Fracture of Ultrafine Grained Magnesium Alloy AZ31 Under Superplastic Deformation Conditions

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Abstract. Equal Channel Angular Pressing (ECAP) with an imposed hydrostatic pressure conducted at a relatively low temperature of 150 ºC was used to obtain exceptional superplastic properties of magnesium alloy AZ31. The mechanism of the significantly improved ductility was studied by examining the fracture surfaces and internal cavities in superplastically deformed specimens by scanning electron microscopy. The micrographs of the fracture surfaces exhibit unusually deep elongated dimples. The micrographs provide an indication that the dimples were formed at an early stage of the tensile test, possibly due to pull-out of relatively large precipitates, as the size of the dimples is comparable with the precipitate size. However, the load-bearing capacity of the ligaments of intact material between the dimples was still high enough to maintain overall integrity of the specimen during the continued tensile test, while the pores formed kept getting elongated along with the extension of the specimen as a whole. The high ductility of the material within the ligaments suggests that further increase of superplastic ductility can be achieved by appropriate heat treatment eliminating large precipitates. We believe that the benefits of the combined effect of increased strength and ductility found for ECAP-induced bi-modal grain structure could then be utilised to an even greater extent.

Introduction

Magnesium alloys have a great potential as structural materials for aerospace, automotive and electronics applications owing to their low density and high strength-to-weight ratio. However, forming of magnesium parts is limited due to a relatively low ductility of magnesium alloys owing to their hcp crystal structure. The development of thermo-mechanical processing leading to superplastic behaviour in magnesium alloys at relatively low temperatures and at acceptable strain rates will further the industrial applications of plastically formed magnesium parts.

To enhance ductility and to establish superplastic properties in magnesium alloys, various methods of thermo-mechanical processing, such as hot-rolling and extrusion, have been used. In the last decade a great deal of attention has been given to Equal Channel Angular Pressing (ECAP), Fig. 1, used as a means of producing uniform distributions of small grains, which is believed to be a prerequisite for superplastic behaviour [1, 2, 3]. However, even for widely investigated magnesium alloys, such as ZK60 and AZ31, there is no consensus concerning the optimum thermo-mechanical processing route leading to enhanced tensile ductility. At the present time there is no agreement on the type of microstructures most favourable for increasing the elongation-to-failure.

Our research on ZK60 [4] and AZ31 [5] has shown that bi-modal microstructure in magnesium alloys is more favourable for extraordinarily enhanced tensile ductility than a uniform small grain structure. However, the mechanism of the significantly improved ductility due to bi-modality of the grain structure is not clear as yet and further investigations are required. In this work a study of superplastic deformation of magnesium alloy AZ31 with a bi-modal structure was carried out by examining the fracture surfaces and internal cavities using scanning electron microscopy (SEM).

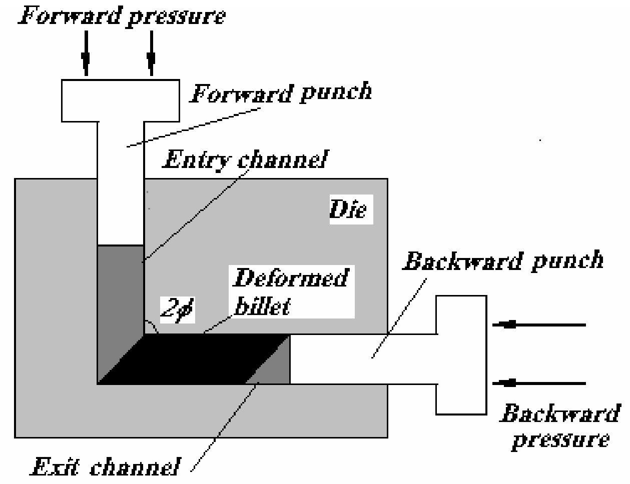


Fig. 1 Schematic illustration of Equal Channel Angular Pressing with applied back pressure.

