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EFFECT OF SEVERE PLASTIC DEFORMATION ON MICROSTRUCTURE EVOLUTION OF PURE ALUMINIUM

WPŁYW INTENSYWNEGO ODKSZTAŁCENIA PLASTYCZNEGO NA EWOLUCJĘ MIKROSTRUKTURY CZYSTEGO ALUMINIUM

Processes of severe plastic deformation (SPD) are defined as a group of metalworking techniques in which a very large plastic strain is imposed on a bulk material in order to make an ultra-fine grained metal. The present study attempts to apply Equal-Channel Angular Pressing (ECAP), Hydrostatic Extrusion (HE) and combination of ECAP and HE to 99.5% pure aluminium. ECAP process was realized at room temperature for 16 passes through route Bc using a die having an angle of 90°. Hydrostatic extrusion process was performed with cumulative strain of 2.68 to attain finally wire diameter of d = 3 mm. The microstructure of the samples was investigated by means of transmission and scanning electron microscopy. Additionally, the microhardness was measured and statistical analysis of the grains and subgrains was performed. Based on Kikuchi diffraction patterns misorientation was determined. The measured grain/subgrain size show, that regardless the mode of deformation process (ECAP, HE or combination of ECAP and HE processes), grain size is maintained at a similar level – equal to d = 0.55-0.59 μm. A combination of ECAP and HE has achieved better properties than either single process and show to be a promising procedure for manufacturing bulk UFG aluminium.

Keywords: Severe Plastic Deformation, ultra-fine grained aluminium, microstructure characterization, ECAP, hydrostatic extrusion

1. Introduction

Grain size can be regarded as a key microstructural factor affecting a lot of aspects of the physical and mechanical behavior of polycrystalline metals. Hence, control over grain size has been recognized as a way to design materials with desired properties. Most of the mentioned properties benefit greatly from grain size reduction. A possible way for microstructure refinement of metals is the use of severe plastic deformation (SPD) [1-3]. Severe plastic deformation (SPD) may be defined as metal forming process in which a large plastic strain is introduced into a bulk metal in order to create ultra-fine grained metals. A unique feature of SPD processing is that high strain is imposed without any significant change in the overall dimensions of the workpiece [3-6].

The most popular SPD methods are: equal-channel angular pressing (ECAP) [7, 8], cyclic extrusion compression (CEC) [9-11] and high pressure torsion (HPT) [12]. Recently the hydrostatic extrusion method (HE) has been used for producing UFG metals [13,14]. Microstructure refinement is also possible using a combination of different SPD methods and their sequential combining, such as CEC + HE [15] or ECAP + HE [16]. Combination of SPD processes or combination of SPD and conventional plastic deformation process allows to receive new properties of material [15, 16]. Additionally such way of deformation creates the possibility of producing useful products. Especially the combination of SPD and hydrostatic extrusion or conventional extrusion process gives such possibility.

TABLE 1

Chemical composition of the Al99.5 (Al1050) [wt%]

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	balance

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The aim of the research was to determine the influence of Equal-Channel Angular Pressing (ECAP), Hydrostatic Extrusion (HE) and combination of ECAP and HE on the microstructure and selected properties changes of polycrystalline aluminium Al99.5.

2. Material and method

The investigations were carried out on the polycrystalline aluminium Al99.5 having an average grain size of 170 μm. The chemical composition of the deformed aluminium is given in TABLE 1.

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 to the Equal-Channel Angular Pressing (ECAP), Hydrostatic Extrusion (HE) and combination of ECAP and HE. ECAP process was realized at room temperature with the strain rate of 0.08 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 processes. The cross section of the ECAP channels was 10x10 mm^2. The value of strain for a single pass through the die gave a strain of 1.15. The samples were pressed using 16 ECAP passes.

Hydrostatic extrusion process was realized with a cumulative strain of 2.68 and strain rate of 24.2-473 s^{-1} just to attain final wire diameter of d = 3 mm. Although the process was realized at room temperature, sample heating induced 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 about deformation process are presented in the TABLE 2.

