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MICROSTRUCTURE AND TEXTURE OF ECAP PROCESSED AlCu4SiMn AND AlCu5AgMgZr ALLOYS

MIKROSTRUKTURA I TEKSTURA STOPÓW AlCu4SiMn i AlCu5AgMgZr WYCISKANYCH W KANALE KĄTOWYM (ECAP)

The superplastic flow occurring in materials of ultra-fine grained structure allows to reducing the drawing temperature. It may be profitable when applied to aluminium alloys like AlCuZr, subjected to superplastic forming. The texture, based on neutron diffraction pole figure measurements of two alloys i.e. AlCu4SiMn and AlCu5AgMgZr during the equal-channel angular pressing (ECAP) is discussed in the paper. Structure observations by means of optical microscopy, transmission electron microscopy (TEM) and electron back scattered diffraction (EBSD) technique for OIM^{TM} and local texture measurements have been applied to obtain a precise and detailed description of structure and orientation changes.

Keywords: ultra-fine grained (nanokrystalline) materials, equal-channel pressing, Al alloys.

Wytworzenie ultra-drobnokrystalicznej mikrostruktury pozwala na obniżenie temperatury płynięcia nadplastycznego. Wykorzystanie tego zjawiska może przyczynić się do uzyskania wymiernych korzyści podczas kształtowania poprzez płynięcie nadplastyczne elementów ze stopów Al takich jak AlCuZr. W pracy przedstawiono wyniki pomiarów tekstury stopów AlCu4SiMn i AlCu5AgMgZr wyciskanych w kanale kątowym (metoda ECAP). Wykorzystano obserwacje struktury za pomocą mikroskopu optycznego, elektronowego (TEM) i skaningowego w zakresie analizy obrazu (OIM^{TM}) oraz tekstury lokalnej (EBSD) w celu szczegółowego opisu struktury i zmian orientacji.

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1. Introduction

Superplastic forming (SPF) has been still interesting for automotive, aerospace and aircraft industries, particularly in the case of AlCuZr alloys (trade name SUPRAL), in which the grain growth is inhibited by Al_3Zr particles [1-4]. Superplastic deformation is observed when the deformation process involves the grain boundary sliding. It occurs at the strain rate inversely proportional to the square of grain size [5]. The direct consequence of that is the tendency to decrease the grain size of such a deformed material.

Recently, the increased interest in materials of the ultra-fine grained (UFG) structure, with the grain size of sub-micrometers order, known as nanocrystalline materials when the grain diameter is below 100 nm, is due to the evident advantages resulting from the development of such a structure and manifested by increased strength properties and greater hardness at higher ductility. The low temperature superplastic flow occurring in these materials allows to reducing the drawing temperature of complicated products [6] and thus may be interesting when aluminium alloys like AlCuZr, used in superplastic forming, are considered.

The paper presents the texture analysis, based on neutron diffraction pole figure measurements and calculated orientation distribution functions of two aluminium alloys i.e. AlCu4SiMn and AlCu5AgMgZr during the equal- channel angular pressing (ECAP). The influence of short time recrystallization on the refinement of grain size is discussed. Structure observations by means of optical microscopy, electron back scattered diffraction (EBSD) technique and transmission electron microscopy (TEM) are used to obtain the most precise and detailed description of structure and orientation changes.

2. Equal-channel angular pressing

Equal-channel angular pressing (ECAP) method [6, 7] consists in successive extrusion steps of a sample through an angular die, which introduces cumulative severe plastic deformation without changing its shape. The extrusion is performed in several passes according to the scheme of equal-channel angular pressing presented in Fig. 1.

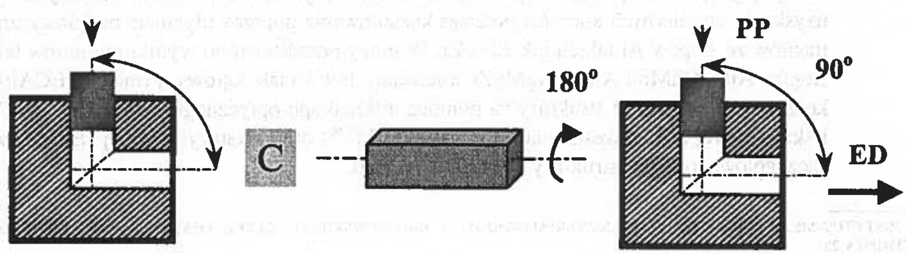


Fig. 1. Scheme of equal-channel angular pressing (ECAP)

The equivalent deformation ε per pass is close to 1 [6]. In route C the sample is rotated 180° around its longitudinal axis between each pass.

