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Severely deformed Cu by using compression with oscillatory torsion method

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Materials

ABSTRACT

Purpose: The present study is aimed at a quantitative description of microstructural parameters as a misorientation angle of Cu deformed by using compression with oscillatory torsion.

Design/methodology/approach: Cu samples were deformed at torsion frequency (f) changed from 0 Hz (compression only) to 1.8 Hz under a constant torsion angle (α) ≈ 8° and compression speed (v)=0.1mm/s. Structural investigations were conducted by using transmission electron microscopy (TEM). Local misorientation have been measured by the Kikuchy pattern technique.

Findings: The results of the investigations have shown that formation of ultrafine structure is seen after deformation at 0.4 Hz, where cells show broad boundaries having a diffuse dislocation arrangement, which transform to high-angle grain boundaries at higher strains (higher value of f). The subgrain interiors also contain dislocation arranged in an irregular shape. For this reason the effects must be explained by movements of dislocation, where dislocation are continuously built up to new boundaries as a deformation increase.

Research limitations/implications: Reported research ought to be completed with EBSD (Electron Backsscattering Difraction) technique.

Originality/value: Grain boundary misorientation is important for describing the microstructure of severe plastic deformation process and may be useful in explanation of ultrafine grains formation.

Keywords: Nanomaterials; Cu; Microstructure; Severe plastic deformation

1. Introduction

One way to develop ultrafine microstructure is through severe plastic deformation (SPD). A description of SPD method using equal-channel angular extrusion (ECAE) and hydrostatic extrusion (HE), have been described in details by others [1-8]. Nano and ultrafine structures obtained by different methods differ from one another in many aspect: the degree of structure refinement, structure homogeneity, and the physical and mechanical properties. However there are no studies on compression with oscillatory compression methods. Compression with oscillatory torsion is a deformation procedure applied to achieve large strains [9] and therefore it should result in nano and ultrafine grains formation [10]. The way of nano and ultrafine grains formation is still unclear. Some recent publications indicate that the formation of nanograins may proceed via mutual crossing of microbands which transform to new grains with large misorientation grain boundaries [11,12]. Another mechanism of refining grains is described in [13,14], where submicron grains are formed via reorganizing the dislocations within the elongated tangled dislocation cells. The present study is aimed at a quantitative description of microstructural parameters as a mean misorientation angle of microareas. Grain boundary misorientation is important parameter for describing the microstructure of SPD processed materials and may be useful in explanation of ultrafine grains formation. For hence microstructure investigations have been carried out by transmission electron microscopy, and local misorientations between adjacent subgrains have been measured by the Kikuchi pattern technique.

2. Experimental details

The grained microstructure of Cu with an average grain size of D=36 μm was deformed at room temperature by using compression with oscillatory torsion. Before deformation the bars of diameters 10 mm and length 15 mm respectively, were thermal treated for two hours at 773 K. The samples were deformed at torsion frequency (f) changed under a constant torsion angle (α) $\approx 8^{o}$ and compression speed (v)=0.1mm/s. A series of stress-strain curves of Cu tested in our experiment are shown in Fig. 1. All samples were deformed to reach a real reduction (ε_h)= 0.7. he details of the deformation procedure have been described elsewhere. Fig. 1 indicates that the compression force (p_{sr}) needed to reach ε_h = 0.7 rapidly decrease with the increase of (f). The mean value of microhardness ($HV_{0,2}$) after deformation at 0 Hz was 120 $HV_{0,2}$ and after processing at 0.4 Hz and 1.8 Hz The mean value of microhardness were 105 $HV_{0,2}$ and 96 $HV_{0,2}$ respectively.

