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
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ULTRAFINE AND NANO-GRAINED ALUMINIUM ALLOYS FORMED BY CYCLIC EXTRUSION COMPRESSION

ULTRADROBNOZIARNISTE I NANO-ZIARNISTE STOPY ALUMINIUM KSZTAŁTOWANE METODĄ CYKLICZNEGO WYCISKANIA ŚCISKAJĄCEGO

1. Introduction

The ultrafine-grained and nanograined materials are produced by Severe Plastic Deformation (SPD) methods mostly from aluminium alloys [1,2]. The relatively low hardening of aluminium alloys facilitates their deformation in the range of very high strains, indispensable for diminishing the microstructure by the SPD processes. A lot of results connected with the production of ultrafine-grained and near-nanocrystalline materials have been obtained by Equal Channel Angular Pressing (ECAP) [3-5], Accumulative Roll Bonding [6-8] and High Pressure Torsion [9,10]. In the present work the Cyclic Extrusion Compression (CEC) method [11] was used for the deformation of aluminium alloys.

The CEC method was invented in 1979 [11]. Since that time such materials as: pure mono- and polycrystalline aluminium, aluminium alloys and copper have been deformed by this method to very large strains, up to the true strain of about $\varphi = 50$ [12 17]. The mechanisms responsible for the formation of ultrafine-grained and nanograined structures were intensively investigated [12,15].

The aluminium alloys deformed by different SPD methods usually show grains with the mean size from 100 nm to about 1000 nm. The mean grain size below 100 nm was reported only occasionally [1,18]. The plastic deformation strongly affects the refinement of the microstructure and increase the strength of the materials. It was found that the evolution of microstructure from the conventional grain size to nanograins requires very high strains and also depends on the kind of the deformed materials. The production of nanocrystalline materials requires attaining a level of deformation, usually much higher than that which could be obtained in conventional plastic working processes. Large deformations also activate the microstructure softening processes. Only materials containing particles or solute atoms are suitable for nanomaterials production, because the soluble atoms prevent the move of grain boundary of the new grains. In the pure materials the structure softening processes usually spoil the effect of grain refinement [1,4,15,19].

The aim of the present study was the investigations of the process of the grain refinement in AlMgSi, AlCu4Zr and AlMg5 aluminium alloys resulting from the exertion of large strains by using the CEC method.

2. Experimental

The AlMg5, AlCu4Zr and AlMgSi aluminium alloys were deformed by the CEC method [11,18] in the range of the true strains $\varphi = 0.4$ - 16 (Fig.1). The deformation was exerted cyclically with the true strain of $\varphi = 0.42$ in a single cycle. The deformation exerted in a single cycle results from the applying of the die chamber diameters equal $d_o = 10$ mm and diameter of the narrowing $d_m = 9$ mm (Fig.1B). The deformation was calculated from the formula: $\varphi = 4n \ln d_o/d_m$, where n – number of the deformation cycles, d_o – die chamber diameters, d_m – diameter of the die narrowing.

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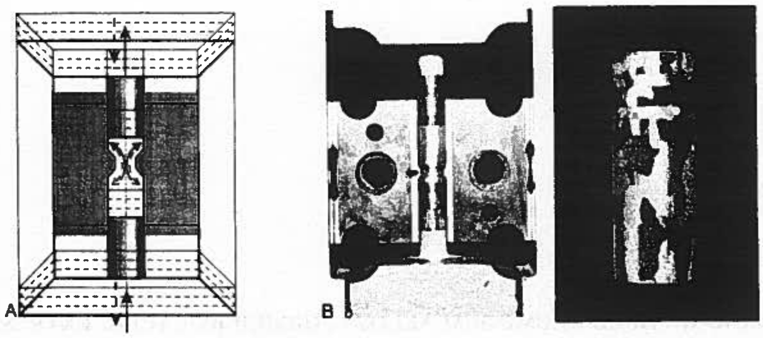


Fig. 1. Cyclic Extrusion Compression method: (A) scheme of the CEC equipment, (B) initial sample from aluminium with large grains and the sample inside the sectional die

Microstructures of the samples were observed on the longitudinal cross sections using transmission electron microscopy JEM 2010 ARP. Thin foils were prepared by electropolishing in the Struers equipment. The mean size of grains and nanograins was determined from the TEM micrographs by the mean chord parameter. The orientation and misorientation of nanograins were determined from the Kikuchi line patterns diffractions using KILIN software [20]. This program determined also the microtexture. The microhardness was measured on the longitudinal cross sections of samples, which were electrolytically polished in the reagent containing: 20% $HClO_4$ + 80%C_2H_5OH. Polishing was performed at about -20°C and 25V. The microhardness was investigated using the PMT3 microhardness tester at the load 100 G.