3. Material and experimental technique

Experiments were performed on two Al alloys: 1) AlCu5AgMgZr alloy [4] — marked D, with chemical composition in weight %: Cu 5.0, Mn 0.2, Ag 0.7, Mg 0.8, Zr 0.4 and grain size d = 10÷20 μm, initially solution treated for 0.5 h at 530°C and then aged 2 h at 250°C and 2) AlCu4SiMn — marked P, consisted of : Cu 4.0, Mn 1.0, Si 0.8, Mg 0.6 and Fe 0.4 [8]. ECAP process was executed by means of INSTRON 6025 testing machine with a specially constructed equipment attached [7]. Final deformations and experimental conditions are presented in Table.

Microstructure observations were carried out by means of Philips CM 20 electron microscope; thin foils parallel to surface 2 (Fig. 2) were prepared by electrolytic thinning.

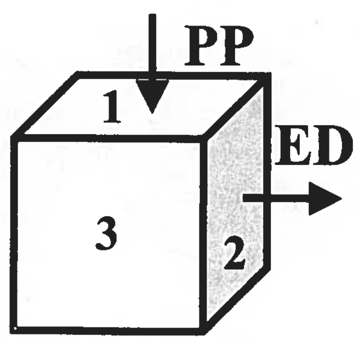


Fig. 2. Orientation of measured samples; texture and structure observation were effected on the surface no 2, PP — pressing direction, ED — extrusion direction (see Fig. 1), in pole figures RD || PP, ND || ED

The changes of crystallographic orientations were studied by the method of pole figures. The measurements of pole figures, with the normal "ND" parallel to the extrusion direction ED and "RD" along the direction of the acting piston (PP in Fig. 2), were performed. The neutron diffraction method at Leon Brillouin Laboratory of CEA/Saclay, France for the global texture (Fig. 3) analysis was applied. For local behaviour studies, the scanning electron microscopy (SEM) equipped with orientation imaging microscopy (OIM^{TM}) software for image analysis and with electron back scattering diffraction (EBSD) technique for local texture determination (Fig. 4) was used.

