The Effect of Strain on the Development of Ultra-fine Grain Structure in 7055 Aluminium Alloy Processed by Equal Channel Angular Extrusion

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Abstract. Grain refinement taking place in a commercial 7055 aluminum alloy under equal channel angular extrusion at a temperature of 250°C, was examined. The material was deformed up to a total strain, ε, of ~12. At $\varepsilon \approx 1$, the development of subgrain bands was found. Upon further straining the average misorientation of deformation-induced boundaries increases; low-angle grain boundaries (LAGBs) gradually convert into true high-angle grain boundaries (≥15°) (HAGBs). At $\varepsilon \approx 4$, a structure consisting of boundaries with low and high angle misorientations was observed. At $\varepsilon \approx 12$, a structure with an average grain size of ~0.7 μm was formed. This size is roughly similar to that for subgrains developed at preceding strains. It was shown that the formation of submicrocrystalline grains occurs through continuous dynamic recrystallization both along initial boundaries and within interiors of original grains as well.

Introduction

Aerospace industry has a great interest in the fabrication of complex parts from novel 7055 aluminum alloy, denoted as 7055 Al herein, due to its high strength combining with sufficiently high fracture toughness, fatigue, ductility and corrosion resistance [1]. At the same time, a low formability restricts the use of the coarse grained 7055 Al. It is known [2] that the formation of a uniform submicrocrystalline (SMC) grained structure in aluminum alloys by severe plastic deformation provides enhanced workability and serves properties.

Recent work has demonstrated that substantial grain refinement in a 7055 Al can be achieved through the introduction of severe plastic deformation via equal channel angular extrusion (ECAE) [3]. However, in these studies not attention has been given to the microstructure evolution resulting in the grain refinement in these studies; examination of the deformed state during severe deformation of 7055 Al seems to have not been carried out as yet.

The general features of the microstructure evolution during deformation processing of aluminum alloys were examined by various works [4-11]. It was reported [4,5] that SMC structure occurs by the extension and compression of initial grain boundaries followed by the discontinuous formation of transverse HAGBs. Other investigations have emphasized the role of continuous dynamic recrystallization (CDRX). The mechanism of CDRX frequently operates in a wide range of metallic materials including aluminum alloys during deformation at elevated and high temperatures [2,6-11]. It is originally associated with formation of low angle subgrain boundaries at early stages of deformation and their gradual transformation into high angle ones with further straining due to accumulation of lattice dislocations [10,11]. New grains result from the gradual increase in misorientation between subgrains during plastic deformation.

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The objective of the present work is to study the microstructural evolution in a 7055 Al subjected to ECAE at 250°C. Special attention was paid to characterizing the origin of fine grained structure and the misorientation characteristics of deformation-induced boundaries with the intent of establishing the mechanism of grain refinement.

2. Experimental Procedure

The 7055 Al with a chemical composition of Al-8.2%Zn-2.1%Mg-2.2%Cu-0.2%Zr-0.09%Mn-0.09%Fe-0.07%Si-0.04%Ni-0.02%Ti-0.08%Cr (in weight %) was manufactured by direct chill casting and then homogenized at 470°C for 24 h and quenched into water. Next, overaging at 410°C for 8 h with following cooling in a furnace was carried out in order to produce homogeneous distribution of coarse seconds phase particles (Fig.1). The alloy had a grain size of ~80 μm. The bars with diameters of 20 mm and length of 100 mm were machined from central part of the ingot parallel to the height direction.

The ECAE processing 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~90° and ψ~1° (as defined by Iwahashi et al.) [12]. Deformation through this die produced a strain of ~ 1 for each pass [12,13]. The pressing speed was approximately 10 mm/s. The rods were extruded to several strains (namely: 1, 2, 4, 8 and 12 passes) at 250°C, and the samples were rotated by 90° in the same direction after each pressing (*i.e.* route B_C) .