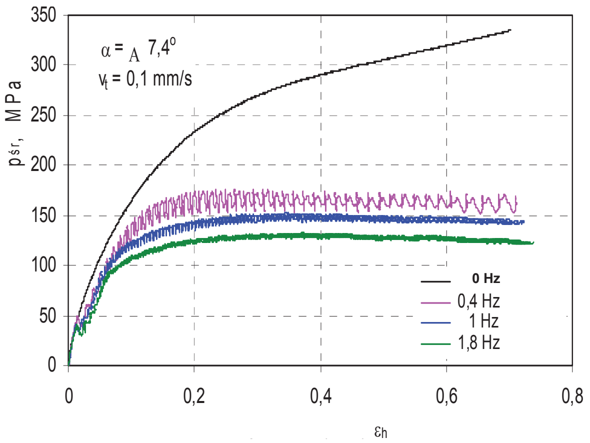

Fig. 1. Dependence of $p_{sr} = f(\varepsilon_h)$ for the investigated Cu [9]

TEM observations were carried out using JEOL 100B operating at 100 kV. Studies using TEM were conducted on samples extracted from the 0.8 radius on the longitudinal and transverse plane section. Kikuchi patterns were obtained to measure the misorientation angle between adjacent grains. The number of analyzed grains was about 40.

3. Results of researches

The investigation of dislocation microstructures formation after change of the deformation mechanism have been analyzed in detail in previous studies [15], which led to identification of three types of microstructure. A characteristic feature of type 1 microstructure is the extended planar dislocation boundaries, which are seen as straight traces. Type 2 microstructure is a three-dimensional cylindrical cell/subgrain structure. Localization of plastic deformation in the shear bands (type 3 of microstructure) was observed in the compressed with oscillatory torsion samples too.

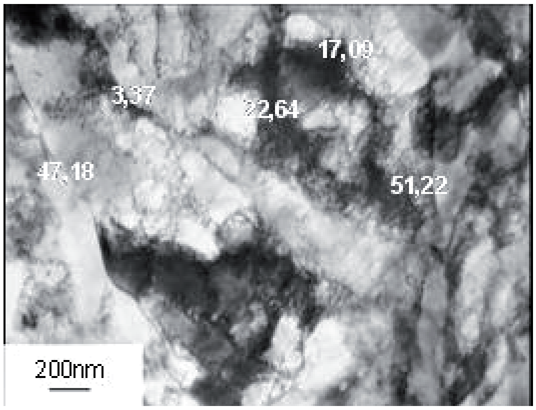

Fig. 2. Development of cells/subgrains structure in Cu deformed at 0.4 Hz. Dislocation cell structure with small and higher value of misorientation. On the left side the grain boundary indicating mobility

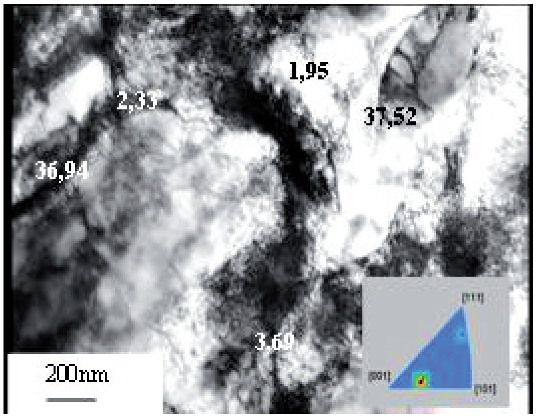

Fig. 3. Development of cells/subgrains structure in Cu deformed at 0.4 Hz. A high density of dislocations with a non-uniform distribution and various value of misorientation

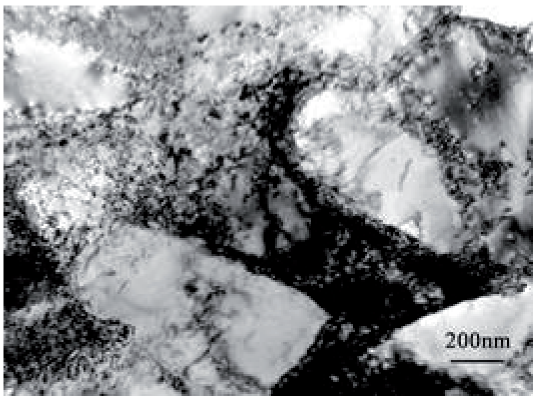

Fig. 4. Development of grains structure in Cu deformed at 1.8 Hz

TEM investigation of shear bands showed that:

