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Influence of ECAP technology on the metal structures and properties

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ABSTRACT

Purpose: The purpose of the paper is the verification of functionality of the ECAP technology at extrusion of the copper, alluminium alloys, magnesium alloys and steel.

Design/methodology/approach: 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.

Findings: The samples of Cu and Al alloys were extruded at room temperature. For the samples of steel and AZ91 alloy was used the two-stage pressing, when the samples were extruded at temperature of approx. $T_1 = 325$ °C and $T_2 = 220$ °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° and extruded again.

Practical implications: Experiments on poly-crystalline copper of the grade C10200, aluminium alloy AlCu2.5Mg and steel P355Q confirmed that the ECAP method is efficient tool for refining of grain.

Originality/value: 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 steel and AZ 91 alloy was 10 x 10 mm and their length was 40 mm.

Keywords: Nanomaterials; ECAP; Microstructure; Mechanical properties

MATERIALS

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 [1-6]. The objective consists in fabrication of structural metallic materials with ultra-fine grain with higher mechanical properties.

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 [7]. 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.

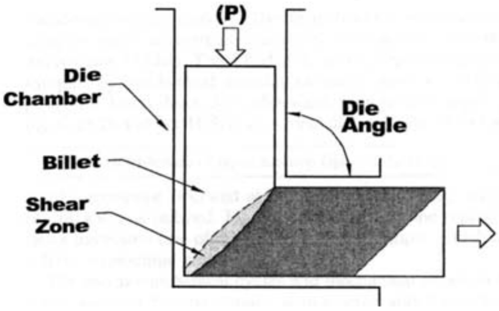


Fig. 1. Schematic of the of ECAP process [1]

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-stagepressing, 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.

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 ^{o}C/3h. Initial shape of the samples and shapes of samples after individual stages of extrusion are shown in Fig 2.

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

The samples were extruded at temperature of approx. 20 ^{o}C. 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}. 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. The substructure of ECAPed Cu samples after 1 and 4 pass is shown on Fig 4.

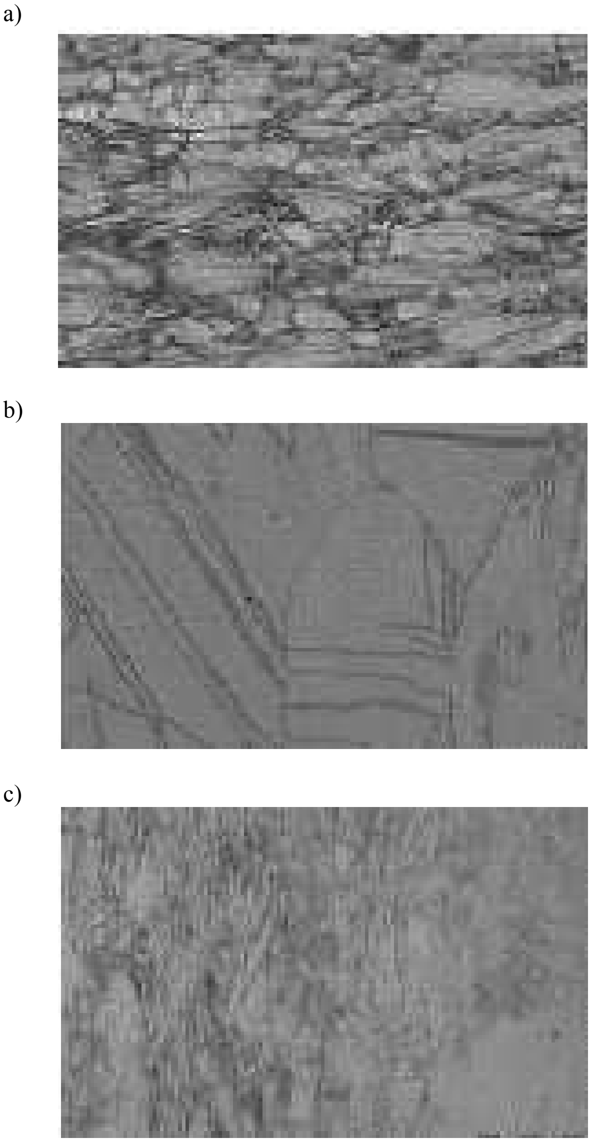


Fig. 3. Development of structure (in longitudinal direction) at extrusion of copper: a – initial structure , b – structure after the 1st extrusion, c – structure after the 4th extrusion

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 4th pass.

