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Structure and properties of copper deformed by severe plastic deformation methods

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Properties

ABSTRACT

Purpose: The main object of this study is to establish the influence of severe plastic deformation on the microstructure evolution and properties of polycrystalline copper Cu99.99.
Design/methodology/approach: Polycrystalline copper Cu99.99 was deformed by cyclic extrusion compression (CEC), equal channel angular pressing (ECAP) and hydrostatic extrusion (HE). Additionally the combination of these methods were applying to the sample deformations. The microstructure and properties of samples after different kinds of severe mode of deformations (SPD) were examined and compared as well as their properties. The microstructure was investigated by optical (MO) and transmission electron microscopy (TEM). The microhardness was measured by PMT3 microhardness tester.
Findings: It was found that increase of deformation diminishing the microstructure and leads to the increase of microhardness of samples.
Practical implications: The results may be utilized for determination of a relation between microstructure and properties of the copper deformed in the severe plastic deformation process.
Originality/value: The results contribute to evaluation properties of the polycrystalline copper deformed to very large strains exerting the typical range of deformations.
Keywords: Microstructure; Properties; SPD processes

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1. Introduction

The severe plastic deformation methods (SPD) [1-7] are used to production of ultrafine (UFG) and nanocrystalline materials (NC).The properties of materials deformed by SPD methods depend on the kind of deformed material, level of deformation, SPD mode of deformation and also in the case of combination of SPD methods or combination of SPD and conventional deformation methods it depends on the deformation path [8, 9].

Polycrystalline copper is one of the most widely studied material due to its special properties allowing applying it in the electrical and power industry.

The grain size, very close to upper boundary of nanometric level of grains, can be obtained in pure copper deformed by SPD methods. It is reported in literature[10-12], that in copper it is possible to obtain an average grain size of about 150-300 nm, which is very close to 100 nm, typical for the nanomaterial size of grains In other kind of pure materials, for example pure aluminium, such diminishing of grains is impossible. The lower, than in aluminium, stacking fault energy of copper is responsible for this phenomenon. Therefore copper belongs to small group of pure materials in which it is possible successfully strongly reduce grain size by severe deformation processes.

Nowadays, very often for materials deformation the combinations of SPD processes or combination of SPD and conventional deformation working processes are applied [13-15]. It allows to receive new properties of deformed materials [13-17]. Additionally such way of deformation creates the possibility of producing selected, useful products.

Especially the combination of SPD and hydrostatic extrusion or conventional extrusion methods brings very good results. For example the conform process is the combination of equal channel angular pressing (ECAP) method and conventional direct extrusion [13, 17]. It allows to fabricate rods and wires from different materials.

In the work the pure copper was deformed by the cyclic extrusion compression (CEC) [3] and equal channel angular pressing (ECAP) [7]. The microstructure and properties of samples deformed by the two methods were compared. Additionally the hydrostatic extrusion was applied for the samples deformed by the CEC, and the microstructure and properties were investigated.

2. Experimental procedure

The investigations were carried out on the polycrystalline copper Cu99.99. The cyclic extrusion compression (CEC) [3] (Fig. 1), equal channel angular pressing (ECAP) [7] and hydrostatic extrusion (HE) [18] were used for sample deformation. In the CEC method the copper samples were deformed in the range of strains φ = 1.3-20.8, with the deformation φ = 0.65 in the single CEC cycle. The deformation by ECAP was performed in the range of strains φ = 4.6-18.4. The samples deformed by CEC next were additionally deformed by hydrostatic extrusion with the true strain φ = 1.83 and strain rate 1.59×10^{1}-6.7×10^{1} s^{-1}.