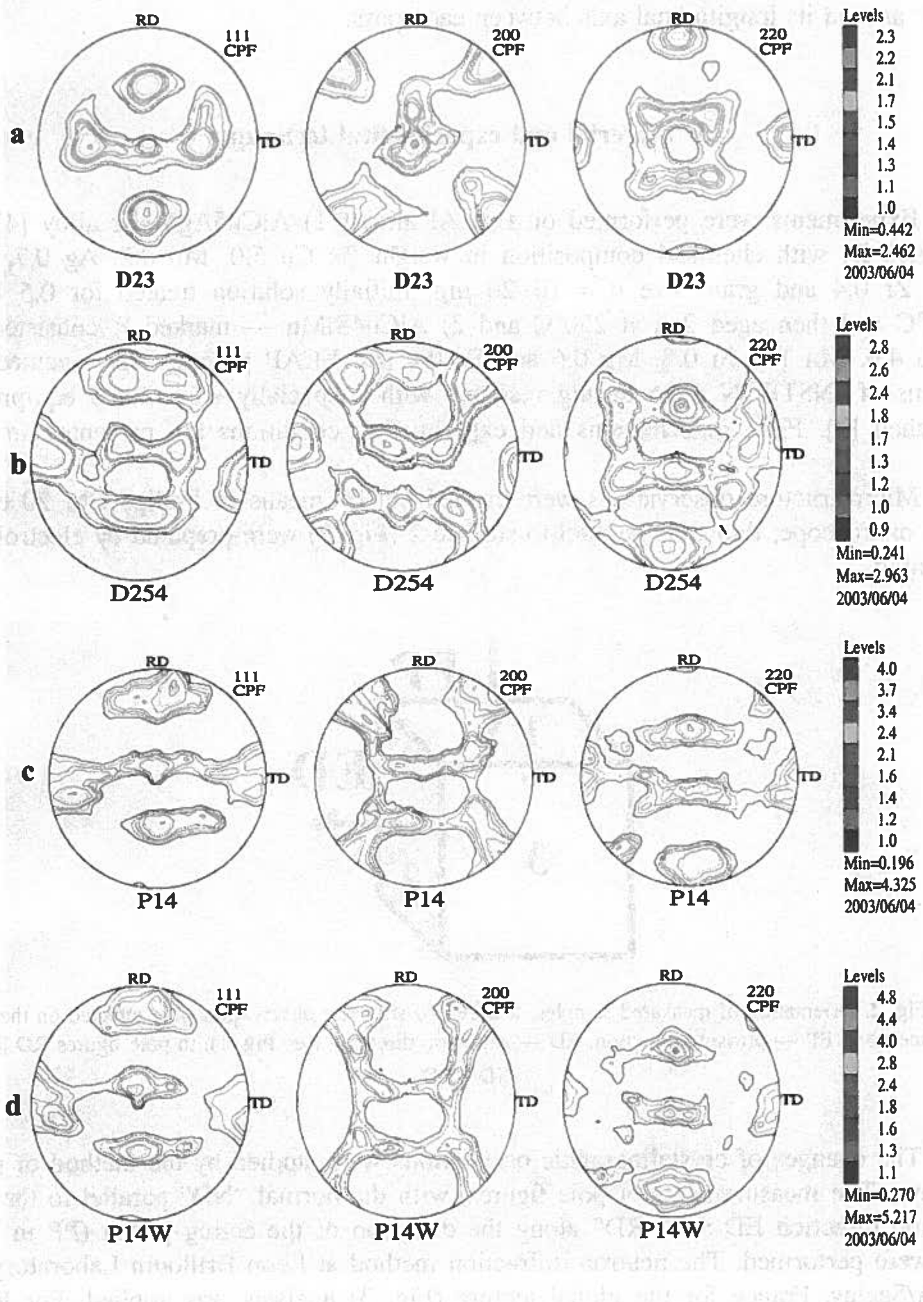
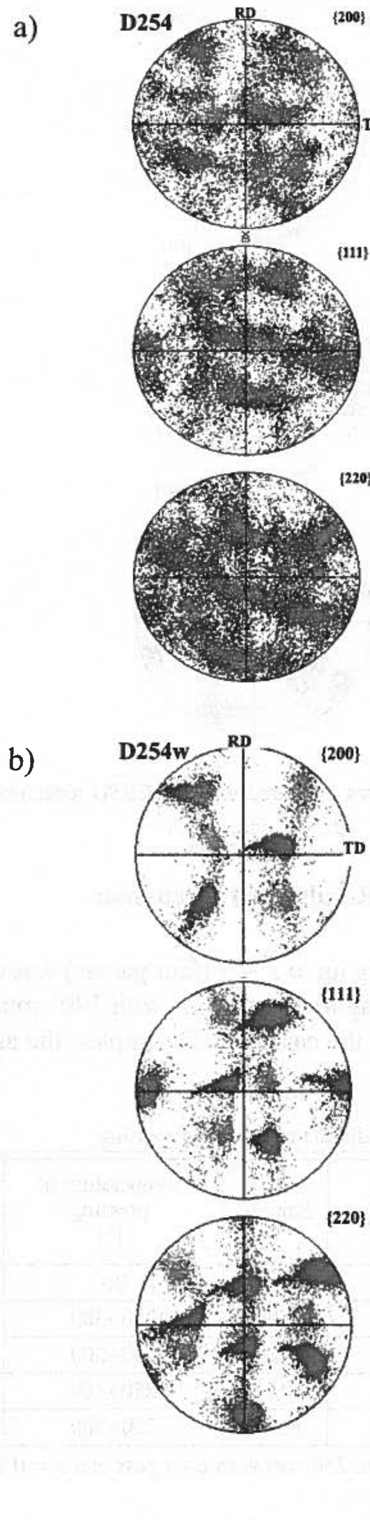


Fig. 3. {111}, {200} and {220} pole figures measured with the neutron diffraction of ECAP processed sample: a) D23, b) D254, c) P14 and d) P14w.