In the second part of the experiment was pressed Al alloy AlCu2.5Mg. The samples were extruded at room temperature. Before pressing the samples were annealed at temperature of 380 ^{0}C [8, 9]. Structure of original samples and that of samples after individual stages of extrusion is shown in Fig.5. Average grain size in transverse direction varied around 150 μm.

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	-	-	-	-	-
AlCu2,5Mg	-	0.25	0.12	-	-	2.6	-	-	97	-	-	-	-

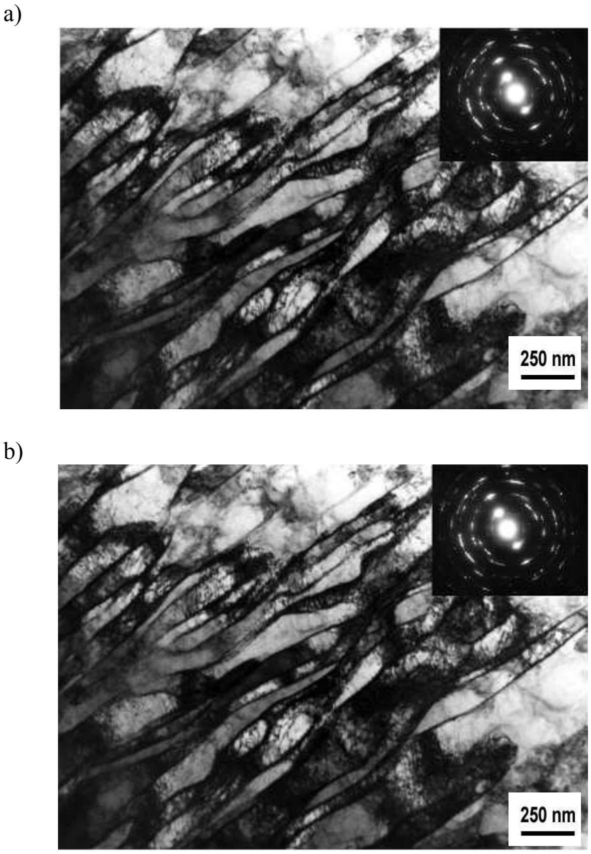


Fig. 4. The substructure of cooper after 1th (a) , 4th pases (b)

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 [10, 11]. During pressing in the channel with a small radius fillet working edges the splits are coming up in the whole lenght of the pressing channel.

Another factor, which influences significantly flow stress and development of microstructure is the angle Φ, which is formed by axis of vertical and horizontal channel. Smaller angle Φ leads to higher shearing stress at each pass. We have checked the size of the angle Φ in the range from 90° to 125° with use of technological route B_C. We have ascertained, that refining of grains is the most efficient (under the same magnitude of deformation), at the angle of 90°. This is given by the fact that two slip planes in the sample make in this case the angle of 60°. For materials, forming of which is more difficult, it is more advantageous to apply the angle Φ = 120° together with higher extrusion temperature.

After particular through pass happened to the comulation of the deformation`s consolidation, which was the basic in the creating substructure [12]. It is demonstrated in Fig 6.

