Original Article

Electron backscatter diffraction (EBSD) microstructure evolution in HPT copper annealed at a low temperature

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Detailed EBSD analysis was performed on copper specimens processed by high-pressure torsion at $P = 6$ GPa for one whole turn and subsequently annealed at a temperature of 100 °C for 15, 30 and 60 min. The basic microstructural parameters (mean grain size, GB statistics, microtexture) were evaluated in the mid-radius areas of the HPT disks. Microhardness of all samples was measured across the two diameters and interlinked to the microstructures observed. Small but noticeable changes of microhardness in HPT copper after annealing were detected. The changes were interlinked to microstructural parameters acquired by EBSD. The relationships obtained are discussed in terms of the microstructure and microtexture evolution during low temperature annealing.

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1. Introduction

The processing of ultrafine-grained (UFG) and nanostructured metallic materials using severe plastic deformation (SPD) \cite{1} is a new and promising method of enhancing the properties of metals and alloys for advanced structural and functional applications \cite{2,3}. Traditionally, there have been two main techniques for producing UFG materials using either equal-channel angular pressing (ECAP) \cite{4} or high-pressure torsion (HPT) \cite{5,6} but other techniques are now available such as accumulative roll bonding (ARB) \cite{7}, multiaxial forging \cite{8}, twist extrusion \cite{9}, plain strain machining (PSM) \cite{10} and others \cite{11}. SPD processing is attractive because the straining is practically unlimited due to the unchanging sample geometry and shape. However, there is a general tendency for a saturation in grain refinement for high melting temperature metals \cite{12} and recovery (and even recrystallization) for low melting materials \cite{13} processed by continuing SPD methods.

There is a considerable interest in creating UFG structure in pure copper and its alloys in order to get the optimum combination of low wear rate and high conductivity. However, pure copper has some distinct drawbacks, such as low strength and low thermostability, which tends to restrict its...
There are numerous reports on the application of SPD for grain refinement of pure copper by ECAP and HPT where the thermostability of ultrafine-grained copper was analyzed. Probably the first comprehensive reports on copper were published in 1999 for ECAP [14] and 2000 for HPT [15]. In the latter report, the microhardness of an HPT disk of 98.5% purity Cu (P = 5 GPa, N = 5 turns) drops down at an annealing temperature of about 180–200 °C. From DSC data with a heating rate of 40 °C/min, the most pronounced peak was detected for the HPT sample strained for N = 1 turn at 220 °C, which corresponds to the microhardness measurements [15]. A similar temperature (~190 °C) of recovery was also reported for higher purity HPT copper (99.9%). High purity copper (99.99%) [16] shows similar behavior when annealing at 134, 269 and 405 °C (0.3, 0.4 and 0.5 of melting point) for 1 h. The microhardness drops down from 130 to 80 HV as the annealing temperature increases from 134 to 269 °C.

There are some reports on low thermostability of UFG copper at room temperature in which recovery processes and grain growth have been detected in a period of one month and one year [17]. Other reports [18] show no significant grain refinement in copper subjected to rolling at liquid nitrogen temperature suggesting that recovery processes take place during rolling.

In practice, the simplest way to achieve UFG copper is by HPT, which permits the processing of disks suitable for experiments on highly strained materials. In an earlier report [19], it was demonstrated that there is a slight increase of Vickers microhardness in HPT copper subjected to low-temperature annealing. Careful monitoring of the microhardness and microstructure in HPT aluminum and copper stored at room temperature for long periods of time [20] has not revealed any significant changes of these parameters. Thus, the present report was undertaken to study microstructure evolution of HPT copper specimens subjected to annealing at 100 °C for 15, 30 and 60 min.

Disks of copper were used as the starting material. The material was purchased from Goodfellow Cambridge Ltd., Huntingdon, UK, and the typical chemical composition was given as (in ppm): Ag 500, Bi < 10, Pb < 50, O 400, other metals <300. The HPT specimens were in the form of disks having diameters of 10 mm and thicknesses of about 1 mm. These disks were processed at room temperature by HPT for a total of N = 1 turn under an applied pressure of P = 6 GPa.

The processing was conducted under quasi-constrained conditions where there is a small outflow of material around the periphery of each disk during processing. Parts of the processed specimens were annealed at 100 °C for a period of 15, 30 or 60 min. During annealing, the HPT disks were placed on the flat surface of a thermocouple and hence the temperature was stable to within ±2 °C. Prior to all measurements, the specimens were mechanically polished on 1000 grit SiC paper followed by a final polish using a diamond suspension containing monocrystalline diamond with a size of 3 μm with subsequent electro-chemical polishing at room temperature using an electrolyte of HNO₃:CH₃OH = 1:3 with a voltage of 10 V.

These samples were employed for microhardness measurements and electron backscatter diffraction (EBSD) analysis.

The microhardness was measured with steps of 0.5 mm along the diameters of the disks using an FM-300 tester equipped with a Vickers indenter using a load of 100 gf and a dwelling time of 15 s.

