Superplasticity in a Zr and Sc modified AA7075 aluminium alloy produced by ECAP

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Abstract

A sub-micrometer grain size was introduced to a Zr and Sc modified AA7075 aluminium alloy using the method of equal-channel angular pressing. Room temperature tensile tests together with microhardness measurements revealed that the refinement of the microstructure had been accompanied by a substantial increase in room temperature strength. The acquired microhardness values were also used to determine the deformation dead zone within the ECAP billet. TEM observations documented a reasonable stability of the microstructure up to the temperature of 623 K. At this temperature, the material exhibited high strain rate superplasticity with an elongation to failure of 520 %.

K e y w o r d s: ultra-fine grained Al alloys, ECAP, high strain rate superplasticity

1. Introduction

Ultra-fine grained (UFG) materials have two principal advantages over their coarse grained counterparts. They generally benefit from enhanced room temperature strength, in accord with the Hall-Petch relation, and at the same time, they may exhibit superplastic behaviour at elevated temperatures. Superplastic forming represents a modern technology, which has already found its commercial applications. Its main advantage consists in the possibility of producing components with a complex shape and uniform thickness in one operation, thus eliminating the need for the assembly of separately made parts and lowering tooling costs [1]. Unfortunately, the relatively low strain rates at which superplastic ductility is achieved in most commercially available materials limit the applications of superplastic forming to low volume production. For this reason, the refinement of the microstructure is of major importance, as it can substantially increase the optimum strain rate for superplastic deformation. New ultra-fine grained metallic materials which exhibit the so called high strain rate superplasticity (HSRSP), defined as superplasticity occurring at a strain rate of 1×10^{-2} s^{-1} or higher [2], therefore have the potential for high volume production as well, e.g. in the automotive industry.

It is well known that severe plastic deformation (SPD) can be used to introduce an ultra-fine grained microstructure to polycrystalline materials. Equal-channel angular pressing (ECAP), first introduced by Segal et al. [3], is at present the most widespread method of SPD. Its advantage lies in the fact that it can be used to prepare relatively large bulk samples of materials with fine, equiaxed grains (a prerequisite for the occurrence of superplasticity) as well as low porosity.

The 7xxx series aluminium alloys (Al-Zn-Mg-Cu) are well known for their high strength and have, for this reason, found many commercial applications. At the same time, fine grained 7xxx series prepared by conventional thermo-mechanical treatments have been shown to exhibit superplastic behaviour at high temperatures and low strain rates (typically 10^{-4}–10^{-3} s^{-1}) [4]. Using an SPD method to refine the microstructure of such a material should result in a higher optimum strain rate for superplastic deformation. Even though the 7xxx series have been the subject of countless experimental studies, the amount of results available on their microstructure and mechanical properties when processed by ECAP remains to

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this day very limited. The aim of the present investigation is therefore to explore the possibility of creating an ultra-fine grained Al-Zn-Mg-Cu-Sc-Zr alloy using ECAP and to study its microstructure as well as its mechanical properties, especially with respect to the potential for HSRSP.

2. Experimental material and procedure

The experimental material was based on a commercial 7075 alloy with the following composition (in wt.%): 5.88 Zn, 2.45 Mg, 1.32 Cu, 0.37 Fe, 0.35 Si, 0.19 Cr, 0.17 Mn, 0.026 Ti and balance Al. For the purpose of this study, the alloy was modified by the addition of 0.2 wt.% Sc and 0.11 wt.% Zr. The material was then processed by ECAP with 6 passes, route B_C, at the temperature of 393 K and the pressing speed of 5 mm/min. The channels had a 90° angle of intersection and a 10×10 mm^2 cross-section. Prior to ECAP, the alloy was subjected to annealing at the temperature of 673 K for 8 hours with subsequent slow furnace cooling (30 K/h), which resulted in a soft, over-aged state. This treatment led to a substantial decrease of the pressing force during ECAP, thus allowing for the processing to be carried out at a relatively low temperature.

