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ISSN 0543-5846
METABK 47(4) 295-299 (2008)
UDC – UDK 539.377:669-122.2:620.17=111

FORMATION OF ULTRAFINE-GRAINED (UFG) STRUCTURE AND MECHANICAL PROPERTIES BY SEVERE PLASTIC DEFORMATION (SPD)

Received – Prispjelo: 2007-12-04
Accepted – Prihvaćeno: 2008-03-23
Original Scientific Paper – Izvorni znanstveni rad

Commercial pure cooper (99,9% Cu) was deformed by equal channel angular pressing (ECAP) using up to 10 passes, route C. The evolution of microstructure and fracture character were observed by OM, SEM and TEM. The mean grain size decreased with increasing deformation, after 10 passes to 100 – 300 nm. TEM analysis suggested the possible nanostructure formation mechanism by the formation of cellular structure in grains, forming of subgrains and then forming of high angle nanograins with random orientation. Fractures of ECAP Cu material after 10 passes had transcrystalline ductile character with dimple morphology.

Key words: severe plastic deformation, ultrafine-grained materials, equal channel angular pressin, TEM, fracture surface

Stvaranje ultrafino zrnate (UFZ) strukture i mehanička svojstva sa intenzivnom plastičnom deformacijom (IPD). Trgovački čisti bakar (99,9%) deformiran je kanalnim kutnim prešanjem (KKP) do 10 provlaka-putanje. C. Razvitak mikrostrukture i karakter loma praćeno je sa OM, SEM i TEM. Veličina zrna se smanjuje povećanjem deformacije poslije 10 provlaka na 100-300 nm. TEM analiza ukazuje mogućnost nastajanja mehanizma nanostrukture stvaranjem celularne strukture u zrnima, nastanak subzrna i zatim obrazovanje nanozrna pod visokim kutem sa slučajnom orjentacijom. Lomovi KKP Cu materijala poslije 10 provlaka imali su transkristalni duktilni karakter sa jamičastom morfologijom.

Ključne riječi: intenzivna plastična deformacija, ultrafino-zrnati materijali, kutno kanalno prešanje, TEM, površina loma

INTRODUCTION

A lot of research works are reported on the development of new nanostructured materials. They have a fine microstructure with the mean grain size less than 100 nm and they manifest excellent physical and mechanical properties. A variety of technologies was developed to prepare nanostructured materials. Some of them were the powder metallurgy (PM) methods. The important phase in these methods is the powder preparation [1-2] and compacting [2]. There are still persisting the problems with the residual porosity of PM materials, the problem of impurities, and the grain growth in the following phases of the production. Severe plastic deformation seems to be a more convenient way to solve the listed problems.

The production of the nanostructure in compact metallic systems was studied e. g. in works [3-4]. The equal channel angular pressing (ECAP) can be the choice. It is pressing of the experimental material trough two right angled (usual 90°) channels of a special die. The ECAP technology allows to obtain the very fine grained microstructure – the nanostructure by multiple pressings trough the die. The development of high angle nano grains in metals and alloys, with specific substructures, arranging dislocations in cells and on grain boundaries was studied and analyzed [5-6]. Statistic evaluations of the heterogeneity of nanostructures produced by plastic deformation were described [7]. Improved mechanical properties can be obtained by severe plastic deformation. High strength and ductility was reported for different systems [8-9]. Extremely fine grains with high angle boundaries were obtained, developing unique superplastic behaviour [10] explained by the mechanisms of grain boundary sliding or by the grains rotation [11-12]. Microstructure and properties of nanomaterials are described in [13-18]. Some new approaches on deformation mechanisms of nanostructured materials are described in [14].

The aim of this work is to analyze the mechanical properties and their relations to the nanostructure developed by SPD for pure Cu using the ECAP technology.