TABLE 2

The deformation path layout

Deformation method	**Cumulative strain, ε**
16 ECAP	18.4
HE	2.68
16 ECAP + HE	21.08

After the deformation, the samples were investigated by means of transmission electron microscope JEM 2010 ARP and scanning electron microscope Hitachi S – 3400N. The thin foils for electron transmission observations were prepared applying the standard technique of electrolytic polishing using the Struers apparatus. The statistical analysis of the grain size was performed using mean chord perimeter. The misorientation was determined using EBSD method. Samples for EBSD were mechanically polished and then electropolished using a 20% perchloric acid -80% ethanol electrolyte cooled to -25°C. The Vickers microhardness, Hv, was measured on polished mirror-like samples surfaces using a standard microhardness tester equipped with a Vickers indenter. The hardness measurements used a load of 100 g.

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

3. Results

The material in as-received state had a coarse grained microstructure with the average grain size of 170 μm. Characteristic feature of the Al99.5 after deformation was occurrence of equiaxed subgrains and grains (Fig. 1b, 2b, 3b). It was found, that some of grains/subgrains have dislocations inside (Fig. 1b, 2b) and some of them are free of dislocations (Fig. 3b). The existence of dislocations inside grains depended among others on the change of the position and inclination of thin foil, however some area did not exhibit dislocations independently to the foil position. Other factor having influence on the dislocation existence is deformation method; a higher density of dislocation was found in the samples after ECAP deformation (Fig. 1b). The presence of the grains free of dislocations indicates the development of intense microstructural renewal processes, which probably takes place during or directly after deformation. The next characteristic feature of microstructure was formation of microbands. The observed microbands were built from elongated subgrains (Fig. 1a, 2a, 3a). In some of them the occurrence of small subgrains was found (Fig. 2a, 3a). The microbands observed in the samples after hydrostatic extrusion propagated to the greater distances than in the samples after ECAP. In the HE processed sample the microstructure contained highly heterogeneous regions (Fig. 3a), whereas the ECAP and ECAP + HE samples had microstructure with well-developed grains (Fig. 1, 2).

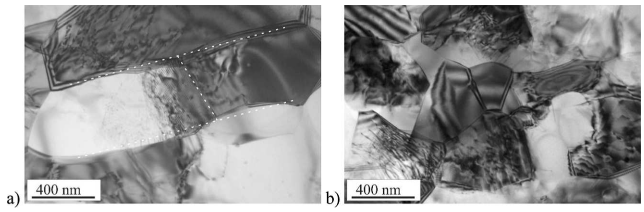

Fig. 1. TEM micrograph of the Al99.5 after 16 ECAP; ε = 18.4

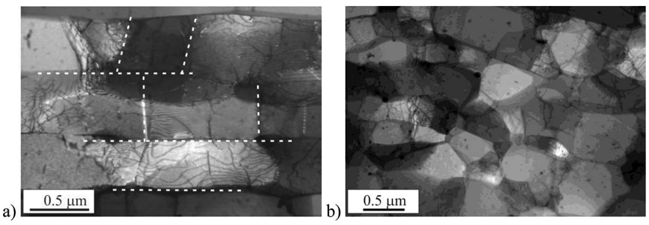

Fig. 2. TEM micrograph of the Al99.5 after 16 ECAP + HE; ε = 21.08

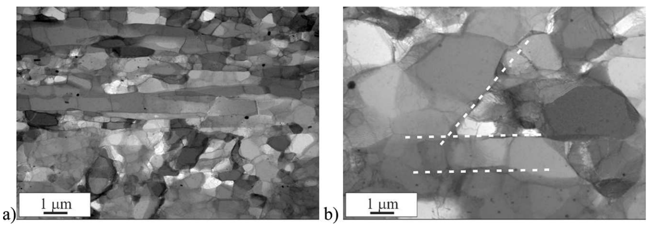

Fig. 3. TEM micrograph of the Al99.5 after HE; ε = 2.68

The measurements of the grain/subgrain size reveal that the sample having an initial in annealed-state grain size of about 170 μm reduced to the grain size below 0.6 μm when it was deformed by ECAP, HE and combination of both. The average grain diameter after deformation in different method is maintained at a similar level – equal to d = 0.55-0.57 μm (TABLE 3).