The objective of the Zr and Sc addition was to enhance the stability of the UFG microstructure at elevated temperatures, which are necessary for superplastic deformation, by means of grain growth retardation. In aluminium alloys, Zr and Sc are known to form fine Al_3 (Zr_xSc_{1-x}) precipitates, which were recently shown to have a strong stabilizing effect on the microstructure. The precipitates form a fine dispersion, relatively stable at high temperatures, which decreases grain boundary mobility, thus providing high resistance to the recrystallization process in the material. A particularly strong stabilizing effect of Zr and Sc was documented in Al-Mg alloys [5, 6]; the material used in the present investigation was expected to exhibit analogous behaviour.

The microhardness of the studied alloy was measured using a Leco M-400-A hardness tester with a Vickers indenter. The measurements were performed on the right side plane of the billet after ECAP (parallel to the pressing direction), with a load of 100 g. The results were used to determine the size and position of the deformation dead zone (DDZ) within the billet, i.e. the zone, where the material was not subjected to shear deformation during all 6 of the passes. For comparison, microhardness was also measured on the material in the soft state, prior to ECAP.

After the ECAP process, tensile specimens with a gauge length of 17 mm and a cross-section of 6×1 mm^2 and 6×0.5 mm^2, respectively, were cut from the above mentioned side plane of the billet, taking into account the position and dimensions of the DDZ. These specimens were then used for tensile tests at room temperature as well as at the temperatures of 623 K and 723 K. The tests were carried out on an Instron 5882 testing machine. At elevated temperatures, the strain rate change method (SRC) was used to investigate the dependence of the strain rate sensitivity parameter m (defined as $\partial \log \sigma / \partial \log \dot{\varepsilon}$) on true strain rate and deformation temperature, in order to determine the optimum conditions for superplastic deformation. Consequently, the material was strained to failure under these conditions, i.e. with a constant rate of crosshead displacement (corresponding to the initial strain rate of 2×10^{-2} s^{-1}) at the temperature of 623 K. During the course of this test, 3 supplementary strain rate changes were performed to once again verify the value of the strain rate sensitivity parameter.

A JEOL JEM 2000 FX transmission electron microscope was used to examine the microstructure. The material was studied in its initial state after ECAP, as well as after 1 hour of annealing at 623 K and 723 K, to simulate the conditions at which the SRC data were acquired. The foils for TEM were prepared by twin jet polishing.

3. Experimental results

At room temperature and an initial strain rate of 1×10^{-4} s^{-1}, a yield stress σ_{02} of 285 MPa, an ultimate tensile strength (UTS) of 388 MPa and an elongation to failure of 3.7 % were recorded. The corresponding deformation curve is shown in Fig. 1. The microhardness of the material prior to ECAP was HV 81, its value after 6 passes at 393 K increased to HV 128. The measurements revealed a marked drop in microhardness at the extremities of the ECAP billet. Figure 2 shows a plot of points with equal microhardness

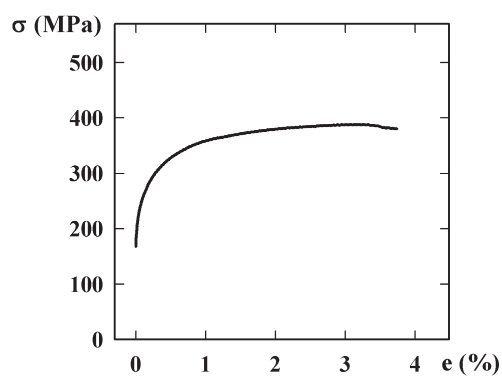

Fig. 1. True stress vs. strain dependence at room temperature and an initial strain rate of 1×10^{-4} s^{-1}.

