Nanostructures and mechanical properties developed in copper by severe plastic deformations

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Abstract

The development of the nanostructure in commercial pure copper and the strength and ductility after severe plastic deformation (SPD) with the technology of equal channel angular pressing (ECAP) are analysed. Experimental results and analyses show that both strength and ductility can be increased simultaneously by SPD. The final grain size decreased from the initial 50 μm by SPD to 100–300 nm after 10 passes. An increase of the ductility together with an increase of strength caused by SPD is explained by a strong grain refinement and by a dynamic equilibrium of weakening and strengthening, and it is visible on the final static tensile test stress-strain charts.

K e y w o r d s: SPD, ECAP, Cu, nanostructured materials, mechanical properties, microstructural parameters

1. Introduction

A lot of research work is 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 were developed to prepare nanostructured materials. Some of them are the powder metallurgy (PM) methods. The important phase in these methods is the powder preparation [1, 2] and compacting [2, 3]. 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 [4, 5]. The equal channel angular pressing (ECAP) can be the choice. It is pressing of the experimental material through two right-angled channels of a special die. The ECAP technology allows to obtain the very fine grained microstructure – the nanostructure by multiple pressings through the die. The development of high angle nanograins in metals and alloys, with specific substructures, arranging dislocations in cells and on grain boundaries was studied and analysed [6–9]. Statistic evaluations of the heterogeneity of nanostructures produced by plastic deformation were described [10, 11]. Improved mechanical properties can be obtained by severe plastic deformation. High strength and ductility were reported for different systems [12, 13]. Extremely fine grains with high angle boundaries were obtained, developing unique superplastic behaviour [14, 15] explained by the mechanisms of grain boundary sliding or by the grains rotation [16, 17]. Microstructure and properties of nanomaterials are described in [18–23]. Some new approaches on deformation mechanisms of nanostructured materials are described in [24].

The aim of this work is to analyse the mechanical properties of copper 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

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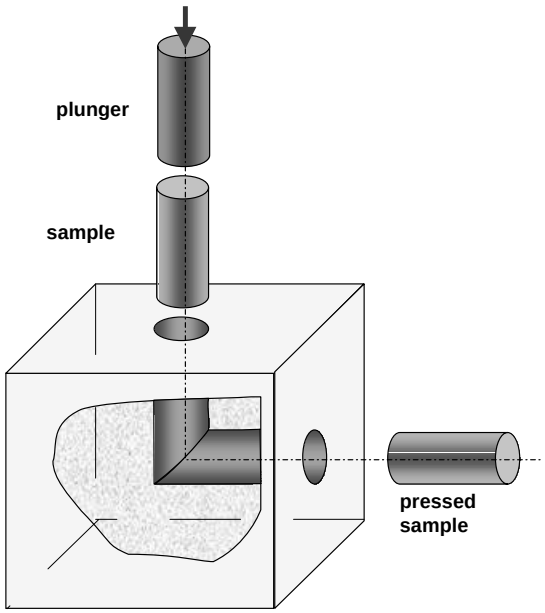

Fig. 1. Scheme of ECAP.

an experimental material. Bars in dimensions of 10 mm in diameter and 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 for ECAP is able to produce a load of 1 MN. The deformed bars were then machined to the form of test specimens (ø 3 mm, 15 mm long, M5) for static tensile testing, hardness testing, metallography and TEM analysis by thin foils.

Table 1. Mechanical properties and grain size of Cu material

$R_p0.2$ [MPa]	R_m [MPa]	A_5 [%]	Z [%]	HV 10	d_z [μm]
270	275	13	65	72	50

3. Results and discussion

The initial values of mechanical properties and grain size of pure copper are in Table 1. 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 (UTS, R_m) are quite low, but the reduction of area (Z) is significant (65 %). The change of strength properties ($R_p0.2$, R_m and HV 10) in dependence on the number of ECAP passes is in Fig. 3. At every next pressing the test piece position was about 180° rotated. Cold working is known to produce an increase in strength and the UTS value after 10 passes increased from 275 MPa to 464 MPa. The hardness HV 10 increased after 10 passes to 128. According to Schiotz [8] we have analysed the dependence of hardening expressed by the yield strength $R_p0.2$ on the grain size using the Hall-Petch equation, Fig. 4. The other hardening contributions were neglected: the Peierls-Nabbaro, substitution and dislocation caused strengthening (R_{PN}, R_S and 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 de-

