Microstructure Development during Equal Channel Angular Extrusion of an Al-3%Cu Alloy

I.Mazurina[1,a], A.Goloborodko[1,b], R.Kaibyshev[2,c], H.Miura[1,d],T.Sakai[1,e]

[1]Department of Mechanical Engineering and Intelligent Systems, UEC, Tokyo (The University of Electro-Communications), Chofu, Tokyo 182-8585, Japan

[2]Institute for Metals Superplasticity Problems RAS, Ufa, 450001 Khalturina 39, Russia

[a]inna@sakai.mce.uec.ac.jp, [b]sasha@sakai.mce.uec.ac.jp, [c]rustam@anrb.ru,
[d]miura@mce.uec.ac.jp, [e]sakai@mce.uec.ac.jp

Keywords: Aluminum alloy, equal channel angular extrusion, severe plastic deformation, fine–grained structure.

Abstract. Microstructural evolution during equal channel angular extrusion (ECAE) was investigated in a coarse-grained dilute aluminum alloy, Al-3%Cu, at a temperature of 250°C. Scanning electron microscopy (SEM) with electron back scattering diffraction (EBSD) and optical metallography (OM) was used to reveal the structural changes in the alloy deformed up to a strain of ε=12. The microstructural evolution at initial and moderate stages of deformation is characterized by the formation of low angle boundaries and deformation bands with moderate misorientations in grain interiors. With further deformation the number and the misorientation of the deformation bands increase, finally leading to the subdivision of original grains by these bands and then the development of fine grains with an average size of about 6 μm at ε=12. The evolution of deformation bands in initial grain interiors and their role on new grain formation are discussed in detail.

Introduction

Equal Channel angular Extrusion (ECAE) is very effective for grain refinement of metals and alloys with submicron levels [1-7]. It was recently shown [8-12] that submicrocrystalline structures are evolved even in pure aluminum and aluminum alloys with high stacking fault energy during hot deformation via ECAE. [8-12] Fine-grain evolution takes place in ECAEed Al alloys containing second phase particles and nano scale dispersoids, e.g. an Al-3%Mg-0.2%Sc [9] in a temperature interval from 473 to 573K, a 7475 Al alloy modified by Zr [10] at temperatures ranging from 523 to 623K and a 2219 Al alloy [11]. The authors [9-11] suggest that high density precipitate particles and coherent dispersoids of Al_3(Zr, Cr and Sc) phase can result in rapid increase in the misorientation of strain induced dislocation boundaries due to strong pinning effect or retardation of dynamic recovery, finally followed by the formation of new fine grains with high angle boundaries (HABs) in high strain. On the other hand, new grain structure evolution was also investigated in pure Al deformed at room temperature and several simple Al-based alloys at temperatures below 200°C. Yamashita [9] and Kaibyshev et al. [12] reported that new fine grains with HABs are formed during ECAE at temperatures only below 200°C. They suggest that hard development of HABs in pure Al and Al-3%Mg alloy can result from frequent operation of dynamic recovery at temperatures of around or above 200°C.

The present work was aimed to investigate the microstructural evolution processes and new fine grain structure formation in dilute Al-3%Cu alloy during ECAE at a rather high temperature, e.g. 250°C. A special attention was paid to examine the misorientation changes of deformation-induced boundaries developed during ECAE.

Material and experimental techniques

An Al-3%Cu (in mass pct) alloy was manufactured by direct chill casting and subjected to solution treatment for 4 h at 520°C. The initial structure consisted of essentially equaxed grains with average grain sizes of ~300 μm. The particles of second θ-phase (Al_2Cu) with an average diameter 0.5-2 μm were uniformly distributed in grain interiors. Samples used for ECAE were cut into cylinders with a diameter of 20 mm and a length 100 mm. The ECAE pressing was carried out in air using an isothermal die with a circular internal cross-section. The channel had an L-shaped configuration with angles ϕ and ψ (as defined by Iwahashi *et al.*) [2] both equal to 90°. Deformation through the die produces a strain of ~1 per pass. The samples were deformed repeatedly to the strains of ~1, ~2, ~4, ~8 and ~12 at a temperature 250°C by using rote A [2,3]. Specimens for microstructure analysis were cut from central part of the pressed samples in parallel to the extrusion direction. Deformed microstructure was examined after etching by a standard Keller solution. Average crystallite sizes were measured by using a linear line intersect method in longitudinal and transverse direction of elongated grains. Orientation imaging microscopy (OIM) and misorientation analysis was carried out by EBSD technique. OIM maps were acquired using a LEO-1530 SEM fitted with automated HKL-EBSD pattern collection system provided by HKL Technology, Inc.

