

Superplasticity of a Sc and Zr modified Al-6%Cu alloy subjected to ECAE

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Abstract. Superplasticity in an Al-6%Cu-0.45%Mg-0.4%Mn-0.16%Sc-0.12%Zr alloy subjected to intense plastic straining through equal-channel angular extrusion (ECAE) was studied in tension at strain rates ranging from 5.6×10^{-4} to $5.6 \times 10^{-3} \text{ s}^{-1}$ in the temperature interval 350-450°C. The alloy had a non-uniform microstructure with an average crystallite size of $\sim 1.2 \mu\text{m}$. The volume fraction of high-angle grain boundaries was about 57%. In spite of small crystallite size the alloy shows moderate superplastic properties. The highest elongation-to-failures of $\sim 320\%$ appeared at a temperature of $\sim 425^\circ\text{C}$ and an initial strain rate of $\sim 1.4 \times 10^{-3} \text{ s}^{-1}$, where the strain rate sensitivity coefficient, m , was about 0.33. The relationship between superplastic ductilities and microstructure stability is analyzed.

1. Introduction

Currently there is great interest in developing new commercial aluminium alloys exhibiting superplastic behavior. The utilization of superplastic blow forming allows producing commercial parts having a complex shape. However, the number of superplastic aluminum alloys is currently limited because of difficulties in producing an ultra-fine grain structure by conventional routes of thermomechanical processing [1]. Recently, it has been shown that extensive grain refinement can be attained in aluminum alloys by imposing intense plastic straining through ECAE [2-6]. Aluminum alloys subjected to ECAE resulted in the formation of a uniform grain structure with an average size of $1 \mu\text{m}$ or even less are capable to exhibit superplastic ductilities at higher strain rates ($>10^{-2} \text{ s}^{-1}$) or lower temperatures [2-7]. Merely achieving an ultra-fine grain size is not sufficient to guarantee that these materials will exhibit high superplastic ductilities, since the grain size needs to remain essentially stable throughout the deformation process [1, 7]. Stability of a microstructure in aluminum alloys under high temperature deformation can be provided by Zr and/ or Sc additions, which form $\text{Al}_3(\text{Sc,Zr})$ nanoscale dispersoids [2-5,7-10]. These dispersoids can act as an effective pinning agent hindering the coarsening of ultrafine grains and suppress dynamic recovery [8]. The ultrafine grained structure stabilized by these nanoscale dispersoids is stable against grain growth under superplastic conditions [9,10].

The present study is dedicated to examine a Zr and Sc modified Al-6%Cu alloy. The aim of this study is to evaluate the potential for using ECAE to attain a fine grain size and superplasticity in the bulk billets of the Zr and Sc modified Al-6%Cu alloy.

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2. Materials and experimental procedures

Aluminum alloy with a chemical composition of Al-6%Cu-0.45%Mg-0.4%Mn-0.16%Sc-0.12%Zr (in weight %) denoted as 1207 Al herein was manufactured by direct chill casting, then homogenized at 510°C for 12 hours and slow cooled in a furnace. The bars with dimensions of 25×25×120 mm³ were machined from central part of the ingot parallel to the height direction and then grinded into rods with a diameter of 20 mm and a length of 100 mm. These rods were repetitively pressed through the ECAE dies at 300°C. An isothermal die with a circular internal cross-section and an L-shaped configuration with angles $\Phi\sim 90^\circ$ and $\psi\sim 1^\circ$, [11,12], was used for the extrusion. Deformation through this die produced a strain of ~ 1.15 in each passage. The rods were pressed 12 times (true strain ~ 13.8) with rotation on 90° in the same direction between each pressing, i.e. route B_c was applied [13].

Following the ECAE, the tensile specimens of 6 mm gauge length and 1.5×3 mm² cross-section were machined from the final rods; the gauge length was parallel to the last extrusion direction. Tension tests were carried out in the temperature interval 350-450°C at strain rates ranging from 5.6×10^{-4} to 5.6×10^{-3} s⁻¹. Each specimen was held at a testing temperature for about 10 minutes in order to reach a thermal equilibrium. The other details of mechanical tests were described previously [14,15].