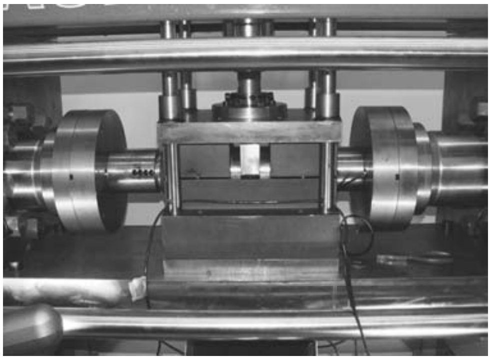

Fig. 1. Cyclic extrusion compression tools mounted at the hydraulic press with the 600 kN capacity

After the deformation, the microstructure of samples was examined by means of optical microscopy (MO) and transmission electron microscopy JEM 2010 ARP (TEM). The Olympus GX51 microscopy was used for microstructures observations, which were performed at the longitudinal cross sections of samples. The thin foils were cut out from the longitudinal sections of the samples and prepared applying the standard technique of electrolytic polishing using the Struers apparatus. The statistical size of subgrains in the microstructure was calculated using the mean chord method.

The microhardness of copper was measured by using PMT3 equipment with the load 100 G.

3. Results and discussion

The optical micrographs (Fig. 2 to Fig. 4) show the evolution of microstructure of copper samples deformed by the CEC method. The band structure was observed in deformed samples. The considerably increase of bands density with the increase of deformation was found . At the initial stage of deformation, bands were arrested inside the copper grains (Fig. 2). They have different inclination, however it was also observed that in some areas bands keep the same direction thorough a few grains. The grains characterize almost equiaxial shape.

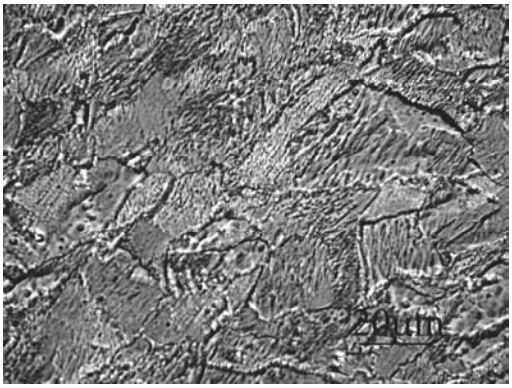

Fig. 2. Microstructure after 10 CEC cycles, φ = 8.9, (MO)

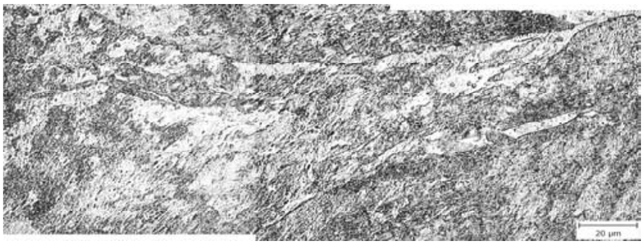

Fig. 3. Microstructure after 20 CEC cycles, φ = 17.9, (MO)

The deformation increase forces the bands propagation thorough the grain boundaries. The long bands were found running at long distances. The shear bands appeared penetrating sample volume, crossing grain boundaries and forming characteristic steps (Fig. 3, Fig. 4).

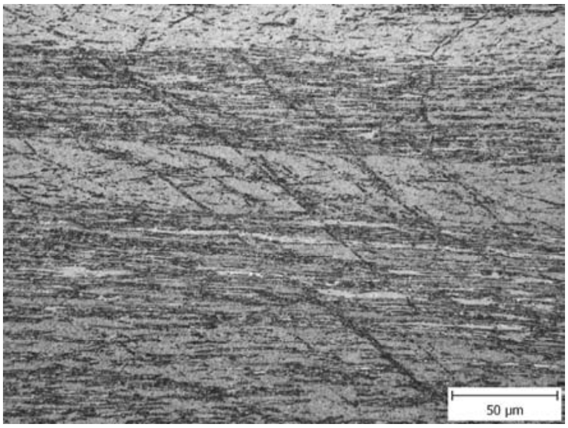

Fig. 4. Microstructure of Cu after 30 CEC cycles, φ = 26.7 (MO)

The bands microstructure were also observed in copper deformed by the equal channel angular pressing (ECAP) method. After the 4 ECAP routs (φ = 4.6) the broader bands were found. They were crossing by the second family of bands inclined at angle of about 50^{o} to the broader bands (Fig. 5). The characteristic steps are visible in the crossing places.

The similar microstructure, with the characteristic stacking of bands grouped into wide bundles, were also observed after the 8 ECAP routs (φ = 9.2) (Fig. 6). In this case, similar as after the 4 routes, the broader bands were cut by the second family of bands, which form distinct offsets at the crossing boundaries .

