Superplasticity in an 7475 Aluminum Alloy Subjected to Equal Channel Angular Extrusion

I.Nikulin[1], R.Kaibyshev[1], T.Sakai[2], F.Musin[1]

[1]Institute for Metals Superplasticity Problems, Khalturina 39, Ufa 450001, Russia (ilya@imsp.da.ru)

[2]Department of Mechanical and Control Engineering, The University of Electro-Communications, Chofu, Tokyo 182-8585, Japan

Keywords: aluminum alloy, equal channel angular extrusion (ECAE), superplasticity,

Abstract. Superplastic behavior of a high-strength 7475 aluminum alloy subjected to intense plastic straining through equal-channel angular extrusion (ECAE) was studied in tension at strain rates ranging from 1.4×10^{-3} to 5.6×10^{-2} s^{-1} in the temperature interval 400-450°C. Microstructure of the 7475 aluminum alloy after ECAE was non-uniform. Volume fraction of fine grains with average size of about 1.2 µm was ~60%. These grains alternate with elongated grains having longitudinal size of ~5 µm. The highest elongation-to-failure of ~470% was attained at a temperature of 425°C and an initial strain rate of 1.4×10^{-2} s^{-1} with the corresponding strain rate sensitivity coefficient, m, of 0.37. It was shown that the main feature of superplastic behavior of the ECAE processed 7475 aluminum alloy is an extensive strain-hardening at initial stages of superplastic deformation attributed to instability of microstructure.

Introduction

The 7475 aluminum alloy (AA7475) is very popular for manufacturing of complex airframe parts due to its high strength combined with sufficiently fracture toughness, fatigue and ductility. The fabrication of frames and thin-walled panels for aerospace applications requires enhanced workability which can be achieved by making this material superplastic [1,2]. Thin sheets from the AA7475 with fine grained structure are currently produced by conventional thermomechanical processing [2]. The alloy with such the structure exhibited high formability under superplastic conditions [2,3]. However, the utilization of this technology in industrial applications is currently limited because of the relatively high temperatures and low strain rates associated with highest superplastic ductilities of the AA7475 [2,3]. The industrial applications of superplastic forming of the AA7475 could be significantly enhanced if the optimum superplastic properties were attained at higher strain rates ($>10^{-2}s^{-1}$). In addition, the conventional thermomechanical processing is not suitable for producing ultrafine grain structure in large bulk billets of the AA7475.

Recently, it has been shown that in numerous aluminum alloys the grain size may be significantly reduced by imposing an intense plastic strain through the process of equal-channel angular extrusion (ECAE) [4-8]. This technique is highly suitable for achieving superplasticity in bulk billets of aluminum alloys. Aluminum alloys having such an ultrafine or even submicrocrystalline structure are capable to exhibit superplastic ductilities at higher strain rates or lower temperatures [4-8].

The present study was initiated to evaluate the potential for using ECAE to attain a fine grain size in the AA7475. The aim of present experiments is to show that ECAE may be used to achieve high strain rate superplasticity at relatively low temperatures in the AA7475.

Material and Experimental Procedure

The material used in the present study was a 0.16%Zr modified version of the AA7475 with a chemical composition Al-6%Zn-2.5%Mg-1.8%Cu-0.16%Zr-0.03%Mn-0.04%Fe-0.03%Si-0.23%Cr (in weight %). The alloy was manufactured by direct chill casting followed by solution treatment at 490°C for 24 h. Then ingot of the AA7475 was cooled slowly within furnace and finally cut into cylinders with 20 mm in diameter and 100 mm in length. The ECAE was conducted using an isothermal die with a circular internal cross-section with the diameter of 20 mm. The channel had an L-shaped configuration with angle equal to 90°. Deformation through this angle produces a strain of ~1 on each passage through the die. The pressing speed was ~10 mm/s. The rods were repetitively pressed through the dies at 300°C to a total strain of ~16, and the samples were rotated by 90° in the same direction between each pressing (i.e. route B_C).

