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# MICROSTRUCTURE OF ULTRAFINE-GRAINED METALS AFTER ECAP

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**ABSTRACT**
*This paper was aimed at verification of functionality of the ECAP technology at extrusion of the copper, alluminium alloys and steel. Experiments were made on equipment, which is demonstrated in the Fig 1. Deformation forces were measured during extrusion, resistance to deformation was calculated and deformation speed was determined approximately. Analysis of structure was made with use of light microscopy and TEM. The samples of Cu and Al alloys were extruded at room temperature. For the samples of steel was used the two-stage pressing, when the samples were extruded at temperature of approx. $T_1$ = 325 $^0$C and $T_2$ = 220 $^0$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. Cross-section of original samples of Cu and Al alloys was 8 x 8 mm and their length was 32 mm and cross-section of original samples of Fe alloy was 10 x 10 mm and their length was 40 mm .*
**Keywords :** Materials, Nanomaterials, ECAP, microstructure, mechanical properties

## 1. INTRODUCTION
New forming technologies, to which the ECAP technology (Fig. 1) belongs as well, are focused on refining of grains by intensive plastic deformations [1,2]. The objective consists in fabrication of structural metallic materials with ultra-fine grain with higher mechanical properties**.**

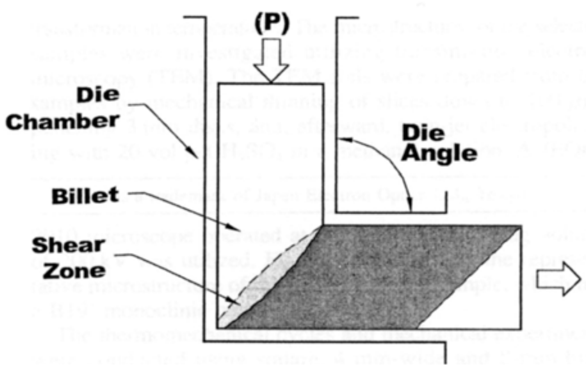



*Figure 1. Schematic of the of ECAP process*

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-grain materials). From the viewpoint of strength properties these components can be represented by sub-grains, grains, lamellas, layers, fibres, etc. For example lamellar pearlite can be considered as nano-composite material, which is formed by ferrite and cementite lamellas with width mostly below 100 nm. The value 100 nm does not have a physical meaning [3]. The term ultrafine-grain material is used also for materials composed of particles below 1 micrometer.

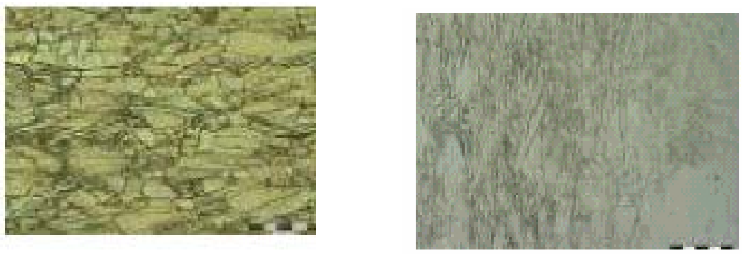
## 2. EXPERIMENTAL TECHNIQUES

The experiment was divided in the three parts. In the first part of experiment was pressed the cooper grade C 10200 (ASTM B152), in the second part was extruded aluminium alloy AlCu2.5Mg and in the final part was pressed the steel P355Q. The chemical composition of all alloys is demostrated in the Table 1. Cross-section of original samples of Cu and Al alloys was 8 x 8 mm and their length was 32 mm and cross-section of original samples of Fe alloy was 10 x 10 mm and their length was 40 mm. The samples of Cu and Al alloys were extruded at room temperature. For the samples of steel was used the two-stage pressing, when the samples were extruded at temperature of approx. $T_1 = 325\ ^0C$ and $T_2 = 220\ ^0C$. 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.

