

High strain rate superplasticity in a commercial Al–Mg–Sc alloy

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Abstract

It was shown that an Al–5.7%Mg–0.32%Sc–0.3%Mn alloy subjected to severe plastic deformation through equal-channel angular extrusion exhibits superior superplastic properties in the temperature range of 250–500 °C at strain rates ranging from 1.4×10^{-5} to 1.4 s^{-1} with a maximum elongation-to-failure of 2000% recorded at 450 °C and an initial strain rate of $5.6 \times 10^{-2} \text{ s}^{-1}$.

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1. Introduction

There is a considerable interest in the application of wrought Al–Mg–Sc alloys in the aerospace industry due to their good weldability, superior corrosion resistance and high strength [1]. The formability of the Al–Mg–Sc alloys has been shown to improve significantly in superplastic state [2,3]. These superplastic alloys are highly suitable for the industrial applications due to the fact that their optimum superplastic properties are exhibited at exceptionally high strain rates ($\geq 10^{-2} \text{ s}^{-1}$) [2–8]. Therefore, the Al–Mg–Sc alloys can be effectively used in the fabrication of airframes, thin-walled and stiffened panels where high workability is required.

Among the novel Al–Mg–Sc alloys the 1570 aluminum alloy, which was designated in the Former Soviet Union and denoted as 1570Al herein, is the most promising material for aerospace applications due to the highest strength attained in non-heat-treatable commercially used aluminum alloys [1]. High mechanical properties of the 1570Al are provided by increased content of Mg (~6%). Recent experiments showed that the highest elongation-to-failure of about 1130% could be achieved in sheets of the 1570Al subjected to extensive cold rolling [3]. However, the cold rolled 1570Al exhibited high superplastic properties with initial unre-

crystallized structure at temperatures over 450 °C, at which the transformation of low angle boundaries to high angle boundaries by continuous dynamic recrystallization took place. At the same time, numerous works [4–8] carried out on the Al–Mg–Sc alloys showed that superior ductilities at high strain rates in a wide temperature range could be attained in Al–Mg–Sc alloy rods with a fully recrystallized structure produced by severe strain through equal-channel angular extrusion (ECAE). However, these works have been focused on the alloys containing $\leq 3\%$ Mg which can find only a limited commercial application. Attempts of these authors to make Al–Mg–Sc alloys with higher Mg content superplastic were unsuccessful. In the previous work [5] it was even assumed that it is difficult to achieve superplasticity in Al–Mg–Sc alloys containing greater than 3% Mg through ECAE processing due to low temperature of solution treatment.

The main aim of the present study is to report the superior superplastic properties of the commercial Al–6%Mg–0.3%Sc–0.3%Mn alloy produced by ECAE. The second objective is to provide a direct comparison of ECAE and cold rolling techniques in the development of superplastic capability of the 1570Al.

2. Material and experimental procedure

The alloy used in the present study was commercial grade 1570 aluminum alloy with a chemical composition

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of 5.76%Mg–0.32%Sc–0.3%Mn–0.2%Si–0.1%Fe (in weight %). The 1570Al was manufactured by direct chill casting followed by solution treatment at 520 °C for 24 h. Then, the 1570Al was 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°. The pressing speed was ~ 10 mm/s. The rods were repetitively pressed through the dies at 325 °C to a total strain of ~ 16 , and the samples were rotated by 90° in the same sense between each pressing (i.e. route B_c).

Tensile specimens were cut parallel to the longitudinal axes of the pressed rods with a gauge length of 6 mm and cross-section of 1.5×3 mm². These samples were pulled to failure in air using a Shimadzu machine (Model AG-G-20 kN), which operates at a constant cross-head speed. Tension tests were carried out in the temperature interval 250–500 °C at strain rates ranging from 1.4×10^{-5} to 1.4 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. The values of the strain rate sensitivity ($m = d \ln \sigma / d \ln \dot{\epsilon}$, where σ is flow stress, $\dot{\epsilon}$ is strain rate) were determined by strain-rate-jump tests [9,10]. The magnitudes of elongation-to-failure were measured by using two scratches within gauge section of samples.

Metallographic analysis was carried out using an Olympus BX60 optical microscope and a Jeol JSM-6400 scanning electron microscope. Microstructures were analyzed in the sections taken from planes containing the longitudinal (tension) and long transverse directions. Samples for evaluation of microstructure parameters by SEM (the average grain size and the grain aspect ratio) were annealed at 170 °C for 4 h in order to decorate grain boundaries with secondary phase particles. These grain-boundary particles were revealed by etching with standard Keller's reagent. The mean grain size was determined by the linear intersect method from measurements of more than 300 grains. A Jeol JEM-2000EX electron microscope was used for the thin foil examinations.