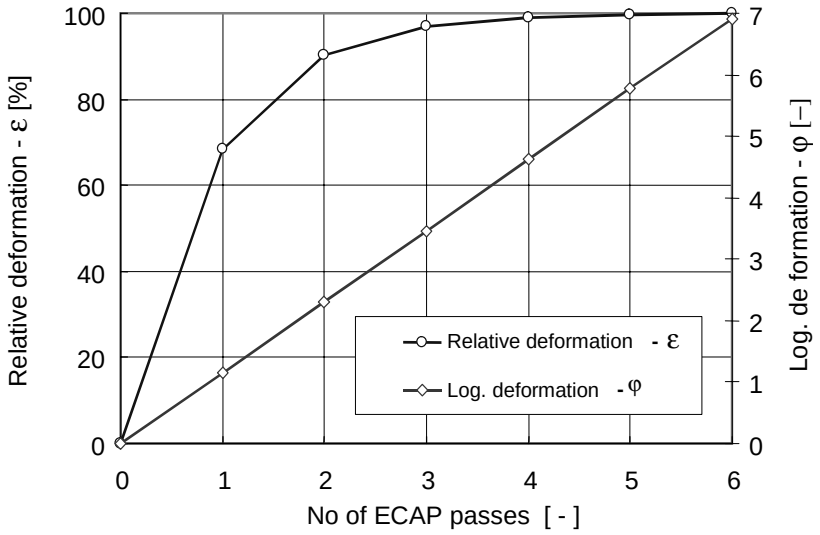

Fig. 2. Deformations during ECAP.

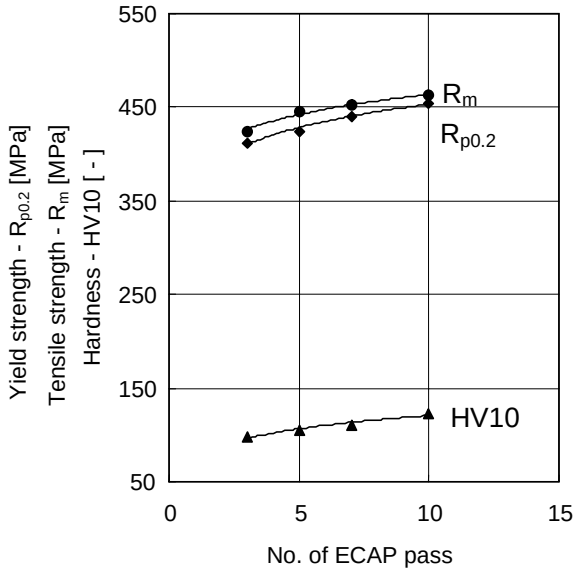

Fig. 3. Strength properties ($R_p0.2$, R_m and HV 10) in dependence on the plastic deformation by ECAP.

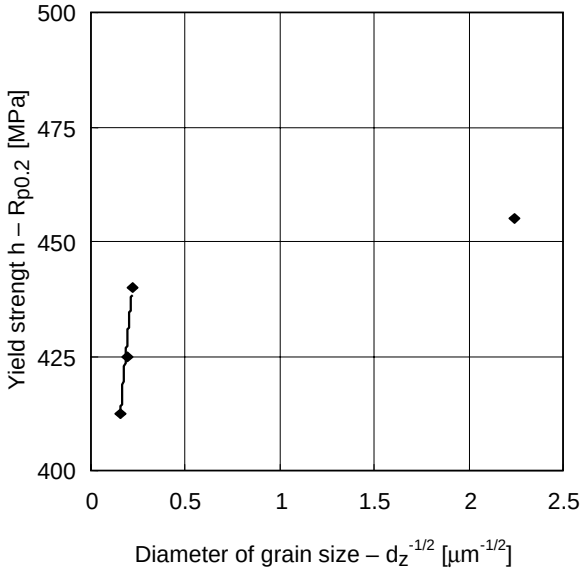

Fig. 4. Dependence of hardening expressed by the yield point $R_p0.2$ on the grain size using the Hall-Petch equation.

