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# Microstructure and properties of dynamically compressed copper Cu99.99

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**Properties**

## ABSTRACT

**Purpose:** The main object of this study is to establish the influence of dynamic compression on the possibility of microstructure refinement in polycrystalline copper Cu99.99.

**Design/methodology/approach:** Polycrystalline copper Cu99.99 was dynamically compressed on a falling - weight - type impact machine with strain rate ranging from 1.75×102 to 2.7×102 $s^{-1}$. After deformation, the samples were tested for microhardness and their microstructure was examined by means both optical and electron microscopy. Additionally, the width of microbands observed in the microstructure was statistically evaluate by using mean chord method. The misorientation of selected microstructural elements was determined using proprietary KILIN software.

**Findings:** It was found that to produce materials with nanometric features is not only possible by exertion of severe plastic deformation methods (SPD) but also by deformation with moderate strains and high strain rates. The demonstrated data show, that in some range the amount of deformation and strain rate can be interchangeable parameters causing similar structural effects.n.

**Practical implications:** The results may be utilized for determination of a relation between microstructure and properties of the copper in the process of dynamic compression.

**Originality/value:** The results contribute to evaluation properties of the polycrystalline copper in the light of achievement of fine – grained microstructure. The obtained results indicated that dynamic compression with high strain rate can be an effective method for microstructure refinement, comparable with SPD methods

**Keywords:** Nanostructure; Dynamic compression; Electron microscopy; Metallography; SPD methods

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## 1. Introduction

Polycrystalline copper is one of the most widely studied materials under the variety of deformation path and over large strains, strain rates and temperatures. Because of their high thermal and electrical conductivity copper and its alloys have found a lot of applications, such as high voltage switches, wires, components of many of the elements in automotive industry, electrical machines, especially electromagnetic motors, generators and transformers, electrical relays, busbars and others [1,2].

The required properties of the materials depend among others on the production method. One of the groups of the methods which allow to obtain material with high strength properties and ultrafine - grained (UFG) and even nano - structure with the grain size below 100 nm, are the Severe Plastic Deformation (SPD) methods [3-15].

The possibility of producing bulk nanomaterial by SPD methods depends on the kind of the initial material, amount of deformation, strain rate, existence of second phases and value of the stacking fault energy. Nanostructures in steels, copper, titanium, zirconium, uranium and theirs alloys were found forming by application of SPD methods. The comparison of different materials showed that when the value of stacking fault energy is higher, the material is less profitable for nanostructure formation. One of such example is aluminium in which due to high value of the stacking fault energy and easy structural recovering, nanostructure is almost impossible to produce.

The bulk nanometric or ultrafine grained materials achieve a new and extraordinary properties different than obtained in the same materials produced by conventional process of plastic deformation. They have not only very small grain size but also specific defect structures, high internal stress, crystallographic texture and often change of phase composition. Also characteristic for nanomaterials is the superplasticity effect [16].

The nanostructures observed in the bulk materials, which were formed through the SPD methods are built from typical dislocation structures with high density of dislocations at the grain boundaries. The large surface of the grain boundaries is the reason of increasing amount of stored energy in nanometric volumes [6].

The alternative to the SPD methods is the deformation with high strain rates ($10^2$ - $10^6$ $s^{-1}$). High strain rates can be applied for example with hydrostatic extrusion process, Hopkinson split bar, ballistic impact, explosive fragmentation, high - speed shaping and forming or dynamic compression. The results obtained from the investigations with high strain rates show the higher value of microhardness than obtained with static deformation and similar structural effects as at SPD methods applications [13, 14].

Characteristic feature of dynamic deformed materials is activation of the deformation in the shear bands, microbands and adiabatic shear bands [4, 14]. Localized shear deformation in the form of a narrow bands generated during dynamic deformation under high strain rates has been a topic of a great interest of decades. Tresca had observed this phenomenon in the nineteen century [17]. Especially interesting is the temperature effect connecting with the adiabatic shear bands, which succession are essential changes in the microstructure [13].