*Table 1. Chemical composition of alloys*

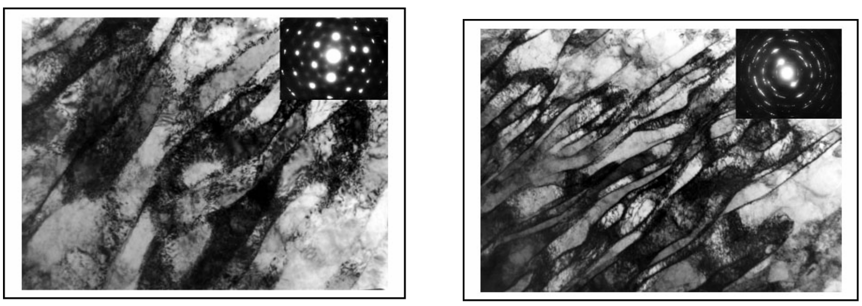
| Alloys | Chemical compositions (%) | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | C | Si | Mn | P | S | Cu | Cr | Ni | Al | Mo | V | Ti | B |
| P355Q | 0.028 | 0.040 | 0.27 | 0.00 9 | 0.015 | 0.06 | 0.06 | 0.03 | 0.004 | 0.013 | 0.004 | 0.177 | 0.005 |
| C 10200 | 0.005 | - | - | 0.003 | 0.005 | 99.95 | - | 0.002 | - | - | - | - | - |
| AlCu2Mg | - | 0.26 | 0.14 | - | - | 2.1 | - | - | 97,5 | - | - | - | - |

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. In the first part of the experiment we have used for extrusion the copper grade C 10200. Original samples were processed by cold forming and they were afterwards annealed at temperature of 600 $^oC$/3h. Initial shape of the samples and shapes of samples after individual stages of extrusion. The samples were extruded at temperature of approx. 20 $^oC$. The samples are ordered from the left to the right according to number of passes. We have measured at extrusion the deformation forces and we have also calculated the pressure needed for extrusion. We have approximately determined the strain rate, which was approx. $2,3\ .10^{-2}\ s^{-1}$ [4]. Structure analysis was made by optical microscopy. Structure of original samples and that of samples after individual stages of extrusion is shown, Fig.2.



*a) b)*

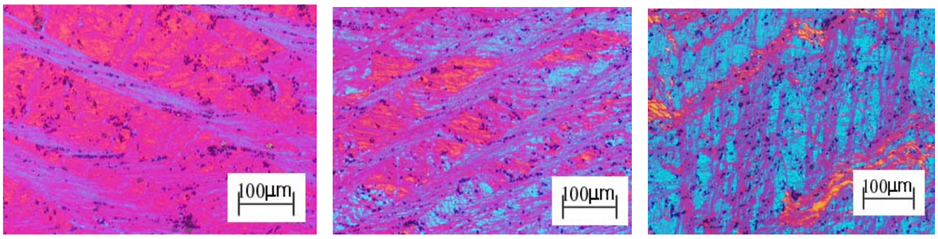
*Figure 2. Development of structure (in longitudinal direction) at extrusion of copper: a – initial structure, b – structure after the 4$^{th}$ extrusion*



*a) b)*

*Figure 3. The substructure of cooper after 1$^{st}$ (a) , 4$^{th}$ pases (b)*

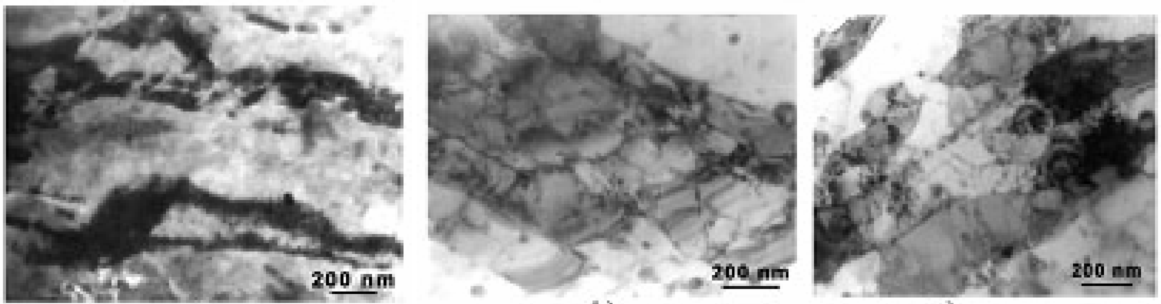
Average grain size in transverse direction was determined by quantitative metallographic methods and it varied around 50 µm at the beginning of extrusion, and around 15 µm at the end of extrusion, i.e. after the 4$^{th}$ pass [5]. In the second part of the experiment was pressed Al alloy AlCu2Mg. The samples were extruded at room temperature. Before pressing the samples were annealed at temperature of 380 $^{0}$C. Structure of original samples and that of samples after individual stages of extrusion is shown in Fig. 4. Average grain size in transverse direction varied around 150 µm.