TABLE 3

The average grain/subgrain diameter measurements

Material	Deformation method	$d_{av.}$, μm
Al99.5	Initial state	170
	16 ECAP, ε = 18.4	0.57
	HE, ε = 2.68	0.55
	16 ECAP + HE, ε = 21.08	0.59

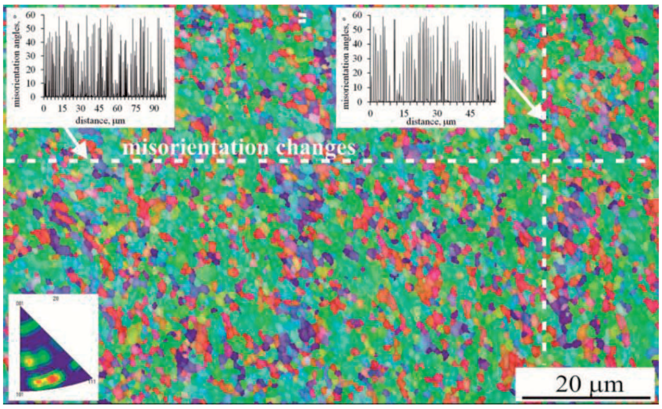

Fig. 4. EBSD map of the microstructure of a sample processed to 16 ECAP passes via route BC and misorientation changes along dashed lines; ε = 18.4

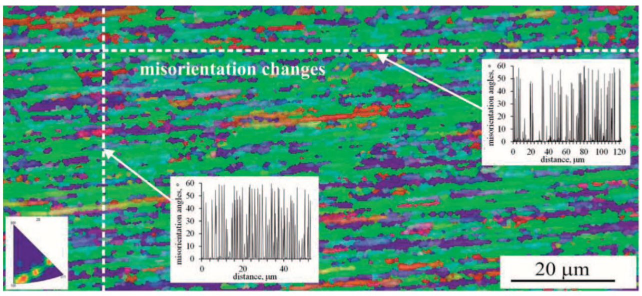

Fig. 5. EBSD map of the microstructure of a sample processed to 16 ECAP + HE and misorientation changes along dashed lines; ε = 21.07

The EBSD maps of the deformed samples show in the ECAP-ed sample equiaxed ultrafine grained microstructure with the large misorientation angles between grains (Fig. 4, 7). Similar in the sample deformed by the compose method ECAP + HE large misorientation angles dominate (Fig. 5, 7). The grains are arranged in the direction of extruding (Fig. 5, 6). The domination of the small misorientation angles was observed after hydrostatic extrusion process. The fraction of large misorientation angles was about 23% (Fig. 6, 7). The occurrence of the large misorientation angles indicate a considerable rotation of the material during the deformation. This phenomenon is also connected with considerable energy storage in the vicinity of newly created grain boundaries. In the conditions of the high strain rates this can lead to a considerable temperature rise of the whole system and the processes of microstructure renewal are intensified. Due to the above, the microstructures with domination of the large misorientation angles could be instable under elevated temperatures in the successive applications.