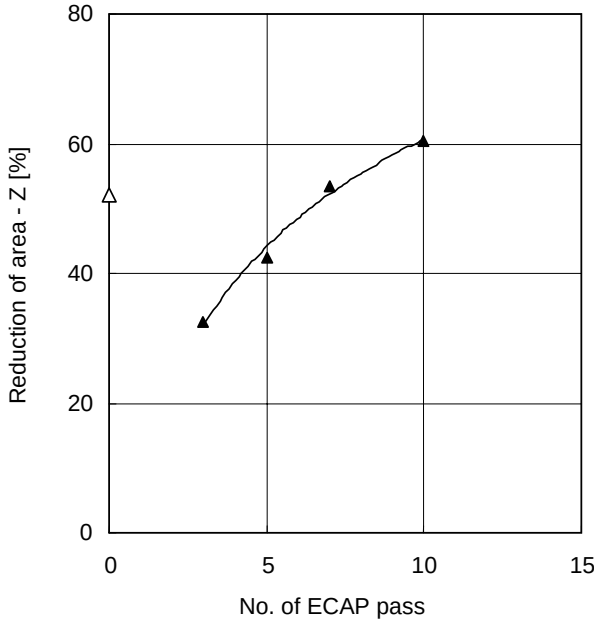

Fig. 5. The dependence of ductility on the deformation represented by the reduction of area (Z).

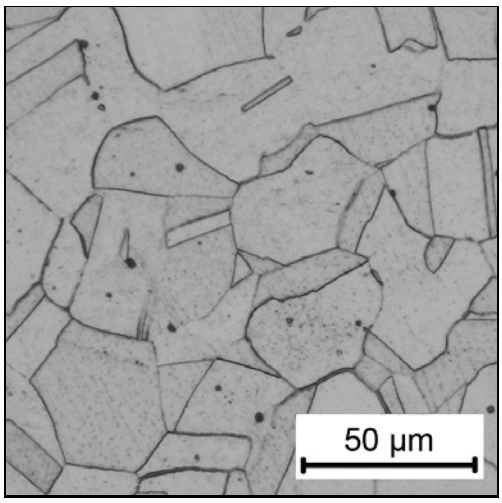

Fig. 6. Microstructure of initial Cu material.

viation agreed with the simulation results and experiments of Schoitz [8].

The dependence of ductility represented by the reduction of area (Z) on the number of passes is presented in Fig. 5. 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 [7], but not with Koch's report [20] 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 analysed the obtained microstructure. The initial grain structure of Cu is presented in Fig. 6 made by light microscope. The grains are equiaxed and the structure is even. After 3, 5, 7 and

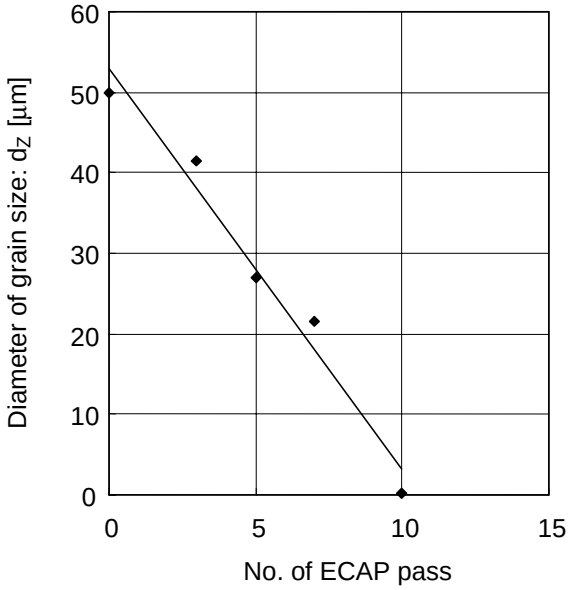

Fig. 7. Decrease of the grain size after the ECAP passes 3, 5, 7, and 10.

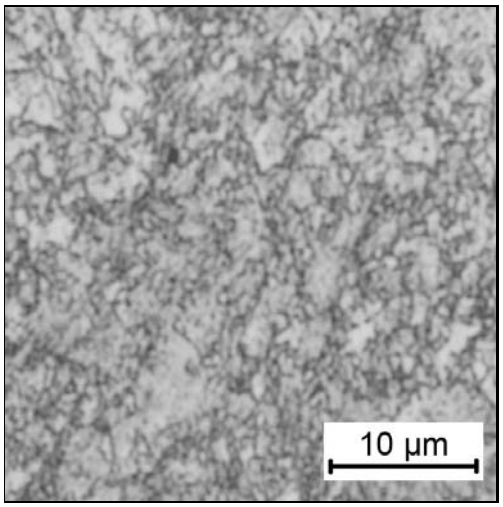

Fig. 8. Microstructure of Cu material after the maximal plastic deformation (10 passes).