- density of microbands increases with the growth of torsion frequency (f). For samples deformed at (f)= 1.6 Hz in some regions strain localization microbands about 0.2 to 8.5 μm thick occur,
- the shear bands contain a more inhomogeneous structure. It is characterized by elongated finer and coarser cells misoriented by more than 15° (the mean misorientation angle was 27.3°), poorly defined boundaries and recovered microareas [15].

The cell/subgrain structures are dominant type of deformed Cu by using compression with oscillatory torsion test. The grains size of these microstructures were studied qualitatively by image analysis and these results will be presented elsewhere. Must be noted that microstructure can be regarded rather as a ultrafine-grained where the crystallite size is between 100 and 1000 nm.

The TEM micrographs (Fig. 2,3) presents a deformed cell/subgrain microstructure at f =0.4 Hz. The Kikuchi patterns that were taken from different areas demonstrated that the cells in many cases are separated by low angle boundary. However, high angle boundaries were found as well. These micrographs demonstrate that there is change of the microstructure from a dislocation cell structure to a subgrain-like structure because misorientation increase between adjacent subgrains.

The transformation from a cell structure to a subgrain can be interpreted in such a way that the dislocations formed in the grain interiors arranged into cell boundaries to minimize their strain-energy. These microstructures have low-angle grain boundary character as they separate cells with small misorientations.

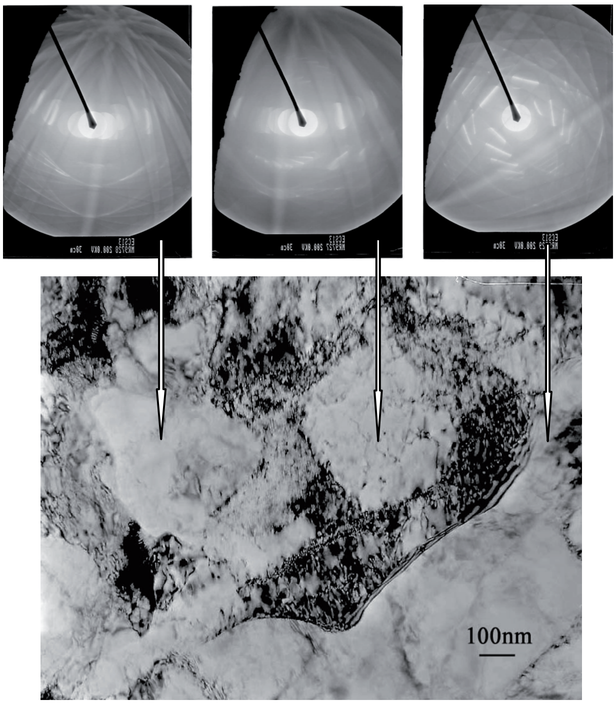

Fig. 5. Microstructure and Kikuchi patterns of Cu after deformation at 1.8 Hz. Kikuchi pattern taken from the area arrows marked

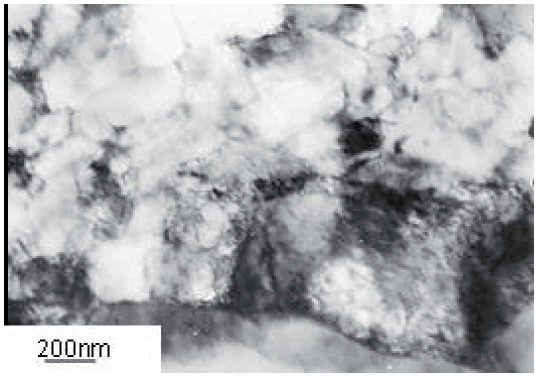

Fig. 6. Development of grains structure in Cu deformed at 1.8 Hz. Recrystalized areas in upper part of the figure