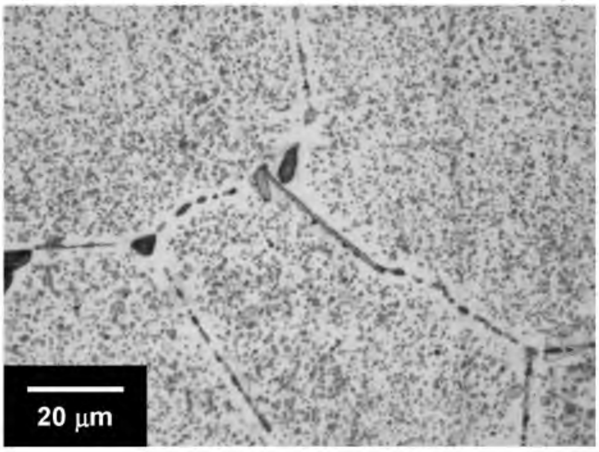

Fig.1 The microstructure of 7055 Al subjected to heat treatment

Following ECAE, small specimens were cut from the central area of pressed rods parallel to the extrusion direction. For the electron-backscattering diffraction (EBSD) analysis these specimens were light electropolished to give a strain-free surface. EBSD orientation maps were recorded using a Jeol JSM-840 SEM fitted with automated HKL-EBSD pattern collection system with an angular resolution of ~1° provided by Oxford Instruments, Ltd. The arbitrary area was automatically scanned with a step size of ~0.15 μm. In the data presented, HAGBs were defined as $\theta \geq 15°$ in misorientation and LAGBs as $3° < \theta < 15°$. HAGBs and LAGBs are depicted in EBSD maps as black and white lines, respectively. Specimens for TEM examination were cut from extruded rods in exactly the same way as for EBSD examinations. Other details of TEM examinations were described in an earlier report [3].

3. Experimental Results

3.1. Microstructure Evolution during Severe Deformation. Fig. 1a. gives an example of the microstructure and associated SAED pattern after a single passage through the die. A single pressing leads to a substantial reduction in the grain size and microstructure now consists of subgrains; as demonstrated by the SAED pattern, the grain boundaries have low angle misorientations. These subgrains are ~1.8 μm in length along the shear direction and ~0.73 μm in thickness. A high density of entangled dislocations is present inside of subgrains. It was observed that the deformed structure is inhomogeneous; two types of structural components could be distinguished within old grains. Deformation bands (DBs) are developed within grains, which were not favorable for multiple slip (Fig. 1b). In the grains, which were favorable for multiple slip on the {111} <110> systems, three-dimensional mixed arrays of LAGBs and HAGBs had evolved. Subgrains are predominant here, and new equiaxed grains can also be found. Subgrains appear homogeneously both within grain interiors and along original boundaries. However, the new grains in the vicinity of the initial boundaries are slightly larger than that within the grain interiors. This suggests a moderate local strain gradient near the old grain boundaries. In general, the population of HAGBs is about 30%. Therefore, in 7055 Al a significant portion of HAGBs is achieved and new recrystallized grains have developed even after ε ~1.

At ε~4 the subgrains interior were free of dislocations (Fig. 2c) and fraction of HAGBs increased to 55%. The fourth pass lead to the occurence of well-defined arrays of deformation-induced boundaries within the interiors of old grains with an orientation favorable for multiple slip (Fig. 2d). These grains contain both recrystallized grains outlined by HAGBs and subgrains delimited by HAGBs in extrusion direction and LAGBs in transverse direction. For other orientations, recovered grains and (sub)grains could be observed within the original grains (Fig. 2d).

