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Microstructure and microhardness of OFHC copper processed by high-pressure torsion

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ABSTRACT

An ultra-high purity oxygen free high conductivity (OFHC) Cu was investigated to determine the evolution of microstructure and microhardness during processing by high-pressure torsion (HPT). Disks were processed at ambient temperature, the microstructures were observed at the center, mid-radius and near-edge positions and the Vickers microhardness was recorded along radial directions. At low strains, Σ3 twin boundaries are formed due to dynamic recrystallization before microstructural refinement and ultimately a stabilized ultrafine grain structure is formed in the near-edge position with an average grain size of ~280 nm after 10 turns. Measurements show the microhardness initially increases to ~150 Hv at an equivalent strain of ~2, then falls to about ~80 Hv during dynamic recrystallization up to a strain of ~8 and thereafter increases again to a saturated value of ~150 Hv at strains above ~22. The delay in microstructure and microhardness homogeneity by dynamic recrystallization is attributed to the high purity of Cu that enhances dislocation mobility and causes dynamic softening during the early stages of straining.

1. Introduction

The processing of ultrafine-grained (UFG) metals, with grain sizes in the submicrometer or nanometer range, has become of major importance over the last two decades [1–3], primarily because these materials generally exhibit superior properties including high strength and, if there is reasonable microstructural stability at elevated temperatures, they provide an opportunity to achieve a superplastic forming capability. Several different procedures are now available for processing UFG metals through the application of severe plastic deformation (SPD) [4–6] but generally most UFG materials are produced using either equal-channel angular pressing (ECAP) [7] or high-pressure torsion (HPT) [8]. In ECAP the sample is in the form of a rod or bar and it is pressed through a die constrained within a channel having a sharp angle near the center of the die whereas in HPT the sample is usually in the form of a thin disk and it is subjected to a high applied pressure and concurrent torsional straining. Although both procedures have been used to produce UFG microstructures in a wide range of metals, the experimental evidence shows that, by comparison with ECAP, processing by HPT produces both small grains and a higher fraction of grain boundaries having high angles of misorientation [9–11].

In practice, the processing of a disk by HPT introduces a potential difficulty in data analysis because the equivalent von Mises strain, ε_{eq}, is given by the relationship [12]

$$\varepsilon_{eq} = \frac{2\pi Nr}{h\sqrt{3}} \quad (1)$$

where N is the total number of turns, r is the radial distance from the center of the disk and h is the height (or thickness) of the disk. It follows from Eq. (1) that the strain varies across the disk from a maximum value at the edge to a strain of zero at the central point where $r=0$. This suggests that the microstructures achieved after HPT will exhibit considerable inhomogeneities but in practice early experiments on pure Ni, conducted using a combination of microhardness measurements, transmission electron microscopy (TEM) and orientation imaging microscopy (OIM), showed that good homogeneity and an equiaxed microstructure was attained

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after processing through 5 turns under an applied pressure of 6.0 GPa [13]. It has been demonstrated theoretically, using strain gradient plasticity modeling, that there is a gradual evolution in the hardness values in HPT processing with increasing torsional straining and ultimately these hardness values achieve an essentially uniform distribution [14].

Early measurements in HPT processing showed that the hardness values increase rapidly around the edges of the disks in the early stages of processing and in the central region the evolution in hardness occurs more slowly although ultimately, after a sufficient number of turns, it is generally possible to achieve similar values of hardness throughout the disks [13,15,16]. This trend, in which the hardness increases to a plateau value, is termed hardening without recovery and it represents the general trend for almost all metals: a detailed description of this type of hardening was given in a recent review [17].

By contrast to this general trend, a comprehensive set of experiments conducted on high purity (99.99%) aluminum showed that the hardness was initially higher, rather than lower, in the central region of the disk but these hardness values decreased with further straining and again there was a gradual evolution towards a plateau hardness [18]. These higher initial hardness values in the centers of the disks matched the smaller grains observed in this region by TEM and the development of larger grains at the disk peripheries was attributed to the occurrence of rapid recovery due to the high stacking fault energy (SFE) in pure Al and the consequent rapid recovery through processes such as cross-slip. The development of a high initial hardness at very low strains and the subsequent decrease in hardness to a lower plateau value is designated softening with rapid recovery [17] and later it was fully documented in experiments on high purity Al where the HPT processing was performed through fractional numbers of revolutions between 1/8 and 1 turn [19,20] or hardness values were recorded on different sectional planes throughout the disks [21]. The occurrence of softening with rapid recovery appears to be almost unique to high purity aluminum because aluminum of lower purity (99.7%) [22] and numerous Al-based alloys [23–33] exhibit conventional hardening without recovery. Nevertheless, there are two recent reports of softening with rapid recovery in the h.c.p. metals of pure (99.9%) Mg [34] and pure (99.99%) Zn [35]. It is important to note that softening with rapid recovery is different from weakening in HPT [17] where all hardness measurements after HPT processing are lower than in the original unprocessed material [36–39].