3. Results and discussion

A typical microstructure of the 1570Al after ECAE processing is shown in Fig. 1. It is seen that a reasonably homogeneous structure was evolved during ECAE. Diffraction spots on selected area diffraction patterns form well-defined rings indicating that the most of deformation induced boundaries are of high-angle nature (Fig. 1a). The average grain size was about 1 μ m. Volume fraction of the unrecrystallized areas was ~ 5 pct. Chains of Al₂FeSi-phase aligned along prior extrusion direction were revealed on the unetched surface as stringers of dark pits (Fig. 1b).

Fig. 2a shows the typical true stress–true strain (σ – ϵ) curves for the ECAE processed 1570Al at an initial strain rate of 1.4×10^{-2} s⁻¹ and temperatures ranging from 250 to 450 °C. The σ – ϵ curves at a temperature of 450 °C and initial strain rates ranging from 1.4×10^{-4} to 1.4 s⁻¹ are depicted in Fig. 2b. Extensive strain hardening takes place initially. After reaching a maximum, the flow stress continuously decreases until failure. Increasing temperature leads to a shift of the peak stress to a higher strain and a reduction in initial work hardening. At temperatures $T < 300$ °C, the peak stress appears at $\epsilon \leq 0.3$ and after that a gradual decrease in flow stress takes place with strain. Localization of plastic deformation resulting in the final necking and fracture was observed in these conditions (Fig. 3). At $T \geq 300$ °C, the peak stress is attained at $\epsilon \geq 1$; extensive softening is observed just before failure. Very uniform deformation visible within the gauge length occurs. As a result, the samples exhibit superior tensile ductilities. A steady-state flow was not found at all examined temperatures despite the fact that the value of elongation-to-failure is high.

The strain rate sensitivity coefficient, m , as a function of true strain is shown in Fig. 4. A weak strain dependence of the m value appears at all studied temperatures except for 250 °C. The m value slightly increases at strains less than the peak strain. Subsequent deformation results in a gradual decrease in the m value. At 250 °C, increasing strain leads to an increase in the coefficient m from 0.22

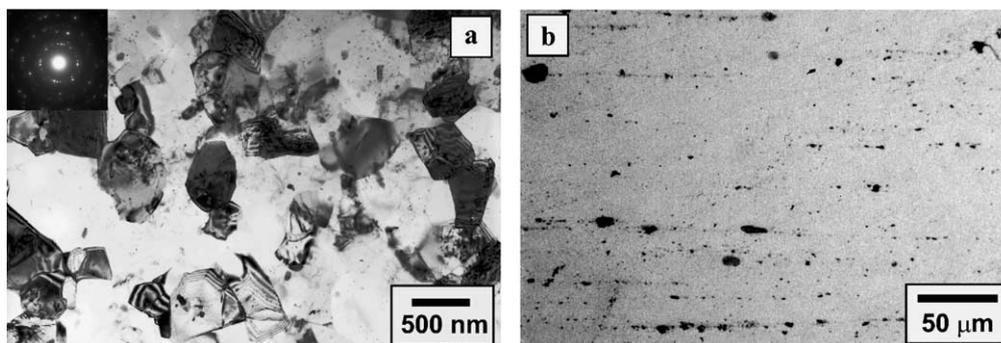


Fig. 1. Microstructures after ECAE with a true strain of ~ 16 at 325 °C: (a) TEM, (b) unetched polished surface.

