Superplastic behaviour and microstructure evolution in a commercial ultra-fine grained Al-Mg-Sc alloy

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Abstract. An Al-6%Mg -0.3%Sc-0.3%Mn alloy was subjected to equal-channel angular extrusion (ECAE) at 325°C to a total strain of about 16 that resulted in an average grain size of about 1 μm. Superplastic properties and microstructural evolution of the alloy were studied in tension at strain rates ranging from 1.4×10^{-5} to 1.4 s^{-1} in the temperature interval from 250 to 500°C. It was shown that this alloy exhibits superior superplastic properties in the wide temperature range 250-500°C at strain rates over $10^{-2}s^{-1}$. The highest elongation to failure of 2000% is attained at a temperature of 450°C and an initial strain rate of 5.6×10^{-2} s^{-1} with the corresponding strain rate sensitivity coefficient of about 0.4. Two different fracture mechanisms were revealed in high strain rate superplasticity. At temperatures higher than 300°C and/or strain rates less than $10^{-1}s^{-1}$, failure took place in brittle manner practically without necking and cavitation played a major role in the failure. In contrast, at low temperatures and/or high strain rates, fracture occurred in a ductile manner suggesting the fracture of samples by localized necking. At these conditions, evidence of very limited cavitation was found in the samples.

Introduction

The aerospace industry has a great interest in developing new aluminum alloys with enhanced service properties. Wrought non heat-treatable Al-Mg alloys containing Sc are attractive candidates for different structural components due to their good weldability, strength, excellent corrosion resistance and ductility [1]. The fabrication of complex-shaped parts for aerospace structures requires enhanced workability. It is possible to improve highly the formability of Al-Mg-Sc alloys by making these materials superplastic [1,2]. Recently, it has been established that the best way to achieve high superplastic ductilities in Al-Mg-Sc alloys is an extensive grain refinement by imposing an intense plastic strain (IPS) through a process such as equal-channel angular extrusion (ECAE). Numerous works [3-6] recently reported a superior ductility of Al-Mg-Sc alloys, with moderate concentrations of Mg (≤3%), subjected to ECAE at exceptionally high strain rates ($\geq 10^{-2}s^{-1}$) that makes the process of superplastic forming highly attractive for commercial application. However, commercial Al-Mg-Sc alloys with Mg less than 4.5% exhibit a relatively low strength and, therefore, their commercial application is restricted toy very specific areas [1]. An attempt by authors of Ref. [4] to make the Al-5%Mg-0.2%Sc alloy superplastic through ECAE at room temperature was unsuccessful due to the cracking of the samples. Therefore, a different procedure should be applied to produce submicrometer grains in the Al-Mg-Sc alloys containing over 3%Mg.

Nowadays the most attractive material belonging to the Al-Mg-Sc system, for aerospace applications, is an Al-6%Mg-0.3%Sc designated in the Former Soviet Union as the 1570 aluminum alloy [1] and denoted as 1570Al herein. The 1570 Al exhibits the highest strength attained in non-heat-treatable commercial aluminum alloys [1] due to an increased content of Mg (~6%) and Sc additions. Recent experiments showed that the highest elongation-to-failure, of about 1000% at high strain rates $\geq 10^{-2}s^{-1}$, was achieved in sheets of the 1570Al subjected to extensive cold rolling [7]. It was found that the 1570Al exhibited high superplastic properties in the unrecrystallized condition at temperatures over 450°C [7] due to the occurrence of continuous dynamic recrystallization, during superplastic deformation, that is typical for Al-Mg alloys [8,9].

The present investigation was initiated in order to evaluate, in detail, superplastic behaviour and to study the low temperature limit for high strain rate superplasticity of the commercial 1570 Al produced by ECAE at elevated temperature. A specific objective of this work is to examine the microstructural evolution, cavitation and fracture during high-strain-rate superplastic deformation.

Material and Experimental Procedure

The 1570 Al with a chemical composition of Al-5.76%Mg-0.32%Sc-0.3%Mn-0.2%Si-0.1%Fe (in weight %) was manufactured by direct chill casting followed by solution treatment at 520°C for 24 h. Then, the alloy was cut into cylinders of 20 mm in diameter and 100 mm in length. The rods were repetitively pressed through an isothermal ECAE die at 325°C to a total strain of ~16. 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 1.5x3 mm^2. These samples were pulled to failure in air using a Shimadzu machine (Model AG-G-20kN) in the temperature interval 250-500°C at strain rates ranging from 1.4x10^{-5} to 1.4s^{-1}. Temperature accuracy was within ±2°C. Each sample was held at a testing temperature for about 30 min in order to reach thermal equilibrium. The values of the strain rate sensitivity were determined by strain-rate-jump tests [10,11]. The magnitudes of the elongation-to-failure were measured by using two scratches within a gauge section of each sample.