The increase of deformation to φ = 18.4 (16 ECAP routs) leads to considerably increase of bands density and their mutual crossing (Fig. 7). As the result of this phenomenon the almost uniform microstructure was formed.

Fig. 5. Microstructure of copper deformed in the 4 ECAP routs (φ = 4.6), (MO)

Samples deformed by the CEC were next hydrostatically extruded with the deformation φ = 1.83. Evolution of microstructure, observed by optical microscopy, after the combined deformation is presented at Fig. 8 to Fig. 10.

After the 10 CEC cycles (φ = 6.5) and hydro-extrusion (φ = 1.83), a few families of the mutually crossing bands were observed (Fig. 8). The elongated to the extrusion direction bands were found. The elongating bands were crossing by shear bands, penetrating the sample at long distances, inclined at angle of about 40º to the extrusion direction and forming distinct offsets at the crossing boundaries. The additional characteristic features of the observed microstructure was short bands placed inside the elongated to the extrusion direction bands and grains.

Fig. 6. Microstructure of copper deformed in the 8 ECAP routs (φ = 9.2), (MO)

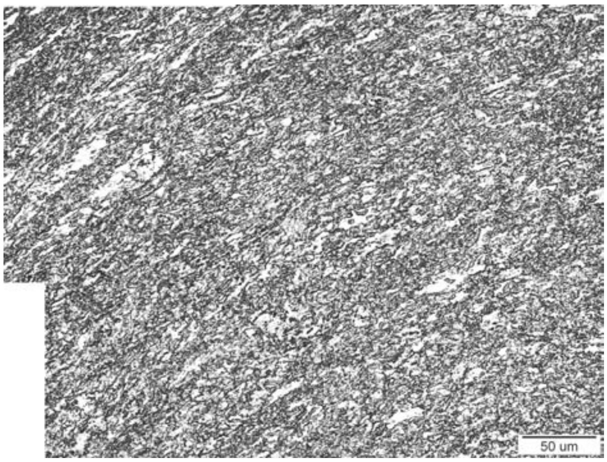

Fig. 7. Microstructure of copper deformed in the 16 ECAP routs (φ =18.4), (MO)

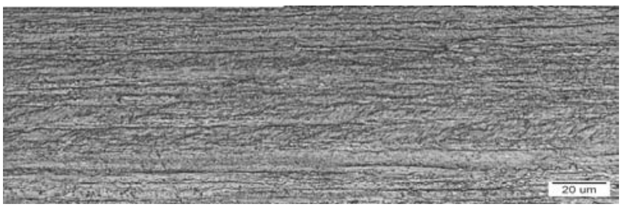

Fig. 8. Microstructure of copper after the combined deformation, 4 CEC cycles (φ = 4.6) and HE (φ = 1.83), (MO)

The example of the microstructure after the 20 CEC cycles (φ = 13) and hydro-extrusion (φ = 1.83) is presented at Fig. 9. The large density of crossing bands with the characteristic shear bands were found. The shear bands are recognized as characteristic deflections of boundaries of crossing bands and penetrate through the whole sample sections. They appeared on the background of elongated bands, and grains. Inside the elongated bands the additional family of short bands were also found.

After the 30 CEC cycles and hydro-extrusion the similar microstructure was found as after the combination of 20 CEC cycles and hydro-extrusion. A large density of mutually crossing bands was observed and characteristic bunches of stacking bands (Fig. 10). Very typical was the formation of shear bands observed in the all investigated samples.

