TEMPERATURE EFFECT ON MICROSTUCTURE DEVELOPMENT IN 7475 ALUMINUM ALLOY DURING ECAP

A.Goloborodko[1], O.Sitdikov[1,2], T. Sakai[1], H. Miura[1] and R.Kaibyshev[2]

[1] Department of Mechanical Engineering and Intelligent Systems, The University of Electro-Communications, Chofu, Tokyo 182-8585, Japan, sasha-g@fedu.uec.ac.jp

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

Keywords: grain refinement, aluminum alloy, equal channel angular pressing (ECAP), hot deformation, continuous dynamic recrystallization

Abstract. The effect of temperature on microstructural evolution under hot ECAP was studied in an as-cast 7475 aluminum alloy at temperatures from 523K to 673K. The samples were pressed by using route *A* up to strains of 12. The average (sub)grain size tends to increase with increasing temperature, e.g. about 0.6μm at 523K and 1.7μm 673K. The misorientation distribution of deformation induced dislocation boundaries shows a single peak type at low misorientation angles after first pass and gradually shifts to higher angles with increasing of strain. Equiaxial grains with high angle boundaries start to be formed after 3 passes and then gradually developed with repeated ECAP, finally leading to full development of a new grain structure with high grain boundaries of about 30° in high strain. Specific features of such microstructure evolution and continuous dynamic recrystallization mechanism are discussed in some details.

Introduction

Metallic materials with ultra-fine grained microstructures have many advantages of mechanical properties, e.g. an increasing of strength [1], low temperature and high strain rate superplasticity [2,3], 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) [4]. 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 [4]. Intense plastic deformation is introduced by simple shear in a thin layer at the crossing plane of channel. There have been several works showing that ECAP is very effective for grain refinement of aluminum alloys with submicron levels [5,6]. However, a great attention has been paid to analysis of the developed ultra-fine grained microstructures and the effect of such microstructures on the resulting properties and the thermal stability [6]. Only a limited number of studies have been dealt with the mechanisms for the formation of ultra-fine grains under intensive plastic straining [1,7,8]. The processes of ultra-fine grain formation and the characteristics of their boundaries formed during ECAP are currently still unclear.

The aim of the present work is to study the evolution processes of fine grained structures and their boundary characters in a modified 7475 aluminum alloy during ECAP. The effect of deformation temperature on the microstructural development was investigated. A special attention was paid to analyze the misorientation distribution of (sub)grain boundaries evolved and how low angle boundaries to transform into high angle ones. The mechanisms of grain refinement were discussed in some details.

Experimental Procedure

The alloy used was a 0.16%Zr modified 7475 aluminum 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 is Al. It was fabricated by direct chill casting and homogenization at 768K for 20h at the Kaiser Center for Technology. The initial microstructure was composed of dendrite lamellas lying parallel to the ingot axis with an average size in the range from 1 to 10mm in longitudinal direction and from 100 to 200μm in transverse direction [8,9]. 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. ECAP was carried out using a circular die in cross-section with a diameter of 20 mm. The die had a channel in an L-shaped configuration with an angle of 90° between the two channels and an angle 90° 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. The samples were pressed repeatedly at temperatures of 523 and 673K up to a strain of 12 by using route A [4,5], 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 –30 °C using a Tenupol-3 twin-jet polishing unit. They were then examined using a JEM-2000FX TEM operating at 200kV. Average crystallite size was measured by linear line intercept method. The misorientation of (sub)grain boundaries were studied using a conventional Kikuchi-line technique [10]. The total number of boundaries analyzed was from 60 to 80 in each sample.

Experimental Results and Discussion

Fig. 1 shows typical microstructures with the associated selected area electron diffraction (SAED) patterns developed at a strain of 12 under ECAP at temperatures of 523 and 673K. It can be seen in Fig. 1 that roughly equiaxed fine 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 after ECAP at the both temperatures. It is concluded from these observations that ECAP of the 7475 Al alloy results in grain refinement at temperatures of 523 and 673K, leading to full development of fine grains separated by high angle boundaries at $\varepsilon = 12$. It should be noted here that increasing of pressing temperature brings about increasing of the grain size newly evolved and reduction of the average size and the volume fraction of second phase particles.

The changes in crystallite size with repeated ECAP at 523 and 673K are summarized in Fig. 2. Elongated crystallites developed at early ECAP were changed to equiaxed ones by repeated pressing and then the crystallite size, d, was measured in parallel and transverse to the elongated substructures developed under ECAP. It can be seen in Fig. 2 that longitudinal crystallite sizes decrease rapidly with increasing of strain before $\varepsilon = 4$ and then approach gradually the average ones in transverse direction in high strain. In contrast, transverse crystallite sizes are roughly constant during ECAP at the both pressing temperatures. Then ECAP can results finally in formation of equiaxed fine grains with an average size of about 0.6μm at 523K and 1.7μm at 673K. It should be noted that the aspect ratio of fine grains developed in high strain is about 1.9 and 1.3 at temperatures of 523 and 673K, respectively. This suggests that increasing of pressing temperature brings about development of more equiaxed crystallite accompanied with increasing the average size.