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EXPERIMENTAL MATERIAL AND METHODS

Commercial pure copper (99,9% Cu) was used as an experimental material. Bars in dimensions of 10 mm in diameter and 80 mm long were pressed using the ECAP die at room temperature. A bar was pressed 10 times. The hydraulic press used for ECAP is able to produce a load of 1 MN. The deformed bars were then machined to the form of test specimens (Φ 3 mm, 15 mm long, M5) for static tensile testing, hardness testing, metallography and TEM analysis by thin foils.

RESULTS AND DISCUSSION

The initial values of mechanical properties and grain size of pure copper are in Table. 1.

Table 1. Mechanical properties and grain size of Cu material before ECAP

$R_{p\ 0,2}$ / MPa	R_m / MPa	A_5 / %	Z / %	HV	d_z /μm
270	275	13	65	72	50

The used copper is coarse grained with a mean grain size of 50 μm, both the yield strength ($R_p0.2$) and ultimate tensile strength (R_m) are quite low, but the reduction of area (*Z*) is significant (65%).

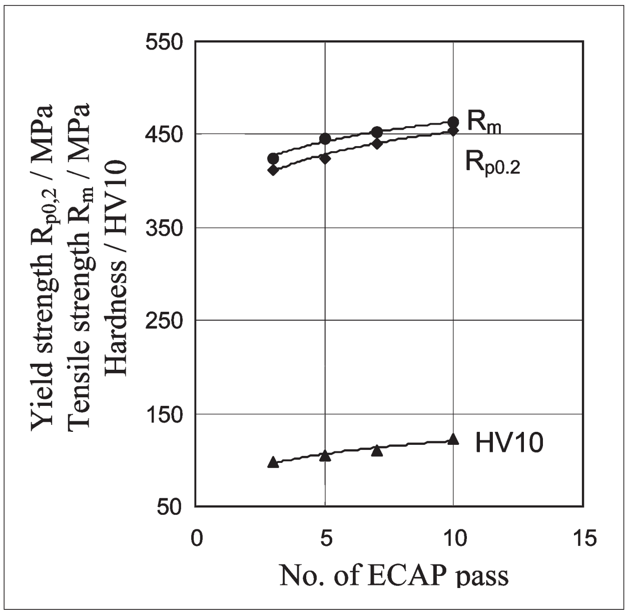

Figure 1. Strength properties ($R_{p0.2}$, R_m and HV 10) in dependence on the ECAP passes

The change of strength properties ($R_p0,2$, R_m and *HV10*) in dependence on the number of ECAP passes are in Figure 1. At every next pressing the test piece position was about 180° rotated. Cold working is known to produce an increase in strength and the Rm value had after 10 passes increased from 275 MPa to 464 MPa. The hardness HV10 increased after 10 passes to 128. According to Schiotz [6] we have analyzed the dependence

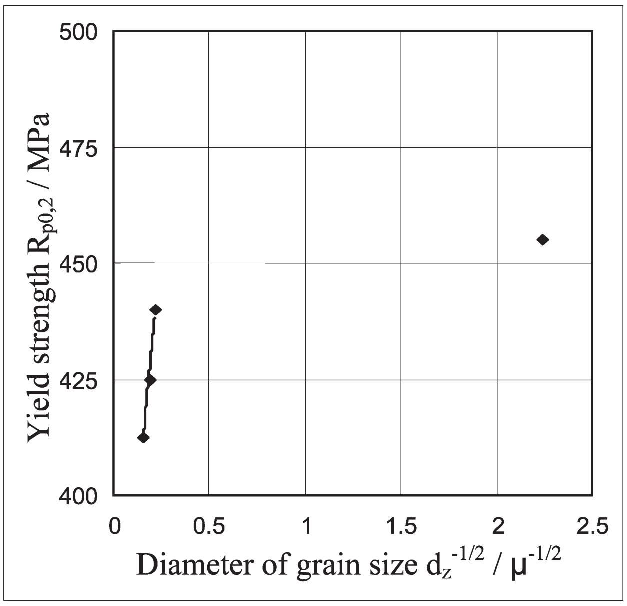

Figure 2. Dependence of hardening expressed by the yield stress point $R_p0.2$ on the grain size using the Hall-Petch equation

of hardening expressed by the yield strength $R_p0.2$ on the grain size using the Hall-Petch equation,

Figure 2. The other hardening contributions were neglected: the Peierls – Nabbaro, substitution and dislocation caused strengthening (R_{PN}, R_S a R_D).