The possibility of microstructure refinement in polycrystalline copper Cu99.99 by dynamic compression is the main objective of the presented study. Dynamic compression experiment have been conducted on cylindrical samples at room temperature.

## 2. Experimental procedure

The investigations were carried out on the polycrystalline copper Cu99.99. Cylindrical samples of diameter $\phi_0$ = 10 mm and height h = 12.5 mm were compressed on a falling – weight – type impact – testing machine. The samples were compressed at the true strain $\varphi$ = 0 - 0.38 and at the strain rate ranging from $1.75\times10^2$ to $2.7\times10^2$ $s^{-1}$. The process was realized at room temperature. The scheme of the falling - weight - type impact - testing machine is presented at the Figure 1.

After the deformation, the samples were tested for microhardness and their microstructure was examined by means of both optical and transmission electron microscopy JEM 2010. The thin foils were cut out from longitudinal sections of the samples and prepared applying the standard technique of electrolytic polishing using the Struers apparatus. The statistical width of the microbands observed in the microstructure was calculated using the mean chord method.

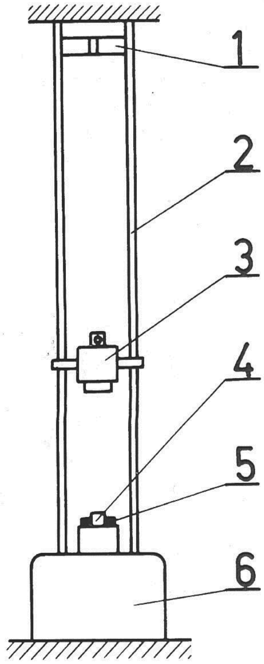



Fig. 1. Falling - weight - type impact - testing machine; 1- beam, 2 - shears, 3 - beater, 4 - sample, 5 - distance ring, 6 - stand

The misorientation of selected microstructural elements was determined using proprietary KILIN software. After the deformation $\varphi$ = 0.38 about 35 misorientation angles were measured and the statistical data was carried out.

## 3. Results and discussion

The optical micrographs show elongated grains cutting by the numerous bands and shear bands (Figs. 2a, 2b, 3a, 3b). The compression direction at the Fig. 2 and Fig. 3 agrees with the perpendicular direction on figures.

Very characteristic is straight curse of bands through the sample cutting grain boundaries (Fig. 3b). The distinct jogs were formed at the crossing boundaries (Fig. 3a). This feature allows identifying these bands as shear bands.

Bands have the inclination to the compression direction of about 45 - 80 degree. Probably such wide spread of the positions of bands is connected with the rotation of the material during deformation process and the internal stresses.

It was found that at the beginning of deformation bands were limited to the single grains (Figs. 2a, 2b). With increase of the deformation the density of bands rise and they propagate to the neighboring grains (Fig. 3b).

Next characteristic features observed in the microstructure, which was found in the compressed copper, was the phenomenon of mutual crossing bands. This effect was observed from the beginning of deformation, but its tremendous development appeared at higher deformations (Fig. 3b).

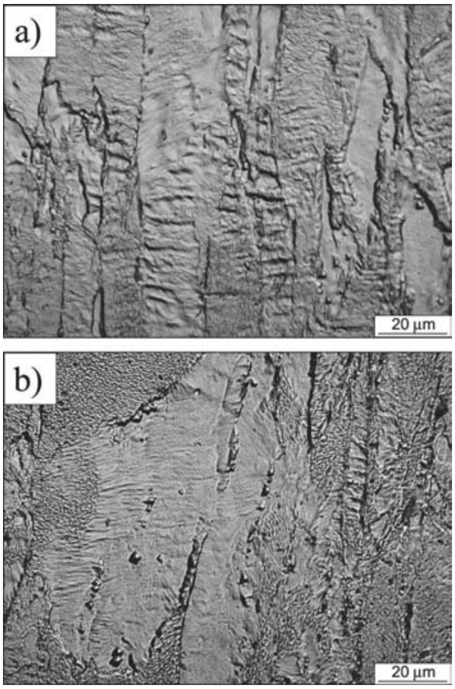



Fig. 2. Microstructures of dynamically compressed copper; a) φ = 0.27, b) φ = 0.3