10 passes the grain size decreased, Fig. 7. The mean values in Fig. 7 were calculated from about 100 grains each. Though the rotation of the bars after every pressing was applied, 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 – 10 passes, the grains are on the limit of the resolution by metallography, Fig. 8. The mean grain

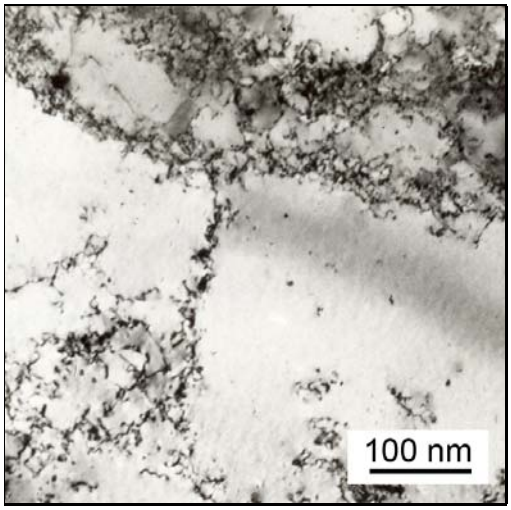

Fig. 9. Forming of subgrains during deformation process.

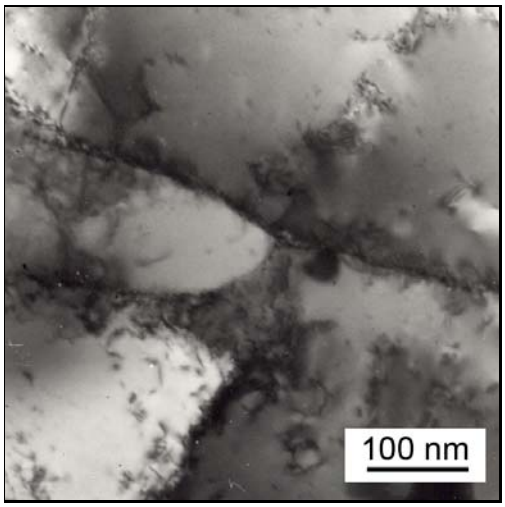

Fig. 10. Nanograins with large angled random orientation.

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, Figs. 9 and 10. The most important phase is the period of change from the cellular structure to high angle grain structure. As described in [7], it is supposed, that the cell walls are thinning and reordering dislocations. 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. We suppose, that the ultra fine grains with high angle

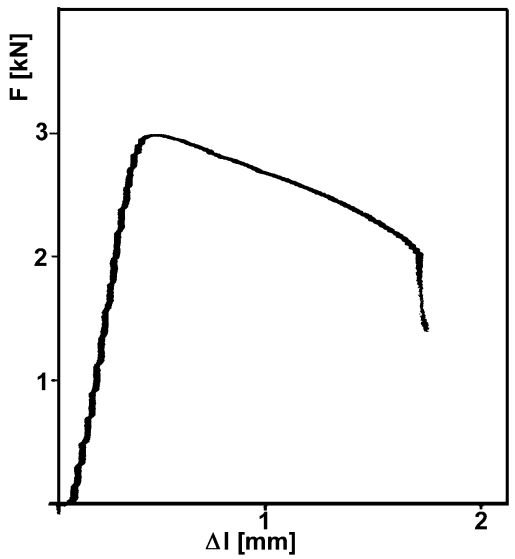

Fig. 11. The load (F)-deformation (Δl) curve.

boundaries at the following 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 [16]. However, superplasticity is a process controlled by diffusion, favoured more at higher temperatures and the question is, why the grain sliding at room temperatures takes place. It is supposed, the unbalanced grain boundaries with the large amount of accumulated energy and high far-reaching stress gradients can support sliding along grain boundaries at low temperatures, too [21]. Our results with the experimental material confirmed this assumption, as well. Load (F)-deformation (Δl) curve after 10 passes is shown in Fig. 11. The curve shows a straight part with the dynamic equilibrium of softening and strengthening – a sign known for superplastic behaviour.

4. Conclusions

The following conclusions can be made:

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. The load-deformation curve of the deformed test specimen after 10 passes showed a straight part, showing superplastic-like deformation mechanism. We suppose sliding and grain rotation. This can explain the increase of the area reduction in tensile tests after severe deformation.

5. 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.

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