The first two contributions have a very low value. It is quite difficult to evaluate the R_D due to structural changes caused by deformation. In the tested range of deformation and grain size the dependence is in good agreement with the Hall-Petch equation.

For the fine grained nanostructure (10 passes) the deformation mechanism deviation agreed with the simulation results and experiments of Schoitz [6].

The dependence of ductility represented by the reduction of area (Z) on the number of passes is presented in Figure 3. The reduction of area was selected as a measure of ductility because it is more sensitive to the local deformation in the neck of the broken tensile test specimen than the elongation to failure. It is important to note that the reduction of area is increasing with the number of passes with a similar trend as strength does. It is quite different as the classic behaviour of metals after plastic deformation. It is in good agreement with the results reported by Valiev [5], but not with Koch's report [13], for nanocrystalline materials with high strength and hardness but low ductility. The increase of ductility is important for the applicability of materials in general, and it is for the tested high strength nanostructural materials, too.

We have also analyzed the obtained microstructure. The initial grain structure of Cu is presented in Figure 4 made by light microscope. The grains are equiaxed and the structure is even. After 3, 5, 7 and 10 passes the grain size decreased, Figure7. The mean values in Figure 5 were calculated from about 100 grains, each. Though, the rotation of the bars after every pressing was applied,

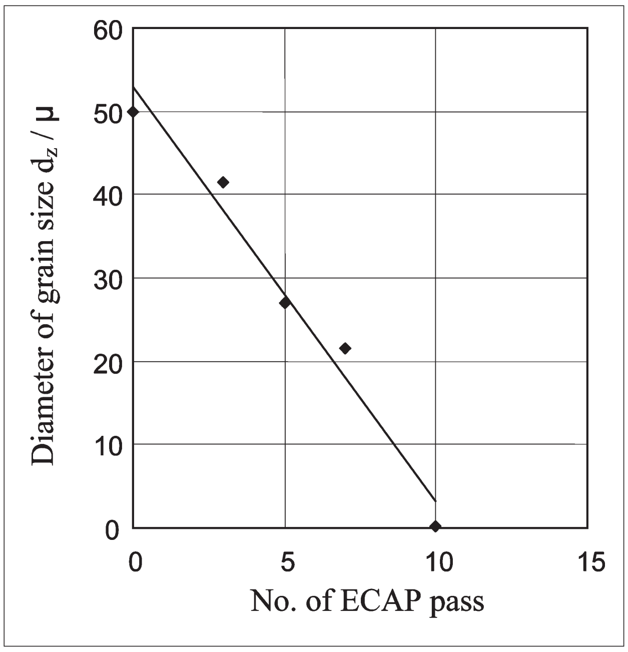

Figure 3. The dependence of ductility on the deformation represented by the reduction of area (Z)