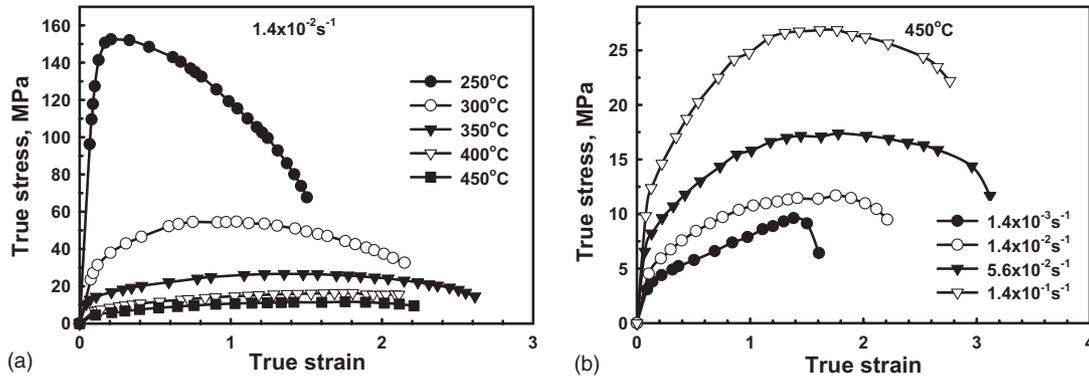


Fig. 2. Effect of temperature (a) and strain rate (b) on the true stress–true strain curves for the ECAE processed 1570Al.

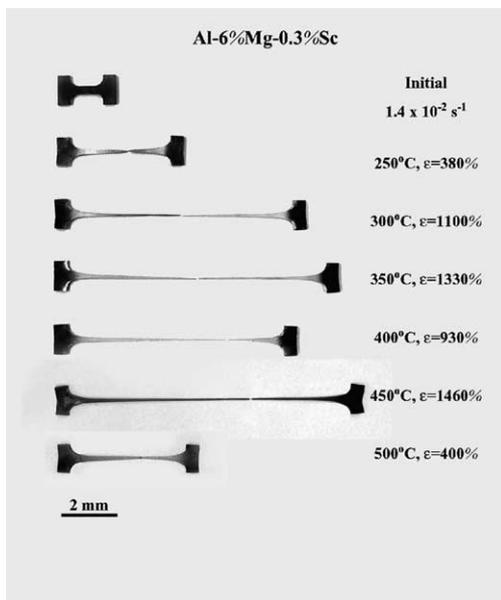


Fig. 3. Samples of the 1570Al after ECAE and pulling to failure at different temperatures and $\dot{\epsilon} = 1.4 \times 10^{-2} \text{ s}^{-1}$.

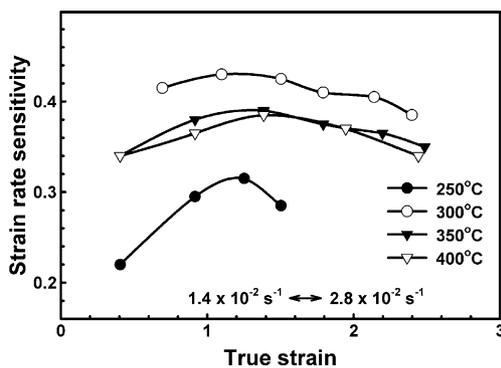


Fig. 4. The effect of true strain on the coefficient of strain rate sensitivity.

to 0.32 followed by a slight decrease. Thus, at 250 °C, low m values can not impede necking by an extensive strain rate hardening and the 1570Al exhibits moderate tensile

ductility. At $T \geq 300 \text{ °C}$, the necking is prevented due to both strain hardening associated with extensive work hardening and strain rate hardening associated with m values greater than 0.33 [10]. As a result, the ECAE processed 1570Al exhibits very high ductilities.

The flow stress taken at a true strain of ~ 0.4 is plotted as a function of initial strain rate on a double logarithmic scale in Fig. 5a. The 1570Al exhibits a sigmoidal relationship between the flow stress and strain rate; three well-known regions of superplastic deformation [9,10] can be identified at all studied temperatures. The variation of strain rate sensitivity, m , and elongation-to-failure, δ , with strain rate are shown in Fig. 5b and c. Experimental data of tensile elongation for cold rolled sheet of the 1570Al [3] are also presented for comparison in Fig. 5c. At $T \leq 400 \text{ °C}$, the elongation-to-failure and the strain rate sensitivity coefficient are found to have maximum in the second region (in which $m \geq 0.33$) and tend to decrease on either side of the strain rate associated with these maximum values. It is seen that increasing temperature results in a shift of the optimal strain rate region for superplasticity (Region 2) to higher strain rates and an increase in the highest values of the m coefficient and tensile ductility. Notably the highest ductilities are observed at lower strain rates comparing with those, at which the highest m values occur. At 450 °C, a continuous increase in the m value with increasing strain rate was revealed in the examined strain rate range despite the fact that a well-defined maximum of elongation-to-failure was found at an initial strain rate of $5.6 \times 10^{-2} \text{ s}^{-1}$. It is apparent, that at 450 °C, the strain rate, at which the highest m value appears, was so high that has not been achieved in the present study.