Numerous reports are now available showing conventional hardening without recovery in pure Cu [15,40–47] although there are two investigations showing the occurrence of softening when post-HPT annealing is conducted at elevated temperatures [35,48]. These results suggest, therefore, that the lower stacking fault energy of Cu prevents the occurrence of softening with rapid recovery as observed in high-purity Al. Nevertheless, a recent report described the use of oxygen free high conductivity (OFHC) Cu in experiments conducted at room temperature in repetitive upsetting-extrusion (RUE) where the material was subjected to repeated upsetting and extrusion [49]. In these experiments, the grain size of the Cu was refined from 150 μm to <2 μm and the microhardness was found to initially increase, reach a peak value and then decrease at low plastic strains to a saturation plateau. This behavior is similar to the softening with rapid recovery observed in the HPT processing of high purity Al and it suggests the same behavior may be observed in OFHC Cu if the HPT processing is conducted at low strains using fractional numbers of HPT turns.

Accordingly, the present investigation was initiated to examine the microstructural evolution in OFHC Cu processed by HPT using optical microscopy (OM), electron backscattered diffraction (EBSD) and the development of microhardness when processing by HPT at ambient temperature to different strain levels. The experiments used a very high purity Cu to facilitate the potential for dynamic recrystallization as reported in an earlier report on processing in the early stages of HPT straining [50]. Also, the use of a high purity material will provide information on the microstructures and microhardness that may be compared with other reports on Cu of lower purity.

2. Experimental material and procedures

The experiments were conducted with an OFHC copper of 99.99+wt% purity as used in an earlier study [50]: the chemical composition is given in Table 1. The material was received as a rod of 10 mm diameter and it was initially annealed for one hour at 673 K and then cut into disks with thicknesses of ~1.0 mm and polished on both sides using abrasive papers to final thicknesses of ~0.8 mm. The processing by HPT was conducted at ambient temperature under quasi-constrained conditions in which there is some limited outflow of material around the periphery of the disk between the two anvils [51]. All processing was conducted using an imposed pressure of 6.0 GPa and a rotation speed for the lower anvil of 1 rpm. The disks were processed through different numbers, N, of 1/4, 1/2, 1, 5 and 10 turns.

The HPT disks were prepared for microstructural examination with mirror-like surfaces using standard metallographic techniques. A Zeiss Axio-Imager optical microscope was used for the OM examinations and the samples were chemically etched at ambient temperature with a mixture of 50 ml of distilled water and 50 ml of nitric acid. Further microstructural evaluation was made using EBSD and this required electro-polishing in a solution of 25% phosphoric acid, 20% ethanol and 10% propanol in water. The grain size and grain boundary structures were analyzed using an HKL Channel-5 EBSD detector interfaced to a JOEL F7001 field-emission scanning electron microscope (SEM) operating at 20 kV. The EBSD data acquisition was made using Flamenco software. All EBSD measurements were performed on the disk plane at three different locations: the center, the mid-radius and a near-edge position. To incorporate microstructure evolution at different length scales, the EBSD maps were constructed using Tango software with step sizes in the range of 0.07–1.0 μm. A minimum misorientation angle of 1° was used to quantify the grain size and grain boundary characteristics and all twins were included in the measurements. The misorientation angle distribution statistics were analyzed by employing a critical misorientation angle of 15° to differentiate between low-angle boundaries (LABs) and high-angle boundaries (HABs). The grain boundaries are presented in the EBSD maps with the LABs shown as thin gray lines, the HABs as solid black lines

Table. 1
Chemical composition of OFHC copper (wt%).

Sample/element	Ag	As	Bi	Cd	Fe	Mn	I	O	P	Pb	S	Sb	Se	Sn	Te	Zn	Cu
OFHC Cu	0.0025	0.0005	0.0001	0.0001	0.001	0.00005	0.001	0.0005	0.0003	0.0005	0.0015	0.0004	0.0003	0.0002	0.0002	0.0001	99.99*

* Minimum guaranteed percentage of Cu.