Microstructure analysis was carried out using an Olympus BX60 optical microscope and a JEOL JSM-840 scanning electron microscope equipped with electron backscattered diffraction (EBSD) hard- and software provided by Oxford Instruments, Ltd. Microstructures were analyzed in the sections taken from planes containing the longitudinal (tension) and long transverse directions. The mean grain size was determined by the linear intersect method from the measurements of more than 300 grains. Cavitation was evaluated by the standard point-count technique.

Results and discussion

The ECAE of the 1570Al led to the formation of a uniform structure with an average grain size of ~1 μm (Fig.1a). A volume fraction of grain boundaries having a high angle misorientation (>15 degree) was about 80 pct. Chains of secondary phase, mainly Al_2FeSi-phase, aligned along the prior extrusion direction were revealed on the unetched surface as stringers of dark pits (Fig.1b).

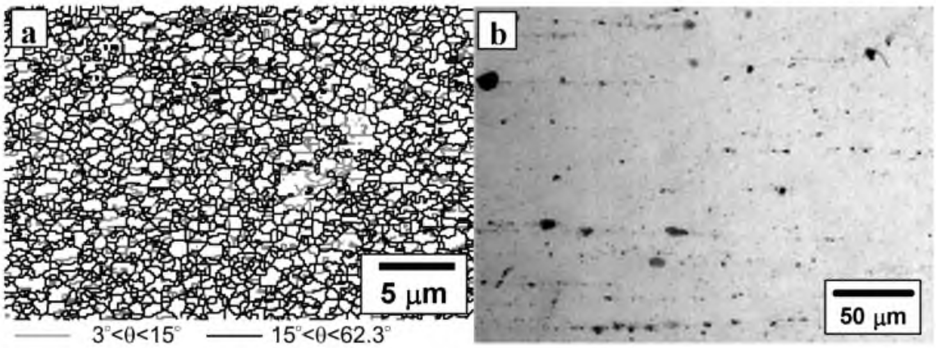

Fig.1. Typical structure in 1570 Al deformed to a strain of about 16 via ECAE at 325°C: (a) EBSD map, (b) unetched polished surface. Note that extrusion axis is horizontal.

Figure 2 shows the typical true stress-true strain curves for the ECAE processed 1570Al at an initial strain rate of 1.4x10^{-2}s^{-1} and temperatures ranging from 250 to 450°C. 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. A steady-state flow was not found at all examined temperatures despite the fact that the value of elongation-to-failure is high.

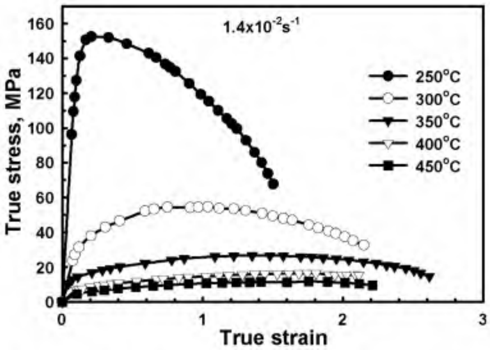

Fig. 2. Effect of temperature on the true stress-true strain curves.

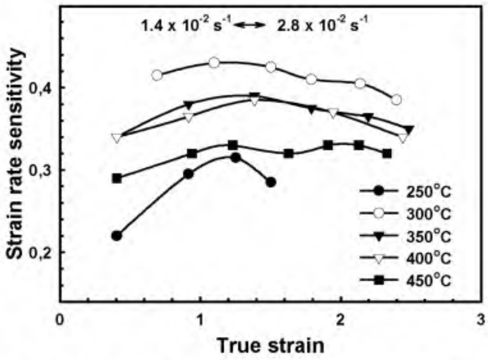

Fig. 3. Effect of true strain on the coefficient of strain rate sensitivity.