It should be noted that at 300 °C, ductility of the 1570Al subjected to ECAE is almost the same as that of the cold rolled 1570Al tested at 475 °C [3]. At 450 °C, the ECAE processed 1570Al exhibits noticeably higher tensile ductilities at higher strain rate in comparison with cold rolled 1570Al [3]. Thus, ECAE of the 1570Al provides an increased ductility and a significant expansion of the temperature domain of superplasticity

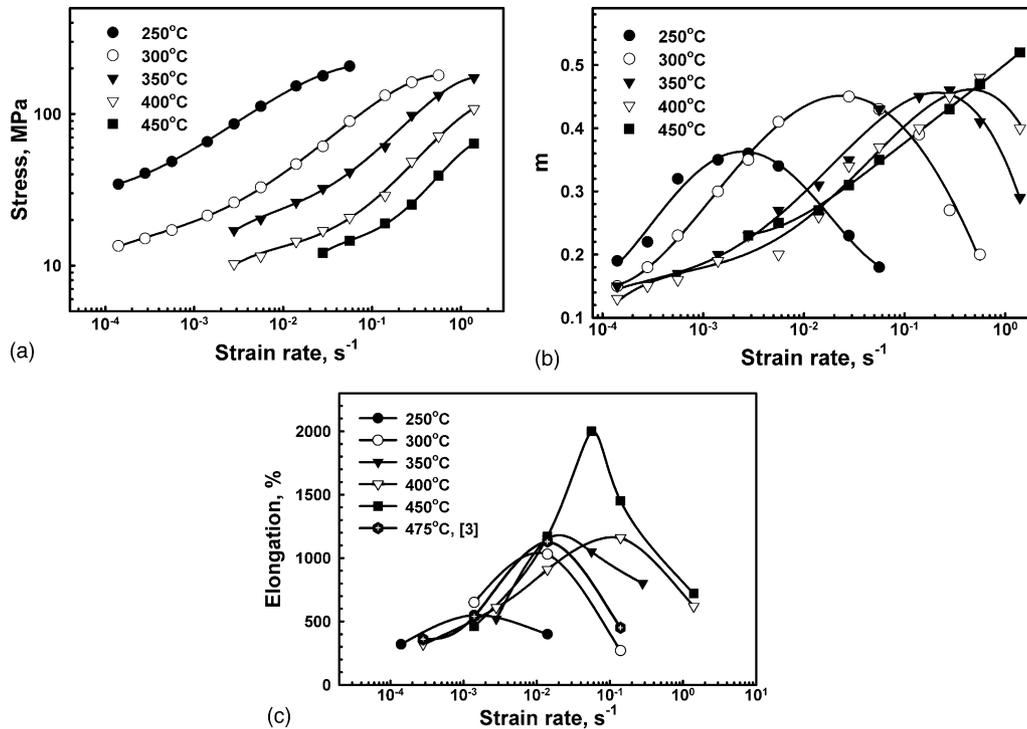


Fig. 5. The variation of flow stress (a), strain rate sensitivity coefficient, m , (b) and elongation-to-failure (c) with strain rate. Datum points for the cold rolled 1570Al [3] are included in (c).

toward low temperatures comparing with state processed by extensive cold rolling.

To evaluate the reproducibility of results, three samples for each testing temperature were pulled to failure at an initial strain rate of $1.4 \times 10^{-2} s^{-1}$ in temperature range of 250–500 °C (Fig. 6). Despite the fact that the dispersion of ductility achieves significant values, the ECAE processed 1570Al exhibits ductility higher than 800% in temperature range of 300–450 °C. Thus, this 1570Al subjected to ECAE shows superplastic properties, which are suitable for commercial applications in different structures.

The microstructure evolution of the 1570Al was examined under conditions of static annealing in grip sections

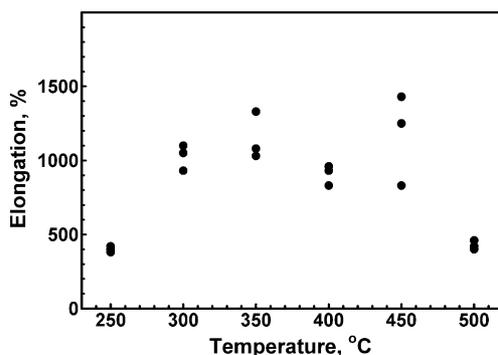


Fig. 6. Temperature dependencies of the elongation to failure at initial strain rate of $1.4 \times 10^{-2} s^{-1}$.

and during superplastic deformation, i.e. dynamic annealing, in the gauge section in the temperature range of 250–450 °C and the strain rate of $1.4 \times 10^{-2} s^{-1}$ (Table 1).

