Effect of pressing temperature on fine-grained structure formation in 7475 aluminum alloy during ECAP

A. Goloborodko , O. Sitdikov , R. Kaibyshev , H. Miura , T. Sakai

Abstract

The effect of pressing temperature on microstructural development under ECAP conditions was studied in an as-cast 7475 aluminum alloy at temperatures from 523 to 673 K. The samples were pressed by using route A up to strains of 12. New equiaxial grains with high angle boundaries (HABS) start to be formed after three passes and then gradually developed with repeated ECAP, finally, followed by fully development of a new fine-grained structure. The average (sub)grain size tends to increase and the aspect ratio decreases with increasing pressing temperature. The misorientation distribution of strain-induced dislocation boundaries shows a single peak type at low angle misorientations until second pass and then gradually shifts to higher angles with increasing of strain. Increase in pressing temperature retards the transformation kinetics from low angle boundaries to high angle ones, while the fraction of the latter as well as the average misorientation approach a same saturation value irrespective temperature in high strain. The mechanism of ultra-fine grain formation during ECAP is discussed.

Keywords: Pressing temperature; Fine-grained structure; Aluminum alloy 7475

1. Introduction

Metallic materials with ultra-fine-grained microstructures have many advantages of mechanical properties, e.g., an increasing of strength [1,2], low temperature, and high strain rate superplasticity [3–5], etc. Several methods available for producing of ultra-fine-grained structure in bulk materials are based mostly on severely large plastic deformation [1]. One of them is equal channel angular pressing (ECAP), originally developed by Segal et al. [6]. The principle of ECAP is that rod-type samples are extruded through special die consisting of the two channel of equal cross-section intersecting at an angle of 90° or higher [6]. Plastic deformation is introduced by simple shear in a thin layer at the crossing plane of channel. ECAP has a great advantage for producing bulk materials with fine-grained structure in comparison with conventional metal working, such as rolling and extrusion, because the dimensions of samples do not change during deformation and the samples can be repeatedly pressed up to strains greater than 10. In case of rolling and extrusion, the dimensions of billet are always reduced with deformation.

There have been several works showing that ECAP is very effective for grain refinement of aluminum alloys with submicron levels [7–16]. A great attention has been paid to analysis of the ultra-fine-grained microstructures developed in high strain and the effect of such microstructures on the resulting properties and the thermal stability [3–5,7–11]. Only a few studies have been dealt with the effect of pressing temperature on microstructure development under ECAP conditions [9,14,16]. Yamashita et al. [9] studied it using pure Al, an Al–3%Mg and Al–3%Mg–0.2%Sc alloys, and reported that increase in deformation temperature results in increasing of grain size and decreasing of misorientation of strain-induced boundaries. It has been shown in [14] that the proportion of high angle boundaries (HABs) evolved in 5052 Al alloy decreases with increase in pressing temperature and reaches only 14% during ECAP at 573 K. In contrast, other authors reported that the fraction of HABs is higher than 60% in Al alloys subjected to ECAP at 473 K [12,13,15]. Thus, the effect of pressing temperature on fine grain formation during hot ECAP is currently a matter of some debate and is not clear.

The present work was aimed to study the effect of pressing temperature on the microstructural development in an as-cast 7475 Al alloy during ECAP following the previous work on the same alloy at 673 K [15]. The evolution processes of fine-grained structures and their boundary characteristics were investigated. A special attention was paid to analyze the misorientation distribution of strain-induced boundaries and how low angle boundaries transform into high angle ones. In addition, the optimum processing route of ECAP for receiving of fine-grained structure is discussed.

2. Experimental procedure

The alloy used was a 0.16%Zr-modified 7475 Al alloy with the following chemical composition (in mass%): 6.04Zn, 2.46Mg, 1.77Cu, 0.23Cr, 0.16Zr, 0.03Si, 0.04Fe, 0.03Mn and the balance Al. It was fabricated by direct chill casting at the Kaiser Center for Technology and then homogenized at 768 K for 20 h. Samples for ECAP were machined parallel to the ingot axis into rods with a diameter of 20 mm and a length of around 100 mm. The initial microstructure was composed of dendritic lamellas lying parallel to the ingot axis with an average size in the range from 1 to 10 mm in longitudinal direction and from 100 to 200 μm in transverse direction [15]. ECAP was carried out using a circular die in cross-section with a diameter of 20 mm. The die had a channel in L-shaped configuration with an angle of 90° between the two channels and an angle 0° at the outer arc of curvature at the point of intersection. These angles lead to a strain of about 1 in each passage through the die. A heating jacket was put on the die, and the pressing temperature was controlled within ±5 K of the setting temperature. The samples were pressed repeatedly up to strains of 12 at temperatures ranging from 523 to 673 K by using route A, i.e. the orientation of billet was not changed at each pass. The pressed samples were quenched in water after each deformation.