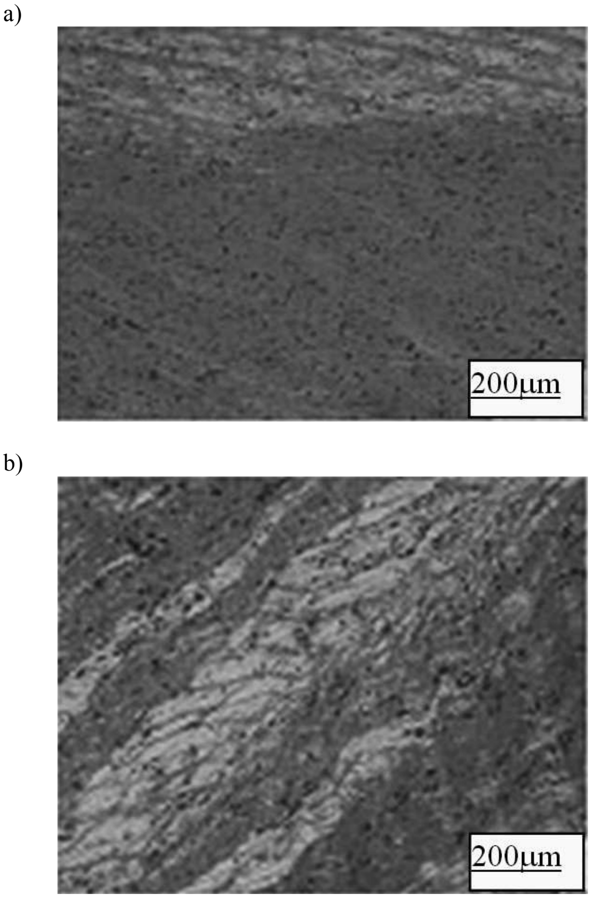


Fig. 5. Development of structure (in longitudinal direction) at extrusion of AlCu2.5Mg: a – initial structure, b – structure after 4th extrusion

In the last part of experiment was pressed steel P355Q. Before pressing the samples were annealed for ECAP. The temperature of anneal was 500 °C and the dwell on anneal temperature was 30 min (Fig 7). After annealing the metallografical examination of structure was made (Fig. 8).

There were used the two-stage pressing, the samples were extruded at temperature of approx. $T_1 = 325^0$C and $T_2 = 220^0$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 9.