a)
D254
RD
{200}
TD
{111}
{220}
b)
D254w
RD
{200}
TD
{111}
{220}

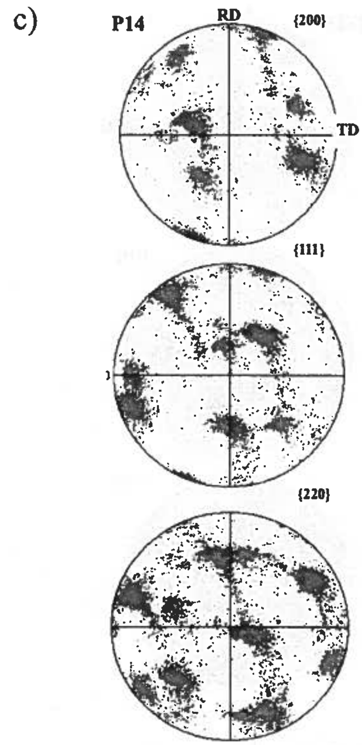


Fig. 4. {111}, {200} and {220} pole figures measured with the EBSD technique of samples: a) D254, b) D254w and c) P14

4. Results and discussion

Multiple procedure of pressing up to $\varepsilon \sim 4$ (four passes) was effected on samples of $10\times10 mm^2$ cross section, according to route C, i.e. with 180° rotation about the sample axis which followed each pass. In the case of D23 samples, the aging procedure during

TABLE

Conditions of ECAP processing

Material	ECAP equivalent deformation ε	Sample	Temperature of pressing [°]	Recrystallization
AlCu5AgMgZr	2.7	D23*	20	—
AlCu5AgMgZr	4.0	D254	250÷300	—
AlCu5AgMgZr	4.0	D254w	250÷300	450°/10 min
AlCu4SiMn	4.0	P14	250÷300	—
AlCu4SiMn	4.0	P14w	250÷300	450°/10 min

D23* — aging during 2 h in 250° between each pass and $\varepsilon = 0.9$ per pass [8]

2 h at 250°C was applied between each pass; for samples D254 and P14 the ECAP procedure was performed at temperature 250÷300°C, D254w and P14w were annealed 10 min in 450°C (see Table).

4.1. Texture measurements

The inspection of pole figures measured by neutron diffraction technique (global texture) of deformed alloys up to $\varepsilon \sim 4$, i.e. D254, P14 in Fig. 3, demonstrates dissimilarities in plane normal distribution due to different alloy compositions; short time recrystallization (10 min in 450°C) results in lowering of dispersion of orientations only (compare P14 and P14w in Fig. 3). Smaller dispersion is observed in sample D23, deformed up $\varepsilon \sim 2.7$ (Fig. 3a) than in sample D254 deformed up to $\varepsilon \sim 4$ (Fig. 3b).

The electron back scattering diffraction (EBSD) technique for local texture reveals more clearly the differences which appear in the texture of annealed samples (Fig. 4a, D254 as deformed and Fig. 4b, D254w as annealed). In short time recrystallized sample i.e. D254w (Fig. 4b), a lower dispersion of orientation and lack of certain components have been observed. Spread of normal planes of P14 sample (Fig. 4c) resembles that of D254w i.e. the recrystallized one and it testifies that P14 recrystallizes under this ECAP hot pressing.

4.2. Structure observations

The structure observed by means of transmission electron microscopy of ECAP deformed up to $\varepsilon \sim 4$ AlCu5AgMgZr alloy (bright field BF and dark field DF photographs of D254 in Fig. 5) presents grains with diameter $d =\sim 1\mu m$, embedded in layers of elongated inclusions. In the AlCu6Zr alloy (Supral 100) it was shown that processing by ECAP refines the grain size to $d \leqslant 2\mu m$ [9]. Similar results of grain refinement ($d =\sim 2\mu m$) had been obtained for 7475 aluminium alloy (AlZn6Mg2Cu2CrZr) by ECAP processing in 400°C up to above 6 by Goloborodko, Sitdikov, Sakai, Kaibyshev and Miura [10]. The grains with width $d_w = 1 \div 5\mu m$, elongated in the transverse direction are presented in the background of randomly distributed inclusions in AlCu4SiMn alloy (bright field BF and dark field DF photographs of P14 in Fig. 6). One can note a significant selective grain growth in transverse direction.