The EBSD analysis was performed using a TESCAN MIRA 3LMH FEI scanning electron microscope equipped with an EBSD analyzer “CHANNEL 5”, and a rectangular grid with scan step of 50 nm was used. The EBSD analysis was performed for regions located near the center of the disk, near the midradius (2.5 mm from the center) and near the edge (~4.5 mm from the center.) The acquired data were subjected to standard clean-up procedures involving a grain tolerance angle of 5 ° and

Fig. 1 – Microstructure (a) and microrotexture (b) of Cu specimen prior to HPT.
3. Experimental results

Fig. 1 presents (a) an initial microstructure and (b) the micro-texture of a copper specimen prior to high-pressure torsion. The average grain size by EBSD was larger than 25 µm with well-defined grain boundaries and twins in the interior of grains. The pole figures in Fig. 1(b) reflect the inherent texture of extruded rods of the primary material.

Fig. 2 shows the inverse pole figure map and grain boundary misorientation distribution of HPT copper processed under a load of \( P = 6 \) GPa for one whole revolution where the EBSD was taken at the mid-radius of the disk. Significant grain refinement with grains elongated along the torsional direction is observed. The mean grain size by EBSD was about 200–300 nm which is a typical grain size for HPT copper. The fraction of low-angle boundaries was below 10%, which is consistent with earlier reports [3]. Fig. 3 represents the integrated Vickers microhardness taken at a distance of \( r/2 \), where \( r \) is the mid-radius of the HPT disk. A slight increase in Hv is observed for HPT copper annealed for 15 min. Although it is in the range of the experimental error, the gain in microhardness is about 4% and it was systematically detected.

An X-ray analysis of HPT-processed copper in Fig. 4 gives a size of coherent domains of 287.5 ± 6.3 nm, which is in good agreement with the EBSD data. Fig. 5 shows a Taylor factor map.
Fig. 5 – Taylor factor map of HPT copper: (a) initial, and after annealing at $T=100^\circ$C for (b) 15 min, (c) 30 min and (d) 60 min.

Fig. 6 – Kernel average misorientation of HPT copper: (a) initial and after annealing at $T=100^\circ$C for (b) 15 min, (c) 30 min and (d) 60 min.
for HPT copper after processing in Fig. 5(a) and after annealing at 100 °C for 15 min in Fig. 5(b), 30 min in Fig. 5(c) and 60 min in Fig. 5(d). The average value of the Taylor factor (TF) decreases slightly from 3.318 for HPT copper to 3.295 for the HPT disk annealed for 15 min. For the sample annealed for 30 min, the TF increases to a value of 3.359 and then it decreases again to 3.317 for the specimen annealed for 60 min. These variations of the Taylor factors are not in agreement with changes in the Vickers microhardness. The Kernel average misorientation (KAM) maps for copper specimens are shown in Fig. 6. The average KAM also increases for the specimens annealed for 15 and 30 min and then slightly decreases for the HPT copper annealed for 60 min.

Table 1 provides a comprehensive summary of all experimental data obtained in this investigation. The area weighted grain size obtained from EBSD shows a gradual increase with annealing time. Also, there is a noticeable increase in the fraction of low-angle boundaries (LAB) for the specimen annealed for 15 min. X-ray analysis revealed a small decrease in the coherent domain size from 193.8 nm (HPT copper) to 185 nm (after annealing for 15 min) and a gradual increase in the microstrain level.

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### 4. Microstructure evolution during low temperature annealing

In pure metals, there are a limited number of factors influencing hardening: the Hall–Petch relationship and the dislocation hardening and texture (corresponding to a change in the Taylor factor). The texture can change during significant changing of the microstructure as in recrystallization and grain growth. During low temperature, annealing at 100 °C it is difficult to expect significant changes in texture and the average values of the Taylor factor for all specimens clearly support this. On the basis of the experimental parameters, it is possible to evaluate the dislocation density either using the average KAM \( \rho = \theta/b \), where \( \theta \) is the average angle in radians, \( b \) is the Burgers vector and \( h \) is the step size of 50 nm in EBSD, or using the microstrain level measured by X-rays \( \rho = 2\sqrt{3}\varepsilon^2/1/2/(b \cdot d) \), where \( \varepsilon^2/1/2 \) is the microstrain and \( d \) is the coherent domain size. The two final columns in Table 1 represent the dislocation densities calculated on the basis of X-ray analysis and EBSD experiments, respectively. The dislocation density calculated from KAM corresponds to the geometrically necessary dislocations (GND) and it is about 3 times higher than the density of the statistically stored dislocations (SSD) estimated from X-ray analysis. Both values show a slight increase in the Cu specimen annealed for 15 min.

### 5. Summary

Integrated microhardness measurements at the mid-radius position show a systematic increase for the HPT copper subjected to low temperature annealing at 100 °C for 15 min. The most correlated parameters that may be responsible for this change are the fraction of low-angle grain boundaries and the size of the coherent domains.

### Conflict of interest

The authors declare no conflicts of interest.

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### References