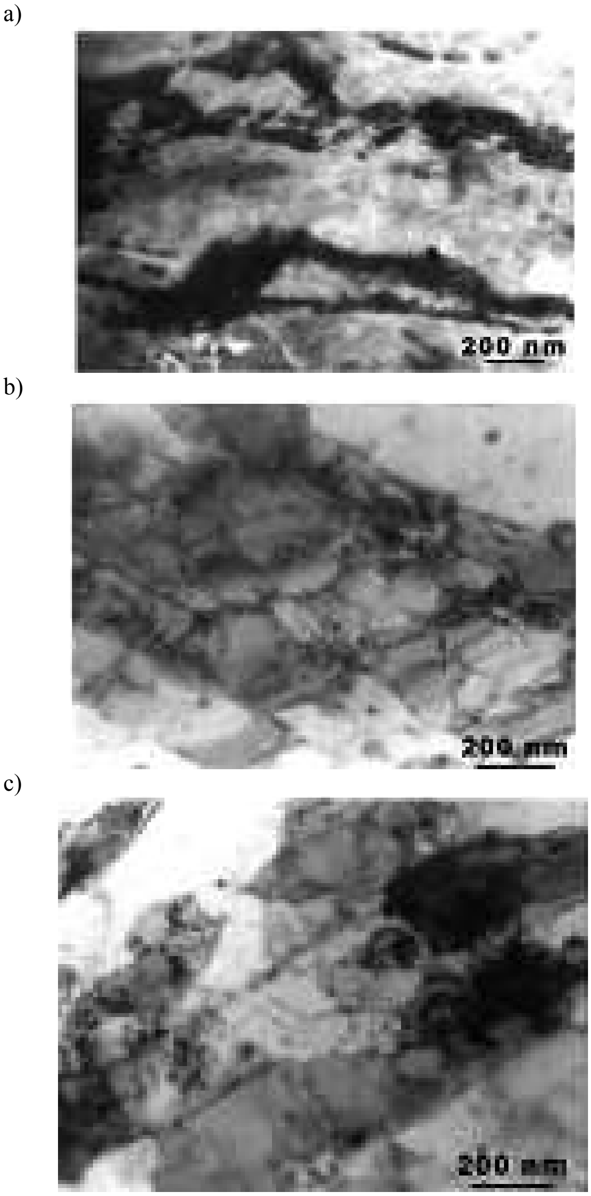


Fig. 6. The substructure of AlCu2.5Mg alloy after 1th (a), 3th (b) and 4th pases (c)

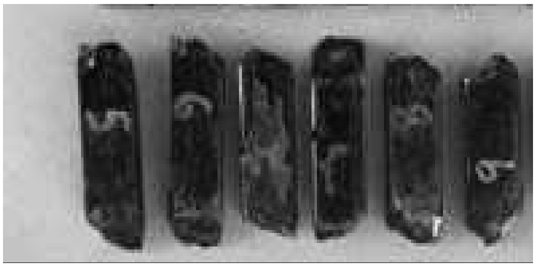


Fig. 7. Steel samples after individual passes with use of the ECAP technology

The structure along the cross section of sample is not equal with expressive areas of shearing deformation [13]. There are rising up with growing deformation (Fig. 9).

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 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.

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 τ_{max} = 620 MPa and then increased. After the 4th extrusion was about τ_{max} = 810 MPa. The samples after application of the ECAP technique, in the form of disc with diameter of 8 mm and thickness of 0.5 mm, were subjected to the penetration test at laboratory temperature. Basic mechanical properties were determined on the basis of penetration test, the principle of which consists in penetration of special punch with spherical surface through the flat disc-shaped sample, which is fixed between the upper holder and the lower die.

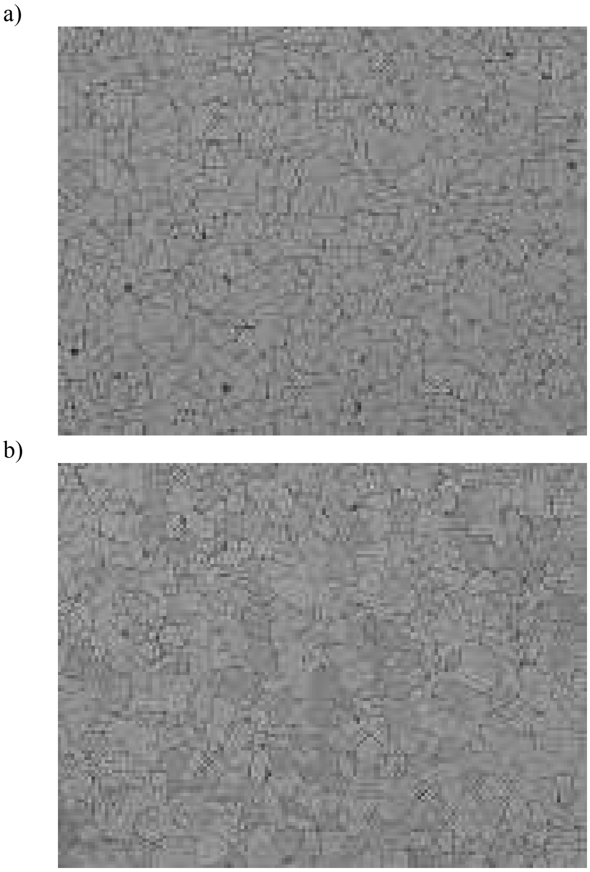
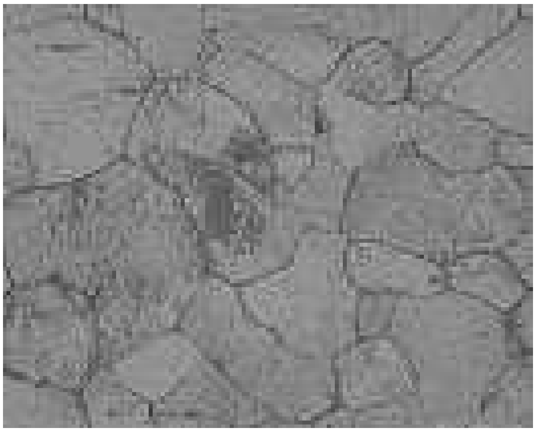