Samples for transmission electron microscopy (TEM) analysis were cut from central places in longitudinal section of the pressed samples in parallel to pressing direction. Specimens for TEM examination were mechanically ground to a thickness of about 200 μm and electropolished in a solution of 30% HNO_3 and 70% CH_3OH at a temperature of 243 K using a Tenupol-3 twin-jet polishing unit. They were then examined using a JEM-2000FX TEM operating at 200 kV. Selected area electron diffraction (SAED) patterns were recorded from areas having diameters of 5 μm. Average crystallite size was measured by a linear line intercept method in longitudinal and transverse directions of elongated crystallites and denoted as, d_L and d_T, respectively. Aspect ratio, AR, was calculated as $AR = d_L/d_T$. In addition, the misorientation of (sub)grain boundaries were studied using a conventional Kikuchi-line technique [17]. The total number of boundaries analyzed was from 60 to 80 in each sample.

3. Experimental results

3.1. Microstructure developed at high strain

Fig. 1 shows typical microstructures with the associated SAED patterns developed at a strain, ε, of 12 during ECAP at temperatures from 523 to 673 K. Fig. 2 shows changes in the average crystallite size and aspect ratio developed with pressing temperature. It is seen in Figs. 1 and 2 that roughly equiaxed crystallites are fully developed accompanying with high density second phase particles. The analysis of SAED patterns shows that mixed networks composed of low and high angle boundaries are evolved at $\varepsilon = 12$ at all the pressing temperatures. Increase in the pressing temperature from 523 to 673 K results in the following changes in microstructures; i.e.: (i) the average crystallite size increases from about 0.6 to 1.7 μm; (ii) the shape of new evolved crystallites become more equiaxed. The aspect ratio of the grains changes from 1.9 to 1.3; (iii) the average size of second phase particles shifts to smaller size under TEM observations, as can be seen in Fig. 1.

It is concluded from Figs. 1 and 2 that ECAP of the as-cast 7475 Al alloy results in significant grain refinement at temperatures ranging from 523 to 673 K, leading to full development of fine grains separated by HABs. Let us study the temperature effect on the process of microstructural evolution taking place during hot ECAP.

3.2. Microstructural evolution process

Microstructural development taking place at 523 and 673 K is comparatively analyzed in some detail. Series of typical TEM microstructures developed at 523 and 673 K are presented in Figs. 3 and 4, respectively. It can be seen that microstructural evolution process depends clearly on deformation temperature. A first pressing at 523 K leads to development of layered substructures having boundaries with medium misorientation angles less then 10° (Fig. 3a). These bands are formed roughly at an angle of 45° to pressing direction. Further straining to $\varepsilon = 3$ results in gradual break-up of the deformation bands followed by (sub)grain formation. Some fine grains having medium to high angle boundaries and also larger (sub)grains are mixedly developed accompanied with high density and rather coarse precipitates (Fig. 3b). After eight passes of ECAP, i.e. at $\varepsilon = 8$, roughly equiaxed grains surrounded by boundaries with medium to high misorientation angles are formed homogeneously (Fig. 3c).

Microstructures developed at 673 K are rather different from those at 523 K, as can be seen in Fig. 4. After a first pressing, a subgrain structure is developed homogeneously and the boundary misorientation is ranged in low angles less than 3° (Fig. 4a). After three passes the misorientation of subboundaries increases accompanied with many secondary phase particles precipitated along the boundaries and in grain interiors. There are some boundaries with misorientation an-

