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Mechanical properties and fracture of nanocopper by severe plastic deformations

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Abstract. The development of the nanostructure in commercial pure copper, its strength and ductility after severe plastic deformation (SPD) with the technology of equal-channel angular pressing (ECAP) are analysed. It is shown that both the strength and ductility can be increased simultaneously by SPD. The final grain size decreased from the initial 50 μm to 100–300 nm after 10 passes of ECAP. The increase of the ductility and strength, caused by SPD, is explained by strong grain refinement and by the dynamic equilibrium of weakening and strengthening.

Key words: severe plastic deformation, equal-channel angular pressing, Cu, nanostructured materials, microstructural parameters.

1. INTRODUCTION

A lot of research has been devoted to the development of new nanostructured materials. Nanomaterials 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 has been developed to prepare nanostructured materials. Some of them are the powder metallurgy (PM) methods. The important stage in these methods is powder preparation and compacting. There are still persisting the problems with the residual porosity of PM materials, the problem of impurities and the grain growth in the following stages of production. Severe plastic deformation seems to be a more convenient way to solve the listed problems.

Production of the nanostructure in compact metallic systems has been studied in [1]. The equal-channel angular pressing is one of the possible choices. It consists of pressing of the experimental material through perpendicular to each other channels of a special die. The ECAP technology permits to obtain very fine grained microstructure or nanostructure of the material by multiple pressing trough the die.

The development of nanograins with high-angle boundaries in metals, alloys and specific substructures and the development of dislocations in cells and on grain boundaries has been investigated in [2–3]. Statistical evaluation of the heterogeneity of nanostructures, produced by plastic deformation, were described in [4]. Improved mechanical properties can be obtained by severe plastic deformation. High strength and ductility for some systems have been reported [1]. Extremely fine grains with high-angle boundaries were obtained, developing unique superplastic behaviour, explained by the mechanism of grain boundary sliding or by grain rotation [5–8].

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

2. EXPERIMENTAL MATERIAL AND METHODS

Commercial pure copper (99.9% Cu) was used as the experimental material. Bars of 10 mm in diameter 70 mm long were pressed using the ECAP die (Fig. 1) at room temperature. A bar was pressed 10 times. A single pressing can be evaluated as a relative deformation according to Fig. 2. The hydraulic press used

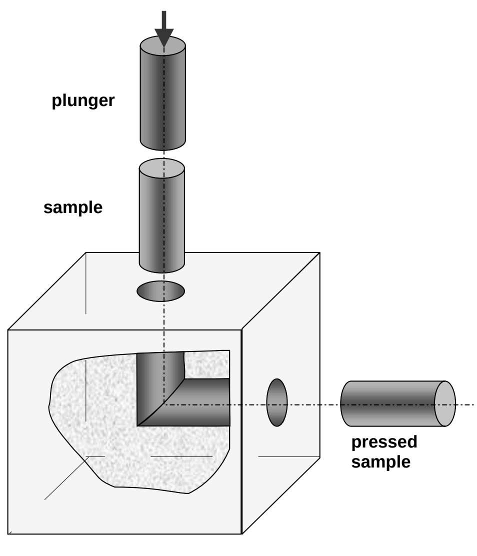

Fig. 1. Scheme of equal-channel angular pressing.

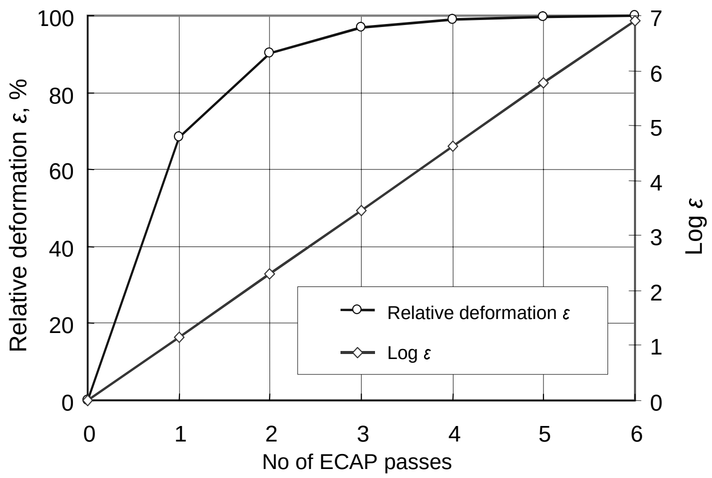

Fig. 2. Deformations during ECAP.

for ECAP is able to produce a load of 1 MN. The deformed bars were then machined to the form of test specimens (with a diameter of 3 mm, 15 mm long, M5) for static tensile testing, hardness testing, metallography and TEM analysis.

