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# Grain refining of Cu and Ni-Ti shape memory alloys by ECAP process

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**Properties**

## ABSTRACT

**Purpose:** This paper was aimed at verification of functionality of the ECAP technology at extrusion of the copper and Ni-Ti alloys.
**Design/methodology/approach:** Cross-section of original samples was 8 x 8 mm and their length was 32 mm. The samples of Cu were extruded at room temperature. For the samples of Ni-Ti alloys was used the two-stage pressing, when the samples were extruded at temperature of approx. $T_1$ = 520°C and $T_2$ = 420°C. In order to increase the concentration of deformation in volume of the sample, the samples were turned around their longitudinal axis by 90 o after individual passes and they were extruded again. Analysis of structure was made by using of light microscopy.
**Findings:** Deformation forces were measured during extrusion, resistance to deformation was calculated and deformation speed was approximately determined. After individual passes there it occurred an accumulation of deformation strengthening ($\tau_3$= 745 Mpa).
**Research limitations/implications:** Microstructure depends on experimental conditions, particularly on number of passes and on rotation of the sample between individual passes.
**Originality/value:** SPD techniques as ECAP proces can be used for non-forrous metals like Ti, Cu. It was approved that elvated temperature provide a succefully conditions for the fine grained final materials obtaining.
**Keywords:** Plastic forming; ECAP technology; Cu and Ni-Ti alloy; Mechanical properties

## 1. Introduction

The extrusion by ECAP method enables obtaining a fine-grain structure in larger volumes. Products made by this technique are characterised by high strength properties (Fig.1), and they can be potentially used at subsequent super-plastic forming. Magnitude of deformation, development of structure and resulting mechanical properties achieved by this technique depend notably on: homologous temperature $T_h$, size of grain $d_z$, deformation speed $\varepsilon^{\circ}$, homologous tension in die (σ/E), density of structural failures, on purity, etc. The obtaining required final structure depends primarily on geometry of tool, number of passes through die, obtained magnitude and speed of deformation, temperature, etc.

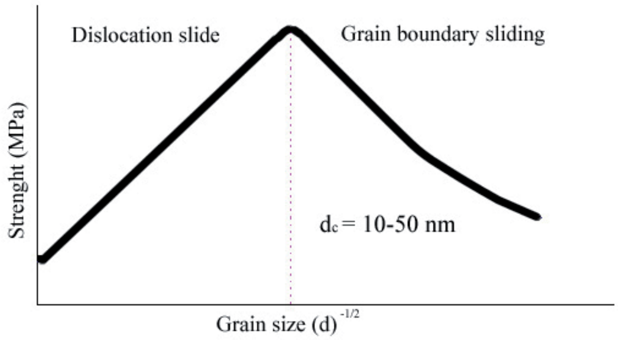



Fig. 1. Predicted dependence of strength on grain size [1]

## 2. Development of structure

Influence of magnitude of plastic deformation on properties of metallic materials is connected with increasing internal energy. Internal energy increases right to the limit value, which depends on manner of deformation, purity, grain size, temperature, etc. As a result of non-homogeneity of deformation at ECAP technique the internal energy gain differs at different places of formed alloy [1,2]. For example the value of internal energy is different in slip planes, at boundaries and inside cells. It is possible to observe higher internal energy also in proximity of precipitates, segregates and solid structural phases. For usual techniques, pure metals, medium magnitude of deformation and temperatures the value of stored energy is said to be approx. around 10 $J.mol^{-1}$ [3].