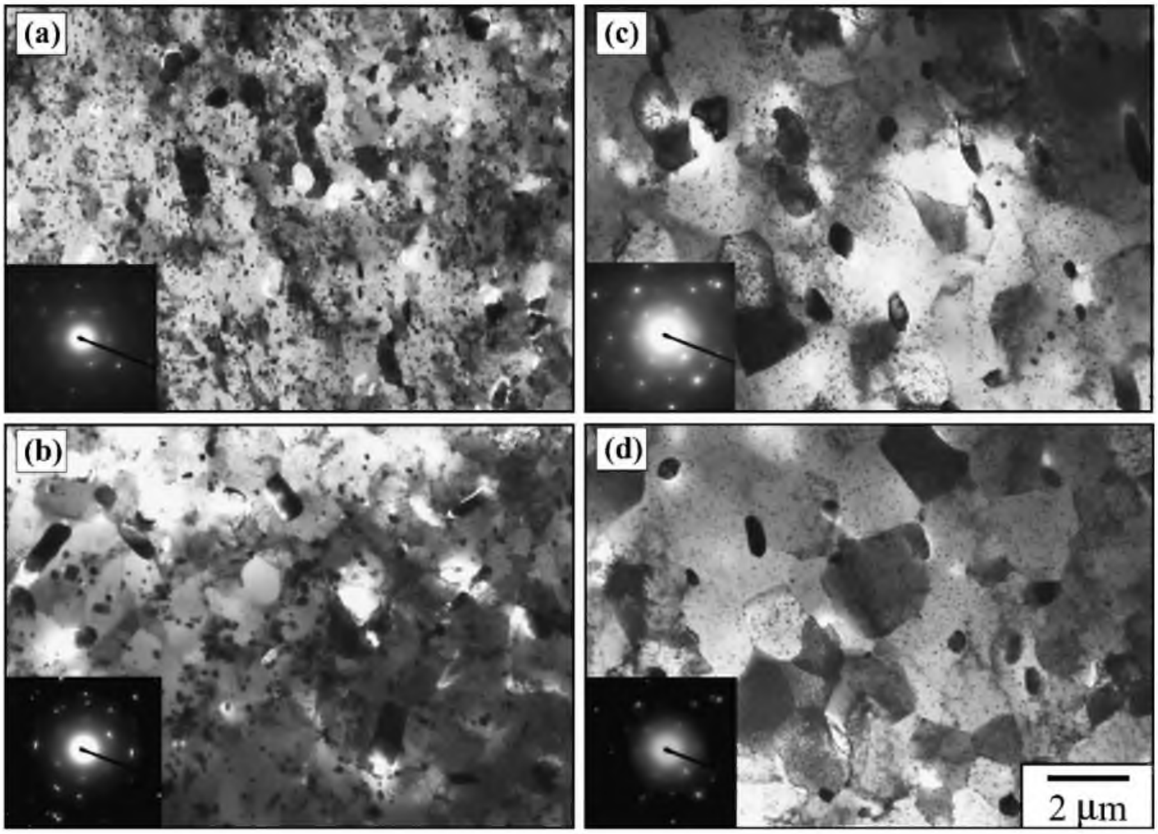

Fig. 1. Typical microstructures and associated SAED patterns for 7475 Al alloy after ECAP. The samples were pressed to a strain of 12 at various temperatures. (a) 523 K, (b) 573 K, (c) 623 K, and (d) 673 K.

gles above 10° evolved, as seen in Fig. 4b. The precipitate particles at 673 K are much finer compared with those at 523 K. After eight passes, roughly equiaxed grains with medium to high angle boundaries are evolved fully in the whole microstructure (Fig. 4c).

Changes in (sub)grains size and the aspect ratio with repeated ECAP at 523 and 673 K are summarized in Fig. 5. Elongated crystallites developed during early ECAP change

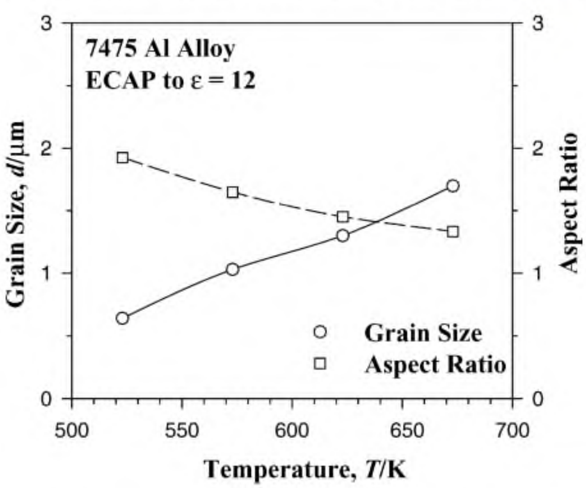

Fig. 2. Changes in (sub)grain size, d, and aspect ratio of new grains developed at a strain of 12 with pressing temperature.

to equiaxed ones by repeated pressing and so the average crystallite size, d, was measured in parallel and transverse to the elongated substructures. It can be seen in Fig. 5a that the transverse crystallite sizes are roughly constant during ECAP at the both temperatures. In contrast, the longitudinal sizes decrease rapidly with increase in strain before $\varepsilon \cong 4$ and approach the transverse ones at high strains. ECAP results finally in formation of roughly equiaxed fine grains with an average size of about 0.6 μm at 523 K and 1.7 μm at 673 K, although the aspect ratio is still large at 523 K.

Fig. 6 shows strain dependence of the distribution of misorientation angles for strain-induced dislocation boundaries developed during repeated ECAP at 523 and 673 K. It can be seen in Fig. 6 that first pass through the die leads to the formation of low misorientation angle boundaries at both the pressing temperatures. Further pressing to $\varepsilon = 4$ shifts the distribution to higher angles and brings about development of a bimodal misorientation distribution with two peaks. The latter appears more clearly at 523 K. This can be resulted from banded substructures evolved at 523 K (Fig. 3). With further repeated ECAP, the fraction of low angle boundaries decreases rapidly and that of high angle ones contrary increases. The misorientation distribution developed at $\varepsilon = 12$ approaches a theoretical distribution for random orientations of fully annealed grains in cubic materials [18], as shown by dashed line in Fig. 6. It contains, however, some large frac-