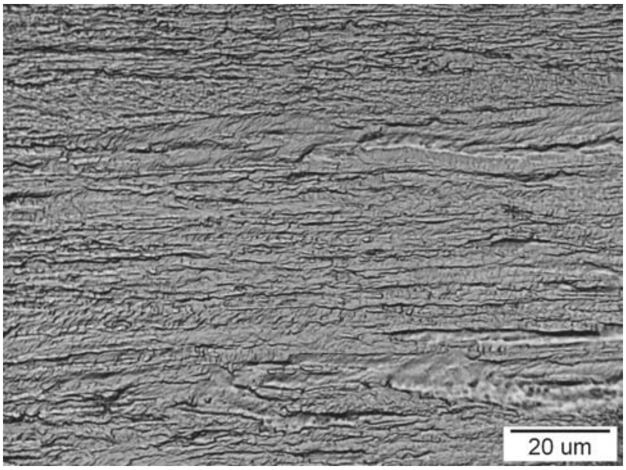

Fig. 9. Microstructure of copper after the combined deformation, 20 CEC cycles ($\varphi = 8.4$) and HE ($\varphi = 1.83$), (MO)

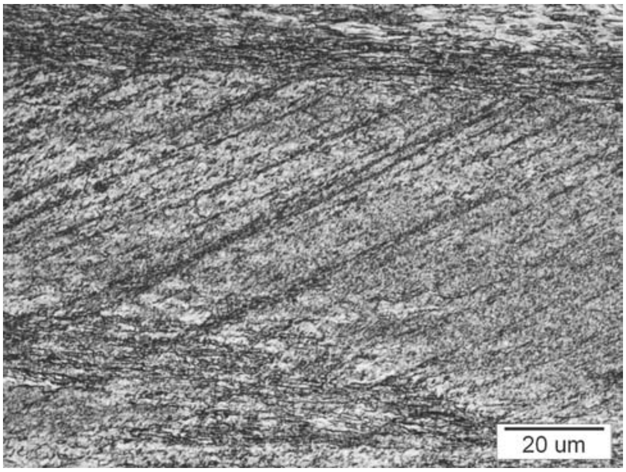

Fig. 10. Microstructure of copper after the combined deformation, 30 CEC cycles ($\varphi = 12.6$) and HE ($\varphi = 1.83$), (MO)

The investigations performed by TEM technique shown that after the cyclic extrusion deformation microstructure contains numerous elongated subgrains and microbands, which density increases with the increase of cyclic deformation (Fig. 11, Fig. 12).

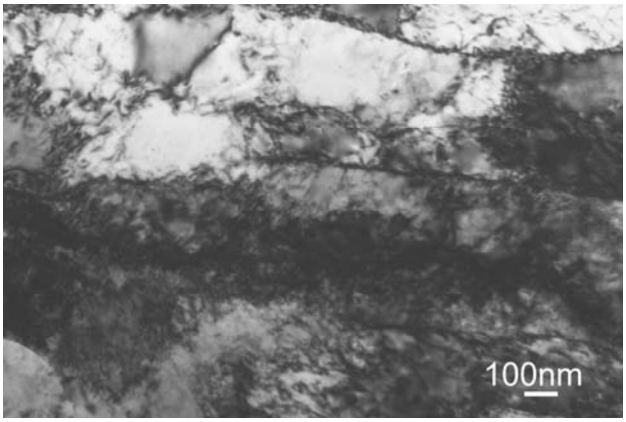

Fig. 11. Microstructure of copper after the, 10 CEC cycles ($\varphi = 6.5$), (TEM)

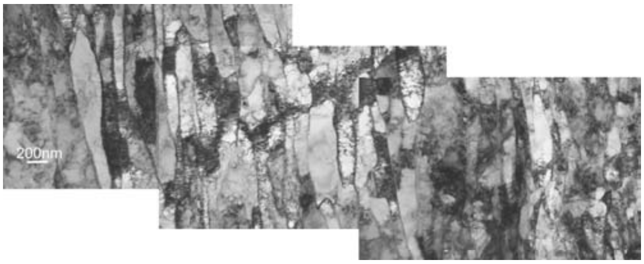

Fig. 12. Microstructure of copper after 20 CEC cycles ($\varphi = 13$) (TEM)

The new features of microstructure were found after the combined deformation cyclic extrusion compression and hydrostatic extrusion. It was observed that still dominating was band microstructure. However inside the bands microstructure the new grains were found. They have been formed during deformation or just after the deformation and result from the dynamic or post dynamic recrystallization processes. This processes initiated the formation of embryos of new grains inside the band microstructure (Fig. 13 to Fig. 16). The new grains characterize almost equiaxial or elongated, to the direction of bands propagation direction, shape and they were almost free of dislocation inside.