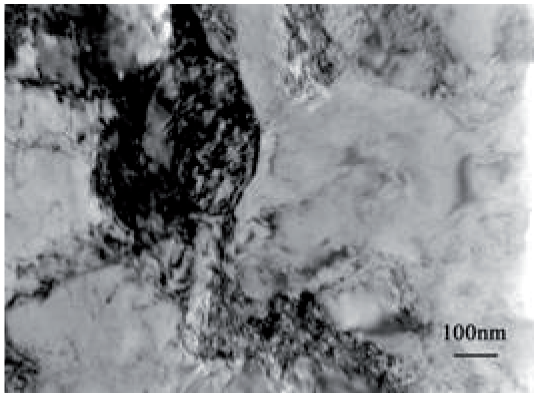

Fig. 7. Development of grains structure in Cu deformed at 1.8Hz

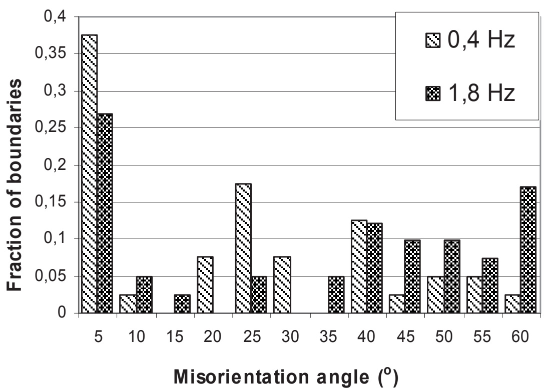

Fig. 8. Misorientation angles distributions developed in Cu after deformation at 0.4 Hz and 1.8 Hz

As the deformation proceeds the dislocation density in the cell boundaries increases, the thickness of the boundaries decreases and the misorientation between the neighboring cells also increases, i.e. the cell boundaries are transformed into high angle grain boundaries. It is well visible in Fig. 2 and Fig. 3.

At a certain strain the microstructure contains low-angle cell boundaries (incidental dislocation boundaries IDBs) and high angle grain boundaries (geometrically necessary boundaries, GNBs) simultaneously. The suitable example of GNBs is double dislocation sheet, DDW. The DDW structure becomes more dominant at larger strains, as shown in Fig. 4 and Fig. 5. These microstructures evolve into high angle grain boundaries.

Well defined subgrain structures delineated by more sharp boundaries with high value of misorientation are visible for deformation at f=1.8 Hz (Fig. 6).

The dislocation density after processing at f= 1.8 Hz both at grain boundaries and within grains are sufficiently high to lead to dislocation rearrangement into a cell-like structure, which may be responsible for further grain refinement (Fig. 6,7).

The misorientation distribution of strain-induced boundaries developed for f=0.4 Hz and f= 1.8 Hz is show in Fig.8. The distribution shows a bimodal shape especially visible for f =1.8 Hz and most boundaries have misorientation of more than 15^{o}, although a big fraction of low angle boundaries is detected too.

The mean misorientation angle after deformation at 0.4 Hz was 20.6^{o}, and after deformation at 1.8 Hz was 30.9^{o} It is visible that the fraction of high value of misorientation gradually increase as the strain increases from 0.4 Hz to 1.8Hz. This leads directly to the evolution of a new fine-grained structure with broad range of misorientation from small to high angle grain boundaries.

4. Conclusions

The formation of sub-grains structure is seen after deformation at 0.4 Hz, where cells show broad boundaries having a diffuse dislocation arrangement, which transform to high –angle grain boundaries at higher strains (higher value of f). The sub-grain interiors also contain dislocation arranged in an irregular shape. For this reason the effects must be explained by movements of dislocation (rearrangements of dislocations), where dislocation are continuously built up to new boundaries as a deformation increase.

Acknowledgements

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