3. RESULTS AND DISCUSSION

Mechanical properties and grain size of asreceived pure copper are listed in Table 1.

The used copper is coarse grained with a mean grain size d_z of 50 μm, both the yield strength ($R_{p0.2}$) and ultimate tensile strength (UTS, R_m) are quite low, but the reduction of the area (Z) is significant (65%). Dependence of the change of the strength properties ($R_{p0.2}$, R_m and HV10) and ductility (represented by the reduction of the area (Z) on the number of ECAP passes) are shown in Fig. 3. By every pressing the test piece position was rotated about 180°. Cold working is known to produce an increase in strength and the value of R_m increased after 10 passes from 375 to 464 MPa. The hardness HV10 increased after 10 passes to 128.

The reduction of the 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 at failure. It is important to note that the reduction of the area increases with the number of passes similarly to the strength. It is

Table 1. Mechanical properties and grain size of Cu

$R_{p0.2}$, MPa	R_m, MPa	A_5, %	Z, %	HV	d_z, μm
270	275	13	65	72	50

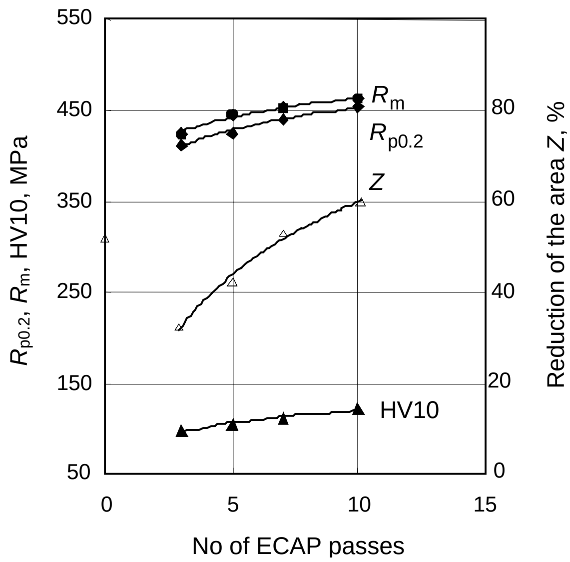

Fig. 3. Dependence of the strength and plastic properties of copper on the plastic deformation by ECAP.

quite different from the classical behaviour of metals after plastic deformation. Ductility is of great importance for high strength nanomaterials.

We have also analysed the obtained microstructure. The initial grain structure of Cu, obtained in optical microscope, is presented in Fig. 4. The grains are equiaxed and the structure is homogeneous. After 3, 5, 7 and 10 passes the grain size decreases (Fig. 5). In Fig. 5, the mean size was calculated taking into account about 100 grains. Although the bars were rotated after every pressing, the grain structure is not homogeneous any more. 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 the properties. Significant changes in the strength can be caused only by grains with high-angle boundaries. After the maximal plastic deformation (10 passes), the grains are at the limit of the resolution by metallography (Fig. 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 the formation of a cellular structure and the formation of subgrains, which are transformed into nanograins with a high angle of random orientation during increasing deformation (Figs. 7 and 8). The most important phase is the period of change from the cellular structure to high-angle grain structure. As it is supposed in [2], cell walls are thinning and dislocations are reordered. It is obvious that the nanograin boundaries are not in equilibrium for the absorbed large deformations of the cellular structure and for the high dislocation density. The ultrafine grains with

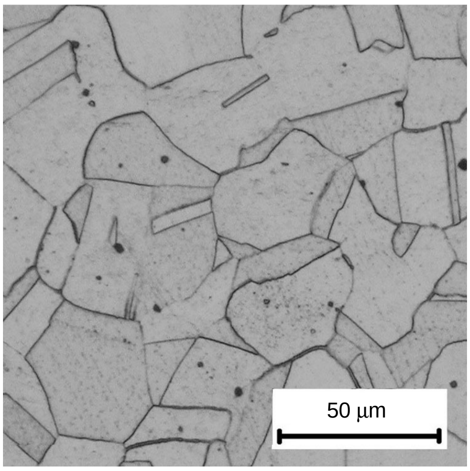

Fig. 4. Microstructure of initial Cu.