The new grains in some cases contained inside some dislocation, but its density was considerably lower than in surrounds. Inside the new equiaxial grains the micro-twins were also often observed (Fig. 17).

The microstructure found in samples at the longitudinal cross-sections, after the equal channel angular pressing contains the microbands and equiaxial subgrains/grains. The size of micro-structure elements was differentiate (Fig. 18, Fig. 19, Fig. 20).

The bands microstructure was very characteristic for lower deformations (Fig. 18 and Fig. 19). The performed observations enable to suggest that with the increase of ECAP deformation the bands microstructure becomes unstable and it transforms into the equiaxial subgrains, which were generally observed after the 16 routes $\varphi = 18.4$ (Fig. 20).

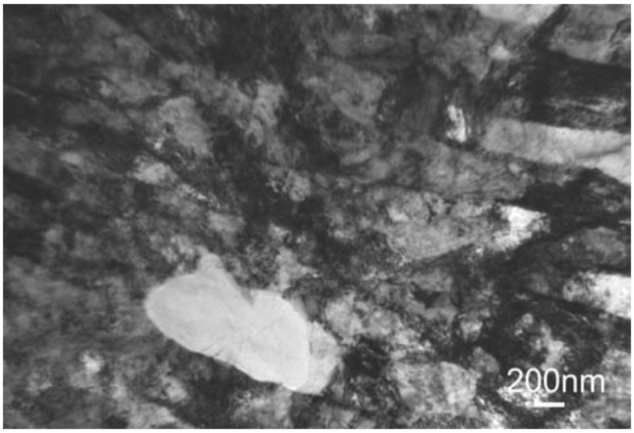

Fig. 13. Microstructure of copper after the combined deformation, 10 CEC cycles (φ = 6.5) and HE (φ = 1.83), (TEM)

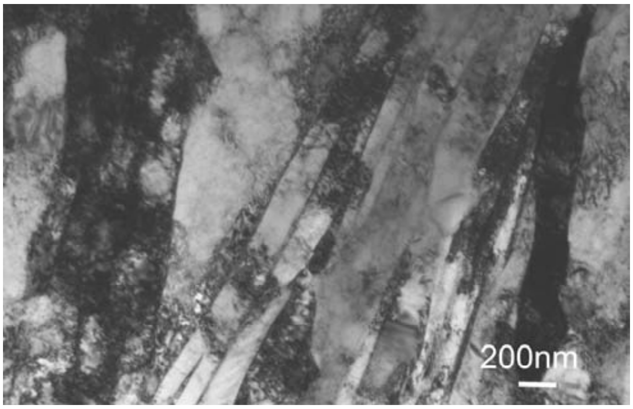

Fig. 14. Microstructure of copper after the combined deformation, 20 CEC cycles (φ = 13) and HE (φ = 1,83), (TEM)

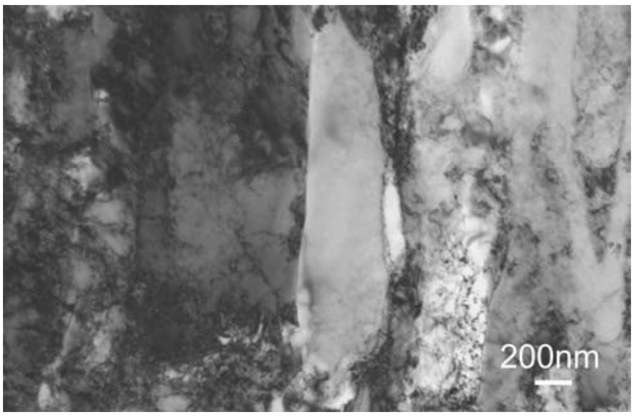

Fig. 15. Microstructure of copper after the combined deformation, 20 CEC cycles (φ = 13) and HE (φ = 1.83), (TEM)