3. Results and discussion

The hardening occurred rapidly during CEC process in the investigated aluminium alloys (Fig.2). At the beginning of deformation a considerable increase of microhardness has been observed. Above the true strain of about $\varphi = 4$ a distinct stabilization of properties appeared. Similar results have been reported also in other works concerning materials deformed to very large strains by the SPD methods [1,2].

As regards the stabilization of properties there appeared considerable changes in the microstructure. The proceeding increase of deformation involved a decrease of grain size. That was most significant in AlMgSi alloy, where nanograins below 100 nm were found (Fig.3a). In the AlMg5 alloy the lowest mean grain size achieved 150 nm.

A large misorientation between nanograins was the next characteristic feature of microstructure concerning the range of properties stabilization (Fig.4). The misorientation considerably increased at the beginning of deformation, correlating with a strong decreasing of grain size. Above the true strain of about $\varphi = 3$ the stabilization and even some drop of the mean misorienation angle was found.

The results suggest that in the range of very large strains the course of hardening does not precisely depend on the evolution of the substructure, observed by TEM. The ultrafine grained and bulk nanostructured materials in the range of large deformations exhibit small changes of properties, but significant changes in some details and features of the microstructure. The changes especially concern the increase of mean misorientation angle and the drop of the grain size.

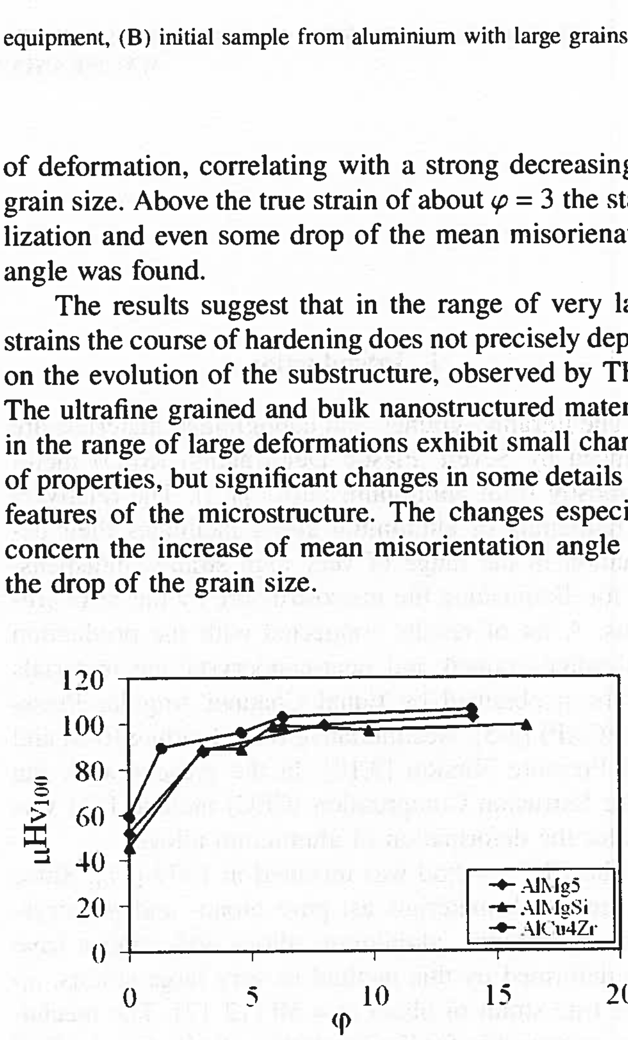
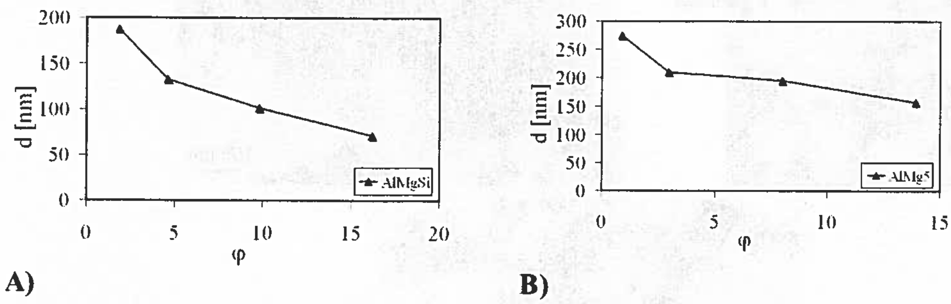