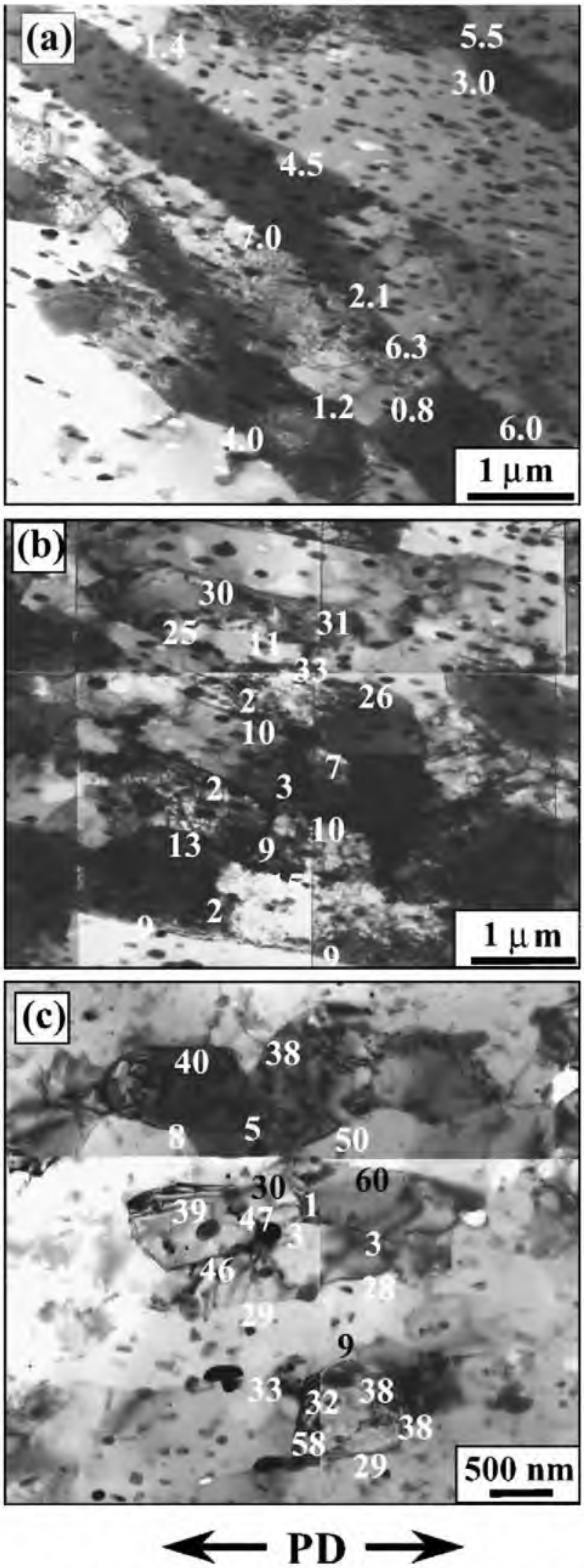

Fig. 3. Typical TEM microstructures evolved during ECAP at 523 K. The samples were pressed up to various strains followed by water quenching. (a) $\varepsilon = 1$, (b) $\varepsilon = 3$ and (c) $\varepsilon = 8$. The numbers indicate misorientation angle in degrees. PD is pressing direction.