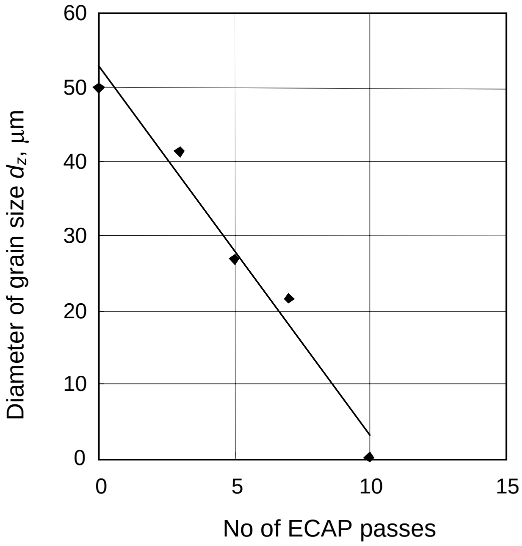

Fig. 5. Decrease of the grain size after 3, 5, 7 and 10 passes of ECAP.

high-angle boundaries by the following deformation lock the dislocation movement and increase the strength. From 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. However, superplasticity is a process controlled by diffusion, favored more at higher temperatures and explains why the grain sliding at room temperatures takes place. It is supposed that the unbalanced grain boundaries with the large amount

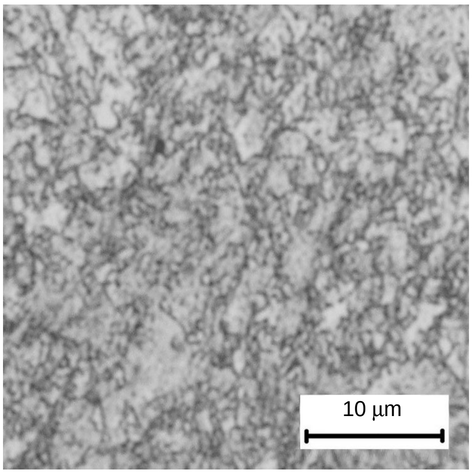

Fig. 6. Microstructure of Cu after the maximal plastic deformation (10 passes).

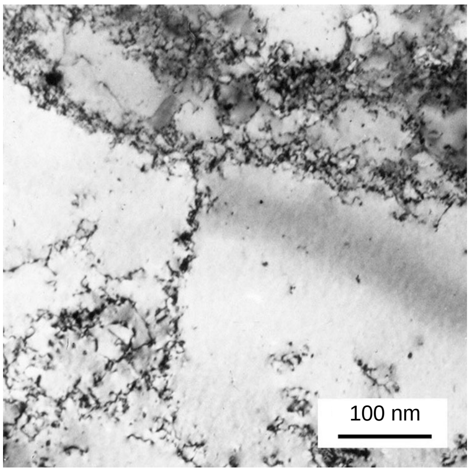

Fig. 7. Forming of subgrains during the deformation process.

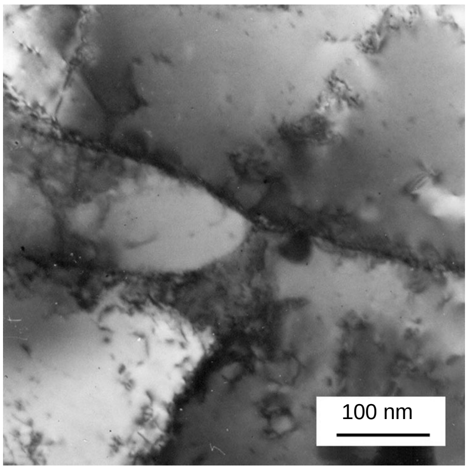

Fig. 8. Nanograins with high-angled random orientation.

of accumulated energy and high far-reaching stress gradients can support sliding along grain boundaries at the room temperature. Our results support this assumption. Load–deformation curve after 10 passes is shown in Fig. 9. The curve shows a straight part with the dynamic equilibrium of softening and strengthening, as typical for superplastic behaviour.