OIM™ observations of recrystallized D254w sample show grain growth in comparison with grains registered by OIM™ of the deformed D254 sample (Fig. 7a and 7b). This grain growth seems to be very selective and results in selecting of certain components in the orientation distribution of the recrystallized D254w contrary to the deformed D254 in which grain orientations are spread and dispersed (Fig. 4). The spread of plane normal distribution of P14 sample is similar to the recrystallized D254w one (compare P14 with D254w pole figures in Fig. 4) and proves relatively large and recrystallized grains observed in P14 sample (Fig. 7c).

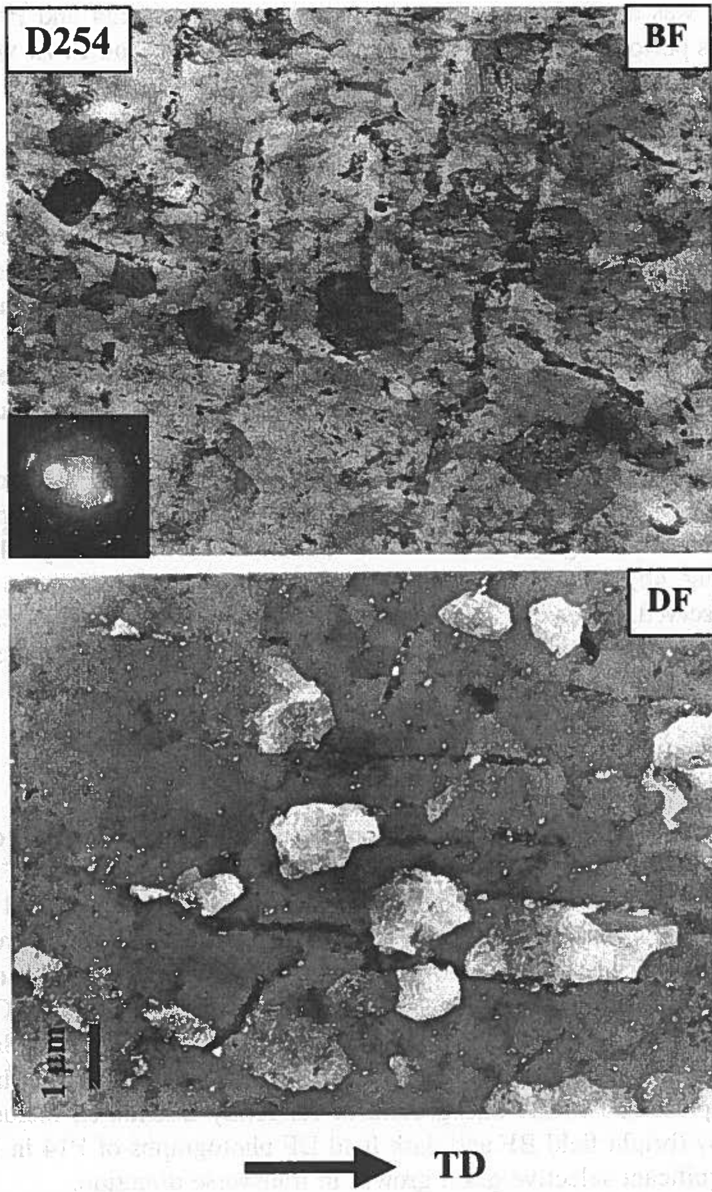


Fig. 5. TEM photographs of AlCu5AgMgZr alloy deformed at 250÷300°C up to $\varepsilon \sim 4$; BF — bright field and DF — dark field (200) images