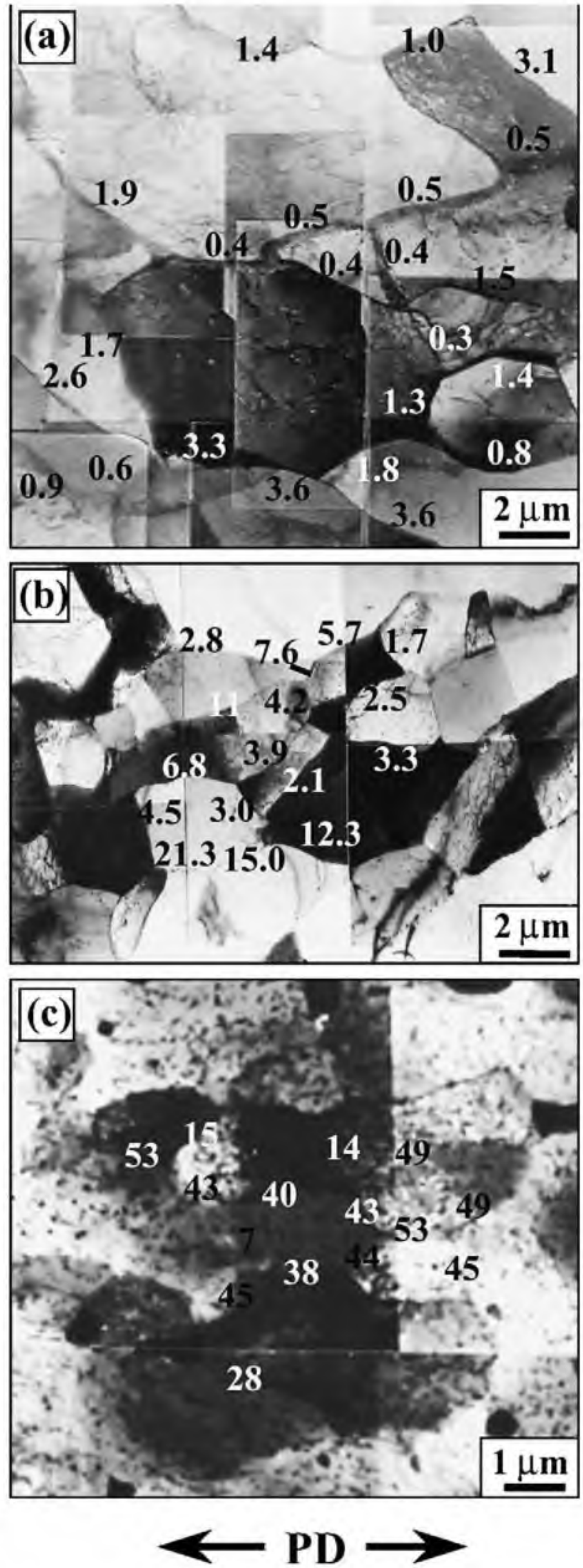

Fig. 4. Typical TEM microstructures evolved during ECAP at 673 K. The samples were pressed up to various strains followed by water quenching. (a) $\varepsilon = 1$, (b) $\varepsilon = 3$, and (c) $\varepsilon = 8$. The numbers indicate misorientation angle in degrees. PD is pressing direction.

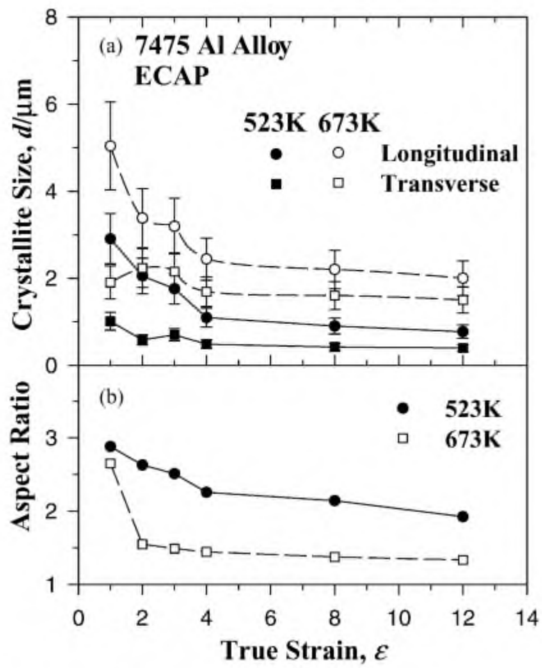

Fig. 5. Changes in (sub)grain sizes, d, with strain during ECAP at 523 K and 673 K. d was measured in parallel and transverse to elongated substructures developed under ECAP.

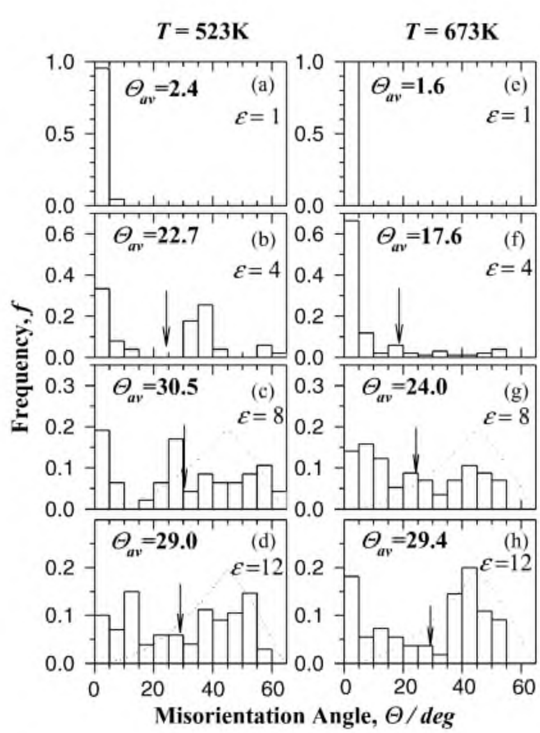

Fig. 6. Change in the misorientation distribution of deformation-induced (sub)boundaries with repeated ECAP for 7475 Al alloy at 523 and 673 K. The broken line indicates the random misorientation distribution predicted by Mackenzie [18]. (a, e) $\varepsilon = 1$, (b, f) $\varepsilon = 4$, (c, g) $\varepsilon = 8$ and (d, h) $\varepsilon = 12$. (a–d) 523 K and (e–h) 673 K.

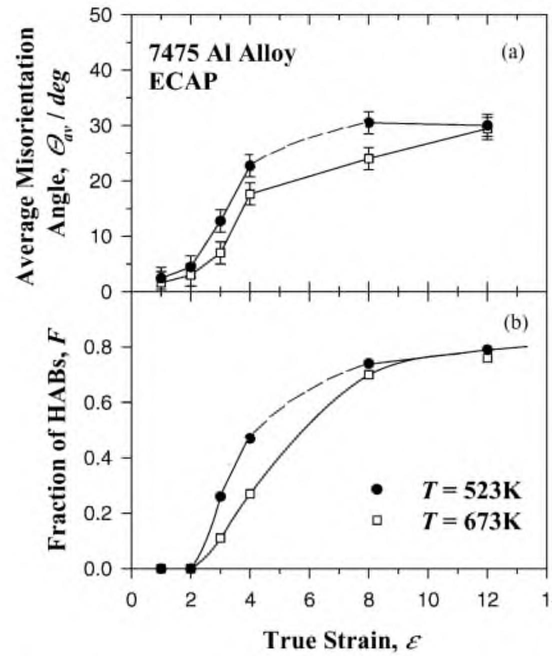

Fig. 7. Changes in (a) average misorientation angle, Θ_{av}, of strain-induced (sub)grain boundaries, and (b) fraction of HAB, F, with repeated ECAP at 523 K and at 673 K for 7475 Al alloy.

tion of low angle misorientations, which is a characteristic of strain-induced grain structure [19].