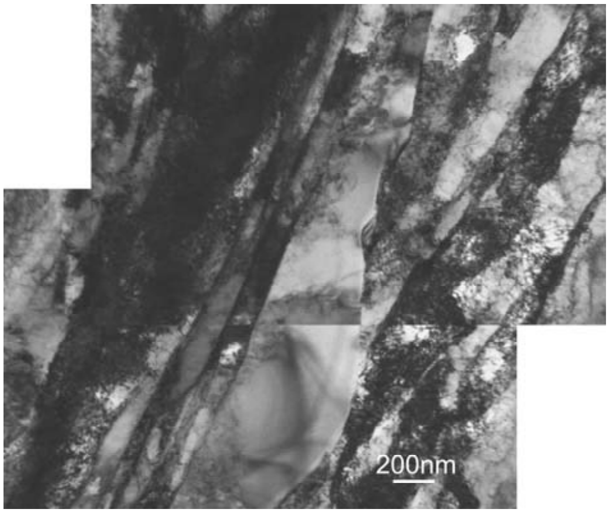

Fig. 16. Microstructure of copper after the combined deformation, 30 CEC cycles (φ = 12.6) and HE (φ = 1.83), (TEM)

Fig. 17. Microstructure of copper after the combined deformation, 30 CEC cycles (φ = 12.6) and HE (φ = 1.83), (TEM)

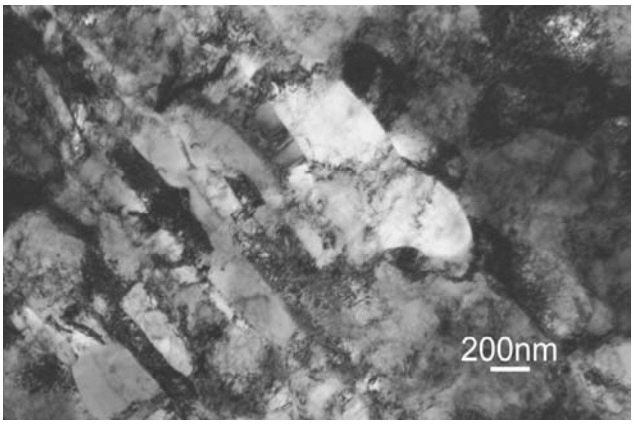

Fig. 18. Microstructure of copper after the ECAP 4 routes (φ = 4.6), (TEM)

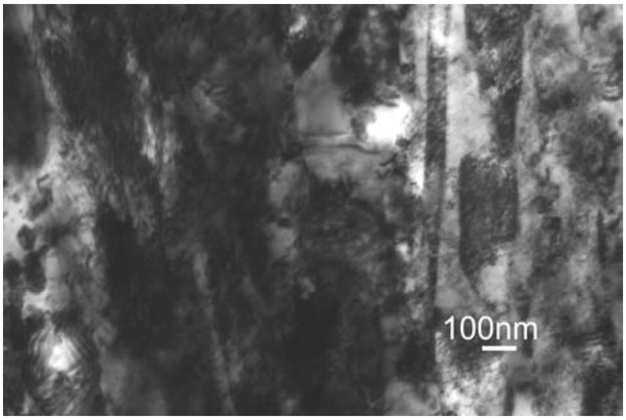

Fig. 19. Microstructure of copper after the ECAP 8 routes ($\varphi = 9.2$), (TEM)

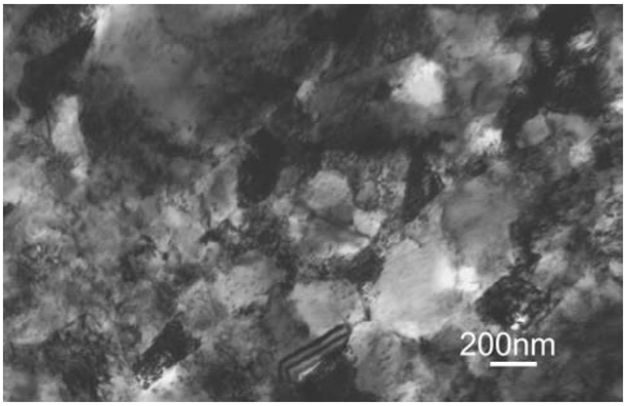

Fig. 20. Microstructure of copper after the ECAP 16 routes ($\varphi = 18.4$), (TEM)

Such evolution of microstructure is probably connected with the considerable increase of deformation energy, which concentrates mostly in band microstructure. On the other side it is possible temperature increase, which favors softening microstructure processes. The bands becomes the privilege places of new grain embryos growing. The observations revealed that at first the new grains spread along the direction of bands propagation (Fig. 13, Fig. 15, Fig. 16). Then they penetrate at the direction perpendicular to the bands propagation. The Fig. 16 showing characteristic front of recrystallization spreading into the neighboring bands. During the following deformation the embryos of new grains are again deformed which leads to the appearance of dislocation inside.