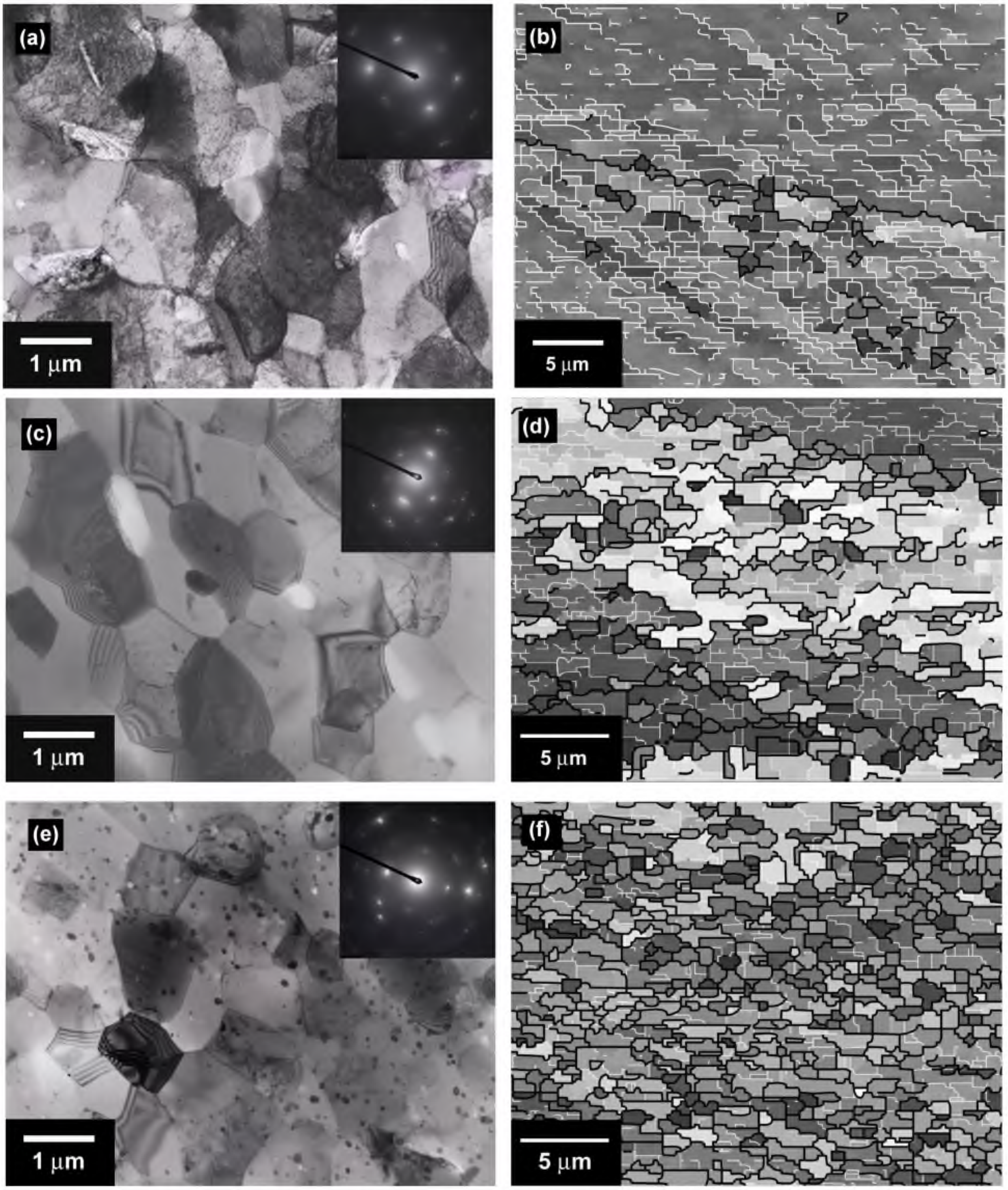

Fig.2 Typical microstructure and EBSD maps of 7055 Al pressed to (a,b) ε~1; (c,d) ε~4; (e,f) ε~12.