Some microstructural characteristics developed in the present alloy, e.g. the average misorientation, Θ_{av}, and the fraction of HABs[1], F, derived from Fig. 6, are presented in Fig. 7. It is seen in Fig. 7a that the average misorientation increases gradually to around 4° after two passes, and then rapidly up to around 13 and 7° at $\varepsilon = 3$ at 523 and 673 K, respectively. Θ_{av} increases at different rates in a medium strain range, i.e. $3 \le \varepsilon \le 8$, and then it approaches a roughly same value of about 30° at high strains irrespective of temperature. On the other hand, changes in the fraction of HABs with repeated ECAP are also similar to those in Θ_{av}. It is interesting to note in Fig. 7b that formation of strain-induced HABs scarcely takes place at $\varepsilon \le 2$ and then rapidly in a strain range of $2 < \varepsilon < 3$ irrespective of pressing temperature. In a medium strain range, i.e. $3 \le \varepsilon \le 8$, however, the kinetics of the formation of HABs accelerates with decrease in pressing temperature. The fraction of HABs approaches almost the same value of about 0.75 in $\varepsilon > 8$.

4. Discussion

4.1. Effect of pressing temperature on microstructural development

The effect of pressing temperature on the microstructure development in the as-cast 7475 Al alloy during ECAP can

[1] High angle boundary means the boundary with misorientation angles more than 15°.

be summarized as follows:

(i) Average crystallite size developed increases with increase in pressing temperature and the crystallite shape becomes more equiaxed with raising pressing temperature (Fig. 2).
(ii) Increase in pressing temperature tends to slow down the transformation kinetics from low to high angle boundaries at medium strain range, i.e. $3 \leq \varepsilon \leq 8$ (Figs. 6 and 7).
(iii) The characteristics of new grain structures developed at $\varepsilon > 8$, i.e. Θ_{av} and F, are almost the same irrespective of pressing temperature (Figs. 6 and 7).

The first results of (i) above mentioned are almost the same as those reported in references. The results of (ii) and (iii), however, are different from some previous data [9,14]. It was shown in [14] that in 5052 Al alloy subjected to ECAP up to a total strain of about 5.6, the fraction of HABs evolved decreases from about 55 to 14% with increasing of temperatures from 323 to 573 K. The rather small fraction of HABs in pure Al and Al alloys can be attributed to higher rate of recovery [9,14]. Additionally, the total cumulative strains used in [9,14] is considered to be rather small, i.e. $\varepsilon \leq 8$. It is evident from Fig. 7 that a significant temperature effect on the stain-induced microstructures appears clearly in a medium strain range, i.e. $3 \leq \varepsilon \leq 8$, while it is not observed in $\varepsilon < 2$ and $\varepsilon > 8$.

Let us discuss the reason why the average misorientation angle and the fraction of HABs increase faster during ECAP at lower temperatures. This is something strange because structure evolution can generally be retarded at lower temperatures. It should be remembered first in Figs. 3a and 4a that a banded substructure with medium angle boundaries is developed just after first pressing at 523 K and, in contrast, almost equiaxed subgrans with low angle boundaries are evolved at 673 K. The authors pointed out in [15] that inhomogeneous deformation characteristics of ECAP leads to formation of deformation bands after first pass through the die. These bands are considered to be developed by relaxation of strain gradients resulting from heterogeneous strain introduced by ECAP. Heterogeneous strains may increase with decrease in temperature so as to collectively maintain compatibility with surrounding grains [20]. Repeated ECAP can result in mutual crossing, increasing of the number and misorientation of such deformation bands, followed by the formation of fine grain structure.

Next, the presence of second phase particles and their effect on structure evolution should be considered to play an important role. It is known [21] that the particles of η-phase ($Mg(Zn_2$, $AlCu)$), S-phase (Al_2CuMg), T-phase ($Al_{32}(Mg, Zn)_{49}$), and the $Al_3(Zr/Cr)$ dispersoids are presented in 7XXX series Al alloy up to about 723 K. It is clearly seen in Figs. 1, 3 and 4 that many particles are precipitated along strain-induced boundaries and can restrict rearrangement of lattice dislocation in short and long range, followed by stabilization of developed dislocation substructures [22,23]. This