a) b) c)

*Figure 4. Development of structure (in longitudinal direction) at extrusion of AlCu2Mg: a – structure after 1$^{st}$ extrusion, b – structure after 2$^{nd}$ extrusion, c – structure after 4$^{th}$ extrusion*

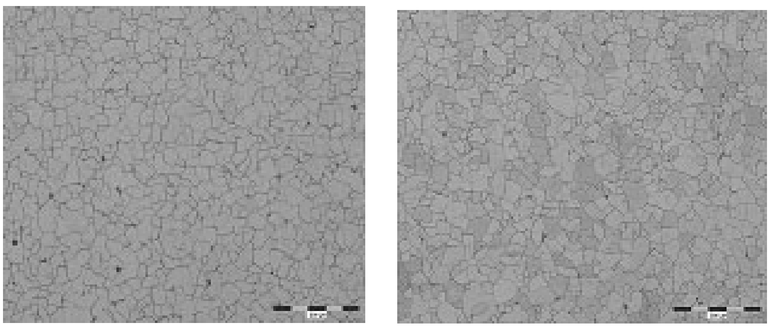
The modification of sample form and sustening solid metal in particular periods of pressing depends on the pasting level and the radius fillet of edges in the pressing channel. During pressing in the channel with a small radius fillet working edges the splits are coming up in the whole lenth of the pressing channel. After particular through pass happened to the comulation of the deformation`s consolidation, which was the basic in the creating substructure. It is demonstrated in Fig. 5.




a) b) c)

*Figure 5. The substructure of AlCu2Mg alloy after 1$^{st}$ (a), 3$^{rd}$ (b) and 4$^{th}$ pases (c)*

In the last part of experiment was pressed steel P355Q. Before pressing the samples were annealed for ECAP. The temperature of anneal was 350 $^{o}$C and the idle period of the anneal temperature was 30 min. After annealing the metallografical examination of structure was made (Fig. 6).



*a) b)*

*Figure 6. Structure of steel P355Q after annealing: a) in longitudinal direction, b) in transverse direction*

## 3. OBTAINED RESULTS AND THEIR ANALYSIS

**For copper:** 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 $\tau_1$ = 658 MPa [6,7]. At the second extrusion it increased to $\tau_2$ = 965 MPa, and at the third extrusion it increased to $\tau_3$ = 1188 MPa.

**For aluminium:** At extrusion with radius of rounding of inside cants ($R_v$ = 2 mm; $R_{vn}$ = 5 mm) the extrusion pressure in die was after the first pass about $\tau_{max}$ = 620 MPa and then increased. After the 4th extrusion was about $\tau_{max}$ = 810 MPa [8].

**For steel:** During pressing the press power was moving due to depending on degree of fulling the die channel. For the 1th sample was $F_{max}$ = 92 kN, for the 2nd sample was $F_{max}$ = 95 kN and for the 3th sample was $F_{max}$ = 123 kN. These powers correspond to these stresses: 1438 MPa, 1484 MPa and 1922 MPa. The press power was going up with growing deformation (the hardening sample). The stability properties are going up with size of deformation (e = 3.54) and during four passes raise double [9]. The tensibility is going down. It is caused due to recovery processes, which were not passed. The tensibility is going up due to softer seen.

## 4. CONCLUSION

Experiments made on poly-crystalline copper of the grade C10200, on Al alloy AlCu2Mg and on steel P355Q have confirmed that the ECAP method is efficient tool for refining of grain. Microstructure depends of experimental conditions, particularly on number of passes and on rotation of the sample between individual passes. The angle between horizontal and vertical part of extrusive channel was for this experiment around $90^0$ for Cu and Al, for steel around 105°. Radii of rounding of working parts of extrusive channel must correspond to conditions for laminar flow of metal.

## 5. ACKNOWLEDGEMENTS

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