Tensile specimens were cut parallel to the longitudinal axis of the pressed rods with a gauge length of 6 mm and cross-section of 1.5×3 mm^2. These samples were pulled in tension to failure in air using an Instron universal testing machine (Model 1185) equipped with a three-zone split furnace. Tension tests were carried out in the temperature interval of 400-450°C at strain rates ranging from 1.4×10^{-3} to 5.6×10^{-2} s^{-1}. Temperature accuracy was within ±2°C. Each sample was held at the testing temperature for about 30 min in order to reach thermal equilibrium. To calculate the true flow stress, the load is measured from the chart and the cross-sectional area calculated from the instantaneous length of the sample assuming both constant volume and a uniform cross-section area [2]. The values of the strain rate sensitivity ($m=d\ln\sigma/d\ln\dot{\varepsilon}$, where σ is flow stress, $\dot{\varepsilon}$ is strain rate) were determined by strain-rate-jump tests [1,2]. Values of elongation-to-failure were measured by using two scratches within gauge section of samples.

All structural studies were carried out on the longitudinal (tension) and long transverse sections of the specimens. The microstructures were examined using a JEOL JSM-840A scanning microscope equipped with an electron back-scattered diffraction (EBSD) hard- and software provided by Oxford Instruments, Ltd. For EBSD examinations, surfaces of samples were electropolished using a 10% perchloric acid in buthanol at ambient temperature and 15 V. Cavitation was evaluated by the standard point-count technique.

Results and discussion

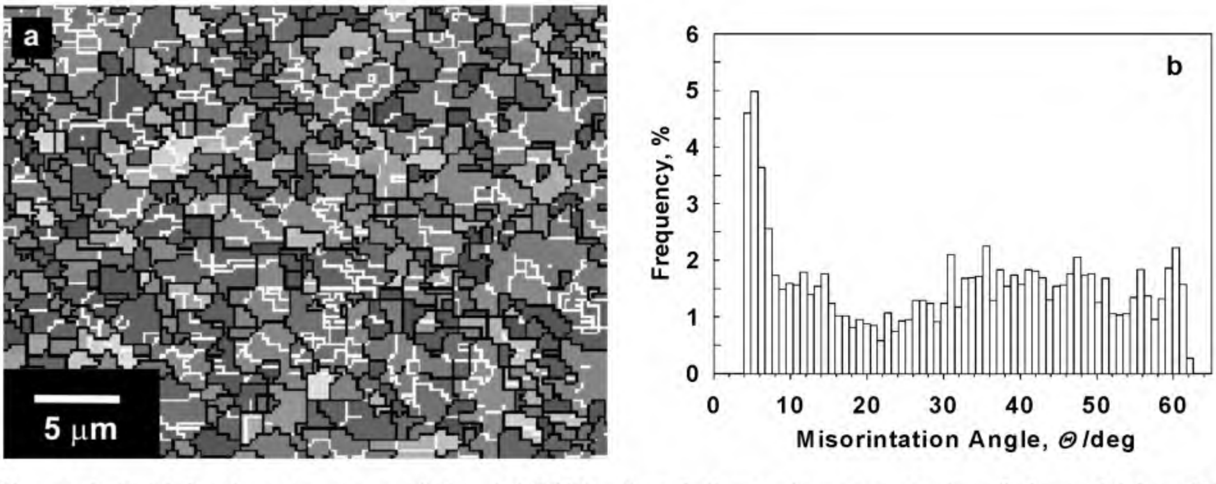

Figure 1. Initial microstructure of the AA7475 after deformation to a strain of about 16 by ECAE processing at 300°C. (a) typical EBSD map and (b) (sub)grain boundary misorientation histogram

Microstructure after ECAE. Typical EBSD map of the ECAE processed AA7475 processed to a strain of about 16 at 300°C is shown in Fig.1a. Black lines and white lines indicate the high-angle boundaries (≥15°) and low-angle boundaries (3–15°), respectively. It is seen that the microstructure of the alloy subjected to ECAE is not homogeneous. In general, the fraction of high-angle boundaries (θ≥15°) being about 70% (Fig.1b) suggests the formation of almost fully recrystallized structure. However, two types of grains distinctly distinguished by shape and size were found. The first structural type is equiaxed grains with an average size of about 1.2 μm. These recrystallized grains outlined by true high-angle boundaries dominate. Their fraction is about 60%. The second structural type is grains with elongated shape. Their longitudinal size ranges from 3 to 7.5 μm, and transverse size is ~2.5 μm. These grains are subdivided by low-angle boundaries on equiaxed subgrains. Most of low-angle boundaries revealed by EBSD technique locate within interiors of such the grains. It is worth noting the size of subgrains within the elongated grains and size of equiaxed grains belonging to the first structural component are essentially the same. The average grain size of overall grains was ~2 μm.