Fig. 8. Structure of steel P355Q after annealing: a) in longitudinal direction and b) in transverse direction

a)



b)

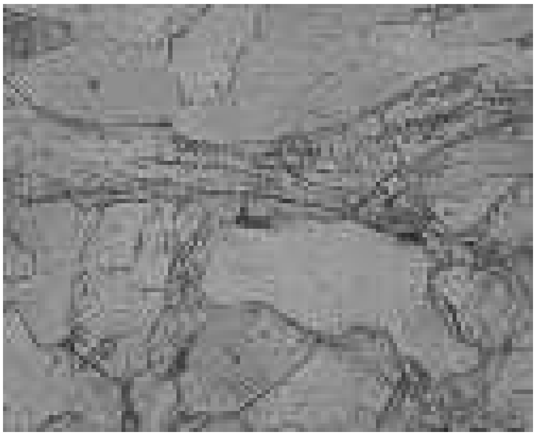


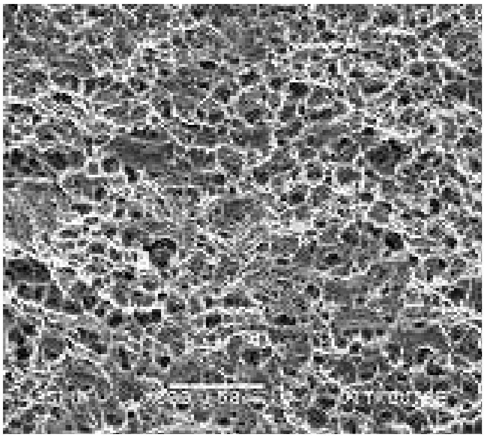
Fig. 9. Development of structure at extrusion of steel P355Q: a – structure after the 1th extrusion and b) structure after the 3th extrusion

On the basis of realised experiments it is possible to state that strength properties of the alloy 2024 obtained by penetration test vary in the range from Rm = 430 to 475 MPa, which demonstrates very good conformity with values of strength properties obtained by classical tensile test (Rm = 460 to 470 MPa).

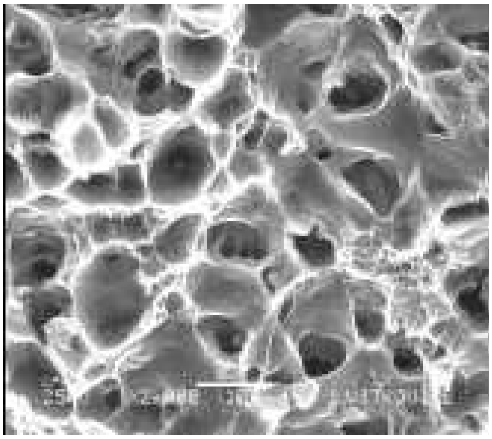
Analysis of fracture areas was made with use of scanning electron microscope JEOL – JSM 5510. From visual viewpoint the fracture area looked as planar and fine-grain with indistinctive shear fractures. It was determined by detail micro-fractographical observation that fracture area was formed exclusively by mechanism of trans-crystalline ductile failure with morphology of various pits – Fig. 10 a. These cavities contained big number of minuscule particles – Fig. 10 b, 10 c.