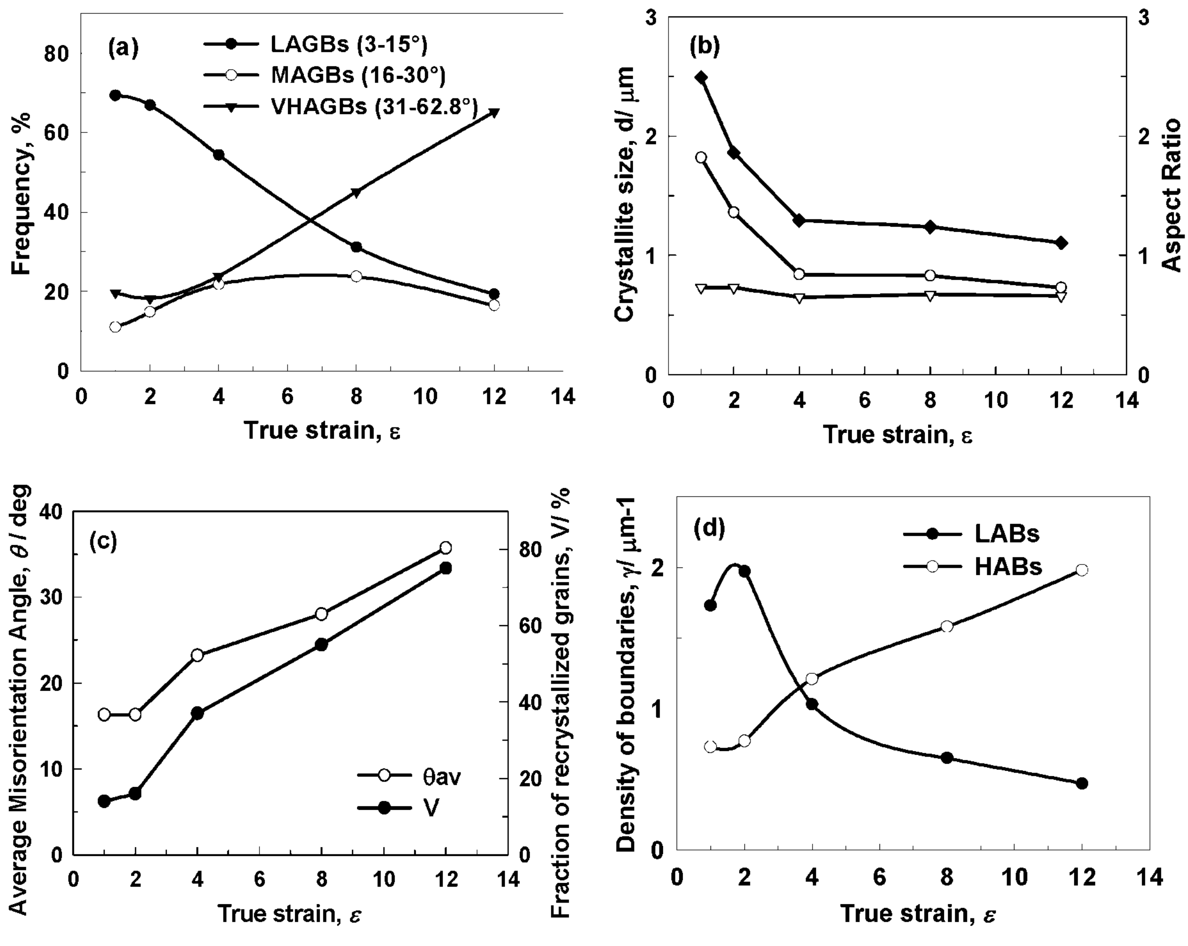

Fig. 3. Effect of strain on (a) population of LAGBs, MAGBs and VHAGBs; (b); the crystallite size, d, and AR; (c) average misorientation of deformation-induced grain boundaries, θav, and fraction of recrystallized grains; (d) density of the boundaries.

It was observed that pressing to a strain of ~8 leads to the formation of a partially recrystallized structure; most of deformation induced boundaries are high-angle in nature. LAGBs were observed within relatively coarse grains. The EBSD and TEM data suggest that the subgrains evolved at earlier strains are replaced by fine grains at higher strains. The average misorientation of ~28° and the population of HAGBs of ~70% suggest that grain refinement is almost complete at this strain. At ε ~8, the volume fraction of grains outlined by HAGBs is about 55%.

After ε~12, the uniformity of the recrystallized structure increases (Figs. 2e and 2f). The fraction of HAGBs attains a value of ~81%. Most of the deformation-induced boundaries are high-angle in misorientation and the recrystallized grains exhibit an equiaxed shape (Fig. 2e). Grain aspect ratio (AR), is close to ~1.1. However, a low fraction of LAGBs was found within deformed microstructure (Figs. 2f).

3.2. Parameters of Deformed Microstructure as a Function of Strain. The effect of strain on the relative frequencies of boundaries is shown in Fig. 3a. These boundaries defined as; low angle (LAGBs 3-15°), medium angle (MAGBs 16-30°) and very high angle grain boundaries (VHAGBs 31-62.8°). As the strain rises the incidence of LAGBs gradually decreases from 70 % to 19% at ε≈1 and ε ≈12, respectively. At ε ≤2 the incidence of VHAGBs decreases due to an increment of the incidence of MAGBs which is decreased at higher strains. No significant densities of boundaries with misorientation greater than 30° are formed up to strains of 4, and these boundaries start saturating at larger strain levels.