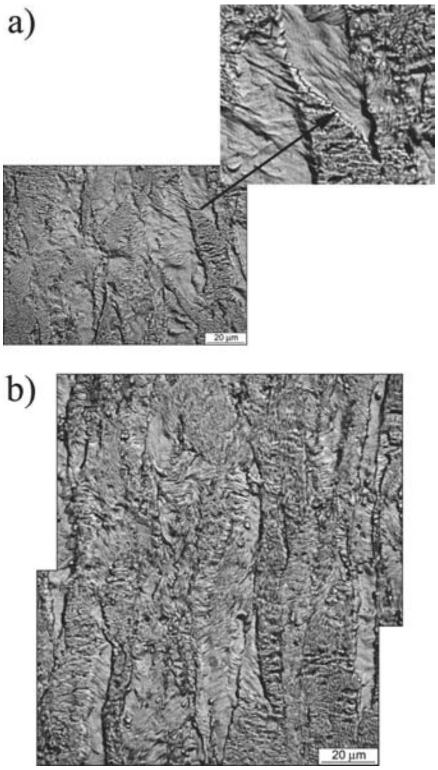



Fig. 3. Microstructures of dynamically compressed copper; a, b) φ = 0.38

The microbands and micro-shearbands on the background of subgrains microstructure were characteristic of dynamically compressed copper (Fig. 4 - Fig. 8). Thick boundless of microshearbands observed in the microstructure proceeds at the considerable distances (Fig. 7).

The walls of microbands contained high density of dislocation (Fig. 4 - Fig. 8). Occasionally, inside microbands characteristic was occurrence of cells microstructure free of dislocation inside (Fig.4 a,4b). The cells observed inside the bands are smaller in size, than the outside the microbands.

The typical feature of dynamically compressed copper was occurrence the second family of microbands which intersect the first family of microbands, forming a characteristic parallelogram microstructure (Fig. 4, Fig. 6). The interior of this type of cells microstructure was free of dislocation and probably create during dynamic recovery processes.

The mutually crossing microbands lead to the formation of micro – and nanovolumes, which could transform to new grains, as it was described and discussed in [6,20,21]. Similar structural effects take place in the samples deformed with enormous strains by the Cyclic Extrusion Compression (CEC) method [6,20].

The increase of deformation (φ = 0.38) cause the enlargement of the amount of the microbands and the reduction of their width (Fig. 7, Fig. 8b). Fig. 10 and Fig. 11 show results of the statistical measurements of the mean width of microbands by the chord mean method. The difference between the width of microbands at comparison to the strain φ = 0.3 and φ = 0.38 is about 15%. The distribution of microbands width show that the majority results are placed in the range between the 100 and 200 n. It suggest essential refinement of microstructure by dynamic compression.

From the literature data the very know phenomenon is that the shear bands were privileged places for nucleation of new grains and nanograins formation [15]. Xu et al. [17] and Mishra et al. [18] proposed a possible mechanism of nanograins formation in the occurrence of shear bands in metals produced by SPD methods. As the grain size reduced to the range of 50 to 200 nm by the localized deformation in the shear bands, the grain boundaries act as primary sources of dislocations. When the dislocations are annihilated in the opposite boundary, the cube is transformed into a parallelepiped. It is necessary for the grain boundaries to rotate back to their initial configuration. The grain boundary rotation is then possible within the deformation process. The rotation of the grain boundaries coupled with shear on a new slip plane guarantee the hold - up of a steady - state equiaxed structure. The authors [17,18] suggest that for nanocrystalline metals the rotation of grain boundaries is a diffusion controlled process.

During the microstructure observations of compressed copper such phenomenon was also searching, however any recrystallized new grains were found.