The electron-backscattering diffraction (EBSD) analysis [14,15] was used to examine a deformed microstructure. Microstructural examination was carried out in parallel sections of the Y-plane [13], i.e. (ED) – (TD) sections of extruded rods and samples pulled to failure. The microstructural evolution was examined under conditions of static annealing in grip sections and dynamic annealing in gauge sections of specimens strained up to failure at a strain rate of 1.4×10^{-3} s⁻¹ and temperatures ranging from 400 to 450°C. In the data presented, HAGBs were defined as boundaries with $\theta\geq 15^\circ$ in misorientation and LAGBs as boundaries having misorientation $3^\circ<\theta<15^\circ$. HAGBs and LAGBs are depicted in EBSD maps as black and white lines, respectively.

3. Experimental results

3.1 Microstructures after ECAE. A typical EBSD map and misorientation distribution for the ECAE processed 1207 Al alloy are shown in Figs. 1a and 1b, respectively. Two types of crystallites are distinctly distinguished. Fine grains having essentially equiaxed shape are outlined by HAGBs; their average size is ~ 1.3 μm in longitudinal and ~ 1.1 μm in transverse directions. This structural component is dominant. The second type of crystallites is highly elongated grains containing

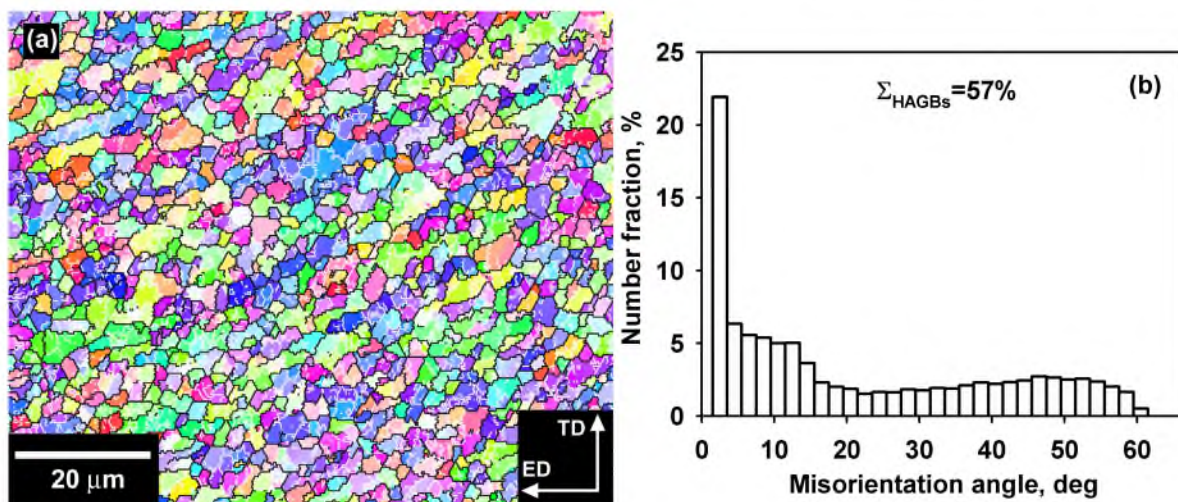


Fig. 1. Microstructure of the 1207 Al after ECAE processing with a total strain of ~ 13.8 at 300°C. (a) typical EBSD map and (b) misorientation distribution.

subgrains in their interiors. Notably the size of these subgrains is similar to one of the new recrystallized grains belonging to first type. The fraction of HAGBs is about 57% (Fig.1b).

3.2 Superplastic behavior. The typical stress – strain curves in the temperature interval 350-450°C at an initial strain rate of $1.4 \times 10^{-3} \text{ s}^{-1}$, and at a fixed temperature of 425°C and strain rates ranging from 5.6×10^{-4} to $5.6 \times 10^{-3} \text{ s}^{-1}$ are shown in Figs. 2a and 2b, respectively. Extensive strain hardening takes place initially. After reaching the maximum, the flow stress continuously decreases until failure and no true steady-state flow occurs. It was found that, all fractured specimens showed strain localization and, therefore, an apparent softening after the stress peak can be attributed to extensive necking in gauge length.