Experimental results

Typical microstructures developed during ECAE at 250°C are shown in Fig.1. It is remarkable to see that the deformed microstructures are highly inhomogeneous. Particularly, microstructures developed after the first passage are mainly characterized by the elongation of initial grains along

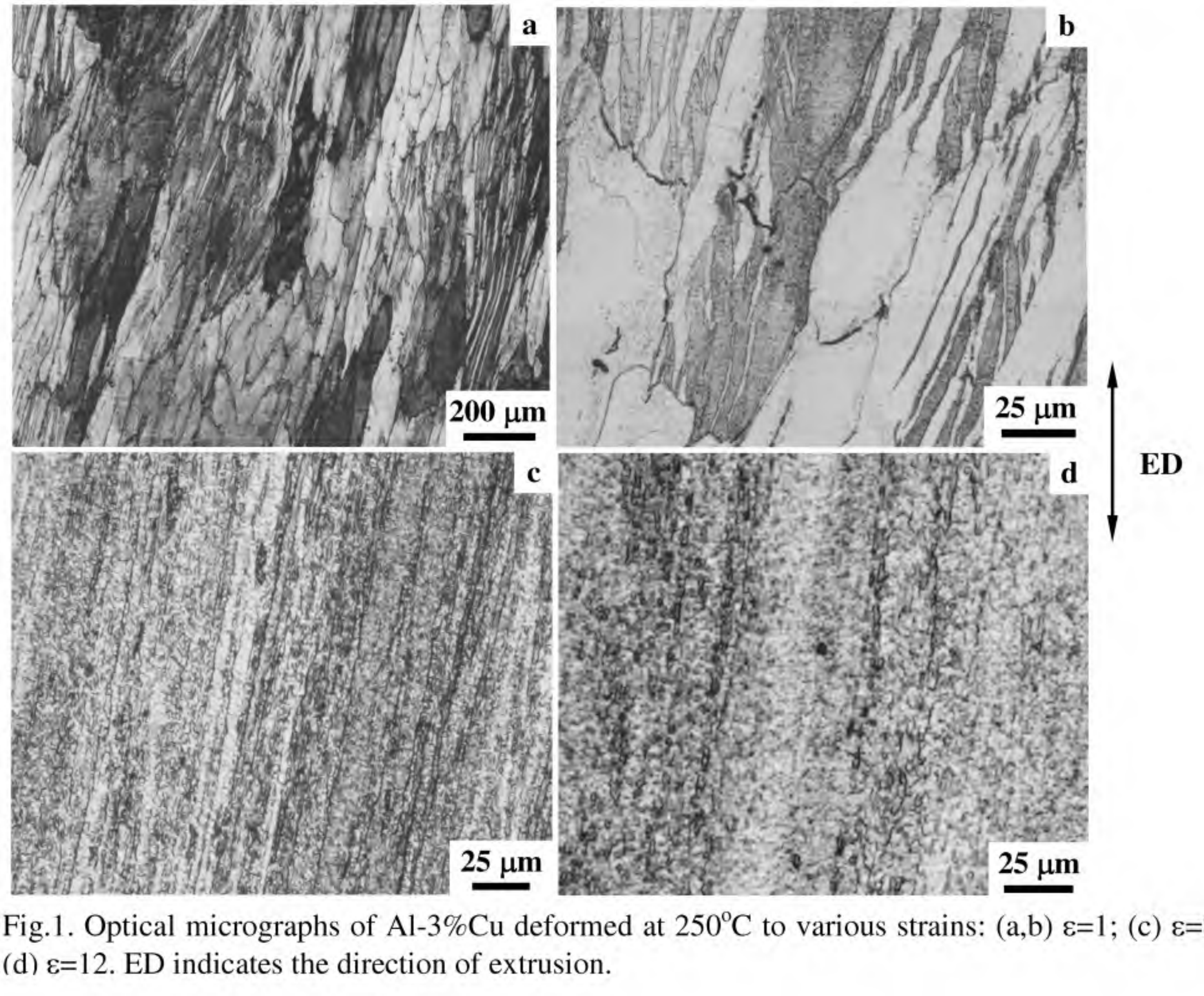

Fig.1. Optical micrographs of Al-3%Cu deformed at 250°C to various strains: (a,b) $\varepsilon=1$; (c) $\varepsilon=8$; (d) $\varepsilon=12$. ED indicates the direction of extrusion.

extrusion direction (ED), in which deformation bands (DBs) are formed heterogeneously (Fig. 1a).