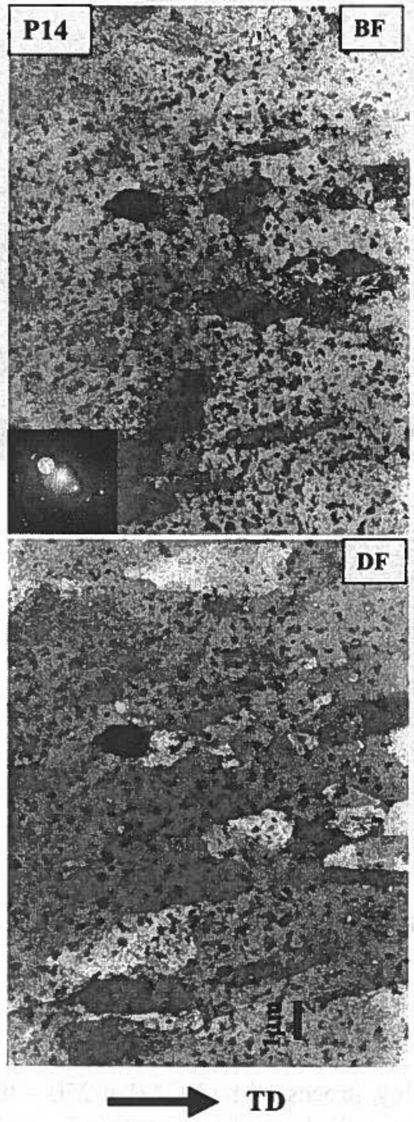


Fig. 6. TEM photographs of AlCu4SiMn alloy deformed at 250÷300°C up to $\varepsilon \sim 4$; BF — bright firld and DF — dark field (200) images

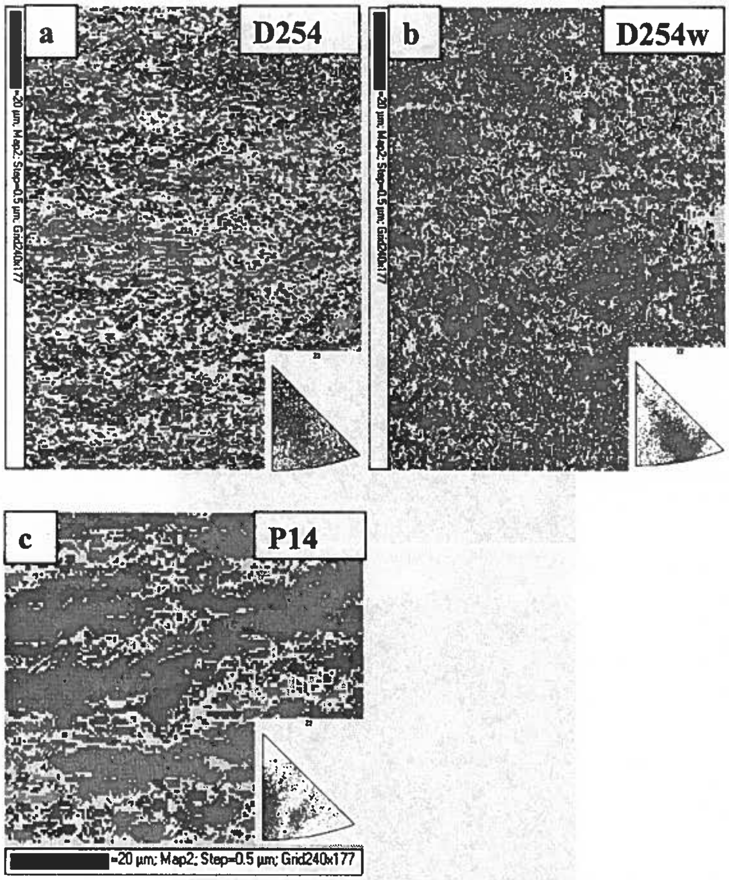


Fig. 7. SEM observations using OIM™ mapping technique: a) D254, b) D254w and c) P14 samples

5. Conclusions

The AlCu5AgMgZr alloy, processed by ECAP at 250 ÷300°C up to $\varepsilon \sim 4$ resulted in final grain size $d =\sim 1\mu$m, which can be important for superplastic deformation.

The elongated grains of width $d_w = 1 \div 5\mu$m are presented in the background of randomly distributed inclusions in AlCu4SiMn, processed by ECAP at 250÷300°C up to $\varepsilon \sim 4$.

The changes of orientation during the ECAP processing deduced from the global texture measurement (neutron diffraction) are strongly dependent on deformation; the both studied alloys behave similarly but the influence of alloying is noted.

The short time recrystallization induces selective grain growth and lowers the orientation dispersion which was observed in EBSD measurements.

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