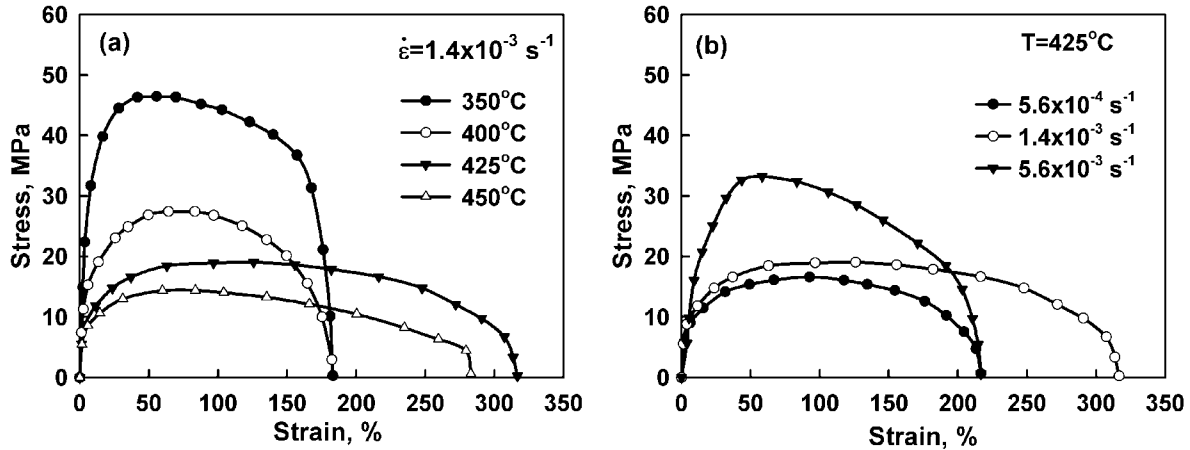


Fig. 2. Typical stress - strain curves of 1207 Al subjected to ECAE. (a) The temperature dependence and (b) the strain rate dependence.

Figs. 3a to 3c show plots of flow stresses, σ , taken at strain $\sim 50\%$, the coefficient of strain rate sensitivity, m , and the elongation-to-failure, δ , as functions of initial strain rates. It is clearly seen that at $T \leq 425^\circ\text{C}$ the σ - $\dot{\epsilon}$ curves show weak evidence of a sigmoidal shape which is typical for superplastic materials [1, 16]. The values of the coefficient of strain rate sensitivity have maximum at strain rates close to 10^{-3} s^{-1} . In the same time, the highest m values do not exceed ~ 0.35 . The highest elongation-to-failure of $\sim 320\%$ (Fig. 3c) with the corresponding m value of ~ 0.33 (Fig. 3b) appears at 425°C and $\dot{\epsilon} \sim 1.4 \times 10^{-3} \text{ s}^{-1}$. An increase or a decrease in strain rate results in decreasing ductility. Thus, we can conclude that the 1207 Al subjected to ECAE exhibits superplastic behavior.

Typical dependencies of the strain hardening coefficient, n , and the strain rate sensitivity coefficient, m , against strain, for the 1207 Al alloy subjected to ECAE are presented in Fig. 3d. It is seen that values of the strain-hardening coefficient decrease to zero with increasing strain. At temperatures below 425°C , the m values tend to decrease with strain, while these values remain essentially unchanged at $T \geq 425^\circ\text{C}$. It is known, that plastic stability in tension occurs if the sum, $n+m$ is high [16,17]. The presence of a neck in a material under tension leads to locally high strain rate and increased strain. As a result, for a high sum of m and n , a sharp increase in the flow stress within necked region occurs providing a resistance to further neck growth. Numerous reports [1,14,16,17] have demonstrated that if $n+m > 0.5$, the fracture does not occur due to unstable plastic flow. In classic superplastic material, the n value is almost zero; the plastic stability is mainly provided by high values of m [1, 16, 17]. In the present alloy extensive strain hardening takes place initially and the m values does not exceed ~ 0.35 . Therefore, stable plastic flow occurs only at strains less than the peak strain due to high value of the strain hardening coefficient. After reaching the peak strain, the $m < 0.35$ value is not enough to inhibit neck development; unstable plastic flow results in material fracture.