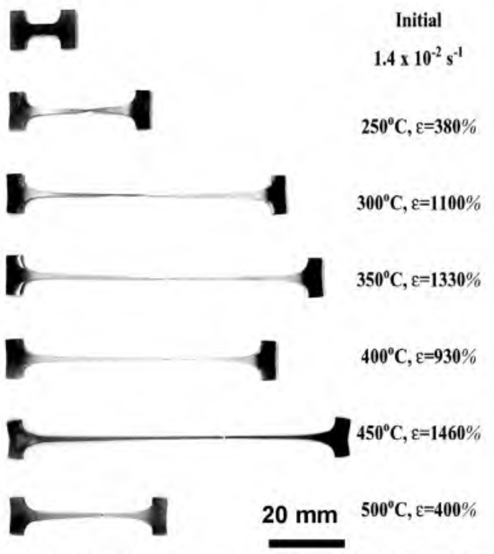

Fig. 4. Samples of the 1570Al after ECAE and pulling to failure at different temperatures and $1.4x10^{-2}s^{-1}$.

The strain rate sensitivity coefficient, m, as a function of the true strain is shown in Fig.3. 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 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≥300°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 (Fig.4).

Variation of the strain rate sensitivity and the elongation-to-failure with strain rate are shown in Fig.5. At T<350⁰C, the elongation-to-failure and the strain rate sensitivity coefficient are found to have maxima in Region 2 (in which m≥0.33 [11]) and tend to decrease on either side of the strain rate associated with these maximum values. It is seen that an increase of the temperature results in a shift of the optimal strain rate region for superplasticity to higher strain rates and in an increase of the highest values of the m coefficient and tensile ductility. Note that at T≥350°C the maximum ductilities are observed at strain rates lower than those corresponding to the highest values of *m*. At 450°C, a continuous increase of *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 $5.6x10^{-2}s^{-1}$. It is evident, that at 450°C, the strain rate corresponding to the highest *m* value is so high that has not been achieved in the present study.

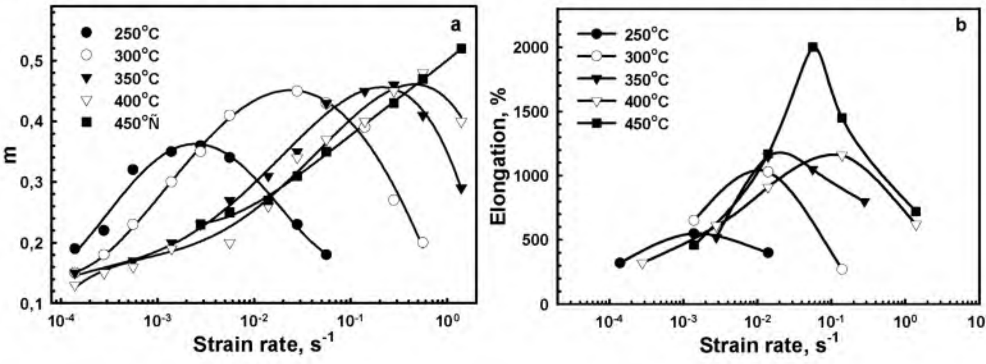

Fig. 5. The variation strain rate sensitivity coefficient, m, (a) and elongation-to-failure (b) with strain rate.

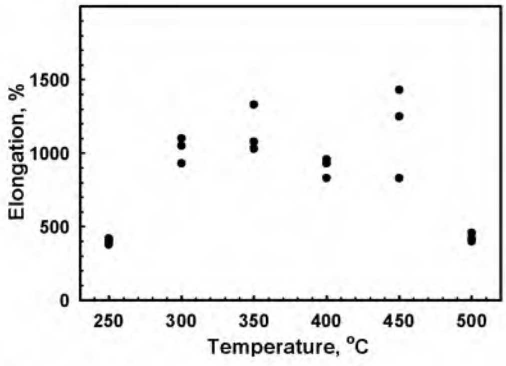

Fig.6. Temperature dependencies of the elongation-to-failure at initial strain rate of $1.4x10^{-2}\ s^{-1}$.

For an evaluation of the reproducibility of results, three samples for each testing temperature were pulled to failure at an initial strain rate of $1.4x10^{-2}s^{-1}$ in the temperature range of 250-500°C (Fig.6). Despite the fact that the variation of ductility reaches significant values, the ECAE processed 1570Al exhibits a ductility higher than 800% in temperature range of 300-450°C. Thus, this 1570Al subjected to ECAE shows superior superplastic properties that are highly suitable for commercial applications in different structures.