## 3. Experimental materials and procedures

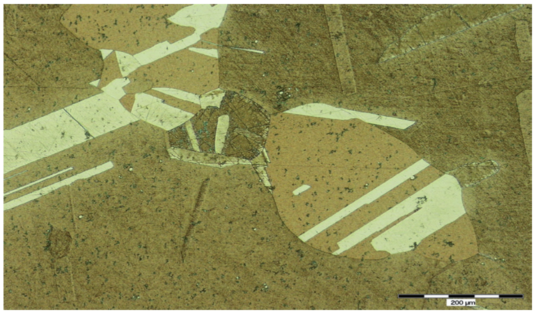
The experiment was divided into three parts. In the first part of experiment the cooper was pressed in the second part Ni-Ti alloy was extruded. Cross-section of original samples of Cu and Ni-Ti alloys was 8 x 8 mm and their length was 32 mm. The samples of Cu were extruded at room temperature. For the samples of Ni-Ti alloy 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 the concentration of deformation in volume of the sample, the samples were turned around their longitudinal axis by 90 $^o$ after individual passes and they were extruded again [4-6].

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 used for extrusion the copper. Original samples were processed by cold forming and they were afterwards annealed at temperature of 870 $^oC$/3h. Initial shape of the samples and shapes of samples as well as their microstrucutres after individual stages of extrusion are shown in Fig 2 and Fig.3, respectively.

Fig. 2. Copper samples after individual passes with using the ECAP technology

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 [7-10]. We measured at extrusion the deformation forces and we have also calculated the pressure needed for extrusion [11]. We approximately determined the strain rate, which was approx. 2,3 $.10^{-2}\ s^{-1}$. The structure analysis was made by optical microscopy. Structure of original samples and that of samples after individual stages of extrusion is shown on Fig. 3.



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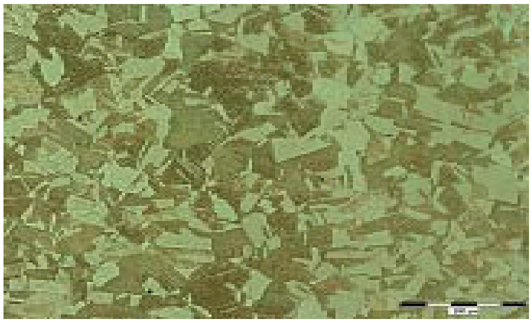

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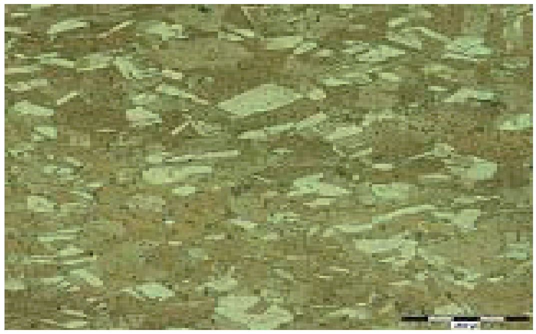
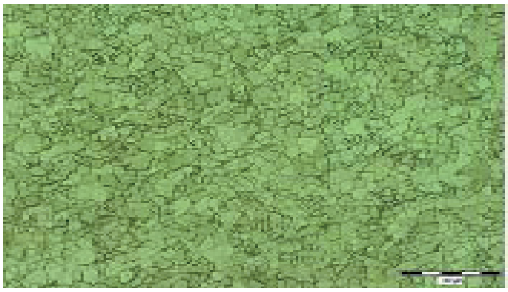


c)

Fig. 3. Development of structure (in longitudinal direction) at extrusion of copper: a – initial structure , b – structure after the 2$^{nd}$ extrusion, c – structure after the 3$^{rd}$ extrusion

After pressing the samples were annealed. The temperature of annealing was 650°C/30 min (Fig. 4). In the second part of the experiment Ni-Ti alloy was pressed. Before pressing the samples were annealed for ECAP. The temperature of annealing was 850 $^oC$ and the idle period of the anneal temperature was 30 min. After annealing the metalografical examination of structure was made (Fig.5), the tensile test on the miniaturized samples and the hardness HV. The fortness of samples after annealing was moving about 308,7 MPa and the hardness from 100 till 104 HV [12]. The structure of Ni-Ti after annealing is more ferrite material with very little grains. The average size of seed is moving about 30 μm.