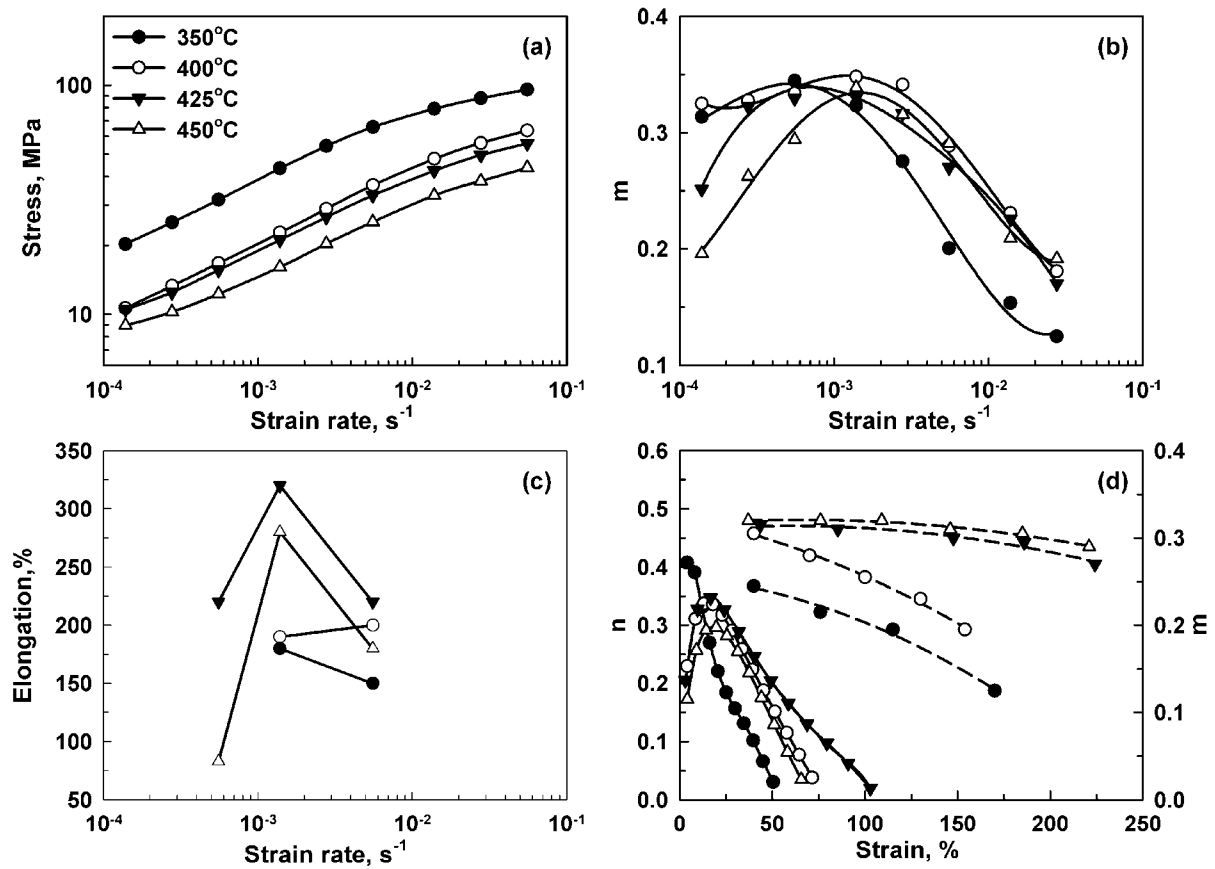


Fig. 3 Strain rate dependence of (a) flow stress, σ , (b) coefficient of strain rate sensitivity, m , (c) elongation-to-failure, δ and (d) variation of the strain rate sensitivity coefficient, (dash line), and the strain-hardening coefficient, n (solid line), with strain. The symbols of temperature defined in (a) are also valid for (b), (c) and (d).

3.3. Microstructural evolution. The microstructural evolution of the 1207 Al alloy subjected to ECAE during static and dynamic annealing was studied in grip and gauge sections, respectively (Fig. 4, Table 1). Under static annealing discontinuous grain growth takes place in grip section (Tables 1) resulting in increased non-uniformity of the deformed structure. Fraction of elongated grains increased under static annealing especially at a temperature of $\sim 450^\circ\text{C}$ (Fig. 4a). Notably the portion of HAGBs after static annealing and ECAE are almost the same (Tables 1). Thus, discontinuous grain growth is not accompanied by the formation of LAGBs. In general, non-uniformity of a structure evolved during ECAE remains under static annealing essentially unchanged; static annealing does not lead to considerable evolution of the microstructure produced by ECAE.