Common feature of the dynamically compressed copper is a large misorientation of microbands with respect to the matrix (Fig. 8a, Fig. 8b). Small misorientation angles were observed only occasionally. Some of the earliest investigations carried out on aluminium alloys compressed by falling - weight - type impact testing machine also show the occurrence of the narrow microbands of the great misorientation to the matrix [4].

The mean misorientation angle for copper deformed to the strain φ = 0.38 was about 33$^{o}$.

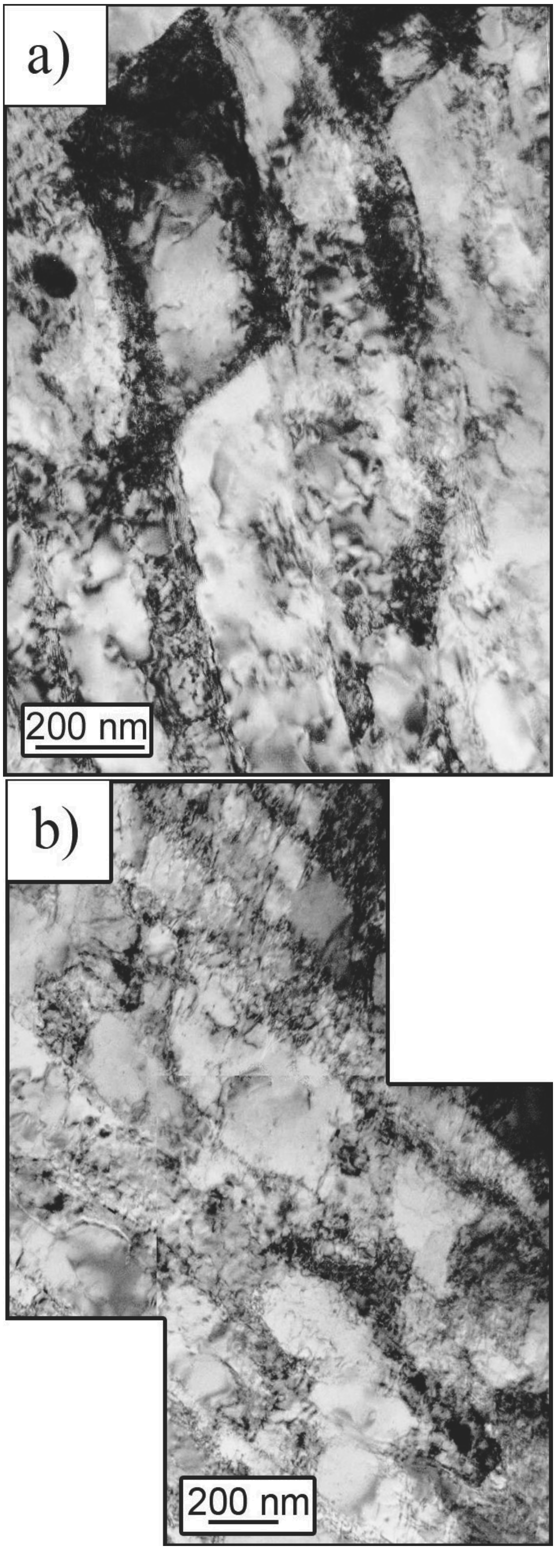



Fig. 4. Characteristic dislocation microstructures; a, b) φ = 0.3

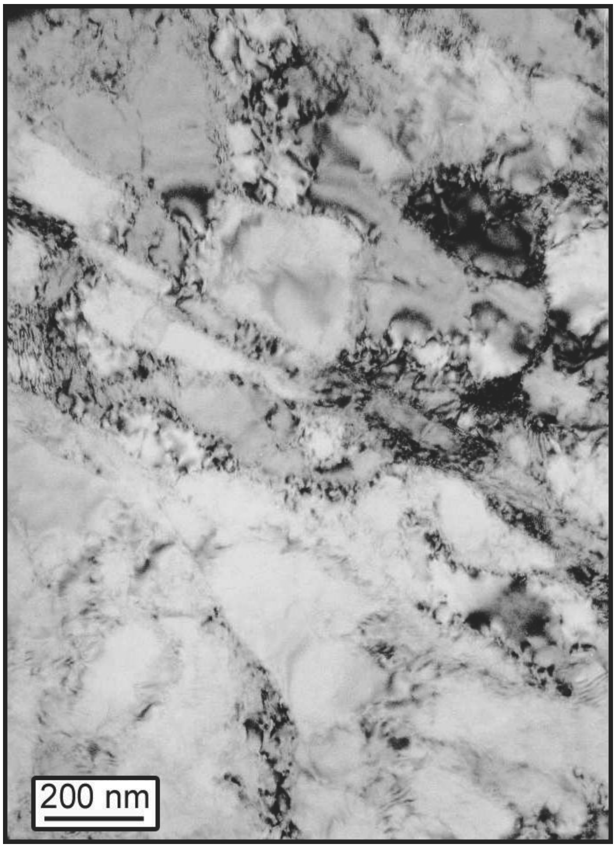



Fig. 5. Thick microbands in the vicinity of cells microstructure, φ = 0.38