The effect of strain on the crystallite size, d, and the aspect ratio (AR) of crystallite defined as the ratio of the crystallite dimension in the shear direction to that in the transverse direction is summarized in Fig. 3b. In the strain interval from 1 to 4 the crystallite size rapidly decreases owing to an increasing fraction of equiaxed (sub)grains. It is worth noting that the transverse size of subgrains remains practically unchanged and the longitudinal size of the elongated subgrains tends to decrease with strain due to the formation of transverse LAGBs (Figs. 2a-2d). As a result, the AR decreases from ~2.49 at ε~1 to ~1.29 at ε~4 (Fig. 3b). Upon further ECAE processing the crystallite size tends to slightly decrease, and AR decreased to ~1.1 at ε~12.

Changes in the average misorientation (θav) and fraction of recrystallized grains, with increasing strain are summarized in Fig. 3c. It can be seen that the average misorientation remains unchanged until $\varepsilon \approx 2$ and then rapidly increases to 23.3° at $\varepsilon \approx 4$, followed by gradual rise with further deformation. A rapid increase in the average misorientation and the fraction of recrystallized grains can be associated with the development of new HAGBs. The data in Fig. 3 clearly demonstrate the rate of grain refinement in 7055 Al. At ε~8, the average misorientation of $\theta \approx 28°$ is close to 30°, that is, to the saturation value for FCC materials under severe plastic deformation [14]. The fraction of HAGBs (*i.e.* MAGBs+VHAGBs) ($\theta > 15°$) attains a value of ~70% (Fig. 3c), which is the critical HAGBs fraction for the granular structure [4].

4. Discussion

The experimental data shows that grain refinement in Al 7055 occurs through a continuous strain-induced reaction, which has elsewhere been termed continuous dynamic recrystallization (CDRX) [11,14]. This mechanism of microstructure evolution during severe deformation in Al 7055 can be summarized as follows:

1. At initial stages of deformation ($\varepsilon \leq 2$) the three-dimensional arrays of LAGBs are developed in original grain interiors due to formation of DBs. These DBs formed roughly parallel to shear direction and contain elongated subgrains with misorientation angles of $5° \leq \theta \leq 15°$.
2. The number and the average misorientation of DBs increase with further deformation by ECAE, leading to fragmentation of original coarse grains into different orientation regions and finally to the development of fine equiaxed grains at high strains.

It was shown in [8,9 15-18] that DBs, such as microshear and kink bands, can be developed by strain heterogeneity taking place due to compatibility requirements of neighboring grains or any intrinsic instability of grain during plastic deformation. Formation of DBs can be strongly dependent on deformation mode and deformation conditions as well as material composition and the initial microstructure of materials. At elevated temperatures, dynamic recovery and grain boundary sliding are frequently operating and so can generally result in difficult development of DBs through relaxation of strain heterogeneity [17,18]. The present Al 7055 alloy, however, contains high density of coarse $T(Al_2Mg_3Zn_3)$ and $S(Al_2CuMg)$ second phases particles [19] which are homogeneously distributed in the grain interior (Fig.1). Probably, the $T(Al_2Mg_3Zn_3)$ and $S(Al_2CuMg)$ phase particles produce deformation gradients providing particle-stimulated nucleation [5,18] in Al 7055. DBs can be easily developed under such severe deformation conditions. Moreover, it has been pointed out in [9,16,17] that inhomogeneous deformation characteristics of ECAE can lead to the formation of DBs due to accommodation of strain gradients evolved by ECAE. Repeated ECAE by route B_C can also introduce DBs in different directions and so results in intersection of DBs.

The microstructural changes of the Al 7055 in an initial strain interval ($\varepsilon \leq 2$) are mainly characterized by homogeneous formation of DBs and dislocation subgrains with LAGBs within individual coarse grains (Fig. 2a, 2b). DBs are continuously formed with increasing the strain to ~2, so that the density of LAGBs rapidly grow (Fig. 4d), leading to a fragmentation of grains in differently misoriented regions. It was recently shown [10,20] that LAGBs are formed at a high rate if they are located in the primary slip planes in the shear direction. The LAGBs aligned in the shear direction exhibit increased misorientation in comparison with transverse LAGBs [21] at low strains. It is apparent, that the formation of LAGBs at ε~2 can be attributed to the easy formation of such LAGBs. Transverse LAGBs form within these elongated subgrains by dislocation rearrangement. This provides the transformation of elongated subgrains into relatively equiaxed subgrains.