The longitudinal size and the apparent width of these bands were over 400 μm and varied from 6 to 40 μm, respectively (Fig. 1b). It has been reported that such DBs are developed in pure Al and an Al-0.13%Mg alloy during ECAE at room temperature [6,8]. Further deformation in strain interval from $\varepsilon=2$ to 8 leads to an increasing number of DBs and formation of new fine grains in the regions with high density of DBs (Fig. 1c). The formation of new grains may result from the intersection or the subdivision of thin DBs due to impingement of the boundaries. With further straining up to $\varepsilon=12$ fine grained microstructures are frequently formed in the whole material (Figs.1d). However, some parts of original grains still remain even at high strains (Fig. 1c,d).

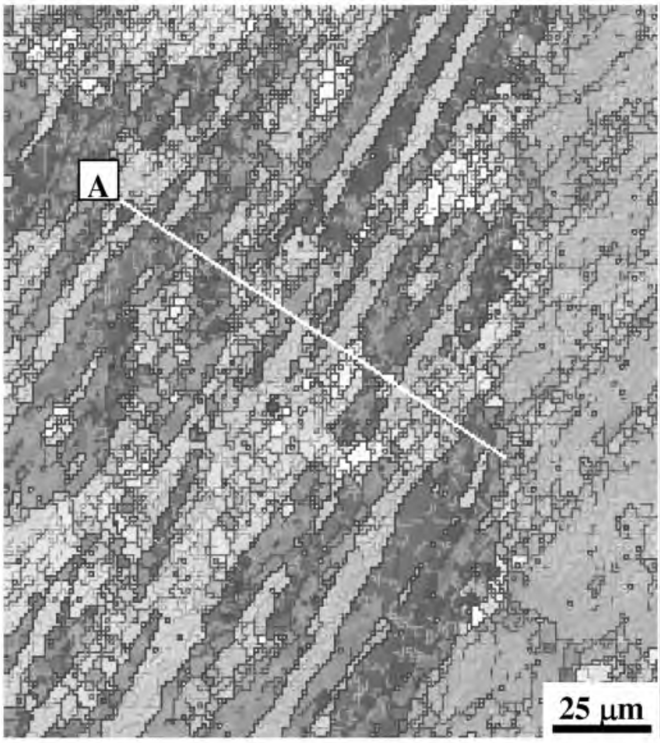

Fig.2 EBSD map of Al-3%Cu deformed to $\varepsilon=2$ at 250°C. Gray and black lines correspond to the boundaries of BDs and new grains with misorientation from 5 to 15° and >15°, respectively.

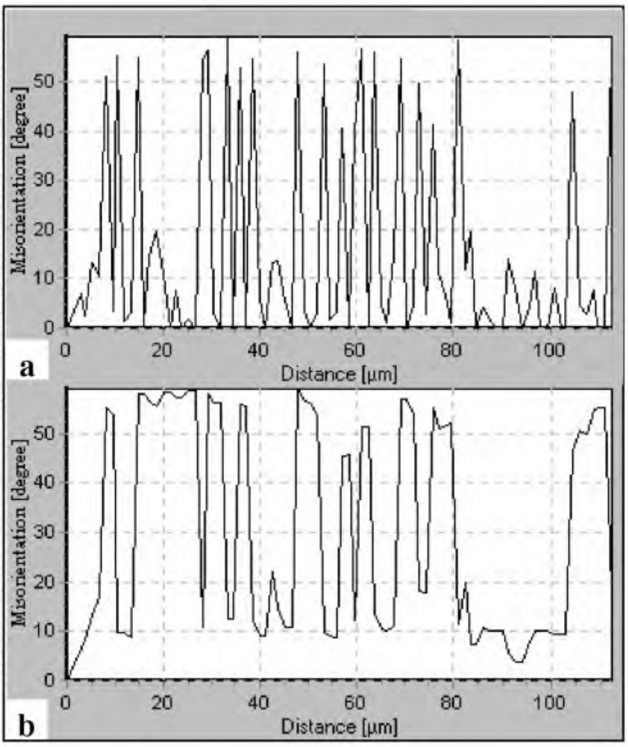

Fig.3 (a) Point-to-point and (b) cumulative misorientations of strain induced boundaries developed along the line A (Fig. 2) in Al-3%Cu deformed to $\varepsilon=2$ at 250°C.