The same regularity appears on both graphs expressing relationship between the mean size of subgrains and deformation, exerted in the the CEC method and in the combination of the CEC and hydro-extrusion (Fig. 21). With the increase of deformation the mean size of subgrains decreasing. The achieved subgrain/grain size in copper is placed between the range 250-350 nm. It could be found that after the combined deformation CEC and hydro-extrusion the mean size of subgrains/grains is only a little higher that after the CEC. There probably result from the intense recovery processes and also recrystallization phenomenon. The appearance of embryos of new grains after the combined deformation is generally connected with the adiabating heating, which is very typical in the case of high strain rate deformation in hydro-extrusion process.

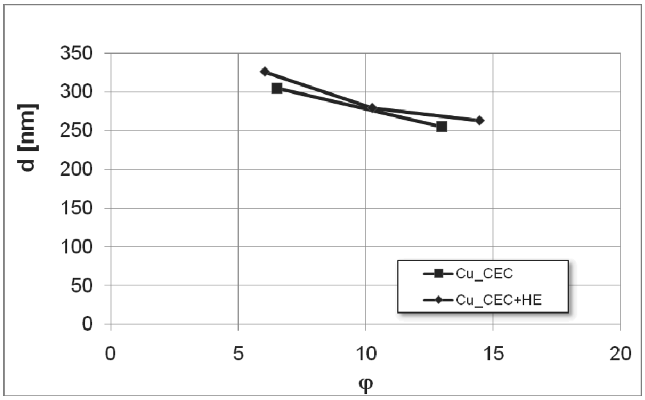

Fig. 21. The relationschip between the mean size of subgrains and deformation exerted by CEC and combination of CEC and HE

Fig. 22 presented the results of microhardness measurement after the CEC, ECAP and combination of CEC and HE and also combination of the ECAP and HE processes. The almost stable microhardness level was found in the deformed copper. The microhardness after the combined deformation is slightly lower that after the CEC and ECAP.

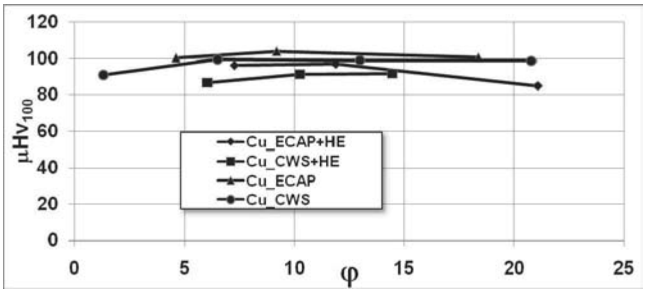

Fig. 22. Microhardness of copper deformed in the different SPD processes and their combinations

The obtained results suggest that the all investigated severe plastic deformation processes bring the similar microstructure features. The very typical is the band microstructure consisting from the elongated bands crossing by the shear bands and additionally by short bands appearing inside the broader bands. The characteristic is also microstructure observed by TEM building from microbands and elongated subgrains.

The achieved properties maintain at the almost same level, which suggests that to fine-crystalline copper produce it is not necessary the deformation increase above the true strain of about $\varphi = 15$.

4. Conclusions

1. Copper deformed by the SPD methods (CEC and ECAP) demonstrate at the initial stage of deformation the band microstructure, which changes into equiaxial subgrains/grains with the deformation increase.
2. After the combined deformation CEC and hydro-extrusion inside the band microstructure the embryos of new recrystallized grains were found.
3. It was found that with the increase of deformation the dimension of subgrains/grains mean size decreased.
4. Microhardness of copper deformed by CEC, ECAP and combination with hydro-extrusion is almost stable in the investigated range of deformations (φ = 4.6-18.4).

Acknowledgements

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