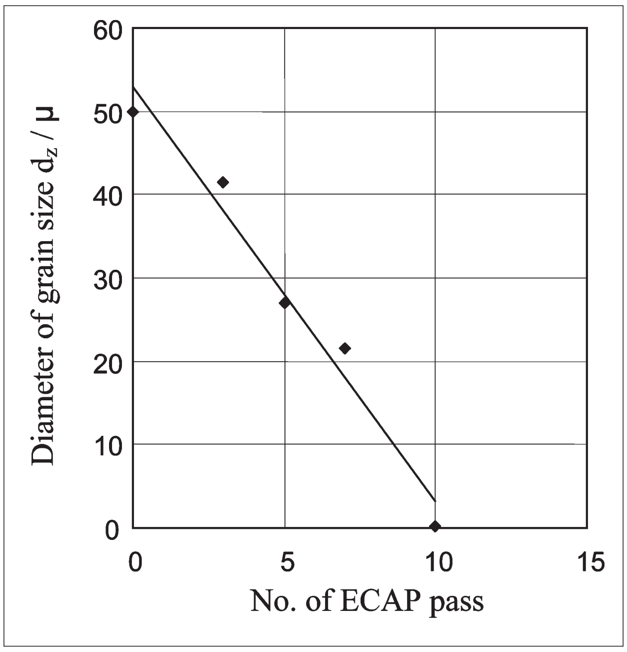

Figure 5. Decrease of the grain size after the ECAP passes)

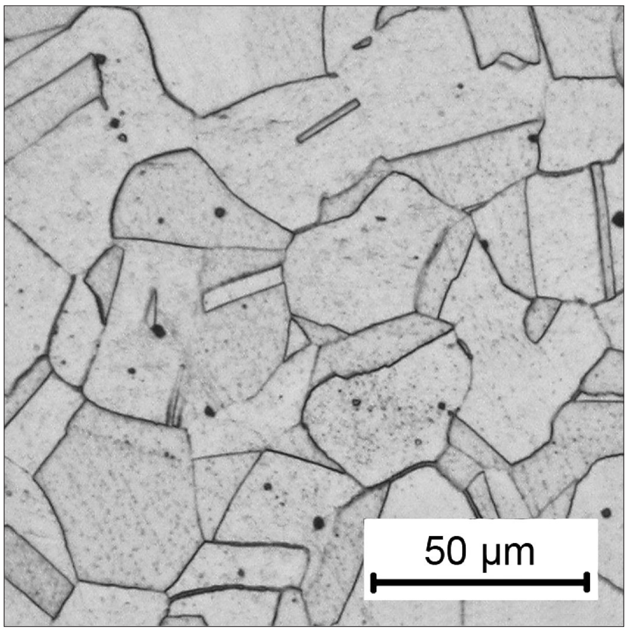

Figure 4. Microstructure of initial Cu material

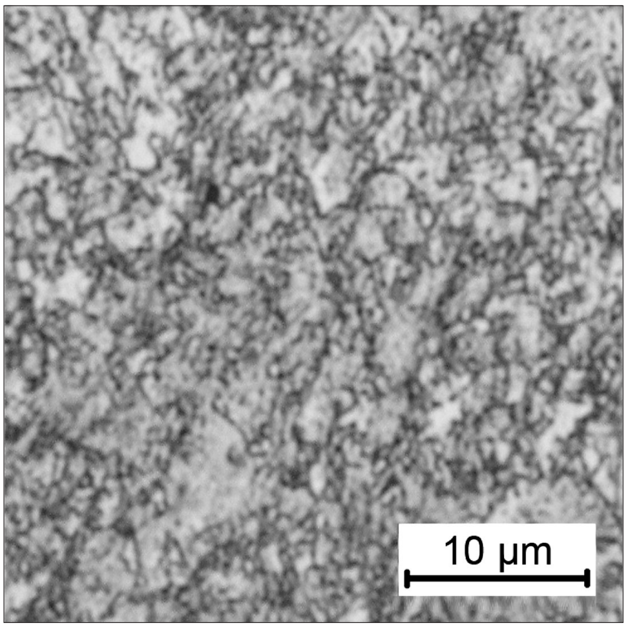

Figure 6. Microstructure of Cu material after deformation of 10 ECAP passes

the grain structure is not even anymore. The grains are elongated in the direction of deformation and with prevailingly high angle boundaries. The heterogeneity can have a negative influence on the stability of properties. Significant changes in strength can be supported by grains with high angle boundaries, only. After the maximal plastic deformation of ten passes the grains are on the limit of the resolution by metallography, Figure 6. The mean grain size was less than 1 μm. We have prepared thin foils to identify the grain size and to monitor the mechanism of grain forming by deformation in the neck. The TEM showed that the mean grain size was from 100 to 300 nm.

