Journal of Achievements in Materials and Manufacturing Engineering
VOLUME 20
ISSUES 1-2
January-February
2007

Ultrafine grained Cu processed by compression with oscillatory torsion

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Received 24.10.2006; accepted in revised form 15.11.2006

Manufacturing and processing

ABSTRACT

Purpose: The aim of this work is a study of Cu microstructure after severe plastic deformation process by using compression with oscillatory torsion test.
Design/methodology/approach: Cu samples were deformed at torsion frequency (f) changed from 0 Hz (compression) to 1.8 Hz under a constant torsion angle (α) ≈8° and compression speed (v)=0.1mm/s. Structural investigations were conducted by using light microscopy (LM) and transmission electron microscopy (TEM).
Findings: The structural analysis made by TEM shows that the process lead to grain refinement by the reconstruction of the banded (laminar) structure, typical for conventional deformation (compression), into a subgrain structure. Deformation at lower (f) and higher (v) were the most effective in refining grains, while using higher (f) and lower (v) may be characterized as a recovered and coarsened structure. This method facilitates obtaining a submicrocrystalline structure with nanocrystalline elements localised in the shear bands' region.
Research limitations/implications: The understanding in refinement of Cu structure could help to modify the process and design deformation parameters.
Practical implications: The knowledge of the characteristic features of unconventionally deformed materials will provide the usefulness of the employed method to produce materials having the desirable functional properties.
Keywords: Nanomaterials; Cu; Transmission electron microscopy; Severe plastic deformation

1. Introduction

The interest in metallic materials in which the size of grain is described in ultra- and manometric scales, results from the assumption that size reduction of grain is conducive to obtaining a material of high strength and good plasticity [1-5]. The technological aspect of submicrocrystalline materials production frequently comes down to employing the same techniques as those used in nanomaterials procesing. The SPD (Severe Plastic Deformation) methods allow to obtain submicron or nano-size grain structure after imposing high levels of strains. A serious limitation is, however, the difficulty in obtaining materials with a significant fraction of the nanocrystalline structure. The microstructure of the materials produced via SPD methods strongly depends on the applied technological parameters, method and kind of used materials. The studies performed on materials deformed by using hydrostatic extrusion (HE) indicate that grain size in pure aluminium and copper has been reduced to below 1μm, whereas in aluminium alloy and titanium, grain refinement to below 100 nm has been achieved [6]. A. P. Zhilyaev et al. [7] examined the grain size in pure nickel after subjecting to deformation by high pressure torsion (HPT) and equal channel angular extrusion (ECAE) methods. Both procedures lead to large refinement of microstructure: the average grain size after HPT was 0.17 μm but after ECAE about 0.35 μm. At the Faculty of Material Engineering and Metallurgy, Silesian University of Technology, the Institute of Modelling Processes and Medical Engineering is conducting research under Professor F. Grosman's management, on the new SPD method – the method of compression with oscillatory torsion. This paper presents selected results of the substructure study concerning on the influence of unconventional mode of deformation on structural processes in copper. The knowledge of characteristic features of unconventionally deformed materials will prove the usefulness of the employed method to produce materials having the desirable functional properties, in accordance with the conception of plastic deformation based on structure forming by force of variable load scheme. The effect of strain path on the plastic properties and structure of materials is less known because an investigations are more difficult and complex [8-11].

2. Experimental details

In this work Cu (M1E grade) was used. The copper samples were annealed at 500°C for 2h to give a grain size about 36 μm. The samples were deformed at room temperature by using compression with oscillatory torsion test. The details of the deformation procedure have been described elsewhere [12,13]. The samples were plastically deformed by apply following parameters: α [°] - torsion angle, f [Hz] - torsion frequency, v [mm/s]- speed compression and ε_h - real reduction.

The samples for structural examination were ground, mechanically polished and finally etched in a reagent containing 30 ml CH_3COOH, 20 ml HNO_3, 30 ml C_2H_6CO. For the microstructure observation, a Olympus GX+70 metallographic microscope was used.

Disks for thin foils were thinned by electropolishing in a reagent containing 600 ml CH_3OH, 340 ml C_4H_9OH i 60 ml 60 % $HClO_4$ Thin foils were examined in a JEOL 100B electron microscope at an operating voltage of 100kV. Studies using TEM and LM were conducted on material extracted from the 0.8 radius.

3. Results of researches

Optical microscopy investigations of Cu revealers well defined slip bands and clusters of slip line after two mode of deformation (Fig. 1,2). Multi system slip is well visible for sample deformed by compression with oscillatory torsion. The dislocation structure developed under compression at ε_h=0.7 demonstrates a banded structure (Fig. 3). An average width of dislocation bands is 0.29 μm [14]. A change of the deformation mode (compression with oscillatory torsion) cause the formation of subgrain structure (Fig. 4). The quantitative measurements of banded structure described in paper [14] demonstrate an increase of dislocation bands width in samples compressed with oscillatory torsion compared to measurements for samples compressed only. The highest values of dislocation bands width were recorded for samples deformed at high torsion frequency value (f) and low compression speed value (v). For instance, the average width of dislocation bands for the deformation parameters of 0.4Hz and 0.6 v, was 0.41μm and for 1.6 Hz and 0.1 v was 1.17μm. The sample deformed by oscillatory compression at 0.4 Hz and 0.1 v exhibit a more extended distribution of dislocation bands width compared to sample compressed (Fig. 5). The obtained results indicate that change of deformation path stabilises the subgrain structure.