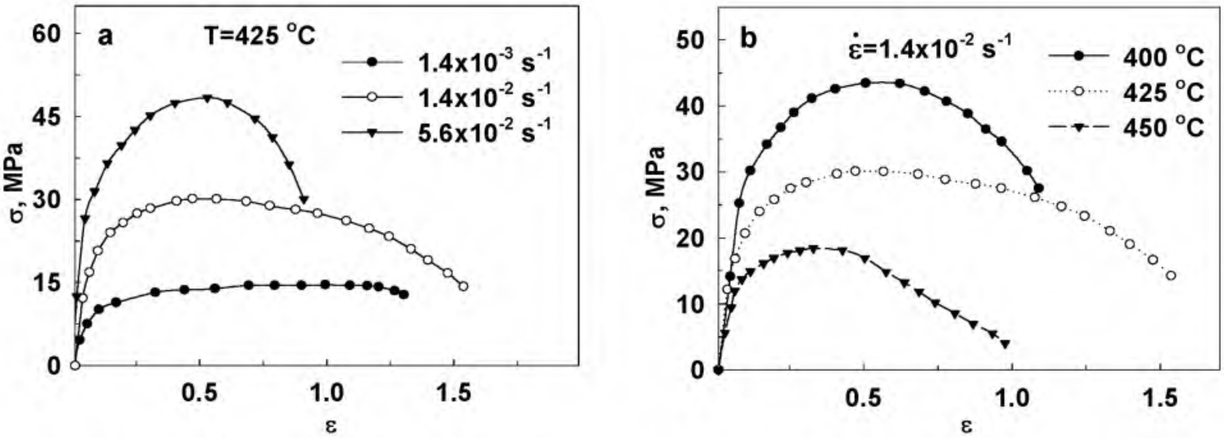

Figure 2. Effect of strain rate (a) and temperature (b) on the true stress-true strain curve for the AA7475.

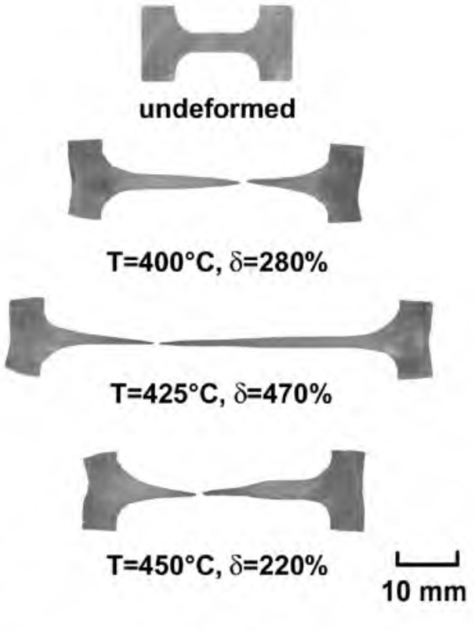

Figure 3. Fractured specimens deformed at strain rate of 1.4×10^{-2} s^{-1} and temperatures ranging from 400 to 450°C.

Superplastic Behavior. The true stress vs true strain curves at initial strain rates ranging from 1.4×10^{-3} to 5.6×10^{-2} s^{-1} in the temperature interval of 400-450°C are shown in Fig.2. Extensive strain hardening takes place initially. After reaching a maximum, the flow stress continuously decreases until failure. No steady-state flow was found. This shape of σ–ε curves is untypical for conventional superplastic materials, in which the flow stress does not dependent on strain [2]. All fractured samples showed unstable plastic flow resulting in premature failure and, therefore, an apparent softening after the stress peak can be attributed to extensive localized necking in gauge length (Fig.3). Notably the ECAE processed AA7475 exhibits low values of yield stress comparing with peak stresses.

Elongation-to-failure, *δ*, and the strain rate sensitivity coefficient, *m*, as a function of strain rate are plotted in Fig.4. The maximum ductility of 470% with the corresponding strain rate sensitivity coefficient of about 0.37 was obtained at relatively high strain rate of 1.4×10^{-2} s^{-1}. Both increase and decrease strain rate lead to decreased m and δ values.