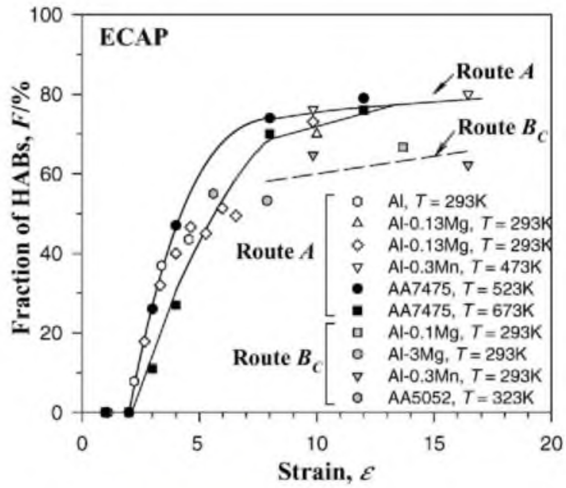

Fig. 8. Changes in fraction of HAB developed with repeated ECAP in Al and Al alloys [12–14,25–28]. Data for ECAP by using route A are indicated by open symbols [12,13,25–28] and solid ones in the present study. The data for the processing route B_C are presented by gray symbols [13,14,26].

may effectively work at lower temperatures, because the number of the precipitate particles increases with decrease in pressing temperature (Fig. 1). It should be considered also that recovery rate taking place during and after ECAP increases and so dislocation annihilation within (sub)grains rather than impinging on the subgrain walls takes place frequently with raising of deformation temperatures. It can be concluded, therefore, that high density precipitate particles and lower recovery rate appearing at lower temperature can result in rapid increase in the misorientation of strain-induced subboundaries, followed by rapid formation of new fine grains with HABs in high strain.

4.2. Relevance of the present results to reference data

The data reported on strain dependence of the fraction of HABs developed in pure Al and Al alloys under ECAP conditions are summarized in Fig. 8 [12–14,25–28]. The materials were subjected to ECAP by using processing routes A and B_C[2] at room and elevated temperatures. The data for ECAP by using route A are indicated by open symbols [12,13,25–28] and solid ones in the present study. The data for process route B_C are presented by gray symbols and a broken line for references [13,14,26]. It is interesting to note in Fig. 8 that the fraction of HABs start to increase at a critical strain, ε_c, of around 3 and approaches a saturation value F_S at high strains in route A. These parameters, ε_c and F_S, do not depend on pressing temperature.

It is interesting to note in Fig. 8 that F_S in route A is larger than that in route B_C. Horita et al. [4,7,8,24] showed that route B_C is the more effective and optimum procedure

[2] Route B_C means that the sample is rotated by 90° in the same sense between each passes.

for receiving a homogeneous microstructure of equiaxed grains with HABs. Recently, Gholinia et al. [13] demonstrated that change in process route from A to B_C results in drop of the fraction of HABs from 60 to 40% in Al–3%Mg. It should be considered that F_S in route A includes not only deformation-induced HABs, but also the original grain boundaries at high strains. The original grain boundary density increases rapidly with deformation in route A due to continuous elongation of original grains along the pressing direction. The original grain structure is changed to pancaked and lamella forms with continued deformation, resulting in an increase in the volume fraction of the original grain boundaries [13,24]. As strain increase, the lamella structure is split up by deformation bands into the elongated submicron grain fragments and the cell blocks, followed by their rotation and the formation of fine grain structures. In contrast, in route B_C the shape of original grains restores every four pressings, leading to roughly the same volume fraction of the original grain boundaries [24]. Therefore, the ECA pressing by using route B_C results mainly in formation of strain-induced HABs.

It is important to consider the reason why HABs start to be developed at $\varepsilon_c > 2$ in route A irrespective of pressing temperature. The value of ε_c in ECAP is rather larger than that in other deformation modes, e.g. rolling [28,29] and multidirectional forging [19,30,31], where $\varepsilon_c \ll 1$. This suggests that deformation-induced formation of HABs by ECAP may be not superior to that produced by other deformation modes. It is interesting to note that almost all the data reported in route A are scattered in the medium strain range from 3 to 8, but they are within the range enveloped by the present results at 523 and 673 K (Fig. 7). Strain-induced HABs formation is suggested to be athermal phenomena and results from deformation characteristics of ECAP.

5. Conclusions

Grain refinement taking place during equal-channel angular pressing (ECAP) in route A was studied by using a 0.16%Zr-modified 7475 Al alloy at temperatures from 523 to 673 K. This investigation revealed some characteristics of strain-induced microstructures developed under ECAP at high temperatures:

1. ECAP results in the formation of fine-grains with medium to high angle boundaries in high strain at all the pressing temperatures. The average crystallite size increases from 0.6 to 1.7 μm and the aspect ratio decreases from 1.9 to 1.3 with increasing of temperature.
2. HABs start to developed after third ECA pressing, i.e. at a critical strain of around 3. The average misorientation angle and the fraction of HABs developed approach a saturation value at high strains of $\varepsilon > 8$ irrespective of pressing temperature.
3. Increase in pressing temperature tends to slow down the transformation rate from low angle boundaries into high angle ones at medium strain range, i.e. $3 \leq \varepsilon \leq 8$.
4. Formation of deformation bands due to high density precipitate particles can take place accompanied by lower recovery rate operating during lower temperature ECAP. These may lead to rapid development of new grains with HABs.