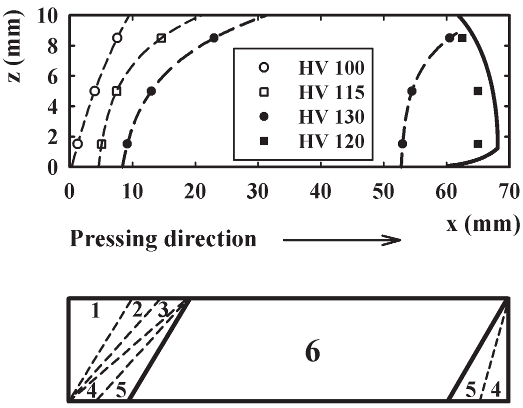

Fig. 2. Microhardness on the side plane of the ECAP billet compared with the theoretical position of the DDZ.

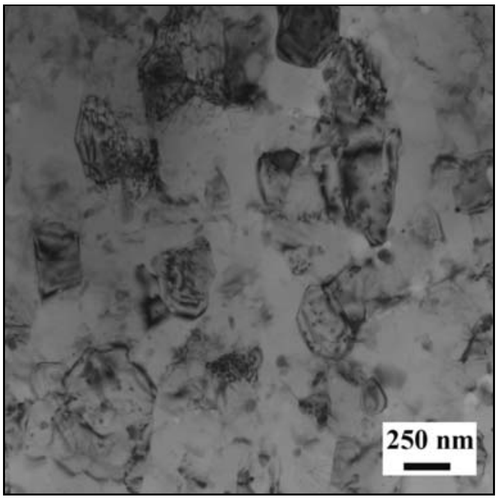

Fig. 3. Microstructure of material in the initial state after ECAP.

values on its right side plane (designated here the x-z plane). The regions with lower microhardness values correspond to the deformation dead zone (DDZ) in the sample. To improve the clarity of the plot, smooth curves were fitted to the experimental data. Microhardness was measured along lines parallel with the pressing direction, in the centre of the billet and 1.5 mm from its edges. The extrapolation of the curves to the regions near the edges only has an approximative character. The lower scheme in Fig. 2 shows the theoretical positions and dimensions of the zones, where the material was subjected to shear deformation during a total of 1–6 passes for the given geometry of the billet. It is based on the calculations of the ECAE 3D software [7].

Figure 3 shows a TEM micrograph of the material in the initial state after 6 passes of ECAP. Typical grain sizes range from 200 nm to 500 nm, the

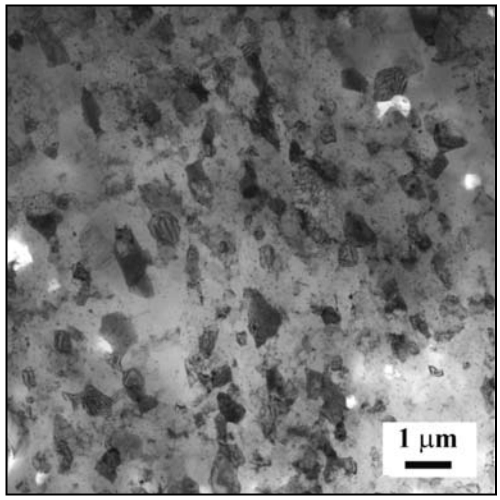

Fig. 4. Microstructure of material annealed for 1 h at 623 K, sub-micrometer grains in a large part of the volume.

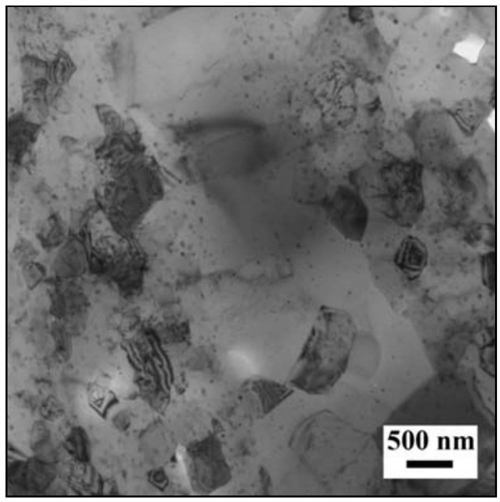

Fig. 5. Microstructure of material annealed for 1 h at 623 K, local bands of coarser grains are starting to appear.