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. The tensibility is going down [14, 15]. It is caused due to recovery processes, which were not passed. The tensibility is going up due to softer seen.

a)



b)



c)

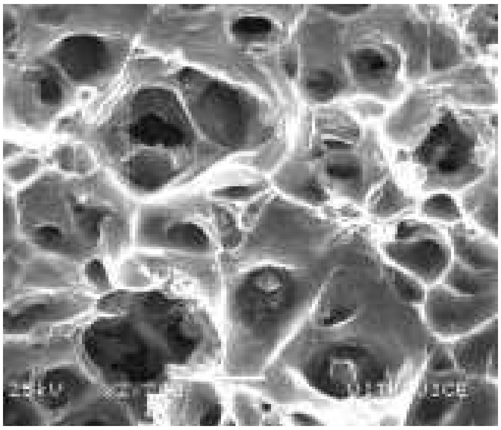


Fig. 10. Fracture areas in the alloy 2024

For magnesium alloy AZ91: Materials made of the alloy AZ 91+T4 (see Fig. 11,12), which were first rolled :

a) by single pass
b) by 3 passes with intermediate heating to rolling temperature

and then pressed were subjected to hot tensile test in order to determinate a possibility of super-plastic behavior. Equal channel angular pressing was made in two stages. The first stage consisted of 4 passes at the temperature 250°C. It was followed by the second stage consisting of 1 pass at the temperature 180°C. The samples were similarly as in previous cases re-heated to the chosen forming temperature in a muffle furnace with connected inert atmosphere Ar_2. After obtaining of the required temperature and a 5-minute dwell at this temperature material was charged into thermally insulated matrix with resistance heating, the temperature of which was identical to that of the chosen forming temperature.

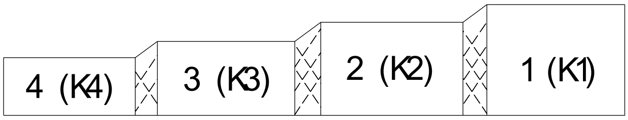


Fig. 11. Shape of samples prior to rolling

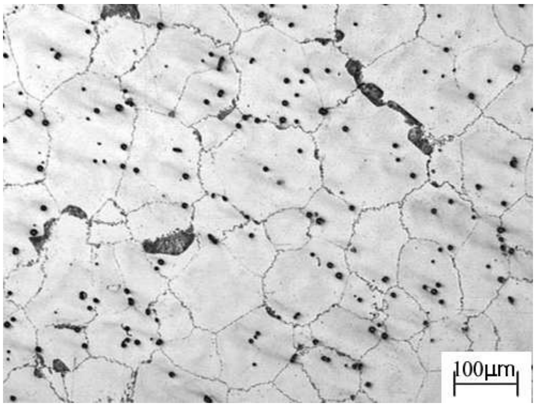


Fig. 12. Alloy AZ91 after T4

3.1. Superplasticity

Temperature used at the tensile test was 250 °C and strain rate was $\dot{e} = 2x10^{-4}$. The samples obtained after processing by ECAP technology were adjusted to the required shape and then subjected to the tensile test (Fig. 14), during which the set temperature was controlled by PID regulator which used a thermo-couple situated directly on the tested sample. Material rolled first in single pass (I, II, III) and then pressed, achieved elongation approx. 200 %, while materials first rolled by several passes and then pressed, achieved elongation up to 413 %. Before the tensile test microstructures of both groups did not differ from each other significantly.