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 $1.4x10^{-2}s^{-1}$. The elongation-to-failure and the time of static annealing in the grip section (in hours) are also indicated.

T, °C	250 0.58h, 380%	300 0.72h, 1100%	350 0.77h, 1330%	400 0.69h, 930%	450 0.67h, 830%	500 0.59h, 410%
L_S, μm	1.0	1.0	1.2	1.3	1.9	7.4
L_D, μm*	1.6/1.4	1.6/1.3	1.9/1.4	3.1/2.0	5.8/3.1	7.1/4.3
AR	1.14	1.23	1.36	1.55	1.87	1.65

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

The microstructure evolution of the 1570Al was examined under conditions of static annealing in grip sections 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 $1.4x10^{-2}s^{-1}$ (Table 1, Fig.7). 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°C to 450°C leads to a static growth of grains from 1 μm to 1.9 μm.

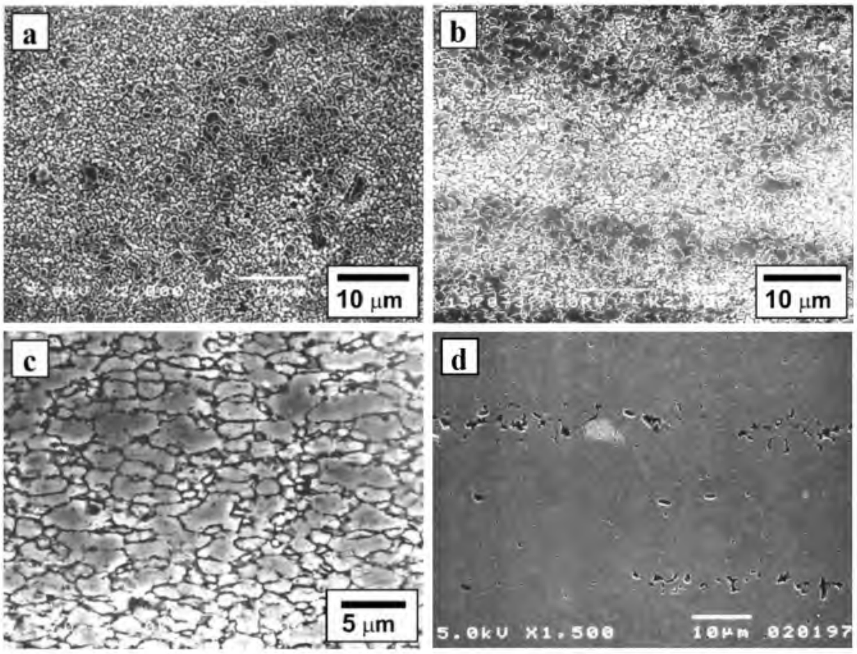

Fig. 7. Microstructural observation of the 1570Al: (a) grip section of specimen at 300°C, (b) grip section at 450°C, (c) gauge section at 450°C and $5.6x10^{-2}s^{-1}$, (d) gauge section at 400°C and $1.4x10^{-2}s^{-1}$. Cavitation near Al_2FeSi inclusions is shown in (d).

Superplastic deformation leads to a remarkable grain growth (Table I). 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.3) is caused by low rate of dynamic grain growth. The aspect ratio value (AR) is typical for conventional superplastic alloys, where a high contribution of grain boundary sliding to the total elongation takes place [10,11]. 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.

