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MICROSTRUCTURE AND HARDNES OF COPPER AFTER PRESSING BY ECAP

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ABSTRACT
The questions about plastic flow of materials at square extrusion through the die (ECAP) were monitored parameters in sequence with following cold rolling in this paper. Prior extrusion samples were subjected to heat treatment for strain hardening as well as original microstructure suppressing. Final microstructures in company with strengthening processes are very important factors influencing the product manufacture qualities. In paper is main attention gazed on utilization of ECAP technology as a possible preliminary way to reach more advantageous mechanical values for next forming by classical technologies. That is why in experimental was applied first ECAP and then cold rolling whereas this connected processing embodied interesting results passing from cold as well as hot tensile tests.
Keywords: commercial pure copper, structure, properties

1. INTRODUCTION
New forming technologies, to which the ECAP (Equal Channel Angular Pressing) technology belongs as well, are focused on refining of grains by intensive plastic deformations. The objective consists in fabrication of structural metallic materials with ultra-fine grain with higher mechanical properties. These structures promise achievement higher mechanical properties in comparison with their coarse-grain equivalents. These concepts are based in particular on notion of validity of the Hall-Petch relationship $\sigma_f = \sigma_o + k.d^{-1/2}$ till the sphere of grains of nanometric dimensions. Another serious problem consists in increase of resistance of fine-grain materials to growth of grain during its treatment at higher temperature or at re-heating to higher temperature, which is in many cases imperative for forming processes and for achievement of the required functional properties of products. Fine-grain materials are the materials, the structure of which consists of components, which have at least one dimension within the range between 100 – 500 nm, these materials are also called ultrafine-grains materials.

2. GRAIN SIZE AND MECHANICAL PROPERTIES OF ULTRAFINE-GRAIN MATERIALS
The fact that strength (hardness) of material increases with decreasing grain size in its structure was known from the early fifties of the last century, when famous Hall-Petch relationship was formulated :

$$Re = \sigma_o + k.d^{-1/2} \quad (1)$$

where

Re is yield value, σ_o is the stress necessary for overcoming of Peierls-Nabarro friction stress, resistance of dissolved foreign atoms, resistance of precipitates from solid solution and lattice defects, k is the constant, the measure of which is the value of shearing stress necessary for release of accumulated dislocations, d is the grain size.

It can be calculated that for grain sizes between 10 – 20 nm the yield value approaches the theoretical material strength. Validity of the relationship (1) has been proved experimentally, with the exception of its validity for large grains and for very fine grains (approx. below 10 nm).
Fine-grain materials are characterised by high density of grain boundaries and other interfaces, which leads to a notion of validity of functioning of high-temperature deformation mechanisms respecting the role of grain boundaries into the zone of lower temperatures. For example at significantly lower homological temperatures the ultrafine-grain materials („nano-crystalline") materials will be deformed by processes, which are controlled by diffusion along grain boundaries [1,2].

2.1. Grain growth and temperature stability of structure
Nano-crystalline materials have due to small size of grains and large surface area a tendency to grain growth. Knowledge of temperature stability of nano-crystalline materials is important from theoretical and technological reasons. From theoretical point of view it will be interesting to compare mechanism of grain growth in nano-crystalline materials and in coarse grain (conventional) materials. Grain growth in conventional materials is described by the equation :

$$\Delta D = D^n - D_o^n = K_o \exp\left(-\frac{Q}{RT}\right)\cdot\tau \qquad (2)$$

where D is grain size after annealing of sample at the temperature T during the time $\tilde{\tau}$, D_o is the initial size of grain, n is exponent of grain growth, K_o is constant, Q is activation energy of grain growth and R is gas constant.

3. EXPERIMENTAL VERIFICATION OF THE ECAP TECHNOLOGY ON COPPER
Achievement of ultrafine-grain or nano-crystalline structure requires true (logarithmical) deformation of approx. 6 – 14, and forming carried out at low homological temperatures. The paper focuses only on preparation of ultrafine-grain Cu with use of severe plastic deformation, particularly with use of the ECAP technology, Fig. 1.

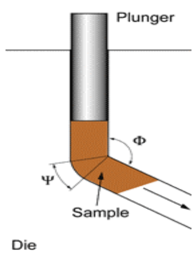


Figure 1. Diagram of ECAP processes

Total accumulated true deformation can be then calculated from the relation:

$$\varepsilon = \frac{2n}{\sqrt{3}}\cdot ctg\left(\frac{\Phi}{2}\right) \qquad (3)$$

where n is number of passes.

The experiments were aimed at determination of extrusion force, pressure necessary in individual stages of extrusion, change of strength properties in dependence on number of extrusions and change of structure. Experiments were made on equipment, the possibilities of which were already presented. We have used for extrusion the copper grade in accordance with the Czech standard ČSN 42 3003. Original samples were processed by cold forming and they were afterwards annealed at temperature of 870 ^{o}C/1h. Initial shape of the samples and shapes of samples after individual stages of extrusion are shown in Fig. 2.