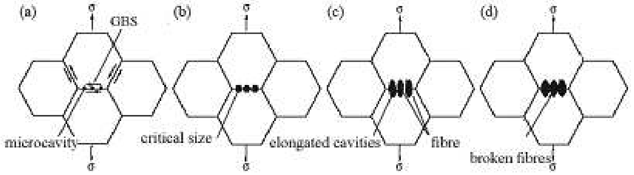


Fig. 13. Mechanism of rupture at superplastic forming

Due to the fact that slip along grain boundaries is under such conditions of deformation in these alloys one of main deformation mechanisms, it seems that micro-cavities were formed at the grain boundaries, which gradually grew into "O"-shaped cavities. These cavities afterwards protruded during plastic deformation as places of concentration of strain, and since they affected these original micro-cavities, as it can be seen in Fig. 13, there occurred elongation of edges of these cavities in the form of fibres, which broke after exceeding their strength. Resulting rupture that occurred at plastic deformation was caused by joining of individual cavities after breaking of elongated fibres.

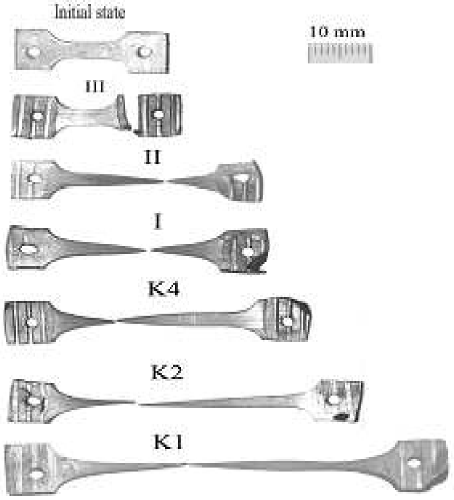


Fig. 14. Smples of AZ91+T4 alloy after ECAP and Hot tensile test

Table 2.
Values of strength and elongation for the alloy AZ 91+T4 after ECAP

Marking of sample	AZ 91 + T4	
	ΔL [%]	Rm [MPa]
I	294	15
II	286	19
III	-	-
K1	418	28
K2	384	32
K4	358	58,7

Table 2 gives obtained values of elongation in individual samples after hot tensile test, where there are apparent the mentioned differences between various methods of rolling applied prior to application of the ECAP technology, which has important influence on final plastic properties of obtained materials. An increase of plasticity with growing applied deformation can be observed at rolling by both methods, i.e. at rolling with single pass and rolling with several passes. In the latter case the obtained ductility was higher, which was probably caused by more homogenous structure obtained byre-crystallisation processes, which at this type of rolling could have developed more than at single-pass rolling.

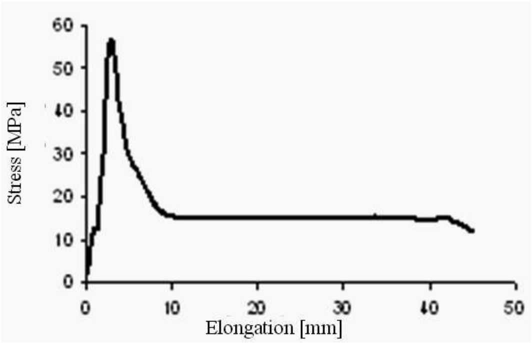


Fig. 15. Diagram of ECAPed AZ91 alloy after hot tensile test

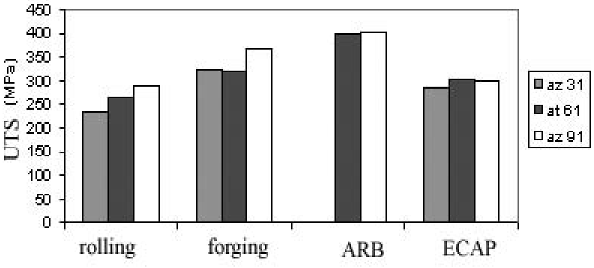


Fig. 16. UTS of Mg alloys at different technologies

Sample taken from the alloy AZ 91 elongated at the temperature of 250°C under constantly applied strain around the value of 15 MPa to rupture, as it is demonstrated in Fig. 15. Comparison of obtained strength in individual types of alloys after application of various forming technologies is shown in Fig. 16. It is evident, that the best method for obtaining the highest values of strength is the ARB process, however, this is achieved at the expense of plastic properties. Contrary to that the ECAP technology is an optimum compromise.