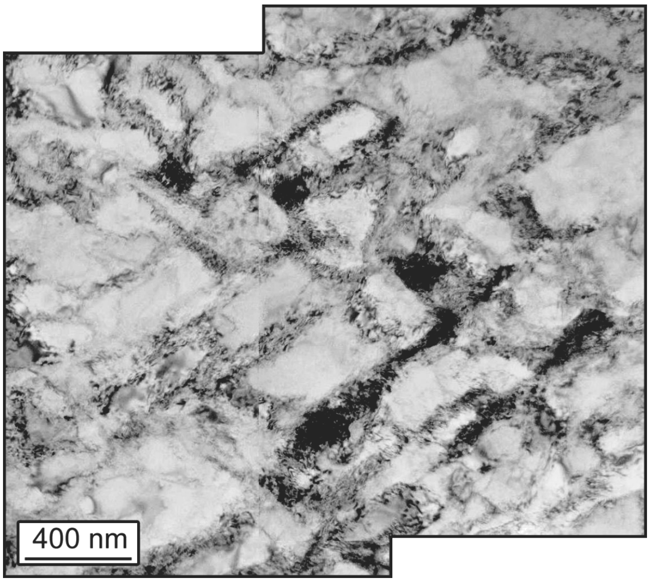



Fig. 6. Mutually crossing microbands, φ = 0.38

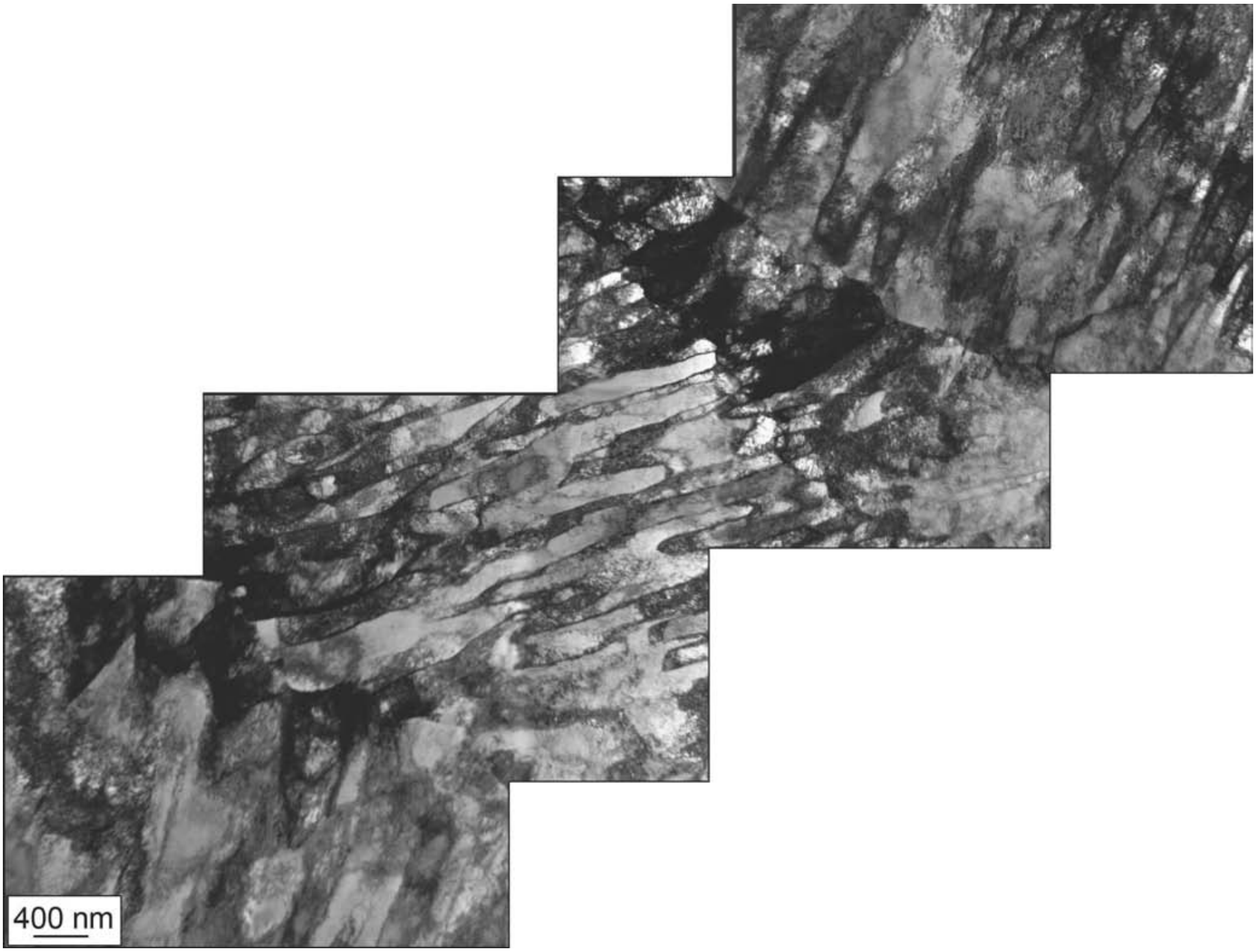



Fig. 7. Bundles of microbands and microshearbands proceeds at the considerable distances, φ = 0.38

The distribution of misorientation angles (Fig. 9) indicated that the portion of the low angle boundaries in comparison to the high angle boundaries is about 30%. The presented results indicated that in the compressed copper large misorientation boundaries dominate. Such microstructure should be very quickly recrystalized in high temperatures during samples annealing directly after deformation due to existence of numerous places with activate high disoriented boundaries. From this point of view, the microstructures produced by SPD methods or other way of deformation, as example by the proposed dynamic compression, probably could be instable under elevated temperatures in the successive applications.

The occurrence of large misorientation angles indicates a considerable rotation of the materials during deformation at the high strain rate and large plastic deformation exerted by SPD methods [2, 5, 6]. This phenomenon is also connected with considerable energy storage in the vicinity of newly created grain boundaries.

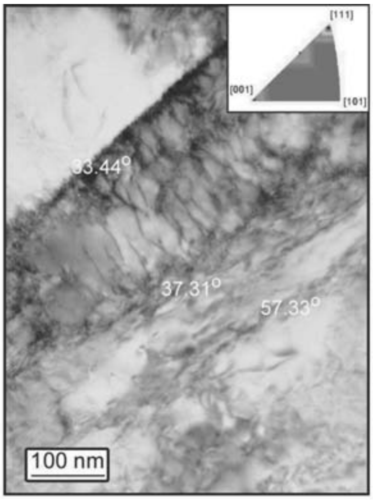
The data of work [19, 20] presented results of copper deformed by SPD methods indicate the domination of large misorientation angles in the ultrafine – or nanograined copper. The summary results for different methods are presented in the Table 1.