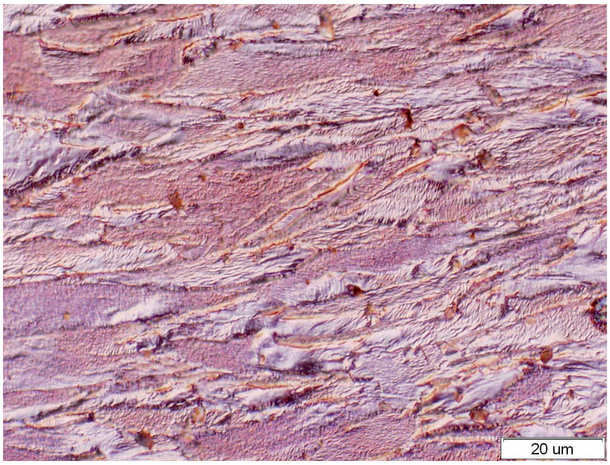

Fig. 1. Slip bands in Cu after compression at ε_h=0.7

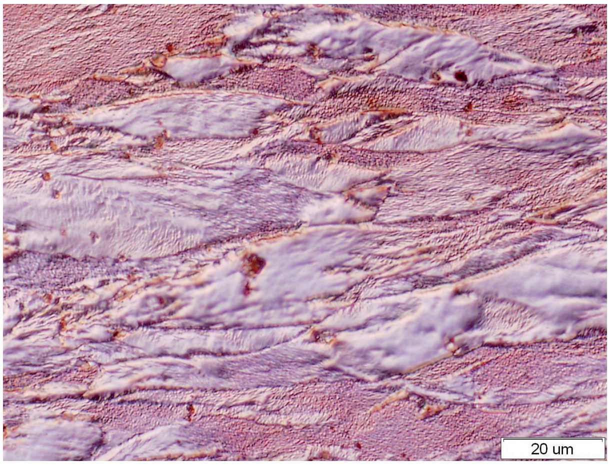

Fig. 2. Microstructure of slip bands in Cu after deformation at Hz= 1.8, $\alpha = 8$, v=0.15, $\varepsilon_h = 0.7$

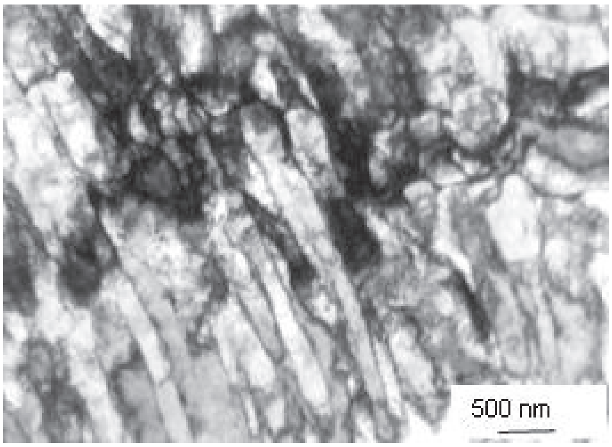

Fig. 3. Substructure of Cu after compression at ε_h=0.7

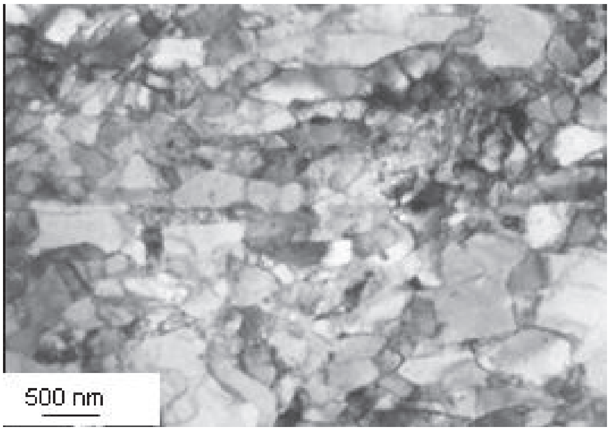

Fig. 4. Substructure of Cu after deformation at Hz= 1.8, $\alpha = 8$, v=0.15, $\varepsilon_h = 0.7$