grains are equiaxed. A high dislocation density can be observed and the image of most grain boundaries is diffuse, testifying to their non-equilibrium character. In the material, which has been subjected to annealing at 623 K for 1 h, the microstructure is still reasonably homogeneous with a sub-micrometer grain size, as documented in Fig. 4. However, in a small part of the volume, bands of grains coarsened to several microns already appeared, see Fig. 5. A more pronounced coarsening of the microstructure can be observed at the annealing temperature of 723 K. Although regions with sub-micrometer grains can still be found in the material, as shown in Fig. 6, in a large part of the

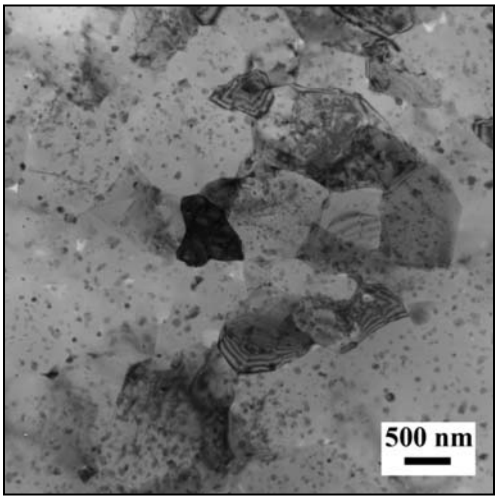

Fig. 6. Microstructure of material annealed for 1 h at 723 K, regions with 500 nm grains can still be found.

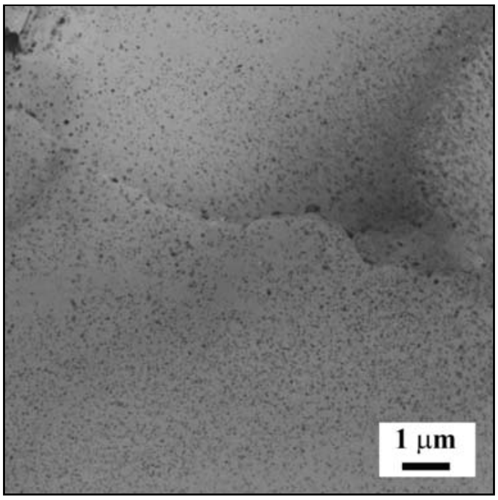

Fig. 7. Microstructure of material annealed for 1 h at 723 K, grains coarsened to 10 μm or more in approx. 70 % of the volume.

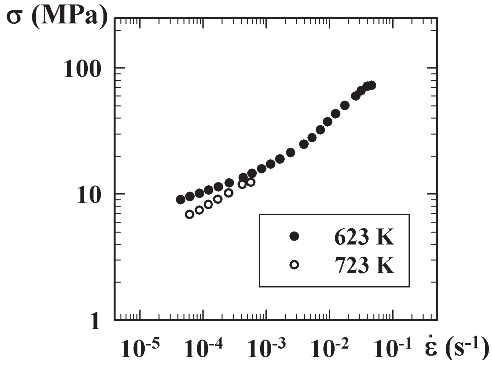

Fig. 8. Dependence of true stress on true strain rate.

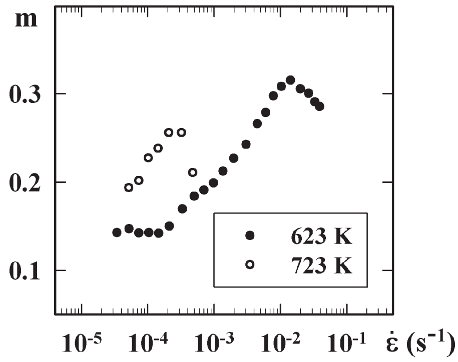

Fig. 9. Dependence of the strain rate sensitivity parameter on true strain rate.