The mechanism of forming grains with high angle boundaries is supposed to be by the forming of a cellular structure, and the forming of subgrains, which are transformed with increasing deformation into nanograins with high angle random orientation Figure 7. and Figure 8.

The most important phase is the period changing the cellular structure to high angle grain structure. As described in [5], it is supposed, that the cell walls are thinning and reordering dislocations. It is obvious that the nano grain boundaries are not in equilibrium for the absorbed large deformations of the cellular structure and for the high dislocation density. We suppose, the ultra fine grains with high angle boundaries at the following

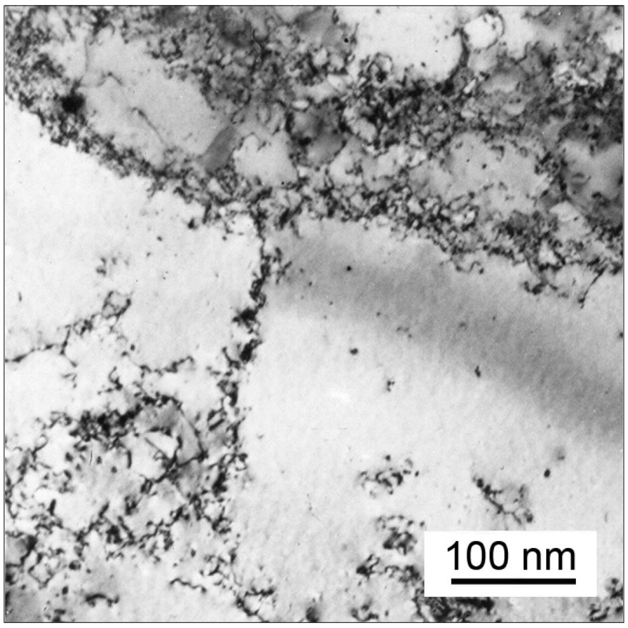

Figure 7. Forming of subrains during deformation process

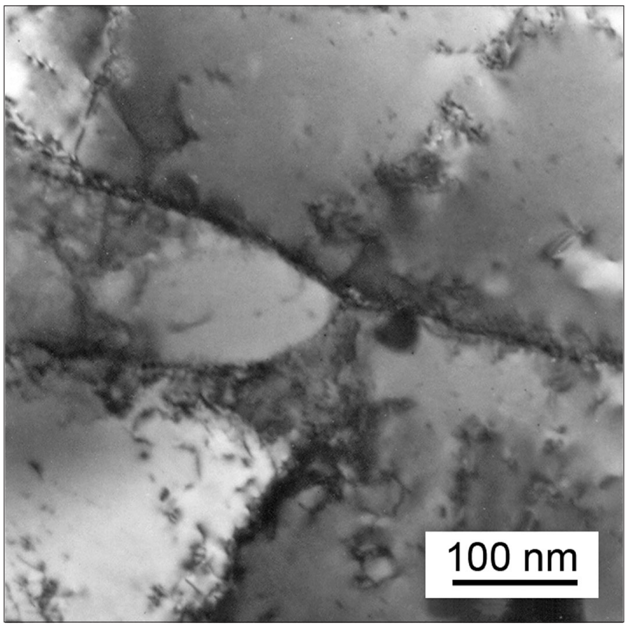

Figure 8. Nanograins with large angled random orientation

deformation lock the dislocation movement and increase the strength. On the other side, the measured increase of plastic properties shows that the nanograins can slide or move by rotation, which can result in high deformation up to a superplastic behaviour. Rotation of grains in Pd deformed by rolling was studied "in situ" by means of TEM, [11].