It is seen that in the 1570Al the ultrafine grained structure produced by ECAE processing exhibits superior stability during static annealing. Increasing temperature from 250 to 450 °C leads to a static growth of grains from 1 to 1.6 μm (Fig. 7a and b). It should be noted that the grain size in the 1570Al after annealing at $T \geq 400$ °C is slightly less than that in the Al–3%Mg–0.2%Sc [5].

Superplastic deformation leads to a remarkable grain growth (Table 1). However, the grain size remains on average less than 4 μm even after 2000% elongation at 450 °C and $5.6 \times 10^{-2} s^{-1}$ (Fig. 7c). It is apparent that the weak strain dependence of m value (Fig. 4) is caused by low rate of dynamic grain growth. Notably the superplastic deformation produces essentially similar grain sizes in the both states of the 1570Al subjected to ECAE processing and extensive cold rolling [3]. The AR value is typical for conventional superplastic alloys, where a high contribution of grain boundary sliding to the total elongation takes place [9,10]. In samples exhibiting lower ductility the AR is higher. It is well known that high values of AR are indicative of an increased contribution of dislocation glide into total deformation.

Examination of unetched surface of the specimens pulled to failure showed that cavitation plays an important role in fracture at temperatures greater than 300 °C. Voids are located in the vicinity of the coarse Al_2FeSi

Table 1

Average grain sizes after static annealing (L_S), and superplastic deformation (L_D), grain aspect ratio (AR) for the samples pulled to failure at a strain rate of $1.4 \times 10^{-2} \text{ s}^{-1}$ and different temperatures

$T, ^\circ\text{C}$	250	300	350	400	450
	0.58 h	0.72 h	0.77 h	0.69	0.67
	380%	1100%	1330%	930%	830%
$L_S, \mu\text{m}$	1.0	1.0	1.2	1.3	1.6
$L_D, \mu\text{m}^a$	1.6/1.4	1.6/1.3	1.9/1.4	3.1/2.0	5.8/3.1
AR	1.13	1.31	1.36	1.55	1.87

The elongation-to-failure and the time of static annealing in the grip section (in hours) are also indicated.

^aNumerator and denominator are grain sizes measured in the longitudinal and transverse directions, respectively.