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 categories of dimples of transcrystalline ductile fracture, analysed at fracture surfaces of both materials (initial and after deformation) by means of SEM: large dimples, formed by the decohesive mechanism in the

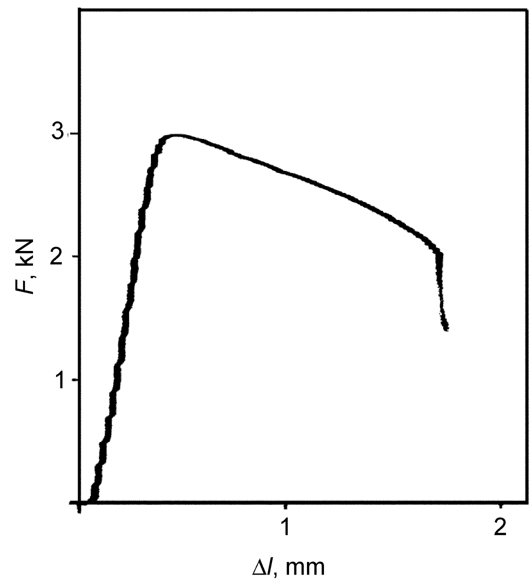

Fig. 9. The load (F) – deformation (Δl) curve.

inclusion–matrix interphase and small dimples which are initiated probably by the dislocation mechanism. We assume that mobile dislocation density in front of the barrier–nanograin boundary is growing up with the deformation passes (1–10). Transform of the dislocations into the multiple unstable crack dislocation, which is the nucleus of cavity initiation, is achieved. Following stages, i.e. growth and cavity coalescence, follow the usual mechanism. Average size of the dimples of both categories was measured from about 200–300 dimples. Size of the dimples is decreased with the deformation passes from 5.2 to 1.1 μm. Fracture surfaces of the Cu material before the test as well as after the maximal plastic deformation (10 passes) are shown in Figs. 10 and 11.

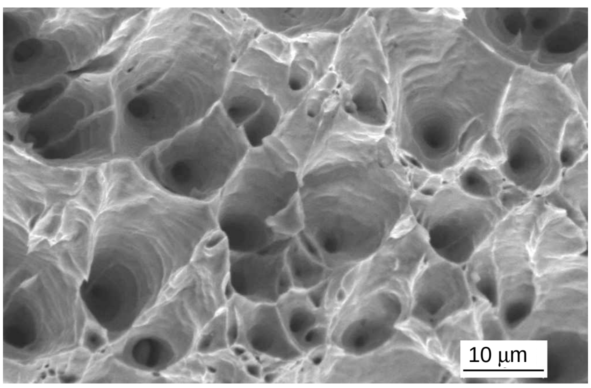

Fig. 10. Fracture surface of initial Cu.

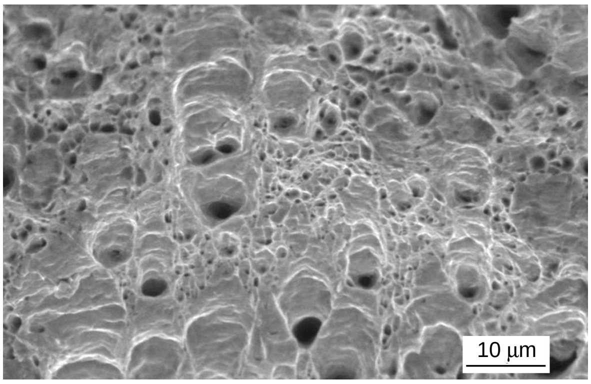

Fig. 11. Fracture surface of Cu after the maximal plastic deformation (10 passes).

4. CONCLUSIONS

The following conclusions can be made.

1. The SPD resulted in an increase both of the strength and ductility.
2. The mean grain size decreased with increasing deformation. After 10 passes of ECAP, the average size of the grains was reduced to 100–300 nm.
3. The load–deformation curve of the deformed test specimen after 10 passes showed a straight part, revealing superplastic-like deformation, sliding and grain rotations. This can explain the increase of the area reduction in tensile tests after severe deformation.
4. TEM analysis suggested a possible nanostructure formation mechanism by the formation of cellular structure in grains, forming of subgrains and then forming of high-angle nanograins of random orientation.
5. Fracture of the initial Cu material as well as of the material after plastic deformations had the transcrystalline ductile character. The size of dimples decreased with the deformation passes from 5.2 to 1.1 μm.

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Nanostruktuurse vase mehaanilised omadused ja purunemine sügavplastsel deformatsioonil

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On vaadeldud nanostruktuurse puhta vase sügavplastse deformatsiooni (SPD) mõju võrdkanalilisel nurkekstrudeerimisel (ECAP) materjali tugevusele ja sitkusele. On tõestatud materjali tugevuse ja sitkuse samaaegse tõstmise võimalus SPD abil, kusjuures tera suurus väheneb kümnekordse ECAP protsessi tagajärjel 50 μm-lt 100–300 nm-ni. Tugevuse ja sitkuse tõusu SPD tulemusena võib seletada terade järsu peenenemise ja tugevnemise/nõrgenemise protsessi dünaamilise tasakaaluga.