Experimental and Results

Alloy AZ31 (3.1wt% Al, 1.0wt% Zn, 0.3wt% Mn) obtained in the form of continuously cast billet was homogenised for 4 hours at 420 ºC. The heat treatment was optimised as described in [6] to obtain a uniform distribution of 10 µm sized particles ($Mg_{17}Al_{12}$) and 1-2 µm sized round precipitates (Mn_5Al_8). The average grain size prior to ECAP was ~640 µm.

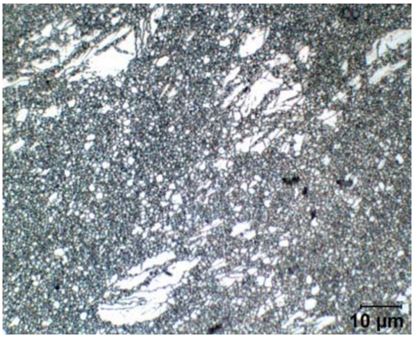


Fig. 2 Bi-modal microstructure of AZ31 after ECAP processing at 150 ºC [5].

ECAP of the samples was performed using a $2\phi = 90^o$ die (cf. Fig. 1), with application of back-pressure. The processing route B_C , which each billet is rotated by 90° about the longitudinal axis in the same direction after each pass, was employed. To produce a bi-modal microstructure, the samples were processed by 6 passes of ECAP at temperature as low as 150 ºC. The pressing of magnesium alloy at such temperature was only possible under high hydrostatic pressure created by application of back pressure (260 MPa in this case) [7]. The two grain populations had distinctly different grain sizes: in the range of 0.5 ± 0.25 µm for small grains and about 16.5 ± 3.5 µm for large grains, Fig. 2. It should be noted that at temperatures above 150 ºC the microstructure consisted of uniform small grains.

Tensile samples with a gauge length of 8 mm and diameter of 4 mm were machined from the ECAP processed billets and tested using INSTRON®4505 machine equipped with an environmental chamber. Elevated temperature tensile tests were carried out at 350 ºC with the strain rate of 10^{-4} s^{-1}. For these testing parameters AZ31 samples exhibited enhanced superplastic behaviour with a record strain-to-failure of 1215%, Fig. 3, while ductility of original samples that did not undergo ECAP at the same testing conditions was only 37%.

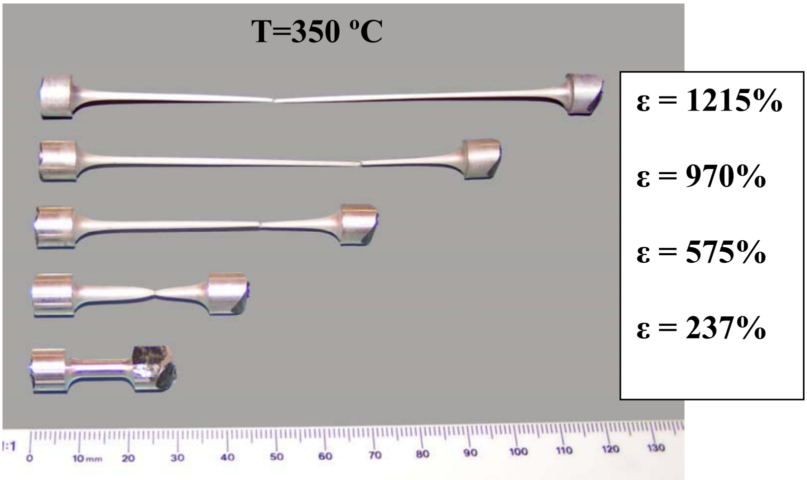


Fig. 3 Original specimen (at the bottom) and tensile specimens tested at 350 ºC under different strain rates. Strain rate increases from 10^{-4} s^{-1} (top sample) to 10^{-1} s^{-1} (second sample from the bottom).