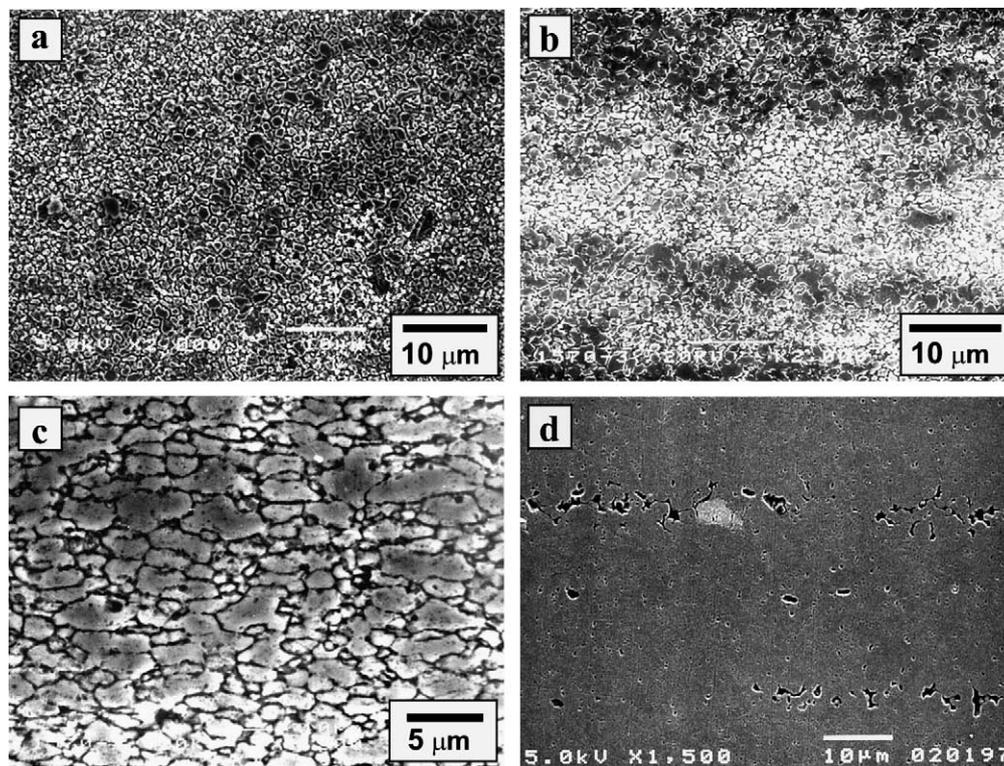


Fig. 7. Microstructural observation of the 1570Al: (a) grip section of specimen at 300 °C, (b) grip section at 450 °C, (c) gauge section at 450 °C and $5.6 \times 10^{-2} \text{ s}^{-1}$, (d) gauge section at 400 °C and $1.4 \times 10^{-2} \text{ s}^{-1}$. Cavitation near Al_2FeSi inclusions is shown in (d).

inclusions forming stringers along the tension direction (Fig. 7d). It is apparent that the nucleation of voids in the vicinity of large inclusions associated with incompatibility of plastic deformation in this region [11] can limit the plasticity resource of the 1570Al. This is in contrast with high purity Al–Mg–Sc alloys, where fracture takes place almost without a cavitation [4–7]. Therefore, it can be expected that the 1570Al containing a reduced amount of Fe and Si can exhibit an enhanced ductility.

4. Conclusions

Thus, the ECAE at 325 °C to a strain of 16 is an attractive way to introduce equiaxed and ultrafine grains in the 1570Al. The alloy subjected to ECAE exhibits

superior superplastic properties with highest elongation-to-failure up to 2000% at 450 °C and initial strain rate of $5.6 \times 10^{-2} \text{ s}^{-1}$. The high elongations achieved at strain rates above 10^{-2} s^{-1} correspond to high strain rate superplasticity in a very wide temperature region. These superior superplastic properties are provided by high stability of ultrafine grained structure produced by ECAE. Such high stability of grains is attributed to the presence of coherent Al_3Sc dispersoids, which are highly effective in pinning of boundaries at temperatures up to 450 °C.

Let us summarize the present results for 1570Al in comparison with those obtained in study [3] as follow. The 1570Al is a material exhibiting high-strain-rate-superplasticity with superior ductilities both in condition with initial unrecrystallized structure obtained by

extensive cold rolling and in condition of recrystallized structure after ECAE with a mean grain size of about 1 μm as well. The difference in the highest values of ductility of the 1570Al in these two structural conditions is not too large. ECAE leads to an expansion of the temperature–strain rate domain of superplastic deformation and allows achieving high ductilities at higher strain rates. Thus, the superior superplastic properties in the 1570Al can be achieved both in the sheets and extruded semi-finished products as well.

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References

- [1] Filatov YuA, Yelagin VI, Zakharov VV. *Mater Sci Eng* 2000;280A:97.
- [2] Sawtell RR, Jensen CL. *Metall Trans* 1990;21A:421.
- [3] Nieh TG, Hsiung LM, Wadsworth J, Kaibyshev R. *Acta Mater* 1998;46:2789.
- [4] Lee S, Utsunomiya A, Akamatsu H, Naishi K, Furukawa M, Horita Z, et al. *Acta Mater* 2002;50:553.
- [5] Furukawa M, Utsunomiya A, Matsubara K, Horita Z, Langdon TG. *Acta Mater* 2001;49:3829.
- [6] Komura S, Horita Z, Furukawa M, Nemoto M, Langdon TG. *Metall Mater Trans* 2001;32A:707.
- [7] Horita Z, Furukawa M, Nemoto M, Barnes AJ, Langdon TG. *Acta Mater* 2000;48:3633.
- [8] Komura S, Furukawa M, Horita Z, Nemoto M, Langdon TG. *Mater Sci Eng A* 2001;297:111.
- [9] Kaibyshev OA. *Superplasticity of alloys, intermetallics, and ceramics*. Berlin: Springer Verlag; 1992. p. 316.
- [10] Pilling J, Ridley N. *Superplasticity in crystalline solids*. London: The Institute of Metals; 1989. p. 214.
- [11] Humphreys FJ, Miller WS, Djazeb MR. *Mater Sci Technol* 1990;6:1157.