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References

[1] R.Z. Valiev, R.K. Islamgaliev, I.V. Alexandrov, Prog. Mater. Sci. 45 (2000) 103.
[2] M.V. Markushev, C.C. Bampton, M.Y. Murashkin, D.A. Hardwick, Mater. Sci. Eng. A234 (1997) 927.
[3] S. Lee, P.B. Berbon, M. Furukawa, Z. Horita, M. Nemoto, N.K. Tsenev, R.Z. Valiev, T.G. Langdon, Mater. Sci. Eng. A272 (1999) 63.
[4] S. Komura, Z. Horita, M. Furukawa, M. Nemoto, T.G. Langdon, Metall. Mater. Trans. 32A (2001) 707.
[5] R. Kaibyshev, T. Sakai, I. Nikulin, F. Musin, A. Goloborodko, Mater. Sci. Tech. 19 (2003) 1491.
[6] V.M. Segal, Mater. Sci. Eng. A197 (1995) 157.
[7] Y. Iwahashi, Z. Horita, M. Nemoto, T.G. Langdon, Acta Mater. 45 (1997) 4733.
[8] Y. Iwahashi, Z. Horita, M. Nemoto, T.G. Langdon, Acta Mater. 46 (1998) 3317.
[9] A. Yamashita, D. Yamaguchi, Z. Horita, T.G. Langdon, Mater. Sci. Eng. A287 (2000) 100.
[10] K. Neishi, T. Uchida, A. Yamauchi, K. Nakamura, Z. Horita, T.G. Langdon, Mater. Sci. Eng. A307 (2001) 23.
[11] S. Lee, M. Furukawa, Z. Horita, T.G. Langdon, Mater. Sci. Eng. A342 (2003) 294–301.
[12] J.R. Bowen, P.B. Prangnell, F.J. Humphreys, in: J.B. Bilde-Sørensen, J.V. Carstensen, N. Hansen, D. Juul Jensen, T. Leffers, W. Pantleon, O.B. Pedersen, G. Winther (Eds.), Proceedings of the 20th Riso International Symposium on Materials Science, Riso National Laboratory, Roskilde, Denmark, 1999, p. 269.
[13] A. Gholinia, P.B. Prangnell, M.V. Markushev, Acta Mater. 48 (2000) 1115.
[14] Y.C. Chen, Y.Y. Huang, C.P. Chang, P.W. Kao, Acta Mater. 51 (2003) 2005.
[15] A. Goloborodko, O. Sitdikov, T. Sakai, R. Kaibyshev, H. Miura, Mater. Trans. 44 (2003) 766.
[16] A. Goloborodko, O. Sitdikov, T. Sakai, H. Miura, R. Kaibyshev, Mater. Sci. Forum 426–432 (2003) 321.
[17] G. Tomas, M.J. Goringe, Transmission Electron Microscopy of Metals, Wiley, New York, 1979, p. 112.
[18] J.K. Mackenzie, Biometrika 45 (1958) 229.
[19] A. Belyakov, H. Miura, T. Sakai, Philos. Mag. A81 (2003) 2629.
[20] P.J. Apps, J.R. Bowen, P.B. Prangnell, Acta Mater. 51 (2003) 2811.
[21] D. Godard, P. Archambault, E. Aeby-Gautier, G. Lapsset, Acta Mater. 50 (2002) 2319.

[22] O. Sitdikov, R. Kaibyshev, I. Safarov, I. Mazurina, Phys. Metal. Metallogr. 92 (2001) 270.
[23] R. Kaibyshev, O. Sitdikov, A. Goloborodko, T. Sakai, Mater. Sci. Eng. A344 (2003) 348.
[24] Z. Horita, M. Furukawa, M. Nemoto, T.G. Langdon, Mater. Sci. Tech. 16 (2000) 1239.
[25] J.R. Bowen, P.B. Prangnell, F.J. Humphreys, Mater. Sci. Forum 331–337 (2000) 545.
[26] P.B. Prangnell, J.R. Bowen, A. Gholinia, in: A.R. Dinesen, M. Eldrup, D. Juul Jensen, S. Linderoth, T.B. Pedersen, N.H. Pryds, A. Schrøder Pedersen, J.A. Wert (Eds.), Proceedings of the 22nd Risø International Symposium on Materials Science, Risø National Laboratory, Roskilde, 2001, p. 105.
[27] J.Y. Chang, J.S. Yoon, G.H. Kim, Scripta Mater. 45 (2001) 347.
[28] O.V. Mishin, D.J. Jensen, N. Hansen, Mater. Sci. Eng. A342 (2003) 320.
[29] A. Gholinia, F.J. Humphreys, P.B. Prangnell, Acta Mater. 50 (2002) 4461.
[30] A. Belyakov, K. Tsuzaki, H. Miura, T. Sakai, Acta Mater. 51 (2003) 847.
[31] O. Sitdikov, A. Goloborodko, T. Sakai, H. Miura, R. Kaibyshev, Mater. Sci. Forum 426–432 (2003) 381.