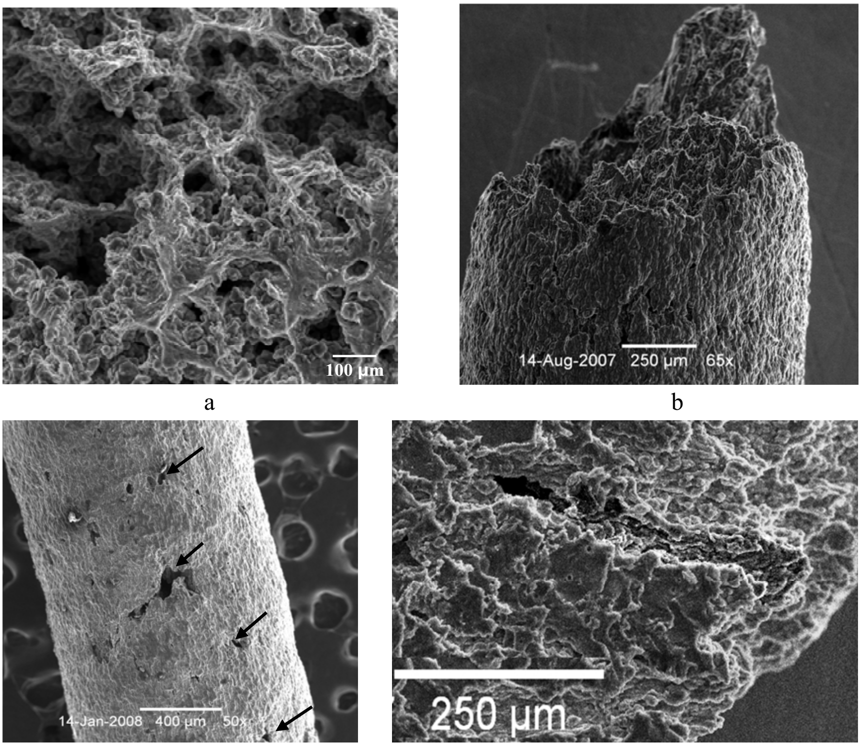
Fracture Investigations

Fracture of the specimens during superplastic tensile tests was studied with regard to three aspects, namely (i) the appearance of the fracture surface and the outer surface of the tensile specimens; (ii) rupture of ductile ligaments; and (iii) specific microstructural features of the specimens tested.

Surface Appearance. The tip of a failed tensile specimen at the rupture site and the fracture surface are shown in Fig. 4. It can be seen that the specimen tip has an atypical asymmetrical shape, Fig. 4b, which could be expected based on the texture produced after ECAP [8, 9]. Unusually deep pores were observed at the dimpled fracture surface, Fig. 4a. The diameter of the pores was comparable with the diameter of large $Mg_{17}Al_{12}$ particles, which were partly cracked during ECAP processing, as was revealed by optical microscopy [6]. The fracture of some particles and their rotation during ECAP led to de-bonding, and large voids were formed at particles/matrix interfaces. These voids grew and coalesced to sizable pores during the subsequent tensile test. Two directions of pore growth, namely parallel with the longitudinal axis of the sample and at an angle to the axis were detected. The growth at an angle to the tensile loading direction, which is known to be ~50º [10], results in cavities exiting at the outer surface of tensile sample, Fig. 4c (indicated by black arrows in the photograph). To determine the depth of longitudinally elongated pores, the tip of the tensile sample was cut lengthwise by a wire spark-erosion cutter. Fortunately, some pores were revealed, Fig. 4d, showing the depth of pore elongation to be about 250μm. This confirms the assumption that the pores were formed at an early stage of the tensile test, most probably due to pull-out of relatively large precipitates at the sites where de-bonding occurred. However, the load-bearing capacity of the intact ligaments between the dimples was apparently high enough to carry the load during the continued tensile test, as the pores were elongated together with the sample.

Smaller secondary fibres were discovered on the surface of the ligaments separated by pores. The spacing between these fibres was comparable with the size of small Mn_5Al_8 precipitates, Fig. 5. The bonds between the matrix and these smaller-size precipitates are more difficult to break. Therefore, these small, secondary fibres were presumably formed on the primary ligaments at late stages of tensile test and did not play a decisive role in tensile ductility.