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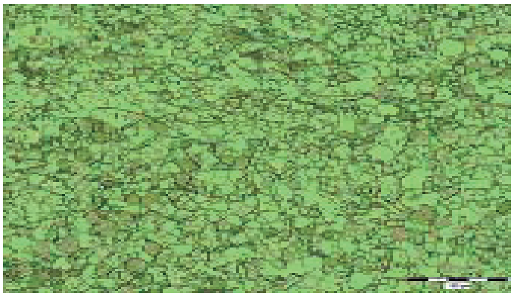

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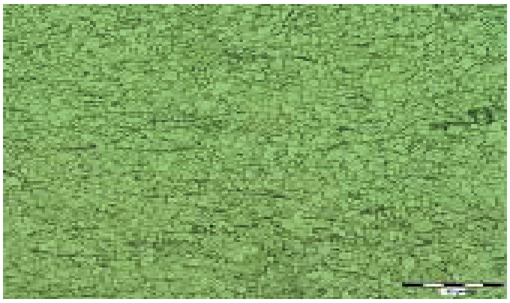


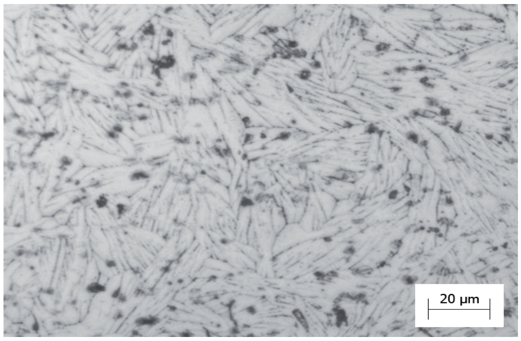
c)

Fig. 4. Development of structure (in longitudinal direction) at annealing after extrusion of copper: a) structure after the 4th extrusion, b) structure after 6th extrusion, c) structure after 9th extrusion

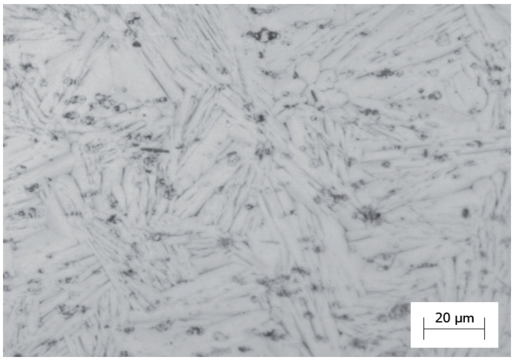
There were used the two-stage pressing, the samples were extruded at temperature of approx. $T_1$ = 520 $^0$C and $T_2$ = 420 $^0$C. Structure analysis was made by optical microscopy. Structure of original samples and that of samples after individual stages of extrusion is shown in Fig 5.

## 4. Experimental results

For copper: After individual passes there it 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 $\tau_1$ = 510 MPa. At the second extrusion it increased to $\tau_2$ = 615 MPa, and at the third extrusion it increased to $\tau_3$ = 745 MPa [13].




a)




b)

Fig. 5. Development of structure at extrusion of Ni-Ti alloy: a) initial structure, b) structure after the 1st extrusion and c) structure after the 3rd extrusion

For Ni-Ti alloy: During pressing the press power was moving due to depending on degree of fulling the die channel. For the 1th sample was $F_{max}$ = 90 kN, for the 2nd sample was $F_{max}$ = 95 kN and for the 3th sample was $F_{max}$ = 125 kN. The press on ejecting punch subject to these powers: 1435 MPa, 1485 MPa and 1920 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 [14-15]. The tensibility is going down. It is caused by recovery processes which were not passed. The tensibility is going up due to softer seen.

## 5. Conclusion

The experiments were made on poly-crystalline copper and on Ni-Ti alloy have confirmed that the ECAP method is efficient tool for refining of grain. Microstructure depends on 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 for Ni-Ti around 105°. Radii of rounding of working parts of extrusive channel must correspond to conditions for laminar flow of metal.

## Acknowledgements

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