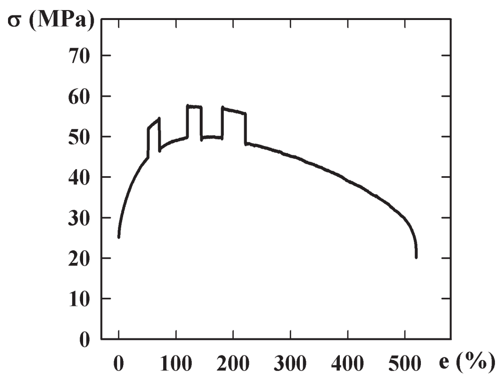

Fig. 10. Dependence of true stress on strain for the material strained at 623 K and the initial strain rate of 2×10^{-2} s^{-1}.

volume (approximately 70 %), grains are coarsened to 10 μm, occasionally even more, as documented in Fig. 7.

The results of the SRC tests at elevated temperatures are presented in Figs. 8 and 9. Figure 8 shows the dependence of true stress on true strain rate in logarithmic scale for the deformation temperatures of 623 K and 723 K. The dependence of the strain rate sensitivity parameter m on true strain rate is shown in Fig. 9. The curves for both temperatures in Fig. 8 show evidence of sigmoidal behaviour, typical for superplastic materials. However, Fig. 9 clearly documents the shift of the curve corresponding to the higher deformation temperature by two orders of magnitude towards lower strain rates. Also, at the temperature of 723 K, the maximum strain rate sensitivity does not reach the value of 0.3, which is generally considered to be the lower limit for superplastic behaviour.

At the lower deformation temperature of 623 K, the parameter m reaches its maximum value of 0.32 at a strain rate exceeding 10^{-2} s^{-1}. When strained at the temperature of 623 K with a constant rate of crosshead displacement corresponding to an initial strain rate of 2×10^{-2} s^{-1}, the material reached an elongation to failure of 520 %, as documented by Fig. 10. The value of the strain rate sensitivity parameter m calculated from the supplementary strain rate changes performed during this test was equal to 0.35. At fracture, the sample showed no visible signs of necking.

4. Discussion

The results of the room temperature tensile test document that the strength of the studied material increased substantially due to ECAP. A UTS of 388 MPa in a 7075 aluminium alloy can be considered as a high value, with respect to the fact that the material was not precipitation hardened (a conventional AA7075 alloy in the O temper exhibits a UTS of 228 MPa). A higher UTS of 677 MPa has been reached by Zheng et al. in a 7050 aluminium alloy after ECAP at 393 K [8]. In this case, however, the strengthening effect of grain refinement during ECAP was combined with precipitation hardening. The recorded room temperature ductility of the ECAP AA7075-Zr-Sc alloy was inferior to that of a conventional 7075 alloy in the O temper. This is in accord with theoretical expectations, as materials produced by severe plastic deformation generally tend to exhibit lower ductility at room temperature. The increase in microhardness from HV 81 to HV 128 confirms again the strengthening effect of ECAP.

The results presented in Fig. 2 prove that a testing method as simple and fast as microhardness measurement can be successfully used to determine the position and dimensions of the DDZ. Such a measurement can therefore serve as a guideline when designing the shape and position of samples, which are to be machined from the ECAP billet for further experiments, e.g. tensile tests. Preparing a sample partly or entirely from the DDZ in the ECAP material would most likely lead to irrelevant results and incorrect conclusions. As shown in Fig. 2, the experimentally obtained size, shape and position of the DDZ qualitatively correspond to the scheme based on the ECAE 3D software. A more precise correspondence is not possible due to the large number of parameters, which influence the formation of the DDZ, but the software does not take them into account. These are especially the effects of friction and backpressure, as well as material parameters such as the strain hardening coefficient or strain rate sensitivity [9, 10].