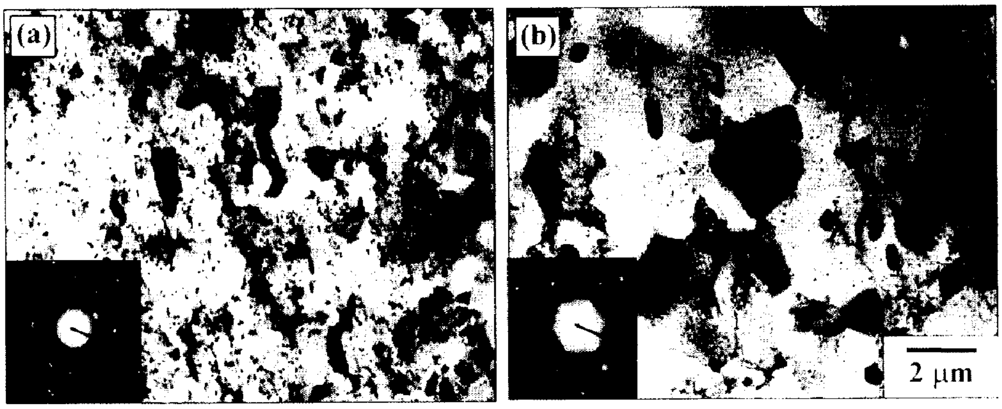

Fig. 1 Typical microstructures and associated SAED patterns for 7475 Al alloy after ECAP. The samples were pressed to a strain of 12 at two different temperatures. (a) 523K and (b) 673K.

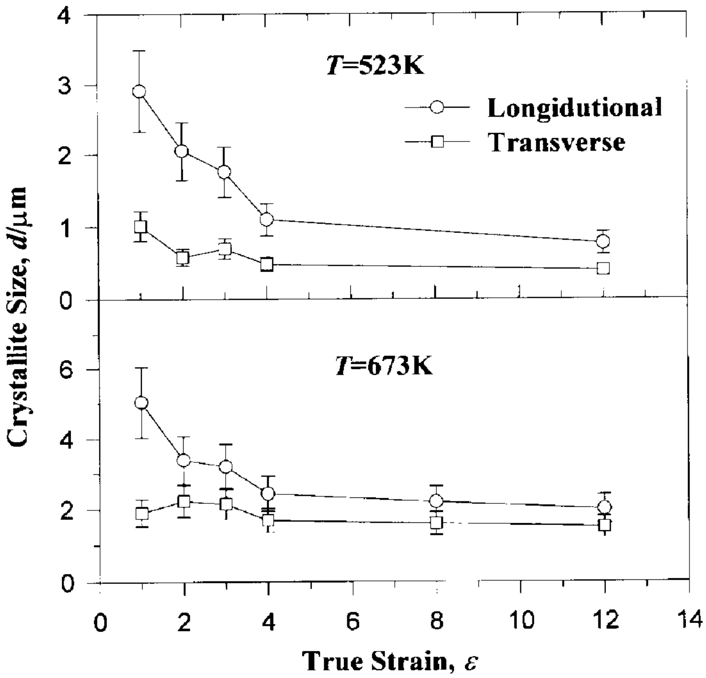

Fig. 2 Changes in crystallite sizes, d, with strain during ECAP at 523K and 673K. d was measured in parallel and transverse to elongated substructures developed under ECAP.