Table 1.
Quantitative changes of large misorientation angles obtained for different deformation methods in polycrystalline copper

| method, deformation size | large misorientation angles fraction, mean grain size [nm] |
|---|---|
| ECAP, φ = 12 | 90 %, d = 210 nm [19] |
| CEC, φ = 14 | 53 %, d = 200 nm [20] |
| HE, φ = 3.79 | 80%, d = 225 nm [21] |
| dynamic compression by special laboratory hammer, φ = 0.38 | 60% (present work) |

It was found that the distinguished orientations are grouped at the <100> - <111> side of the basic triangle (Fig. 8a, Fig. 8b). The TEM investigations were performed at the longitudinal sections of samples therefore the distribution of the microtexture is connected with the orientations perpendicular to the sample axis and therefore it is impossible identified the real texture of compressed copper, which should be connected with the <110> orientation, typical for the compression.

a)




b)

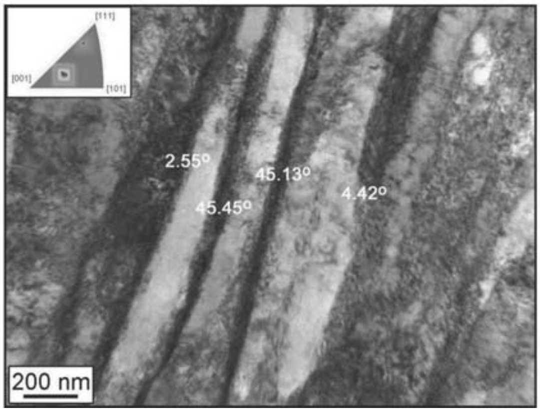



Fig. 8. Characteristic dislocation microstructures with misorientation angles, $\varphi = 0.38$

The microhardness of polycrystalline copper is presented at Figure 12. The results show copper hardening throughout the whole investigated range of deformation, starting from 80 $\mu Hv_{100}$ and attaining near 110$\mu Hv_{100}$ after the deformation of $\varphi = 0.38$ (Fig. 12).

The monotonic hardening of copper is due by balance between the hardening and softening processes, which are active during the dynamical compression. The continuous increase of microhardness indicated, that the hardening processes prevailed in the deformed copper.