It can be assumed that - should it be possible to eliminate large precipitates by heat treatment - the elongation-to-failure can be raised even further.



a b

c d

Fig. 4 SEM micrographs of the superplastically deformed tensile sample

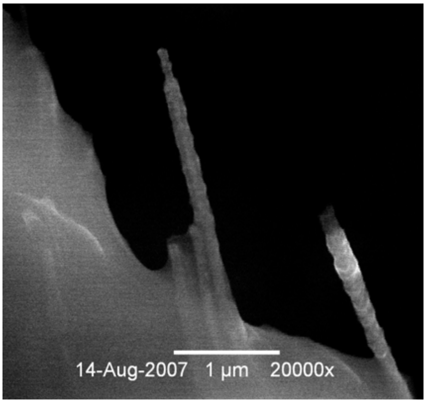
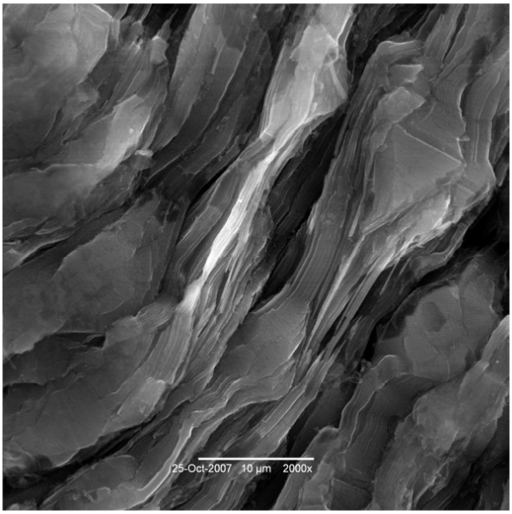


Fig. 5 Secondary fibres with spacing ~ 1 µm.

Ductile Fracture of Ligaments. Apparently the fracture of ligaments between the pores had an extremely ductile character with the cross-section of ligaments reduced to the order of 50-100 nm at the fracture location, which is typical for ductile rupture. After superplastic deformation the visible bulk of the sample had a lamellar structure composed of fibres or ligaments oriented parallel to the tensile axis, Fig. 6a. There is little variation in the width of the ligaments and the majority appeared to have a very high aspect ratio, Fig. 6b,c. Large areas with a pronounced "wood fibre" structure were observed, with places where the individual fibres were visibly delaminated and/or fractured, Fig. 6b. The fibres clearly form during the tensile elongation process and extend *en masse* up to the point where some fibres break and de-lamination is beginning to predominate.



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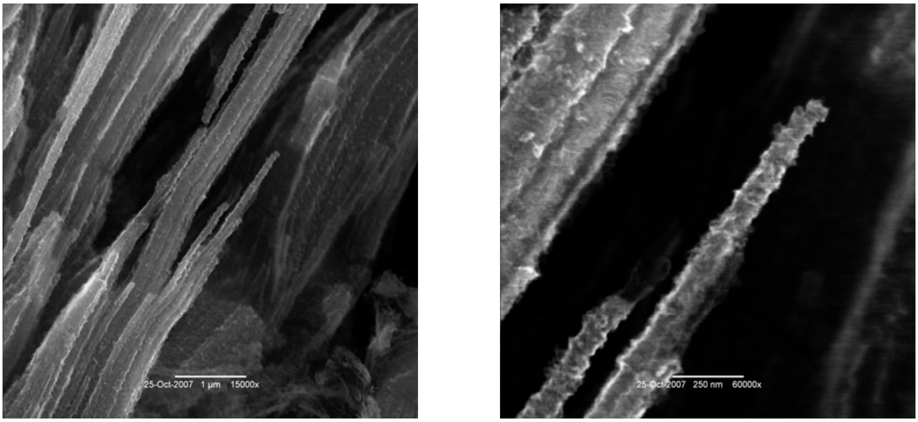

b

c

Fig. 6 Micrographs showing ductile rupture of ligaments

Specific Microstructural Features. In the case of texture favourable for activation of basal slip, the latter will be a predominant mode of deformation and failure of hcp metals. This is the case with the ECAP processed AZ31, in which a pronounced texture, with basal planes oriented at 45º to the ECAP direction, is formed [1, 8, 9], cf. Fig. 7.