The elongation-to-failure at an initial strain rate of 1.4×10^{-2} s^{-1} as a function of temperature is given in Fig.5. The maximum elongation appeared at 425°C and tended to reduce both with increasing and decreasing temperature. Notably, at T>425°C, the elongation-to-failure sharply decreased to 180%.

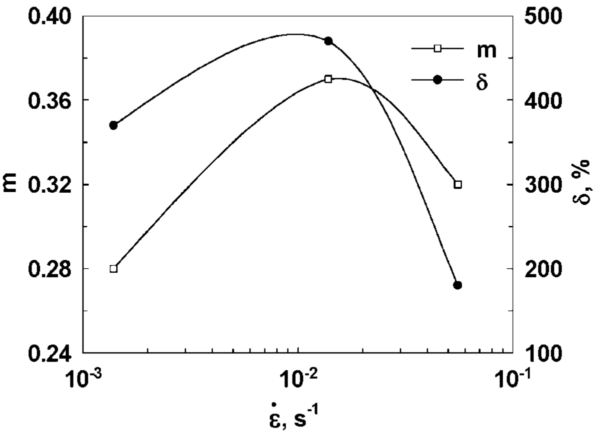

Figure 4. The variation in elongation-to-failure, δ and coefficient of strain rate sensitivity, *m* with strain rate.

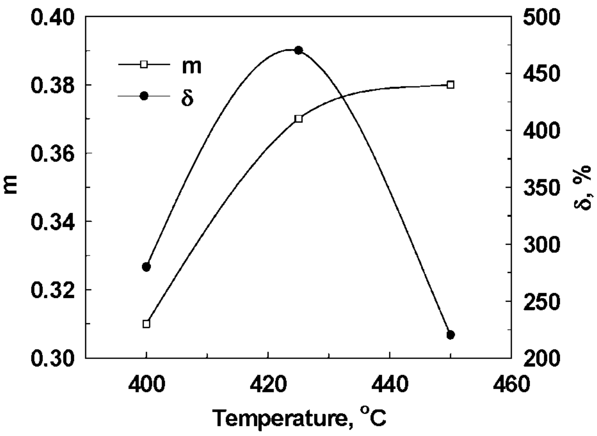

Figure 5. The coefficient of strain rate sensitivity, *m*, and elongation-to-failure, *δ*, as a function of temperature at $\dot{\varepsilon}\sim1.4\times10^{-2}$ s^{-1}.

Microstructural evolution. The microstructural evolution of the AA7475 was examined under conditions of static annealing in grip section and during superplastic deformation, i.e. dynamic annealing in gauge section, in the temperature range 400-450°C at an initial strain rate of 1.4×10^{-2} s^{-1}. It is seen that initial grains tend to grow under static annealing in the temperature range of 400-450°C (Table 1). Remarkable growth the fine recrystallized grains belonging to the first structural component is observed at T≥400°C. In the same time, average size of overall grains increases insignificantly with temperature.

Superplastic deformation leads to significant growth of the fine recrystallized grains. Grains belonging to the second structural component are essentially stable under superplastic conditions; transformation of low-angle boundaries to true high angle boundaries takes place within the elongated grains (Fig.6b). In general, superplastic deformation leads to an increase of average grain size by a factor of 2 or even higher (Table 1). As a result, fraction of high-angle boundaries attains 90% at 425°C and $\dot{\varepsilon}=1.4\times10^{-2}$ s^{-1}. It is apparent, that extensive initial strain hardening is attributed to grain growth under dynamic annealing [2]. Dynamic grain growth terminates superplastic flow and restricts superplastic ductilities.

Cavitation under superplastic deformation in the AA7475 was measured on the samples pulled to failure at an initial strain rate of 1.4×10^{-2} s^{-1} and various temperatures (Table 1). In the temperature range 400-450°C the AA7475 exhibited limited cavitation. Cavities have a jagged shape suggesting the plasticity-controlled cavity growth mechanism [9]. Typical example of cross-sectional view of the specimen pulled to failure at optimum conditions of superplastic deformation is shown in Fig.6c. One can see that the cavities form short stringer toward tension direction. Careful observation of the specimen cross-section near fracture surface showed that the formation of bridges between cavity stringers in the transverse direction was not found. Notably, the volume fraction of the cavities is less than 2% at all condition of the superplastic deformation examined.