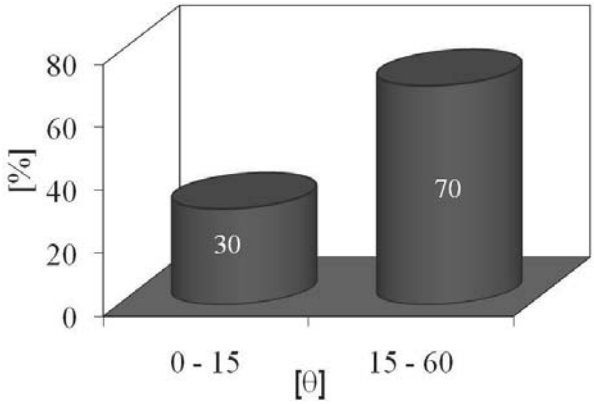



Fig. 9. Distribution of misorientation angles

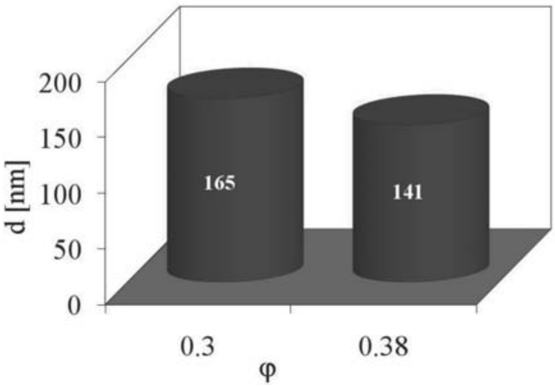



Fig. 10. Mean width of microbands as a function of deformation

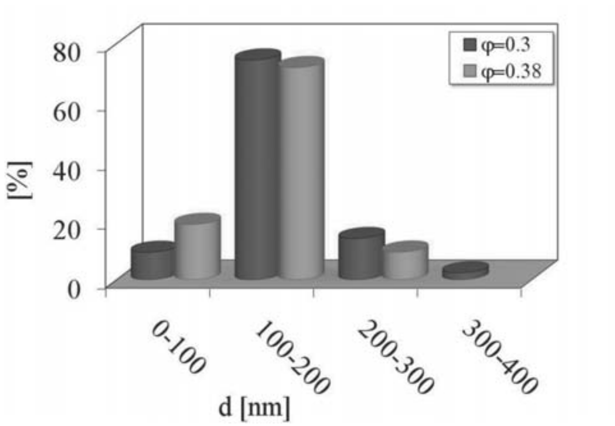



Fig. 11. Distribution of width of microbands

The presented dislocation microstructures (Fig. 4 - Fig. 7) show, that the main deformation mechanism develop by the microbands and microshearbands and it essentially contributes to the hardening due to high density of dislocations both in microbands boundaries and inside the microbands. Following parameters which contribute to the hardening are narrow microbands. Some portion in the global level of sample hardening has the multiplication of the dislocations in the matrix. The detailed observation of the copper microstructure indicates some changes of dislocations arrangement inside the microbands. Especially formation of thick walls perpendicular to microbands boundaries is the result of such phenomenon. This is connected with the development of the softening processes (as recovery) and can influence on the lowering hardening level.

The similar results and description of hardening - softening balance in the deformed materials has been discussed and presented in several scientific works [22 - 26].

The results concerning dynamic deformed materials and presented by the Y.Tirupataiah and co - workers [26] show higher properties of the dynamic deformed materials than deformed with low and moderate strain rate.

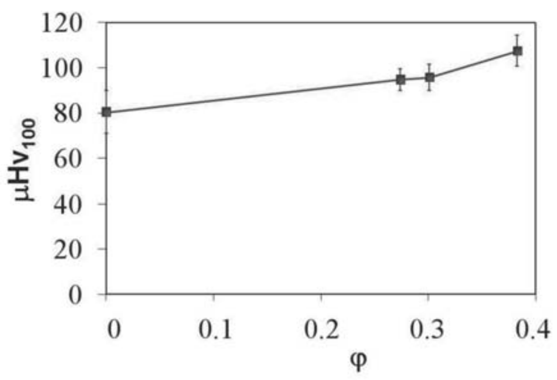



Fig. 12. Microhardness of polycrystalline copper Cu99.99

High properties of ultrafine- grained materials are in agreed with Hall - Petch relationship. In the conditions of very small grain size (smaller than 100 nm) this relation can be inversed. This is the reason of the great interest in the physics of the strength and plasticity of materials with nanometric size. The inversion of the Hall - Petch relationship probably is conditioned by a possible change in the mechanisms of plastic deformation. The initiation and movement of dislocations may become impossible in nanometric grains [16].

## 4. Conclusions

1. The bands and shear bands with characteristic band intersection and formation of distinct jogs on the intersected boundaries are characteristic features of microstructure observed by optical microscope.
2. Thick microbands of the great misorientation with respect to the matrix are typical microstructure of dynamically compressed copper.
3. Occurrence the second family of microbands intersecting the first family forms a characteristic parallelogram microstructure free of dislocation inside.
4. The reduction of the mean width of microbands with increasing of deformation was observed. After the deformation of $\varphi = 0.38$ the mean width of microbands was about d = 140 nm.
5. Dynamic compressed copper hardening throughout the whole investigated range of deformation, starting from 80 $\mu Hv_{100}$ and attaining near 110$\mu Hv_{100}$ after the deformation of $\varphi = 0.38$.

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