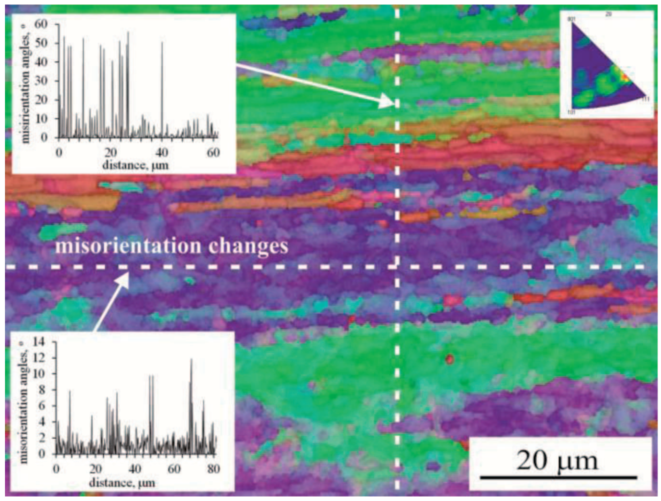

Fig. 6. EBSD map of the microstructure of a sample hydrostatic extruded and misorientation changes along dashed lines; ε = 2.68

The inverse pole figures (IPF) corresponding to the EBSD maps (inserts in Fig. 4-6) show fiber-like texture of deformed materials. In Figure 4, after only ECAP deformation, the fiber texture presents maxima at poles lying at [011] - [112] circle and close to [158] and to [001] poles. On the other hand, when HE process is operating, the maxima are observed only along [011] - [112] circle with no fiber close to [0 0 1] pole (Fig. 5, 6). In case, when both plastic deformation process are active, the strongest intensity peak is close to [011], while when only HE operates, the maximum is close to [112].

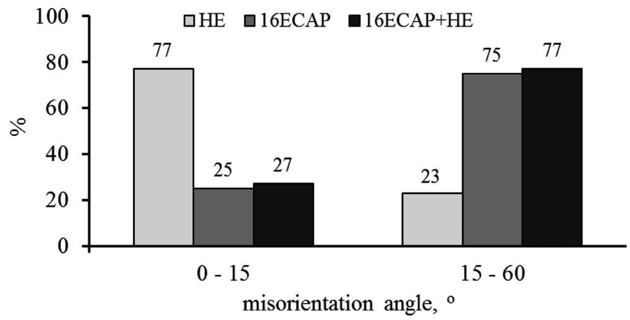

Fig. 7. Misorientation angles distribution

The nearly double increase of microhardness after deformation compared to the initial state was observed, from 21 HV0.1 to about 40 HV0.1. It was found, that HE of ECAP-ed samples leads to an increase of the microhardness level of about 15% as compared to the samples after ECAP process (Fig. 8).

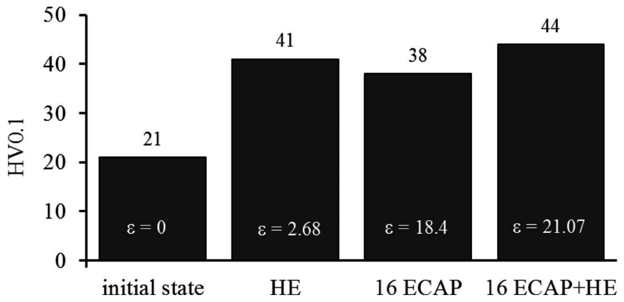

Fig. 8. Influence of the strain value of the microhardness level

The obtained results show that after deformation $\varepsilon = 2.68$ properties of aluminium stabilized. Increase of the strain and change strain path by applying a combination of ECAP + HE methods or ECAP process did not result in significant changes in the value of microhardness and also grain size. This phenomenon proves the achievement of balance between the hardening and renewal processes by the material.

4. Conclusions

It is possible to obtain ultrafine grained aluminium with the grain size below 0.6 μm and dominance of large misorientation angles using ECAP and combination of ECAP and HE. The measured grain/subgrain size show, that both the deformation in the ECAP, HE process and combination of ECAP and HE methods lead to a situation where grain size is maintained at a similar level – equal to 0.55-0.59 μm. The grain refinement during deformation results in the increase of the microhardness level. A combination of ECAP and HE has achieved better properties than either single process, and show to be a promising procedure for manufacturing bulk homogenous UFG aluminium.

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