Fig. 2. Hardening of aluminium alloys deformed by CEC

It was found that in the range of strains between $\varphi = 4$ - 14 in the deformed aluminium alloys prevailed ultrafine grained microstructure. With the increase of deformation the fraction of structure with nanostructural features increases. Above $\varphi = 14$ the nanostructure occupied the largest volume of AlMgSi samples (about 75% of sample volume). Grains smaller than 100 nm with a large misorientation were observed also in the AlMg5, AlCu4Zr alloys, but in these alloys generally the ultrafine-grained microstructure prevailed (Fig.5). The results of misorientation presented at Fig.5. show that large misorientation above 15° is visible at some segments of boundaries and at the remaining part the small misorientation below 15° appears. The basic triangle in the left upper corner at Fig.5a shows that in the investigated area the < 110> orientation is prevailed. The result agrees with the texture typical for the compressed state of stresses, which is dominating in the CEC process.



A) B)

Fig. 3. Decrease of the mean grain size d with an increase of strain φ in; (A) AlMgSi and (B) AlMg5

The diminishing of the mean grain size and the increase of the number of large, misoriented boundaries were the main characteristic features of large deformations. These features suggest development of special mechanisms providing the formation of a nanostructure. The performed observations show that the deformation mainly occurs in the formation of macro- and micro-shear bands. Their number considerably increases with the increase of strain and they mutually cross, which leads to their partition into small pieces. If the width of microbands is nanometric, the formed pieces become nanovolumes from which the nanograins can be created.

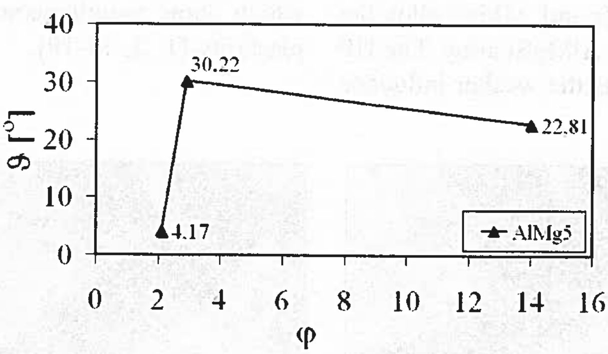


Fig. 4. The mean misorientation angle θ of grain boundaries in AlMg5 alloy versus CEC deformation φ

The idea of geometrical mode of the formation of nanograins was presented in the works concerning the CEC deformation [15-17]. The geometrical mechanism of microstructure refinement is schematically shown in Fig.6. Inside the mutually crossing microbands the rectangular nanovolumes are formed (Fig.6B), which can be transformed into more equal shapes by the activation of the microstructure softening mechanisms.

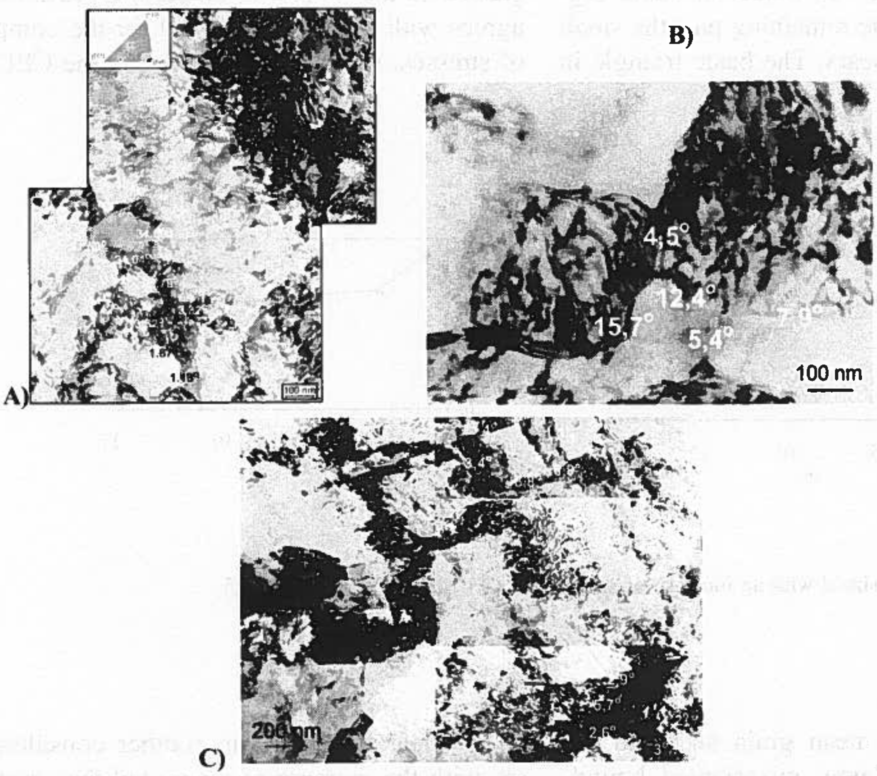


Fig. 5. Nanograins in; (A) AlMgSi alloy after φ = 16, (B) AlCu4Zr alloy after φ = 14, (C) AlMg5 alloy after φ = 14