A typical OIM map of Al-3%Cu deformed to $\varepsilon=12$ and corresponding misorientation profiles are presented in Figs. 2 and 3, respectively. Fig. 3 presents a distribution of typical point-to-point ($\Delta\theta$) and cumulative (point-to-origin $\Sigma\Delta\theta$) misorientations developed along the line A showed in Fig. 2. $\Delta\theta$ defines a relative difference of crystal orientation between two adjacent scan points with a step of 1 μm. The misorientation analysis shows that network of low and moderate angle boundaries are inhomogeneously developed in early stage of deformation (Fig. 2). The moderate angle boundaries with misorientation angles ranging from 10 to 15 degrees may correspond to those of DBs evolved during ECAE. The boundaries of DBs can transform to high angle ones and the new fine crystallites are developed along these boundaries. It should be noted that the misorientation of boundaries of DBs rapidly rises and ranges from 20 to 50° even at $\varepsilon=2$ (Fig. 3a). As result, microstructure developed at $\varepsilon=2$ consists of the region of new fine grains with HABs and the parts of original grains with LABs or boundaries of DBs with moderate angle (Fig. 2). In Fig. 3b point-to-origin misorientation is plotted as a function of distance along the line A. Here, it is seen that misorientation within the DBs interiors, which are limited by HABs defined in Fig. 3a, does not change significantly or remains nearly constant. It is interesting to note that new grains are frequently evolved in the regions enclosed by DBs with HABs, as shown in Fig.2.

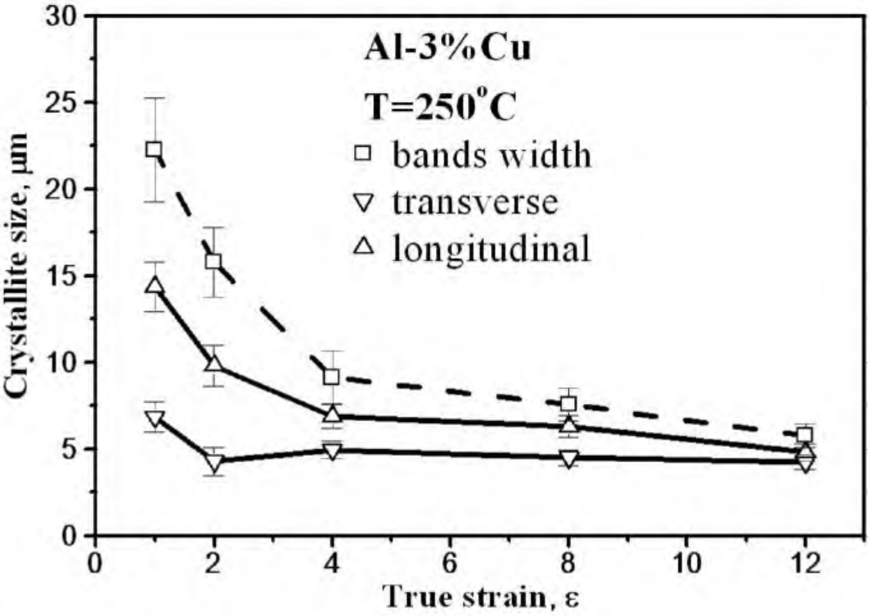

Fig. 4. Changes in crystallite size for Al-3%Cu. Crystallite size was measured in parallel and transverse to elongated substructures developed by ECAE at 250°C.

Strain dependence of the average grain sizes in the regions of newly developed grains is represented in Fig. 4. The width of DBs is also plotted against strain in Fig.4. The average sizes of crystallite were measured in the transverse and longitudinal directions. It is seen that the longitudinal size of grains and the width of DBs tends to decrease rapidly with increasing of strain and approaches a same value of about 6 μm at ε=12. It is noted here that the average width of DBs is about twice of crystallite size measured in the transverse direction, which is roughly constant in a strain interval from ε=2 to 8.