It is obvious that at ε >2, a decrease in density of LAGBs (Fig. 4d) occurs due to their conversion into HAGBs. The recrystallized grains replace subgrains evolved over a small number of passes through continuous transformation of their boundaries. Dislocation glide occurs within the subgrain interiors [20]. Mobile dislocations move across subgrains and are absorbed by sub-boundaries resulting in an increase in their misorientation. As a result, subgrains convert to SMC equiaxed grains outlined by true HAGBs. It is concluded, therefore, that the process of development of new fine grains can be a series of deformation-induced continuous reactions, which is similar to CDRX [8,9,17,18].

5. Summary

Grain refinement taking place in commercial aluminum alloy 7055 subjected to ECAE at T=250°C was examined. The formation of new grains during ECAE occurs through continuous dynamic recrystallization. At the initial stage of deformation (ε ≤2), three-dimensional arrays of LAGBs evolve homogeneously both within interiors of initial grains and along old boundaries as well. High density of coarse second phase particles accompanied by lower recovery rate operating during lower temperature ECAE leads to rapid development of DBs initially. Further deformation leads to a gradual transformation of LAGBs into HAGBs at a high rate. A new fine-grained microstructure with an average crystallite size of about 0.7 μm develops at strains above ~ 8.

References

1. I. N. Fridlyander: Met.Sci.Heat Treat., 1 (2001),p. 5.
2. R.Z. Valiev, T.G. Langdon: Prog. Mater. Sci., 51 (2006), p. 881.
3. R. Kaibyshev, T. Sakai, I. Nikulin, F. Musin, A. Goloborodko: Mater. Sci. Tech., 19 (2003), p. 1491.
4. F.J. Humphreys, P.B. Prangnell, J.R. Bowen, A. Gholinia, C. Harris: Phil. Trans. R. Soc. Lond., 357A (1999), p. 1663.
5. P.J. Apps, J.R. Bowen, P.B. Prangnell: Acta Mater., 51 (2003), p. 2811.
6. A. Gholinia, F.J. Humphreys, P.B. Prangnell: Acta Mater., 50 (2002) , p. 4461.
7. A. Gholinia, P.B. Prangnell, M.V. Markushev: Acta Mater., 48 (2000), p. 1115.
8. O. Sitdikov, T. Sakai, A. Goloborodko, H. Miura, R. Kaibyshev: Phil. Mag., 85 (2005), p. 1159.
9. A. Goloborodko, O. Sitdikov, T. Sakai, R. Kaibyshev, H. Miura: Mater. Trans., 44 (2003), p. 766.
10. S. Gourdet, F. Montheillet: Mater. Sci. Eng., A 283 (2000), p. 274.
11. H.J. McQueen: Mater. Sci. Eng., A 387–389 (2004), p. 203.
12. Y. Iwahashi, Z. Horita, M. Nemoto, T.G. Langdon: Acta Mater., 45 (1997), p. 4733.
13. K. Nakashima, Z. Horita, M. Nemoto, T.G. Langdon: Acta Mater., 46 (1998), p. 1589.
14. A. Belyakov, T. Sakai, H. Miura, K. Tsuzaki.: Phil Mag., 81 (2001), p. 2629.
15. X. Yang, H. Miura, T. Sakai: Mater. Trans., 44 (2003), p. 197.
16. A. Goloborodko, O. Sitdikov, R. Kaibyshev, H. Miura, T. Sakai: Mater. Sci. Eng., A381 (2004), p. 121.
17. O. Sitdikov, T. Sakai, E.Avtokratova,R. Kaibyshev,Y. Kimura, K. Tsuzaki: Mater. Sci. Eng., A444 (2007), p. 18.
18. F.J. Humphreys, M. Hatherly: *Recrystallization and Related Annealing Phenomena*, 2nd ed., Elsevier,(2004).
19. Ch. Mondal, A.K. Mukhopadhyay: Mater. Sci. Eng., A391 (2005), p. 367.
20. O. Sitdikov, R. Kaibyshev, I. Mazurina, D.R. Lesuer: Mater. Sci. Eng., A334 (2002), p. 104.
21. Y. Fukuda, K. Oh-ishi, M. Furukawa, Z. Horita, T.G. Langdon: Acta Mater., 52 (2004), p. 1387.