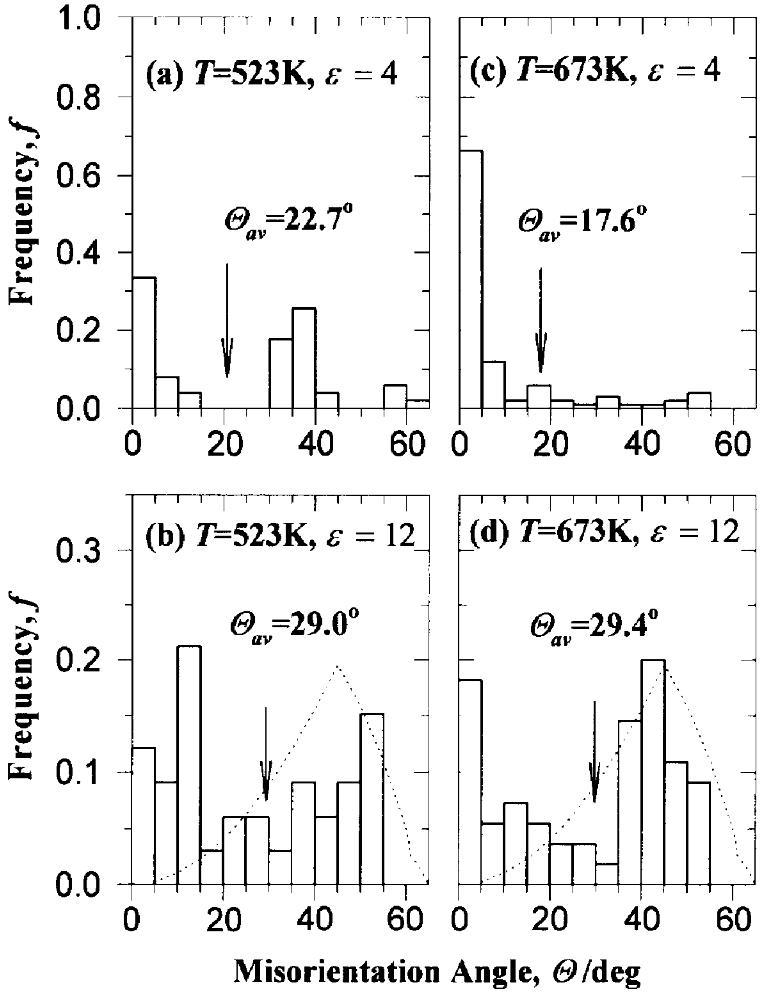

Fig. 3 Changes in misorientation angle distribution for strain-induced boundaries of 7475 Al alloy during ECAP. Broken line indicates the theoretical misorientation distribution for random orientation of fully annealed grains [11]. (a) $T = 523$K, $\varepsilon = 4$; (b) $T = 523$K, $\varepsilon = 12$; (c) $T = 673$K, $\varepsilon = 4$; (d) $T = 673$K, $\varepsilon = 12$.

Fig. 3 shows strain dependence of the distribution of misorientation angles for strain-induced dislocation boundaries developed under repeated ECAP at temperatures of 523 and 673K. It can be seen in Fig. 3(a) that pressing to a strain of 4 at 523K brings about development of a bimodal misorientation distribution with two peaks appearing at angles below 5° and around 35°. With repeated ECAP, the misorientation distribution shifted toward the region of high angles. After ECAP at $\varepsilon = 12$, the fraction of low angle boundaries decreases rapidly and that of high angle ones in the contrary increases, leading to an average misorientation of around 29° (Fig. 3(b)). It is also interesting to note in Fig. 3(b) that a new peak appears at moderate angles misorientation, i.e. $\Theta \approx$ 10-15°. Changes in the misorientation angle distribution developed under ECAP at 673K are

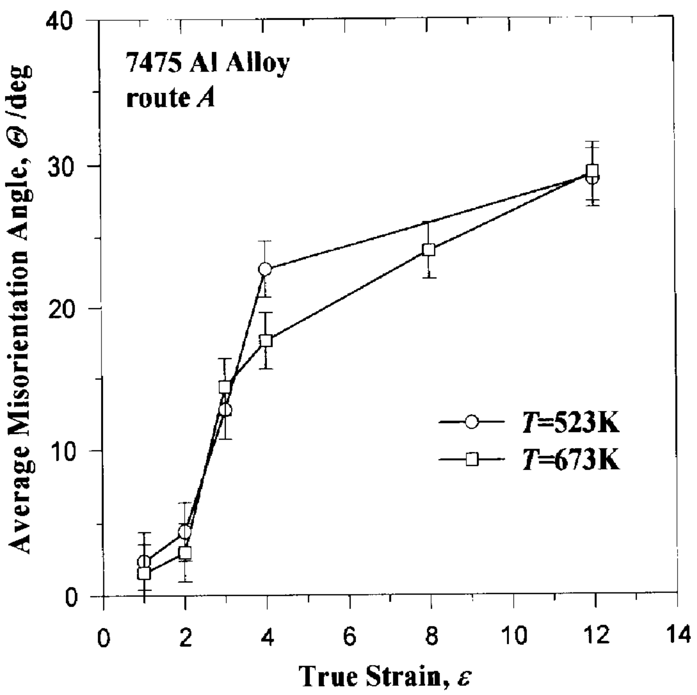

Fig. 4 Changes in average misorientation angle, Θ_{av}, of strain-induced (sub)grain boundaries with repeated ECAP of 7475 Al alloy at 523 and 673K.