4. Conclusions

Experiments made on poly-crystalline copper of the grade C10200, on Al alloy AlCu2.5Mg 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.

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References

[1] I. Karaman, The effect of severe marforming on shape memory characteristics of a Ti rich NiTi alloy processed using ECAP, Metal Material Transaction 34A (2006) 2527-2539.

[2] A.M. Camacho, M. Marin, L. Sevilla, R. Domingo, Influence of strain hardening on forces and contact pressure distributions in forging processes, Proceedings of the 11th Contemporary Achievements in Mechanics, Manufacturing and Materials Science CAM^{3}S'2005, Gliwice-Zakopane 2005 (CD-ROM).

[3] M. Greger, S. Rusz. Possibilities of aluminium extrusion with use of the ECAP method. Proceedings of the IX International conference: Aluminium in transport 2003, Fotobit, Kraków, 2003, 165-169.

[4] I.J. Beyerlein, R.A. Lebensohn, C.N. Tomé, Ultrafine Grained Materials II. Proceedings of the International conference TMS, Seattle, 2002, 585-589.

[5] P. Kwapulinski. Magnetic properties of amorphous and nanocrystalline alloys based on iron, Journal of Materials Processing Technology 157-158 (2004) 735-742.

[6] L. Čížek, et al. Structure and properties of the selected magnesium alloys. Proceedings of the 10th International Scientific Conference "Achievements in Mechanical and Materials Engineering" AMME'2001, Gliwice-Zakopane, 2001, 75-78.

[7] J. Adamczyk, M. Opiela, Non-homogenity plastic strained austennite of high strenght low alloy steel, Proceedings 9th International Scientific Conference "Achievements in Mechanical and Materials Engineering" AMME'2000, Gliwice-Sopot-Gdańsk, 2000, 5-10.

[8] R.D.Herling, M.T. Smith, Superplastic microstructure of modified AA-5083 alluminum alloy processed by equal channel angular extrusion. Ultrafine Grained Materials. The Minerals, Metals and Materials Society, 2000, 83-87.

[9] H. Akamtsu, T. Fujinami, Z. Horita, T.G. Langdon. Influence of rolling on the superplastic behavior of an Al-Mg-Sc alloy after ECAP, Scripta Materialia 44 (2001) 759-764.

[10] M. Dollár, A. Dollár. On the strength and ductility of nanocrystalline materials. Journal Materials Processing Technology 153-158 (2004) 491-495.

[11] J.R. Davis, Metals Handbook, Desk. Edition, Second Edition, ASM International, The Materials Information Society, USA, 1998, 560- 564.

[12] B.K. Min, H.W. Kim, S.B. Kang, Influence of Al_3Sc precipitate on the formation of high angle grain boundary during accumulative roll bonding in Al-0.2wt% Sc alloy, Proceedings of the 13th International Scientific Conference on “Contemporary Achievements in Materials Science”. CAMS 2005, Gliwice-Wisła 2005, 115-119.

[13] T. Ungar, I. Alexandrov, M. Zehetbauer, Ultrafine- grained microstructures evolving during severe plastic deformation, Journal of Metallurgy 52/ 4 (2005) 34-36.

[14] J. Raab, Z. Ruslan, I. Valiev, T. Lowe, T. Yuntian, Continuous processing of ultrafine grained Al by ECAP- Conform, Materials Science and Engineering A 382 (2004) 30-34.

[15] R. Kocich, M. Greger, Structure characteristics after hot rolling of AZ91 alloy, 6. Scientific Conference “Material in Engineering”, Herlany, 2005, 81-86 (in Czech).

[16] M. Greger, R. Kocich, L. Čížek, L.A. Dobrzański, M. Widomská, A. Silbernagel, The structure and properties of chosen metals after ECAP, Journal of Achievements in Materials and Manufacturing Engineering 18 (2006) 103-106.