A R C H I V E S O F M E T A L L U R G Y A N D M A T E R I A L S
Volume 59 2014 Issue 1
DOI: 10.2478/amm-2014-0051

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EFFECT OF SEVERE PLASTIC DEFORMATION ON MICROSTRUCTURE AND PROPERTIES OF POLYCRYSTALLINE ALUMINIUM Al99.5

WPŁYW INTENSYWNYCH ODKSZTAŁCEŃ PLASTYCZNYCH NA ZMIANY MIKROSTRUKTURY I WŁAŚCIWOŚCI POLIKRYSTALICZNEGO ALUMINIUM Al99,5

Polycrystalline aluminium Al99.5 was deformed through the combination of equal-channel angular pressing (ECAP) by B route (4, 8 and 16 passes) and then by the hydrostatic extrusion (HE) using the cumulative way of deformation, just to the achieving the final wire diameter d = 3 mm. The microstructure of samples was investigated by means light microscopy (LM). Additionally the microhardness measurement and the tensile test were performed to determine the level of aluminium hardening. The texture was determined by using the Brucker Advance D8 equipment.

The aim of the research was to determine the influence of severe plastic deformation exerted in the process of equal-channel angular pressing (ECAP) and hydrostatic extrusion (HE) on the microstructure and properties of polycrystalline aluminium Al99.5.

The microstructure observations both after the HE process and the combination of ECAP + HE revealed the elongated to the extrusion direction grains and numerous bands and shear bands. The bands and shear bands most clearly revealed at the perpendicular section. The performed investigations showed that with the increase of the deformation the aluminium level hardening increase. The highest properties of tensile strength - UTS = 218 MPa and microhardness level HV0.1 = 46 were obtained after 8 ECAP + HE.

Keywords: aluminium, severe plastic deformation, texture, mechanical properties, microstructure

Polikrystaliczne aluminium Al99,5 odkształcono w procesie równokanałowego kątowego prasowania (ECAP) i kolejno wyciskania hydrostatycznego (HE). W procesie ECAP zastosowano 4, 8 i 16 przejść według drogi Bc, z kolei proces wyciskania hydrostatycznego prowadzono w sposób kumulacyjny, aż do osiągnięcia końcowej średnicy drutu d = 3 mm. Tak odkształcone aluminium poddano obserwacjom mikrostruktury przy zastosowaniu mikroskopu świetlnego. Dodatkowo przeprowadzono pomiary mikrotwardości próbek metodą Vickersa i przeprowadzono próbę jednoosiowego rozciągania w celu określenia umocnienia aluminium. Teksturę wyznaczono za pomocą dyfraktometru rentgenowskiego Brucker Advance D8.

Celem pracy było określenie wpływu intensywnych odkształceń plastycznych wywieranych w procesie wyciskania hydrostatycznego (HE) i równokanałowego kątowego prasowania (ECAP) na mikrostrukturę i właściwości polikrystalicznego aluminium Al99,5.

Obserwacje mikrostruktury Al99,5 zarówno po procesie HE, jak i po kombinowanym procesie ECAP + HE ujawniły występowanie wydłużonych ziaren w kierunku wyciskania oraz licznych pasm i pasm ścinania. Pasma ścinania najbardziej uwidoczniły się na przekroju prostopadłym do kierunku wyciskania. Przeprowadzone pomiary wykazały, że ze wzrostem odkształcenia następuje wzrost poziomu umocnienia aluminium. Po 8 przejściach ECAP i kolejno wyciskaniu hydrostatycznym uzyskano najwyższy poziom wytrzymałości na rozciąganie i mikrotwardości, odpowiednio: Rm = 218 MPa, HV0,1 = 46.

1. Introduction

Due to their special physical and mechanical properties, ultrafine- (UFG) and nano-grained (NG) materials produced by severe plastic deformation (SPD) methods have been subject of many research works [1-7]. SPD processes can be defined as metal forming process in which a very high level of strain is applied to the material during processing [2]. The possibility of producing bulk nanomaterial by SPD methods depends among other on the kind of the initial material, amount of deformation, strain rate, existence of second phases and value of the stacking fault energy. Privileged to nanostructures formation are alloys. The comparison of different materials shows that with the increase of stacking fault energy the material is less susceptible for nanostructure formation. Aluminium is the material with high value of the stacking fault energy (SFE), therefore is less susceptible for grain size reduction because of the easy microstructural recovering, preventing the formation of nanostructures [3, 8-10].

The most popular SPD methods are: equal-channel angular pressing (ECAP) [1, 2], cyclic extrusion compression (CWS) [3, 4], high pressure torsion (HPT) [5], hydrostatic

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extrusion (HE) [6-10] and others. Microstructure refinement is also possible using a combination of different SPD methods and their sequential combining, such as CWS + HE [11, 12] or ECAP + HE [12].