roughly the same as those at 523K (Figs. 3(c) and 3(d)), although that at $\varepsilon = 12$ does not show any peak at around $\Theta \approx$ 10-15° (Fig. 3(d)). It is interesting to note in Fig. 3 that the fraction of high angle boundaries above 15° evolved at $\varepsilon = 4$ is about 50% and 30% at 523 and 673K, respectively. This suggests the formation of high angle boundaries takes place more rapidly during lower temperature ECAP. This may result from a larger volume fraction of second phase particles at 523K (see Fig. 1). It should be noted in Figs. 3(b) and 3(d) that the misorientation distributions developed at $\varepsilon = 12$ are roughly similar to a theoretical distribution for random orientations of fully annealed grains [11], although the fraction of low angle boundaries is still relatively large. The latter should be a characteristic of strain-induced grain structures, which contains many low angle dislocation boundaries evolved during deformation.

Change in the average misorientation, Θ_{av}, of strain-induced boundaries with repeated ECAP is shown in Fig. 4. It is seen in Fig. 4 that the average misorientation increases gradually to around 4° before 2 passes and then rapidly up to around 22° and 17° at $\varepsilon = 4$ at 523 and 673K, respectively. This suggests that formation of high angle boundaries may be affected not only by deformation temperature itself, but also by second phase particles, which change also clearly with temperature (Fig. 1). With further deformation at the both temperatures, the average misorientation increases gradually and approaches a same value of around 30° at $\varepsilon = 12$. This suggests that strain-induced high angle boundaries are frequently evolved only after three passages of ECAP, i.e. $\varepsilon \geq 3$, in route *A*.

Summary

Grain refinement taking place during ECAP by using route *A* was studied in a modified 0.16%Zr 7475 aluminum alloy at temperatures of 523 and 673K. This investigation revealed some characteristics of strain-induced microstructures developed under ECAP.

1. Network composed of low to high angles boundaries is fully developed at $\varepsilon = 12$. The average crystallite size increases from 0.6μm to 1.7μm with increasing temperature, although the aspect ratio of elongated substructures decreases from 1.9 to 1.3.

2. The average misorientation of strain-induced dislocation boundaries increases gradually before 2 passes and then rapidly after 3 ones, i.e. $\varepsilon \geq 3$, followed by gradual rises to a saturation value of around 30° irrespective of deformation temperature.

3. The misorientation angle distribution of strain-induced dislocation boundaries shows a single peak at early ECAP and bimodal one at medium to high strains, and finally approaches a randomly orientated one of fully annealed grains in high strain. It may conclude that fine grains formation under ECAP can result from a kind of strain-induced continuous reaction, that is essentially similar to continuous dynamic recrystallization.

Acknowledgements

The authors acknowledge with gratitude the financial support received from the Light Metals Educational Foundation of Japan and the International Science and Technology Center under Project no. 2609. One of the authors (A.G.) would like to express his hearty thanks to the Japanese Government for providing the scholarship. One of the authors (O.S.) wishes to thank to the Japan Society for Promotion Science for providing scientific fellowship.

References

[1] R.Z. Valiev, R.K. Islamgaliev and I.V. Alexandrov: Progress Mater.Sc. Vol. 45 (2000), p. 103

[2] S. Lee, P.B. Berbon, M. Furukawa, Z. Horita, M. Nemoto, N.K. Tsenev, R.Z. Valiev and T.G. Langdon: Mater.Sc.Eng. Vol. A272 (1999), p. 63

[3] K. Neishi, T. Uchida, A. Yamauchi, K. Nakamura, Z. Horita and T. G. Langdon: Mater.Sc.Eng. Vol. A307 (2001), p. 23

[4] V.M. Segal: Mater. Sci. Eng. Vol. A197 (1995), p. 157

[5] Y. Iwahashi, Z. Horita, M. Nemoto and T.G. Langdon: Acta Mater. Vol. 46 (1998), p. 3317

[6] A. Yamashita, D. Yamaguchi, Z. Horita and T.G. Langdon: Mater.Sc.Eng. Vol. A287 (2000), p. 100

[7] O. Sitdikov, R. Kaibyshev, I. Safarov and I. Mazurina: Phys.Metal.Metalloved. Vol. 92 (2001), p. 270

[8] A. Goloborodko, O. Sitdikov, T. Sakai, R. Kaibyshev and H. Miura: Mater.Trans. (in press)

[9] R. Kaibyshev, O. Sitdikov, A. Goloborodko and T. Sakai: Mater.Sc.Eng. Vol. A344 (2003), p. 348

[10] G. Tomas, M.J. Goringe: *Transmission Electron Microscopy of Metals* (Wiley, New York 1979), pp.112

[11] J.K. Mackenzie: Biometrika Vol. 45 (1958), p. 229.