Thus, nucleation and growth of the voids does not play a significant role in failure of the AA7475 alloy subjected to ECAE processing.

Table 1. Average grain size after static annealing, L_s, and superplastic deformation, L_d, the volume fraction of cavities for the AA7475 strained up to failure at $\dot{\varepsilon}=1.4\times10^{-2}$ s^{-1} and various temperatures. The elongation-to failure and the time of static annealing in the grip section (in min) are also indicated.

T, [°C]	400 $\varepsilon=1.1$ (34)	425 $\varepsilon=1.5$ (37)	450 $\varepsilon=0.97$ (34)
L_s [μm]	2.1	2.1	2.3
L_d [μm]	2.3	2.6	2.8
V [%]	1.6	2	1.8

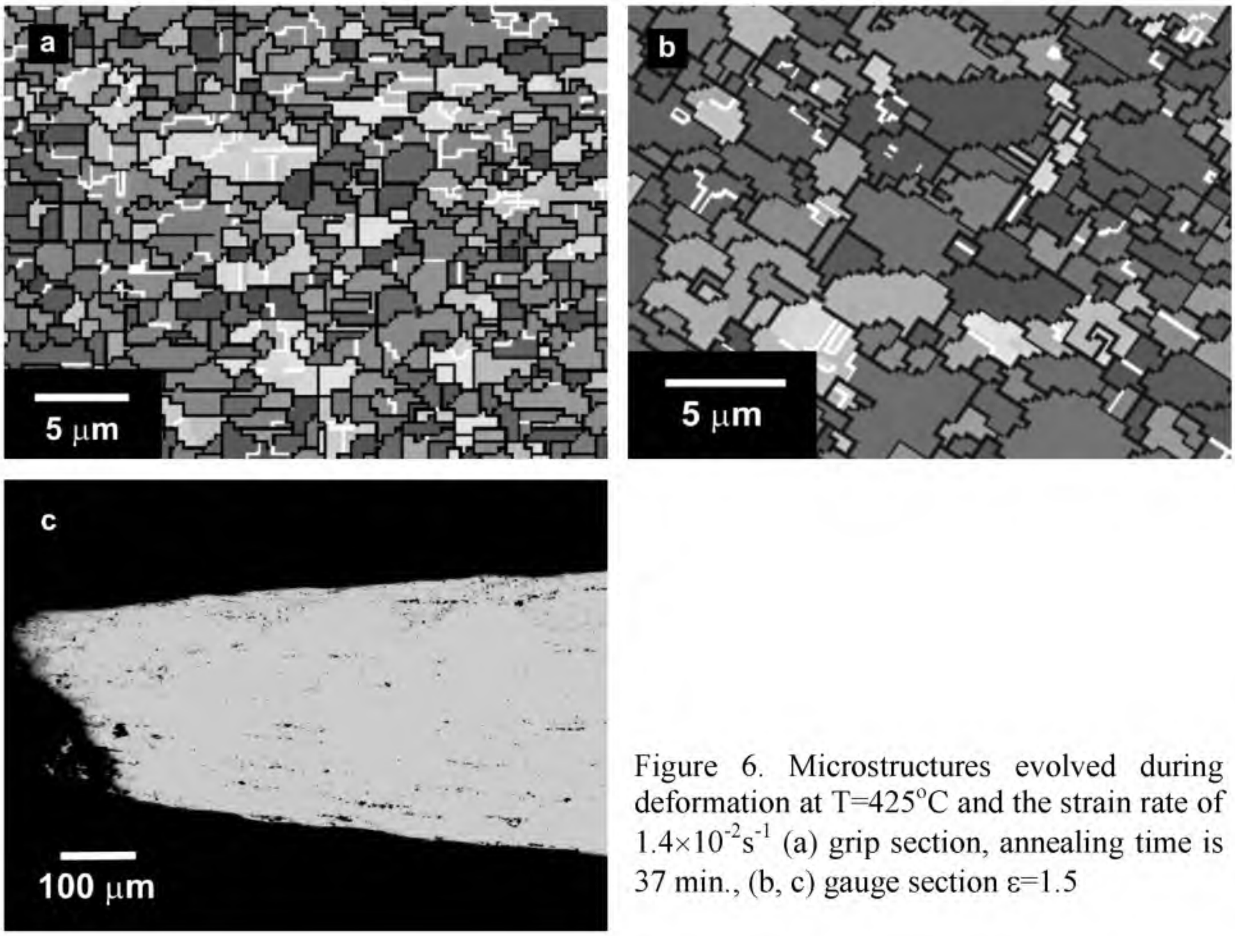

Figure 6. Microstructures evolved during deformation at T=425°C and the strain rate of $1.4\times10^{-2}s^{-1}$ (a) grip section, annealing time is 37 min., (b, c) gauge section $\varepsilon=1.5$

Let us summarize the obtained results for the AA7475 as follow. The present study demonstrated the feasibility for producing ultra fine grain size in the 0.16%Zr modified AA7475 in as-cast condition using intense plastic straining. The average grain size of the alloy was reduced from 100 μm in as-received material to 1.2 μm through the ECAE conducted at 300°C to a total strain of 16. The ECAE processed AA7475 showed optimum superplastic properties at strain rates over $10^{-2}s^{-1}$ and temperatures appreciably less than that for the AA7475 subjected to conventional TMP [2,3]. The AA7475 subjected to ECAE demonstrated highest superplastic ductility of 470%. Taking into account that the required ductility for most forming operations usually is less than 300%, the elongations obtained in present study can be useful for industrial applications.

It should be noted that the moderate values of the elongations-to-failure are attributed to heterogeneous microstructure produced by ECAE at the present conditions. Therefore, it can be expected that the optimization of the parameters of the initial heat treatment as well as the ECAE processing route, such as temperature, path or strain rate, can lead to uniform microstructure providing enhanced superplastic ductility in the AA7475.