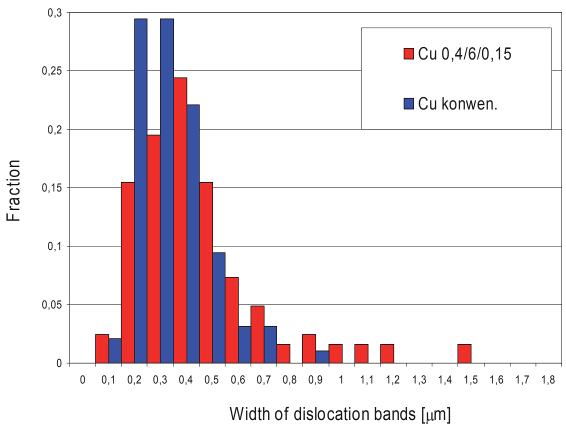

Fig. 5.Width of dislocation bands distributions developed in Cu after deformation at 0.4 Hz, $\alpha = 8$, v=0.15,ε_h = 0.7 and after conventional deformation (compression)

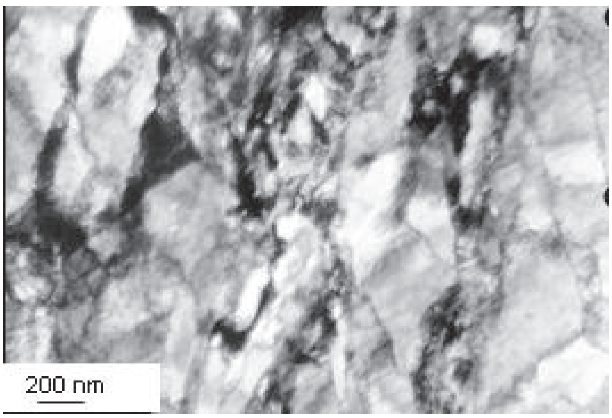

Fig. 6. Substructure of Cu after deformation at Hz= 1.8, $\alpha = 8$, v=0.15, ε_h = 0.7. Different subgrain size in banded and matrix structure

The excellence of the subgrain structure (equiaxial subgrains, with distinct boundaries) depends of the process parameters. In the case of samples deformed at low values of (f), a cellular dislocation structure is observed. An increase of the value of (f) favours the creation of a distinct subgrain structure with low dislocation density inside the subgrain. The relaxation processes observed during deformation at high value of (f) contribute however to a growth of the subgrain size (Table 1).

Table 1.
Dislocation cell/ subgrain diameter (μm) depending on apply parameters

$\varepsilon_h = 0.6 / \alpha = 8$ / v=0.15		
f=0.4	f=1	f=1.8
0.31	0.35	0.40

The process of compression with oscillatory torsion ensures to obtain a structure with different grain sizes (Fig.6) as a result of shear bands generation. Inside the shear bands the crystallites achieve relatively small sizes of ca. (50-350 nm) against the remaining matrix, which guarantees the submicrograin structure. The bimodal nature of formed structures is particularly well visible for samples deformed at high values of (f), where the subgrain attains a relatively big size (Table 1) and the frequency of banded structures (shear bands) increases [15]. At the same time, the thermally activated processes which take place in the matrix are also pass onto the shear bands, thus causing of crystallites size increase, as proven in [15]. Fine, most frequently elongated crystallites, which are formed inside shear bands, are characterised by a misorientation value exceeding 15°. Detailed studies of misorientation values between individual areas of crystallites will be presented elsewhere. At the same time, the matrix is characterised by low- and high-angle boundaries, where with an increase of the parameter (f), the frequency of high-angle boundaries increases.

Deformation process which accumulate considerable structural defects cause the partial recrystalization (Fig.7). Moreover, the process of adiabatic material heating during the deformation, favors the recrystalization phenomenon.

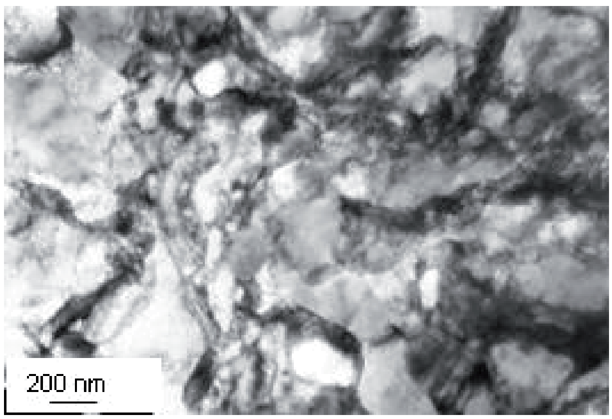

Fig. 7. Substructure of Cu after deformation at Hz= 0.4, $\alpha = 8$, v=0.15, $\varepsilon_h = 0.7$

Should be noted that the recrystalization which eliminates the accumulated deformation contribute to the retardation of grain size reduction, by which the effect of reduction in grain size of Cu is much slower when compared to other materials, e.g. aluminium alloys or ferritic steels.

4. Conclusions

Based on the conducted research it has been proven that:
A change of deformation path one's attention to the way of substructure forming. The process lead to grain refinement by the reconstruction of the banded (laminar) structure, typical for conventional deformation, into a subgrain structure, which is characterised by a misorientation value about 15^o. The method of oscillatory compression facilitates obtaining a submicrocrystalline structure with nanocrystalline elements localised in the shear bands' region.

Acknowledgements

This work was supported by the Polish Ministry of Science and Information Technology (project no. 7 T08A 059 28)

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