The bulk nanometric or ultrafine grained materials achieve new and extraordinary properties different than obtained in the same materials produced by a conventional process of plastic deformation. They have not only very small grain size but also specific defect structures, high internal stress, crystallographic texture and often change of phase composition. Also characteristic for nanomaterials is the superplasticity effect [6-9, 12, 13].

This paper presents the results of Al99.5 deformed in the process of ECAP and subsequently hydrostatically extruded. The aim of the research was to determine the influence of severe plastic deformation exerted in the process of equal-channel angular pressing (ECAP) and hydrostatic extrusion (HE) on the microstructure and properties changes of polycrystalline aluminium Al99.5.

2. Experimental procedure

The investigations were carried out on the polycrystalline aluminium Al99.5 having an average grain size of 110 μm. The chemical composition of the deformed aluminium is taken down in TABLE 1.

TABLE 1
Chemical composition of the Al99.5 (Al1050) [%weight]

Cu	Mg	Mn	Si	Fe	Zn	Ti	Al
0.05 max	0.05 max	0.05 max	0.25 max	0.40 max	0.07 max	0.05 max	99.5

Before the deformation, the samples were annealed in the sylite furnace at the temperature of T = 500°C during 2 hours and then cooling with the furnace.

The samples were subjected through the combination of equal-channel angular pressing (ECAP) and then by the hydrostatic extrusion (HE). ECAP process was realized at room temperature with the processing speed of 8×10^{-2} s^{-1} using a die with a 90° angle between the channels and route Bc in which the sample was turned 90° around its axis between consecutive process. The cross section of the ECAP channels was $10\times10mm^2$. The value of strain for a single pass through the die gave a strain of 1.15. The samples were pressed using 4, 8 and 16 ECAP passes.

Hydrostatic extrusion process was realized with a cumulative strain of 2.68 to attain finally wire diameter of d = 3 mm. Although the process was realized at room temperature, sample heating induces by the high strain rates was possible. Therefore the samples were water cooled at the exit of the die in order to minimize the effect of temperature on properties and microstructure. Detailed information's about deformation process are presented in TABLE 2.

After the deformation, the samples were investigated by means light microscopy (LM) OLYMPUS GX 51. The investigations by means light microscopy were performed on the longitudinal and cross sections of the samples. The samples were cut out and then mechanically ground and polished using diamonds pastes and colloidal suspension of SiO_2. The used technique for showing microstructure and deformation effects was anodizing in the Barker reagent. The samples were observed in the polarised light. To establish how severe plastic deformation (ECAP and HE) influences the properties of the polycrystalline aluminium Al99.5, the microhardness HV0.1 was measured and the tensile test was performed. The microhardness measurements were performed on the polished longitudinal sections of the samples perpendicularly to the sample axis. The tensile test was performed at the room temperature with constants strain rate of the order of $10^{-3}s^{-1}$. Additionally the texture was determined by using the Brucker Advance D8 equipment with CuKα radiation.

TABLE 2
The deformation path layout

Deformation method	Cumulative strain, ϕ
HE	2.68
4ECAP+HE	7.28
8ECAP+HE	11.88
16ECAP+HE	21.08

The ECAP and HE process were realized in the Institute of High Pressure Physics of the Polish Academy of Sciences in Celestynów.

3. Results and discussion

Selected micrographs obtained by using light microscope after hydrostatic extrusion and compose mode of deformation ECAP+HE are presented in Fig. 1.

Characteristic features of aluminium after hydrostatic extrusion and after compose mode of deformation ECAP+HE were elongated in the extrusion direction grains (Fig. 1a, c). Also typical is bands and shear bands occurrence which were observed both in the transverse (Fig.1b, d) and longitudinal section of the samples (Fig. 1a, c). The observed shear bands propagate to great distances, what is especially seen in Fig. 1 b, d. The effect of shear bands propagation is the presence of grain boundary deflection in the place of shear bands intersection what is particularly seen in Fig. 1a.

Deformation with the compose mode of deformation (ECAP + HE) resulted in microstructure homogenization, which was confirmed by microhardness on the samples axis measurements (Fig. 4).

Texture test results are shown in Fig. 2 and summarized in TABLE 3. It was identified the occurrence of an axial texture type <111>. This type of texture is characteristic for the cylindrical samples deformed in the extrusion, drawing or tensile deformation, connected with the tensile stresses. For aluminium deformed in the process of hydrostatic extrusion (HE) there is an additional component of the axial texture type <100>, probably indicating occurrence of the recrystallization processes. After compose mode of deformation ECAP + HE additional component of texture type {123}<111>was observed. Cylindrical component is strongest for the variant 8 ECAP + HE, for the variant 16 ECAP +

HE cubic texture component is visible and appears renewal process microstructure by the dynamic recrystallization, which causes weakening of the cylindrical texture component.