The grain size obtained in the studied material after ECAP documents that this method of severe plastic deformation can be efficiently used to refine the microstructure of an Al-Zn-Mg-Cu alloy well below the micrometer level. The development of a relatively homogeneous microstructure with equiaxed grains can be attributed to the use of route B_C, which is known to have this effect given a sufficient number of ECAP passes [8]. For this reason, route B_C was chosen for the present investigation in the first place. The non-equilibrium state of grain boundaries and high dislocation densities resulting from the severe deformation represents typical microstructural characteristics of materials after ECAP. The stability of the UFG microstructure up to the temperature of 623 K shows the influence of the Zr and Sc additions. The fine-grained microstructure of a material of similar composition (also an AA7075 alloy), which was the subject of an earlier study [11], and contained no Zr or Sc, was stable only up to the temperature of 573 K. At higher temperatures, an onset of very rapid grain growth was recorded, with grain sizes exceeding 100 μm at 673 K. It should also be pointed out, that the material with Zr and Sc used for the present study underwent ECAP at a lower temperature than the previously studied alloy (393 K instead of 473 K). A higher driving force for grain growth can therefore be expected in the material due to a greater amount of stored energy, which is likely to act against the stabilizing effect of Zr and Sc, making it seem less pronounced.

The strain rate dependence of the strain rate sensitivity parameter m is consistent with the observations of the microstructure. The lower value of the maximum m as well as the shift of its position towards substantially lower strain rates at the temperature of 723 K can be attributed to the coarsening of the microstructure, which was documented by TEM. Nevertheless, the stability of the microstructure at 623 K is sufficient for the occurrence of high strain rate superplasticity with an elongation to failure exceeding 500 %. This confirms again the advantage over the previously studied material with no Zr or Sc, as it reached a maximum elongation to failure of only 200 % at 573 K and an initial strain rate of 8×10^{-4} s^{-1} [11]. Xu et al. achieved HSRSP in an ECAP 7034 alloy containing 0.2 wt.% Zr with an elongation to failure exceeding 1000 % at 673 K, due to a good thermal stability of the microstructure [12]. However, in [12], as well as in [11], the temperature of ECAP was 473 K, i.e. higher than the ECAP temperature of our AA7075-Zr-Sc alloy. This supports the hypothesis that a lower ECAP temperature acts against the stabilizing effect of the Al_3 (Zr_xSc_{1-x}) precipitates. At the temperature of 623 K and an initial strain rate of 1×10^{-2} s^{-1}, Xu et al. recorded an elongation to failure of 500 %, which is in accord with the results for the ECAP AA7075-Zr-Sc alloy obtained in the present study. The strain rate sensitivity parameter m in the vicinity of 0.3 can be considered as relatively low for a superplastic alloy, nevertheless, such values have already been reported for similar materials [11, 12]. Above all, it should be emphasized that despite the low strain rate sensitivity, the studied material does exhibit high strain rate superplasticity at a relatively low temperature, which represents a very attractive combination with respect to its eventual commercial applications.

5. Conclusions

The following conclusions can thus be drawn from this study:

1. The grain size of a Zr and Sc modified 7075 aluminium alloy was reduced to 200–500 nm by equal channel angular pressing, which resulted in a microhardness increase from HV 81 to HV 128 as well as a UTS of 388 MPa.

2. Microhardness measurements were successfully used to determine the deformation dead zone (DDZ) in the ECAP billet.

3. The ultra-fine grained microstructure was stable up to the temperature of 623 K, which documents the stabilizing effect of the Zr and Sc additions. At this temperature, the material exhibited high strain rate superplasticity. At an initial strain rate of $2 \times 10^{-2}\ s^{-1}$, an elongation to failure exceeding 500 % was achieved.

4. At 723 K, a coarsening of the microstructure was observed, caused most likely by a high driving force for grain growth due to the relatively low temperature of ECAP. This driving force is expected to act against the stabilizing effect of Zr and Sc.

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