Under dynamic annealing conditions, substantial grain growth takes place (Table 1). The grain size increases in 2-3 times during superplastic deformation. In addition, grains tend to elongate along tension direction. Increased values of grain aspect ratio AR_d [3,4] suggest significant contribution of dislocation glide to total deformation [1,16], and may be indicative for decreased contribution of grain boundary sliding (GBS) to total elongation which is supported by a decrease in the m value with strain at these temperatures (Fig. 3d) [1,16]. Superplastic deformation provides the uniformity of the microstructure (Fig. 4b). The fraction of the HAGBs structure increases in comparison with those in grip section (Table 1, Fig. 4a and 4b). At 425°C , the formation of almost recrystallized structure was revealed (Fig. 4b).

Table 1. Average crystalline size, L_s and L_d , the grain aspect ratios, AR_s and AR_d and fraction of HAGBs after static annealing and superplastic deformation, respectively.

	T [°C]		
	400	425	450
Local strain in gauge sections, [%] (the equivalent time of static annealing in grip sections, [h])	180 (0.57)	320 (0.83)	280 (0.77)
L_s [μm]*	1.9/1.8	1.8/1.6	1.7/1.4
AR_s	1.06	1.13	1.21
L_d [μm]*	2.5/2.1	3.2/2.5	3.1/2.2
AR_d	1.19	1.28	1.41
Σ_{HAGBs} (grip section) [%]	43	55	55
Σ_{HAGBs} (gauge section) [%]	61	77	73

*Numerator and denominator are grain sizes measured in the longitudinal and transverse directions, respectively.

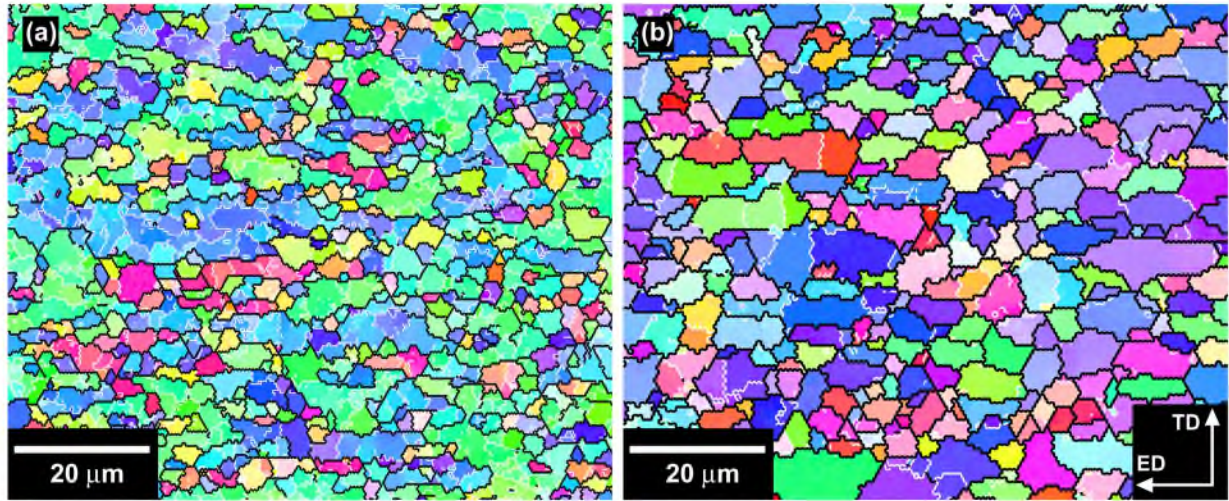


Fig. 4. Microstructures evolved during superplastic deformation: (a) grip section at 450°C, and (b) gauge section at 425°C and $1.4 \times 10^{-3} \text{ s}^{-1}$.