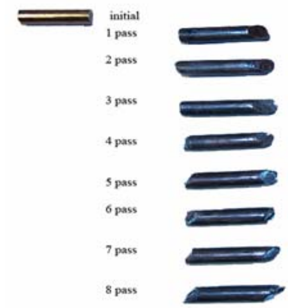
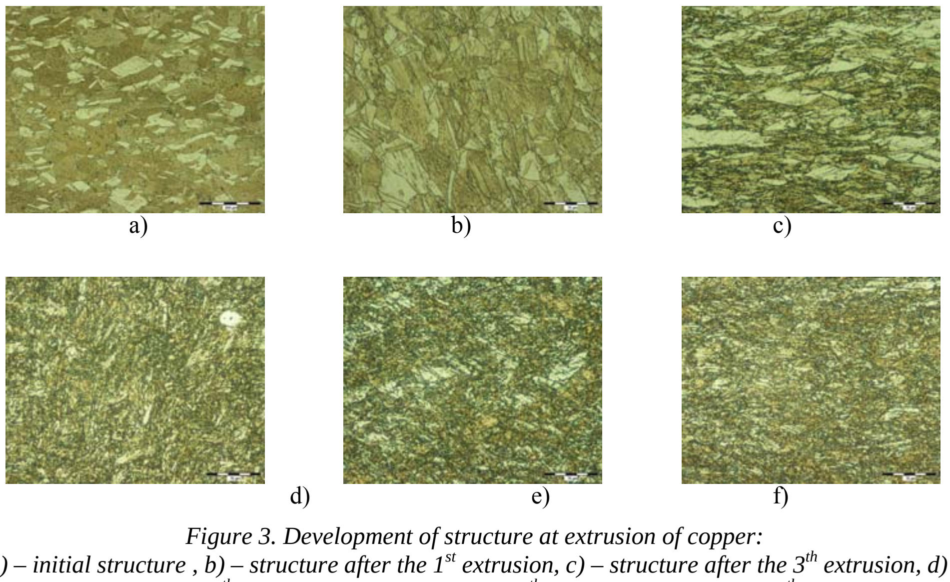


Figure 2. Copper samples after individual passes with use of the ECAP technology

Section of original samples was ϕ 12 mm. The samples were extruded at temperature of approx. 20 ^{o}C. In order to increase concentration of deformation in volume of the sample the samples were after individual passes turned around their longitudinal axis by 90^{o} and they were extruded again. The samples are ordered from the left to the right according to number of passes.
At extrusion were measured the deformation forces and pressure needed for extrusion was calculated. The strain rate was approximately determined, which was approx. 2,3 .10^{-2} s^{-1} [3, 4]. Structure analysis was made by optical microscopy. Structure of original samples and that of samples after individual stages of extrusion is shown in Fig. 3.



a) b) c)

d) e) f)

Figure 3. Development of structure at extrusion of copper:
a) – initial structure , b) – structure after the 1st extrusion, c) – structure after the 3th extrusion, d) - structure of the 5th extrusion, e) structure after 6th extrusion and f) after 8th extrusion

Individual grains were elongated by main deformation in longitudinal direction. Average grain size in transverse direction was determined by quantitative metallographic methods and it varied around 50 μm at the beginning of extrusion, and around 5 μm at the end of extrusion, i.e. after the 8th passes . HV hardness changed in dependence on number of passes through a die – see Fig. 4.

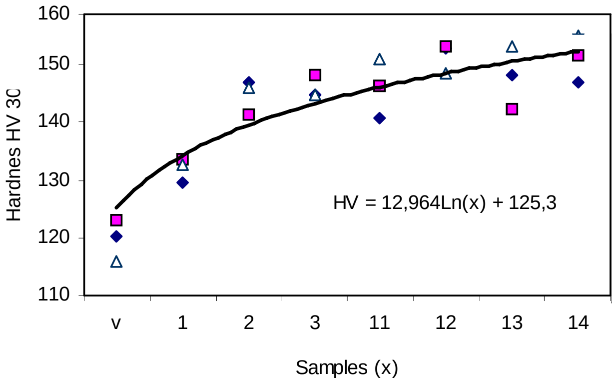


Figure 4. Hardness of individual samples after extrusion

3.1. Obtained results and their analysis

After individual passes there has occurred an accumulation of deformation strengthening, e.g. at extrusion with radius of rounding of inside cants (R = 0,5) the extrusion pressure at the beginning varied around τ_1 = 658 MPa. At the second extrusion it increased to τ_2 = 965 MPa, and at the third extrusion it increased to τ_3 = 1188 MPa. Increase in pressure at constant dimensions of samples corresponds to the increase if deformation resistance. Distinctively higher values of resistance to deformation and also strengthening at extrusion are related to high absolute value of octahedral stress.

4. CONCLUSION

Experiments made on poly-crystalline copper of the grade 42 3003 have confirmed that the ECAP method is efficient tool for refining of grain. This process enabled obtaining of grain size of matrix of approx. 5 µm. Microstructure depends of experimental conditions, particularly on number of passes and on rotation of the sample between individual passes. Convenient angle between horizontal and vertical part of extrusive channel is around 90°. Radii of rounding of working parts of extrusive channel must correspond to conditions for laminar flow of metal.

5. ACKNOWLEDGEMENTS

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