Conclusions

1. A 0.16%Zr modified AA7475 was subjected to ECAE at 300°C with a total strain of 16. This processing resulted in non-uniform microstructure with an average grain size of about 2 μm.
2. The highest elongation of 470% was recorded at temperature of 425°C and a strain rate of 1.4×10^{-2} s^{-1} with corresponding strain rate sensitivity coefficient, m, of 0.37.
3. Superplastic deformation led to significant grain growth and limited cavitation. Average grain size was about 2.6 μm after 470% elongation at 425°C. Volume fraction of cavities was about 2%.

Acknowledgements

The financial support by the International Science and Technology Center under Project no. 2609 and Light Metal Education Foundation are gratefully acknowledged

References

[1] O. A. Kaibyshev: *Superplasticity of Alloys, Intermetallides, and Ceramics* (Springer-Verlag, Berlin 1992).
[2] J. Pilling, N. Ridley: *Superplasticity in crystalline solids* (The Institute of Metals, London 1989).
[3] J. Xinggang, C. Jianzhong and M. Longxiang: Mater.Sci.Tech. Vol 9 (1993), p. 493
[4] S. Komura, Z. Horita, M. Furukawa, M. Nemoto and T.G. Langdon: Metall.Trans. A, Vol. 32A (2001), p. 707.
[5] S. Lee, P. B. Berbon, M. Furukawa, Z. Horita, M. Nemoto, N. K. Tsenev, R. Z. Valiev and T. G. Langdon: Mater.Sci.Eng., Vol. A272(1999), p. 63.
[6]. R. Z. Valiev, D. A. Salimonenko, N. K. Tsenev, P. B. Berbon and T. G. Langdon: Scr. Mater., Vol. 37 (1997), p.1945
[7] F. F. Musin, R. O. Kaibyshev, Y. Motohashi, T. Sakuma and G. Itoh: Mater. Trans., Vol. 43 (2002), p. 2370.
[8]. F. F. Musin, R. O. Kaibyshev, Y. Motohashi: Hot deformation of aluminum alloys III, TMS ed. by., Z. Jin, A. Beaudoin, T. A. Bieler and B. Radhakrishnan (2003), p. 221
[9] J. Chadek: Creep of Metallic Materials (in Russian) (Min, Moscow 1987)
[10]. S. L. Semiatin, D. P. Lelo, E.B. Shell: Acta Mater. Vol 48 (2000), p. 1841.