4. Discussion

The present study demonstrates the feasibility to make the Zr and Sc modified 1207 Al alloy superplastic through extensive grain refinement by ECAE. The ECAE at 300°C to a total strain of ~ 13.8 produces bulk billets containing a partially recrystallized structure with an average grain size of $\sim 1.2 \mu\text{m}$. The 1207 Al subjected to ECAE exhibits the highest tensile elongation of about 320%, and the corresponding strain rate sensitivity coefficient of ~ 0.33 at a strain rate of $\sim 1.4 \times 10^{-3} \text{ s}^{-1}$ and a temperature of $\sim 425^\circ\text{C}$.

It is apparent that a non-uniform microstructure produced by ECAE influence strongly on superplastic properties of the presented alloy. It was recently shown [9] that discontinuous static recrystallization takes place in fine-grained aluminum alloys in which the fraction of HAGBs is $< 64\%$ as in the present case. The low fraction of HAGBs in the deformed structure yields the formation of the non-uniform microstructure under static annealing just before superplastic deformation. The uniform fine grain structure is necessary for frequent operation of GBS by shifting of the grain groups along the common grain boundary surfaces [1, 16]. In the present study the coarse elongated grains appears under static conditions. The GBS can not operate in such structure in the easiest way. The low values of the coefficient of strain rate sensitivity (Fig. 3b and 3d) are the main reason for moderate superplastic ductilities achieved in this alloy. Fracture occurs

in this material due to unstable plastic flow. Thus, the present 1207 Al alloy showed marginal superplastic properties due to occurrence of discontinuous grain growth in the initial partially recrystallized structure under static annealing.

5. Conclusions

1. It was found that ECAE is suitable for the formation of the ultra-fine grained structure in the bulk billets of the Zr and Sc modified 1207 Al alloy.

2. The 1207 Al subjected to ECAE exhibits limited superplasticity. The maximum elongation-to-failure of ~320% and a corresponding strain rate sensitivity coefficient of ~0.33 were found at a temperature of 425°C and an initial strain rate of $1.4 \times 10^{-3} \text{ s}^{-1}$.

References

- [1] J. Pilling, N. Ridley, Superplasticity in crystalline solids, The Institute of Metals, London, 1989.
- [2] S. Komura, Z. Horita, M. Furukawa, M. Nemoto, T.G. Langdon: Metall. Mat. Trans., 32A (2001), p. 707.
- [3] F.Musin, R.Kaibyshev, Y.Motohashi, G.Itoh, Metall.Mater.Trans.: 35A (2004) p. 2383.
- [4] R.Kaibyshev, K.Shipilova, F.Musin, Y.Motohashi: Mater.Sci.Tech., 21 (2005) p.408.
- [5] F.F. Musin, R.O. Kaibyshev, Y. Motohashi, T. Sakuma and G. Itoh: Mater. Trans., 43 (2002) p. 2370.
- [6] K.T. Park, H.J. Lee, C.S. Lee, W.J. Nam, D.H. Shin: Scr. Mater., 51 (2004) p. 479.
- [7] Z. Horita, M. Furukawa, M.Nemoto, A.J.Barnes, T.G Langdon: Acta Mater., 48 (2000) p. 3633.
- [8] F.J. Humphreys, M. Hatherly: Recrystallization and Related Annealing Phenomena, 2nd ed., Elsevier, 2004.
- [9] H. Jazaeri, F.J. Humphreys: Acta Mater., 52 (2004) p. 3251.
- [10] M Ferry, N.E. Hamilton, F.J. Humphreys: Acta Mater., 53 (2005) p. 1097.
- [11] T. Aida, K. Matsuki, Z. Horita, T.G. Langdon: Scr. Mater., 44 (2001) p. 575.
- [12] V.M. Segal: Mat. Sci. Eng., A197 (1995) p. 157.
- [13] Z. Horita, M. Furukawa, , M. Nemoto, T. G. Langdon: Mater.Sci.Tech., 16 (2000) p 1239.
- [14] I. Nikulin, R. Kaibyshev, T. Sakai: Mater. Sci. Eng., A 407 (2005) p.62.
- [15] R. Kaibyshev, T. Sakai, I. Nikulin, F. Musin, A. Goloborodko: Mater.Sci.Tech. 19 (2003) p.1491.
- [16] O.A. Kaibyshev, Superplasticity of Alloys, Intermetallics, and Ceramics; Springer-Verlag, Berlin, 1992.
- [17] J. W. Edington, K. N. Melton, C. P. Cutler: Prog. Mater. Sci., 21 (1976) p. 61.