Discussion

The present results shows that grain refinement takes place in the Al-3%Cu deformed by ECAE up to strains of 12 at 250 °C. Mechanism of fine grain formation can be directly associated with evolution of deformation bands (DBs) during ECAE. First passage of ECAE results in formation of DBs with low to moderate angle misorientations. DBs are inhomogeneously formed not only in macroscopic scale, but also microscopically in the initial grain interiors. The number of DBs and their intersections increase with further deformation to high strains. Such DBs are very stable and persistent against deformation. Their boundaries with low to moderate misorientation angle can transform into HABs faster compared with those of subgrain structure with LABs. This may accelerate the formation of a new grain structure with HABs. As a result, new grains with a size of 6 μm were firstly observed in the regions containing high density DBs. It can be concluded that microstructural evolution occurs via strain induced continuous reactions, that is similar to continuous dynamic recrystallization (cDRX).

All of these results are similar to characteristics of cDRX for pure Al [6], Al-0.13%Mg [8] deformed by ECAE at room temperature as well as 7475 Al alloy [10] deformed in the same conditions as the Al-3%Cu. It was also reported in these works that grain refinement process was related to evolution of deformation bands, which appeared inhomogeneously due to development of strain gradient during deformation. It is found from the present results described above that formation of DBs also takes place in the investigated Al-3%Cu alloy ECAEed at 250°C. At early stages of deformation DBs boundaries have moderate misorientation and then, rapidly transform into HABs with further deformation (Fig. 3a). On the other hand, the crystal orientation of DBs and one of the matrixes are frequently alternated at the boundaries of DBs (Fig. 3b). It is also noticeable that misorientation changes within bands interiors are rather constant. The subdividing process of initial coarse grains by DBs brings about a progressive reorientation of matrix inside these bands. Such DBs may be similar to microhear bands observed in cold-rolled Al-0.13%Mg [14] and multidirectional forged 7475 Al alloy at 490°C [15], where they were associated with high strain heterogeneity developed during deformation. The appearance of strain gradient in the present Al-3%Cu alloy even at high temperature of 250°C may be associated with coarse second phase precipitates distributed inhomogeneously (Fig. 1). It should be also noted that numerous microshear bands were formed in the regions rich by second phase particles. This factor is clearly responsible for much more rapid development of both the density and misorientation of new microshear bands formed at lower strains. Consequently, particles may retard or prevent any relaxation of strain gradients. This is considered to be another factor promoting evolution of microshear bands during high temperature deformation.

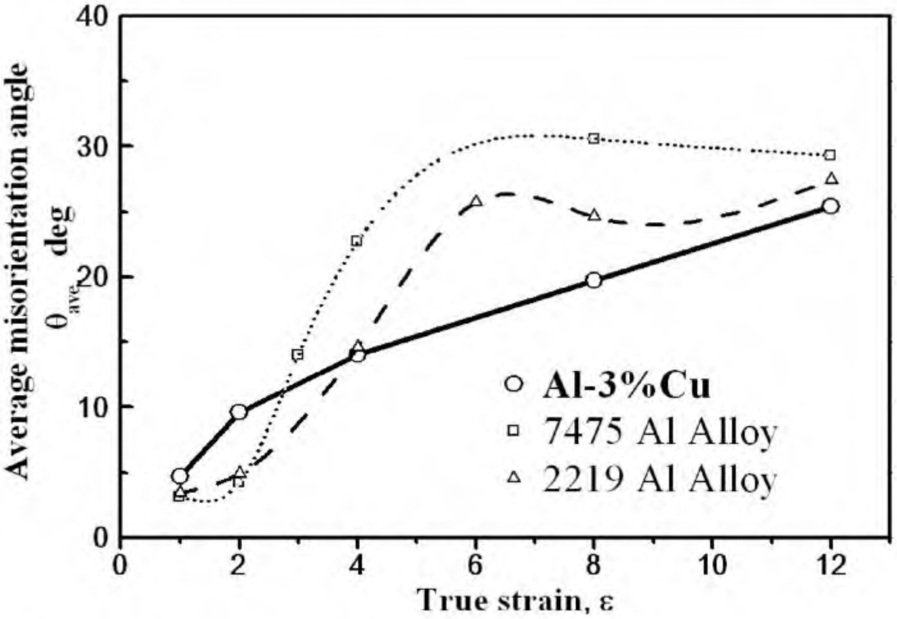

Fig. 5 Changes in average misorientation angle θ_{ave} of deformation induced boundaries with strain for Al-3%Cu deformed at 250°C