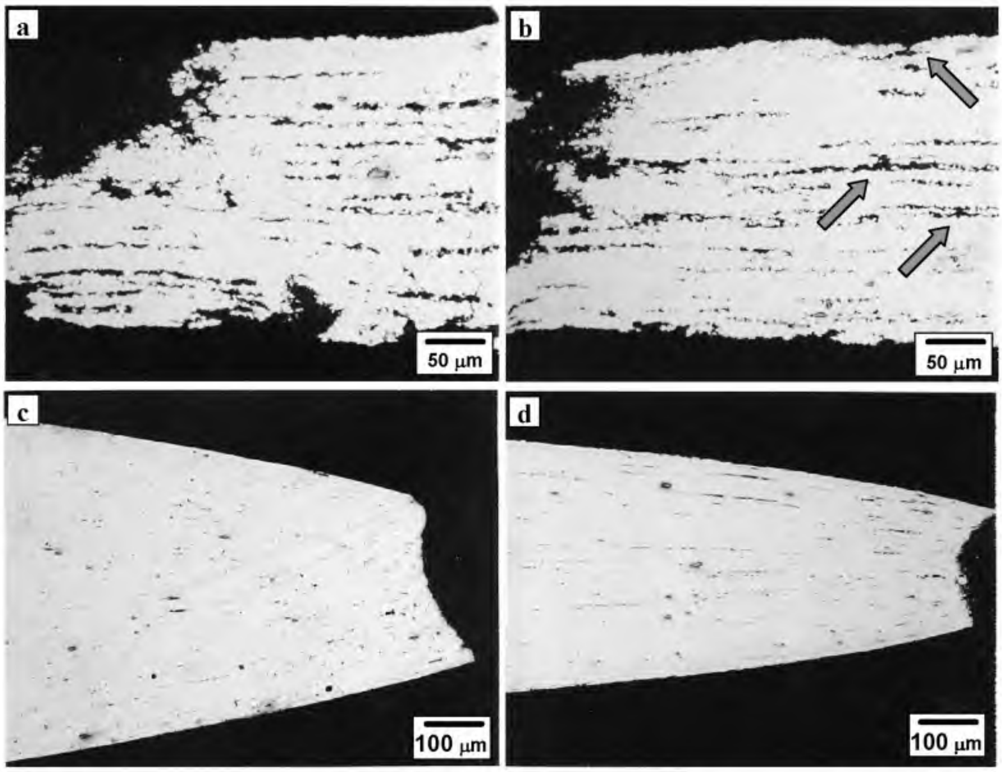

Fig. 8. Cross-sectional views of samples strained to failure near fracture tip. (a) 350°C, $1.4x10^{-2}s^{-1}$; (b) 400°C, $1.4x10^{-1}s^{-1}$; (c) 250°C, $1.4x10^{-2}s^{-1}$; (d) 400°C, $1.4x10^{0}s^{-1}$. Arrows indicate the cavity stringers interlinkage and the bridges between cavities and free surface.

Table 2. The volume fraction of cavities, V, for the samples pulled to failure at a strain rate of $1.4x10^{-2}$ s^{-1}. Areas located in 1mm, 5mm and 10mm from the fracture surface were analyzed.

T, °C	250 420%	300 1100%	350 1330%	400 930%	450 830%	500 410%
V_{1mm}, %	0.14	1.0	5.2	4.9	13.2	11.4
V_{5mm}, %	0.1	0.4	2.1	2.0	5.3	6.7
V_{10mm},%	0.0	0.1	0.53	0.13	1.2	4.0

Examination of unetched surfaces of the specimens pulled to failure showed that cavitation starts to play an important role in fracture at temperatures greater than 300°C (Table 2). Voids are located in the vicinity of the coarse Al_2FeSi-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 [12], 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 [3-6]. Therefore, it can be expected that the 1570Al containing a reduced amount of Fe and Si can exhibit an enhanced ductility. It is worth noting that a sufficient amount of cavities formed during deformation does not lead to a drop of superplastic properties. It is evident, that an extensive cavitation takes place only on the late stage of superplastic deformation, since the quantity of the cavities quickly decreases with a distance from the fracture surface (Table 2).

Cross-sectional views of near-fracture-surface regions of the samples strained to failure are shown in Fig.8. It is seen that two types of fracture are distinctly distinguished. At T>300°C and/or strain rates less than 10^{-1} s^{-1}, the fracture occurs abruptly in a brittle manner practically without necking (Fig.8a,b), suggesting that the cavitation plays a major role in the failure. The formation of bridges between the free surfaces and cavity stringers located near them as well as an interlinkage of the cavity stringers in the transverse direction initiates the pseudo-brittle fracture which occurs without a strain localization. The second type of fracture mechanism is observed at temperatures less than 300°C and/or strain rates over $10^{-1}s^{-1}$. In these conditions the change of the fracture mechanism from the one caused by porosity to the ductile type takes place. The alloy hardly has any cavitation and the samples break down with localized necking (Fig. 8c,d).

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 the highest elongation-to-failure up to 2000% at 450°C and an initial strain rate of $5.6x10^{-2}s^{-1}$. 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 the high stability of ultra-fine grained structures 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. The results suggest that the development of submicrometer grained structure in the commercial Al-Mg-Sc alloy enables superplastic deformation at high strain rate and low temperature making the process of superplastic forming commercially attractive for the fabrication of high-volume components.

Acknowledgements

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