The diminishing of grains should strongly influence the level of the alloys hardening. The Hall-Petch relation for the investigated alloys was presented at Fig.7. The course of the relation is shown for three alloys AlCu4Zr, AlMg5 and AlMgSi. In the all cases the linear increase of hardening has been found. The only difference is connected with the slope of diagrams to the ordinates axis. It can be visible that for the AlCu4Zr and AlMg5 alloy the slope is much sharper than for the AlMgSi alloy. The HP diagram for AlMgSi alloy suggests the weaker influence of grain size on the properties than in the other alloys. However the AlMgSi alloy contains the largest fraction of the nanograins microstructure and the observed behavior can be probably connected with the weakening of the material due to the formation of the very large number of new nano- and ultrafine- grains. This result is in agreement with the data of nanomaterials behaviour, which show simultaneously high hardening and good plasticity [1, 2, 17-19].

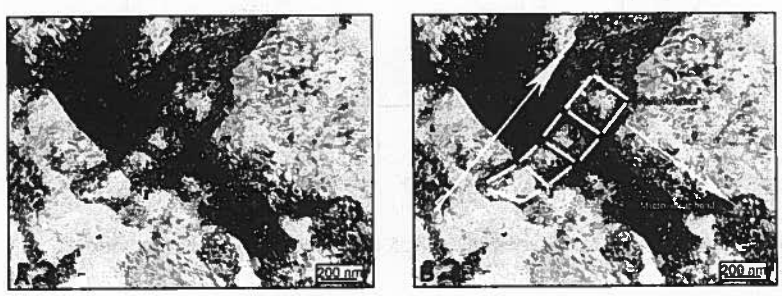


Fig. 6. The geometrical mechanism of microstructure diminishing; (A) mutually crossing microbands, (B) formation of nanovolumes in the area of crossing microbands

In the performed investigations it has been observed that nanograins have some density of dislocation inside (Fig.5), but much lower in comparison to the areas of extended boundaries of nanograins and microbands. In the areas of crossing microbands, inside nanovolumes, it seems that the density of dislocations still decreases (Fig.5c, Fig.6). Probably in the areas of crossing microbands mechanisms of microstructure softening is stronger and its progress leads to the lowering of alloy hardening. The effect of the diminishing of the AlMgSi slope of the HP diagram can be connected with this phenomenon.

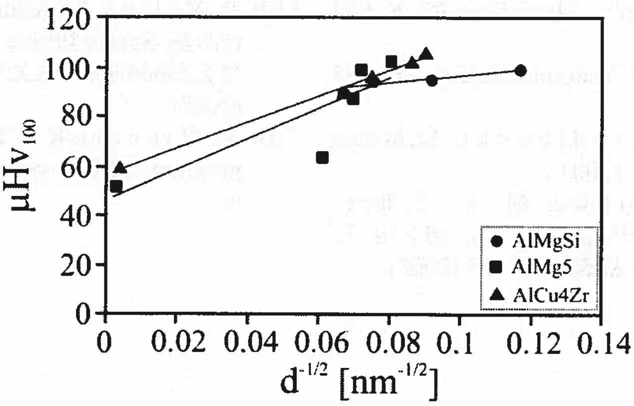


Fig. 7. Hall-Petch relation for aluminium alloys deformed to large strains by the CEC method

The obtained results suggest that transformation of a microstructure from conventional to nanostructure in the bulk materials deformed by SPD processes depends both from the level of the exerted strain and also strongly on the kind of the deformed material. The precursor material should have features preventing the growth of nanograins. The literature data and the earlier investigations proof that the presence of precipitates or solute atoms, which prevent the move of nanograin boundaries and favour the stabilization of nanostructures, is very desirable [4,15,19].

4. Conclusions

1. The ultrafine grained and nanograined materials can be produced from aluminium alloys using the CEC method. In the AlMgSi alloy, after the deformation of about $\varphi = 16$, the nanostructure has been found inside about 75% of the samples volume.
2. The balance between the hardening and softening processes have a great influence on the final microstructure and properties of the deformed aluminium alloys. The considerable increase of the fraction of nanostructural grains results in a hardening reduction.
3. The microstructure of aluminium alloys deformed by the CEC method depends on the value of deformation. The microstructure is mixed and contains grains with the size below and above 100 nm. At lower strains the fraction of micro-grained microstructure is much higher than nano-grained microstructure. With the increase of deformation the nanostructure fraction increases.

Acknowledgements

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