Fig. 5 shows changes in the average misorientation angle of deformation induced boundaries measured by EBSP for Al-3%Cu alloy ECAEed at 250° C. The data measured by TEM for 2219 Al [13] and 7475 Al [10] alloys ECAEed at the same conditions are plotted here for comparison. The average misorientation of Al-3%Cu tends to increase continuously with strain and reaches a value of 24° at $\varepsilon=12$. In contrast, the average misorientation of 7475 and 2219 Al alloys rise rapidly at initial stages of deformation from $\varepsilon=2$ to 3 and reaches a saturation value of about 30° with further deformation. The differences in the rate of increase in misorientation may be related to development of microstructures in these alloys. It has been clarified in [14] that the boundary misorientations of microshear bands developed in cold-rolled Al alloys also increase continuously with progress of deformation. It can be concluded that development of microshear bands is directly associated with new grain refinement.

Summary

Equal-channel angular extrusion of Al-3%Cu alloy with total strain of about 12 provides the formation of uniform new fine-grained structure with an average grain size ~6 μm at 250°C. New fine grains are developed by initial coarse grain subdivision due to formation of microshear bands having moderate angle misorientation. The number and average misorientation angle of these boundaries increase with deformation, finally leading to development of a new fine-grained structure. Presence of the particles causes heterogeneity in deformed structure. Frequent formation of microshear bands takes place rapidly due to high density of precipitates.

Acknowledgments

This work, supported by the DAAD (German Academic Exchange Service), was performed in part at the Institute of Physical Metallurgy and Metal Physics, IMM RWTH - Aachen, Germany. One of the authors (I.M.) would like to express hearty thanks to the Japanese government for providing the scholarship.

References

[1] F.J.Humphreys, P.B. Prangnell, J.R. Bowen, A. Gholinia, C. Harris: Phil. Trans. R. Soc. Lond. Vol. A357 (1999), p. 1663.
[2] Y. Iwahashi, Z. Horita, M. Nemoto, T. G. Langdon: Acta Mater. Vol. 45 (1997), p.4733.
[3] P.F. Meier and S.K. Estreicher: Mater. Sci. Forum Vol. 83-87 (1992), p. 119.
[4] Y. Iwahashi, Z. Horita, M. Nemoto, T.G. Langdon: Acta Mater. Vol. 46 (1998), p. 3317.
[5] S. Ferrase, V. Segal, K. Hartwig, R. Goforth: Metall. and Mater. Trans. A Vol. 28 (1997), p.1047.
[6] S. D. Terhune, D. L Swisher, K. Oh-Ishi, Z. Horita, T. G. Langdon and T. R. McNelley: Metall. and Mater. Trans. A Vol. 33 (2002), p. 2173.
[7] A. Gholinia, P. B. Prangnell and M. V. Markushev: Acta Mater. Vol. 48 (2000), p. 1115.
[8] P.J.Apps, J.R.Bowen, P.B.Prangnell: Acta Mater. Vol.51 (2003), p. 2811.
[9] A. Yamashita, D.Yamaguchi, Z.Horita, T.G.Langdon: Mat. Sci. Eng. A Vol. 287 (2000), p. 100.
[10] A.Goloborodko, O.Sitdikov, R.Kaibyshev, H.Miura, T.Sakai: Mat. Sci. Eng. A Vol. 381 (2004) p.121.
[11] O.Sitdikov, R.Kaibyshev, I.Safarov, I.Mazurina: Phys. Metal. Metallogr. Vol. 92 (2001), p. 2629.
[12] R. Kaibyshev, I. Mazurina, I. Denisova and O. Sitdikov: Hot Plastic Deformation of Al Alloys III, TMS (The Minerals, Metals & Materials Society), (2003), p. 151.
[13] R.Kaibyshev, I.Mazurina: Mat.Sci. Forum Vols. 467-470 (2004), p.1251.
[14] P.J.Hurley, F.J. Humphreys: Acta Mater. Vol. 51 (2003), p. 1087.
[15] O.Sitdikov, T.Sakai, A.Goloborodko, H.Miura, R.Kaibyshev: Phil. Mag. Vol.85 (2005) in press.