are both equal to the shear stress τ. The principal stresses σ_1 and σ_3 are tensile stress and pressure stress, respectively.

The intergranular deformation is more important than the transgranular deformation in the severe deformation. Slipping and twinning are the two main deformation mechanisms in material plastic deformation; the refinement of metal material in SPD can be explained by the above two mechanisms. However, the formation of large angle grain boundary in SPDed material is not easy to explain using the traditional dynamic recrystallization theory. Subsequently, the continuous dynamic recrystallization and geometric dynamic recrystallization contribute to the understanding of the formation of large angle grain boundary in individual materials during SPD. Hence, the movement and migration of grain boundary in SPD are worth further studying. Assuming that the approximately cubic grains have a regularly annular distribution on the transverse section of the torsion sample, the loaded deformation on the grain boundary is proportional to the strain. Assuming the grain boundaries act as the primary sources of dislocations, an idealized cubic grain is transformed into a parallelepiped with the angle of inclination under the influence of torsion shear. With the strain increasing, an equiaxed grain structure becomes a parallelepiped under the impact of shear stress. This above analysis is in agreement with the hypothesis of two-stage deformation by Beygelzimer [27]. In the hypothesis, some accidental multi-scale rotative motions, similar to turbulent motions in liquids, take place in the material's severe plastic deformation.

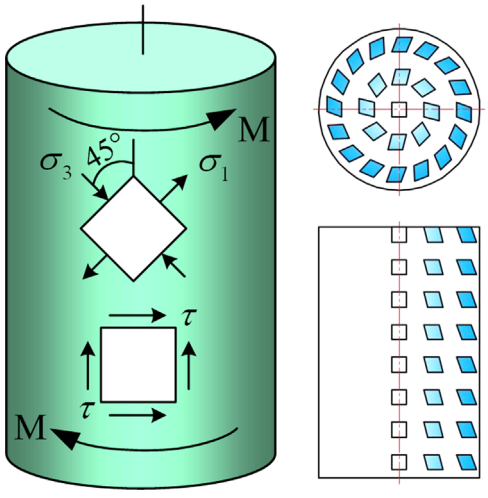

Fig. 1. Schematic diagram of stress and grain-boundary rotation in torsion deformation.

3. Simulation and experimental procedures

The finite element software ABAQUS was used to simulate the torsion deformation of the copper sample, which was 5 mm in diameter and 35 mm in length. The data of the stress–strain curve was from reference [16]. The calculations of only half of the circumferential section (*rz* plane) were adequate for model establishment. The number of initial mesh (four nodes generalized axisymmetric element with a twist, CGAX4) was 5000.

Commercial pure copper bars (99.7 in wt%) were preconditioned to obtain a fully recrystallized microstructure with an average grain size of 20–40 μm and a hardness of ~40 HV by annealing at 650 °C for 2 h followed by furnace cooling. The starting bars were machined into torsion samples with dimensions 5 mm in length and 35 mm in diameter. The samples were deformed in torsion at ambient temperature by rotations of 3.86, 7.72, 11.58, 15.44, and 19.30 turn, corresponding to the maximum equivalent strains of 1, 2, 3, 4, and 5 at the sample surface (assuming the same length), respectively. Torsion deformation was achieved in a wire torsion testing machine XC-10 under the torsional rate of 30 r/min. Fig. 2 shows the photograph of torsion samples. The torsional helix increases with increase in torsional turns.

The equivalent strain in torsion can be calculated as [28]

$$\gamma = 2\pi Nr/l, \quad \varepsilon = \gamma/\sqrt{3} \tag{1}$$

where ε is equivalent strain, γ is shear strain, N is the number of rotation, r is the radial position in the sample, and l is the sample length.

The transverse and longitudinal segments were wire-cut from the positions of processed bars (Fig. 3). Segments were performed by coarse grinding and fine grinding at water-proof abrasive papers, and then mechanically polished with diamond powder. For optical microscope (OM) observations, the samples were etched with a solution containing $FeCl_3$, HCl and H_2O (with a ratio of 1:3:20) for 10 s. An OM of Olympus PMG3 was used to observe the macrographs.

The thin foils for TEM observations were cut from the longitudinal section of the torsion samples, mechanically ground to about 40 μm, and finally thinned using a precision ion polishing system (Model 691 PIPS, GATAN Inc.). The microstructure observations were

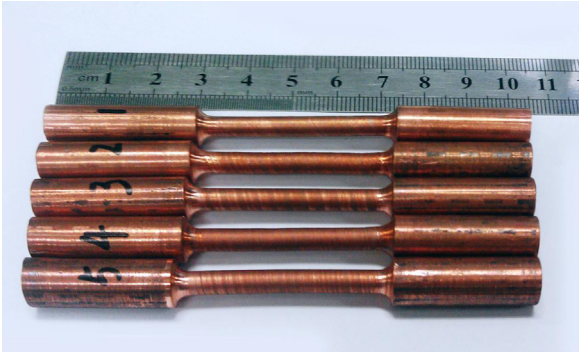

Fig. 2. Photograph of torsional deformed samples.