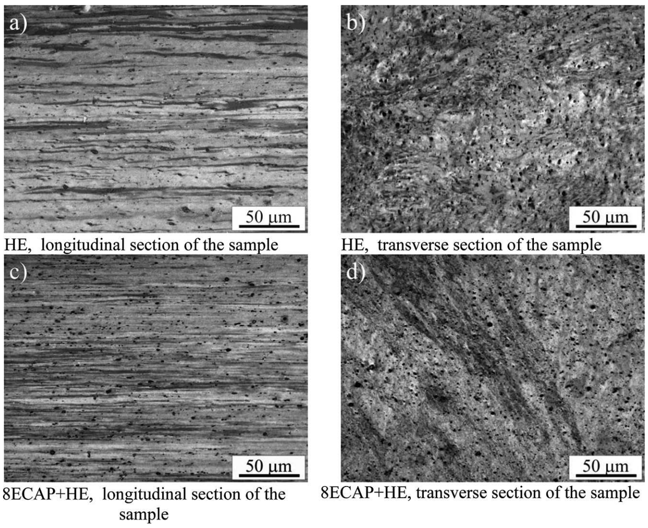

Fig. 1. Microstructures of deformed aluminium, a, b) after hydrostatic extrusion, $\phi = 2.68$, c, d) after combined mode of deformation 8 ECAP + HE, $\phi = 11.88$

It can be concluded that with increasing of compose deformation ECAP + HE originally evolves a cylindrical texture, which under the strong influence of the recovery process weakened as a result of the cubic texture occurrence.

TABLE 3

Texture analysis

Deformation method	Texture components
HE (ϕ = 2.68)	axial texture: <111>and <100>
4ECAP+HE (ϕ = 7.28)	{123}<111>, axial texture: <111>
8ECAP+HE (ϕ = 11.88)	{123}<111>, axial texture: <111>
16ECAP+HE (ϕ = 21.08)	axial texture: <111>and<100>, {123}<111>

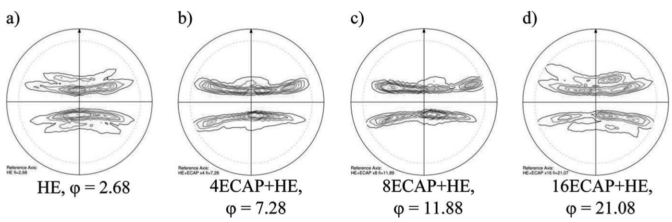

Fig. 2. Texture analysis, {111}pole figure

The microhardness measurements presented in Fig. 3 show increase of microhardness level with increase of strain. After HE nearly double increase of microhardness was found in comparison to the initial state, microhardness increase from 21 HV0.1 to 41HV0.1. The maximum value of microhardness equal - 46 HV0.1 achieved a sample after 8 passes ECAP and subsequent hydrostatic extrusion.

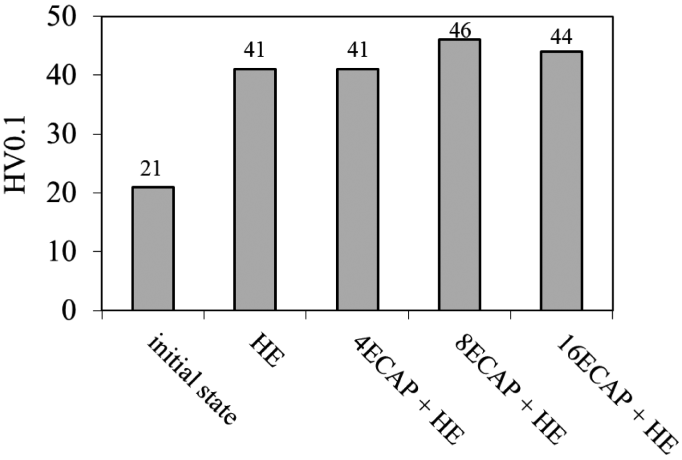

Fig. 3. Influence of strain value on the Al99.5 microhardness

Obtained results show, that after the strain ϕ = 2.68, the properties of Al99.5 have stabilized. Increase of the strain and change strain path by applying a combination of ECAP + HE methods did not result in large changes in the value of microhardness. This phenomenon proves the achieving by the material balance between the hardening and renewal processes.

The microstructure observations and the distribution of the microhardness along the samples sections show, that after compose mode of deformation ECAP + HE the distributions of the properties is more homogenous than after hydrostatic extrusion HE (Fig. 1, 4).