According to the deformation theory a ductile fracture mechanism is created by initiation, growth and cavities coalescence. Initiation of cavities is the most important. There are two dimples categories of transcrystalline ductile fracture analyzed at fracture surfaces of both materials (starting and after deformation) by means of SEM: large dimples, formed by decohesive mechanism in the

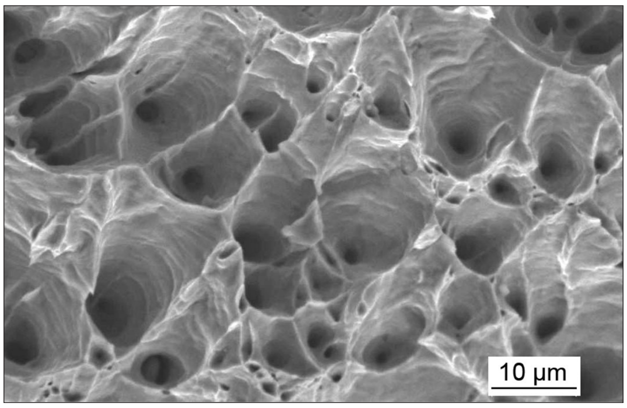

Figure 9. The fracture surface of the intial Cu material

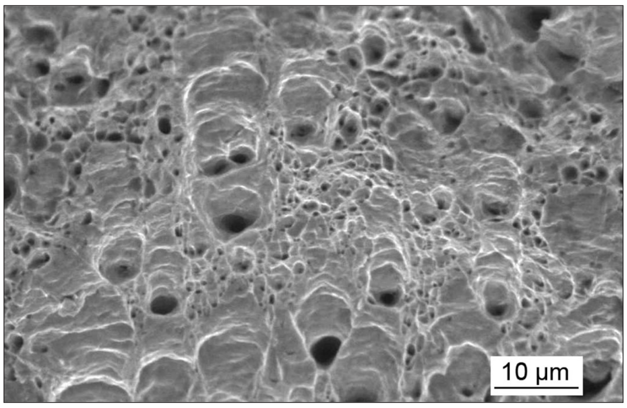

Figure 10. The fracture surface of the Cu material after 10 passes of ECAP

interphase inclusion / matrix and small dimples which are iniciated probably by dislocation mechanism. We assume, that mobile dislocation density in front of barrier - nanograin boundary is growing up with the deformation passes (1 -10). The coalescence of the dislocations into the multiple unstable crack dislocation which is the nucleus of cavity initiation is achieved. Following stages e. i. growth and cavity coalescence are going by the usual mechanism. Average size of the both dimple categories was measured from the cca 200-300 dimples. Dependence of dimple size on ECAP passes is listed in Table 2. Dimples size decreased with the deformation process from 5,2 to 1,1 μm, Figure 9 and Figure 10.

Table 2. Dimple sizes for the initial and the ECAP passed Cu material

Number of passes	0	3	5	7	10
Dimple size [μm]	5,4	3,1	2,3	1,9	1,1

CONCLUSIONS

The following conclusions can be made from experiment:

1. The changes in grain size and yield strength followed in some range the Hall-Petch equation.
2. The SPD resulted in an increase of both strength and ductility.

3. The mean grain size decreased with increasing deformation, after 10 passes to 100 – 300 nm.
4. TEM analysis suggested the possible nanostructure formation mechanism by the formation of cellular structure in grains, forming of subgrains and then forming of high angle nanograins with random orientation.
5. Fractures of ECAP Cu material had transcrystalline ductile character with dimple morphology. Size of dimples decreased with the deformation passes from 5.2 to 1.1 μm,

Acknowledgements – The authors are grateful to the Slovak Research and Development Support Agency (Project No. APVV-20-027205) the Slovak Grant Agency for Science (Grant No. 2/5142/25) for the support of this work.

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Note: The responsible translator for Englisch Language is K. Sulleiová, Institute of Materials Research of Slovak Academy of Sciences, Košice, Slovakia