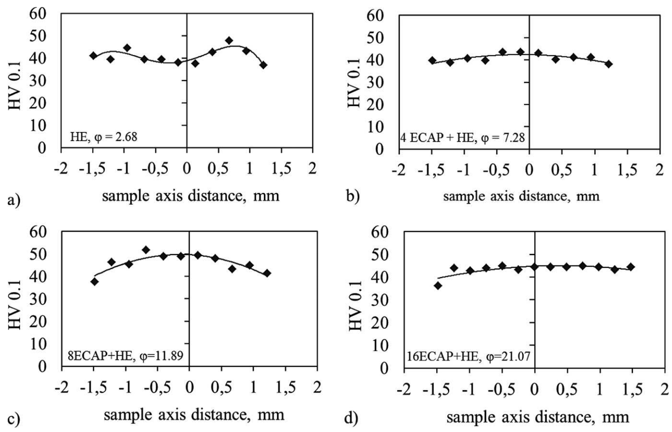

Fig. 4. Microhardness distributions on the sample axis; a) HE, $\phi = 2.68$, 4 ECAP + HE, $\phi = 7.28$, 8 ECAP + HE, $\phi = 11.89$, d) 16 ECAP + HE, $\phi = 21.07$

In the case of aluminium deformed in the hydrostatic extrusion process, the highest value of microhardness was measured at the distance of 500-600 microns from the edge of the sample. Both at the central part and edges of the samples, the microhardness level was lower of about 20% (Fig. 4a). In the case of the samples deformed in the process of ECAP+HE differences in microhardness level between central part and edges of the samples are definitely smaller. The largest difference in the microhardness on the sample axis distribution was observed in samples deformed with the combination 8ECAP + HE – differences in microhardness don't beyond of 10% (Fig. 4c). After deformation 4ECAP + HE and 16ECAP + HE microhardness distribution is very uniform, the value of microhardness in the different areas of the sample is different from each other by a several units (Fig. 4 b, d).

The results obtained from the tensile test confirmed considerable strengthening of the polycrystalline aluminium after hydrostatic extrusion process – UTS and YS increase almost three times with comparison to the initial state: UTS = 184MPa, YS = 96MPa, the four times decrease in elongation was observed – up to 8,6%. The combination of ECAP + HE resulted in a greater strengthening than after the hydrostatic extrusion process. The highest level of hardening while maintaining elongation of 8% was achieved in aluminum after deformation 8 ECAP + HE (ϕ = 11.89) - UTS = 218MPa. Compared to the material deformed only in hydrostatic extrusion process an increase of UTS almost 20% was observed (Fig. 5).

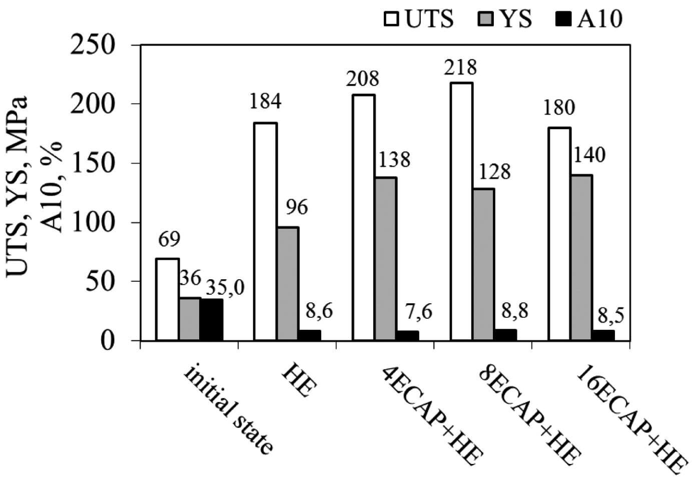

Fig. 5. Influence of strain value on the Al99.5 properties

After cumulative way of deformation 16 ECAP + HE the tensile strength decreased to the level of UTS = 180 MPa, comparable to that after hydrostatic extrusion process. Simultaneously, there was an increase of YS to a 140MPa, elongation was at the level of 8,5%. Probably the strain limit value, which has resulted in an increase of mechanical properties is ϕ = 11.89, after this strain a large amount of defects in the microstructure of the material is accumulated. With further deformation microstructure renewal processes can occur, material is no longer able to be strengthened.

4. Summary

Microstructure observations of polycrystalline aluminium Al99.5 both after hydrostatic extrusion and compose mode of deformation ECAP + HE revealed elongated in the extrusion direction grains and bands and shear bands occurrence which were observed both in the transverse and longitudinal section of the samples.

The microhardness measurements and the results obtained from the tensile test show increase of the properties level with increase of strain. Both the highest microhardness level and UTS achieved a sample after 8 passes ECAP and subsequent hydrostatic extrusion and attain the value of: 46 HV0.1 and UTS = 218 MPa.

The microstructure observations and the distribution of the microhardness along the samples sections show, that after compose mode of deformation ECAP + HE the distributions of the properties is more homogenous than after hydrostatic extrusion HE.

Acknowledgements

The Statutory Activities no 11.11.180.653 supported this work.

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Received: 10 May 2013.