Using argon plasma etching, the morphology of slip in the ECAP processed AZ31 during uniaxial tensile loading can, to some extent, be observed in the SEM. Some tensile test samples were placed in a GATAN PECS (precision etching/coating system) and slope-cut at 45º to the longitudinal axis for several hours. This resulted in a relatively deep smooth slope cut into the specimen, with a very large adjacent area of the sample (several mm^2 in area) more generally etched by the 6 kV argon plasma beam. A normal view of a cut at 45º to the tensile axis of the tensile specimen produced by plasma etching is presented in Fig. 8. A schematic sketch shows the geometry of the cut and the direction of view. The terrace-like pattern observed in many regions of the sample can be interpreted as a result of crystallographic slip on the basal plane of the hexagonal Mg structure. Detailed analysis of the plasma-etched material by SEM is the subject of an ongoing study to be reported elsewhere.

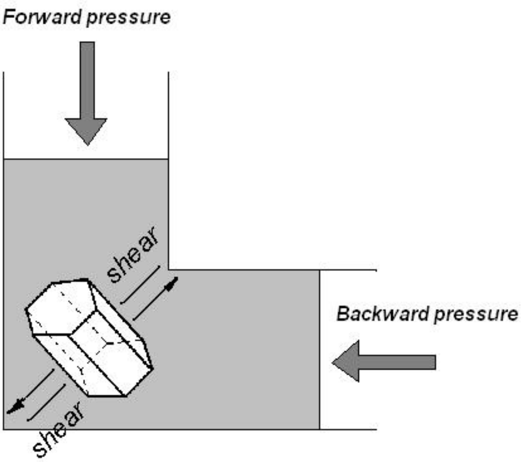

Fig. 7 Texture in an ECAP processed specimen: orientation of basal plane at 45º to the pressing direction, after Ref. [1].

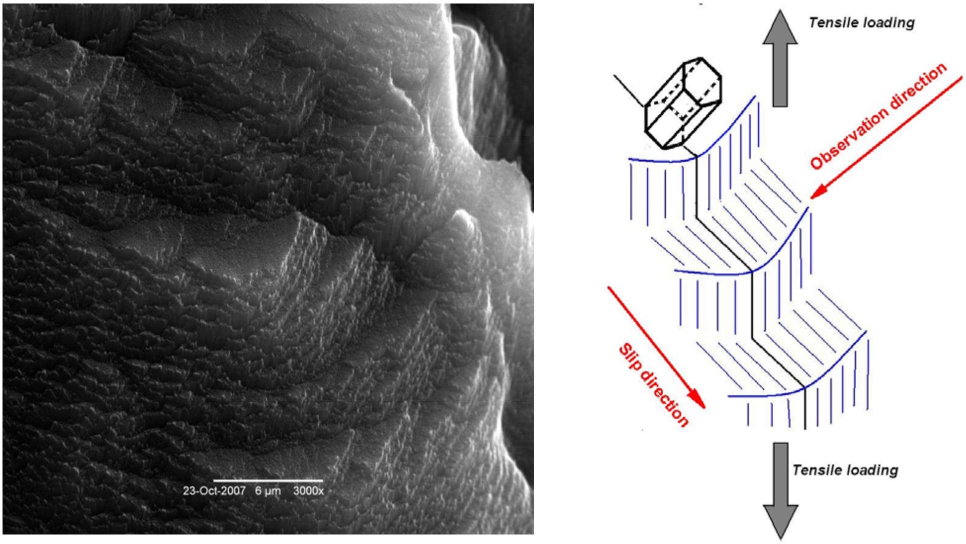

Fig. 8 Terrace-like morphology of the fracture surface reflecting crystallographic slip: SEM micrograph (left) and a schematic drawing (right)

Summary

Processing of magnesium alloy AZ31 by low temperature ECAP with back pressure was shown to lead to a record strain-to-failure in tensile test performed at the temperature of 350 ºC and strain rate of 10^{-4} s^{-1}. Fracture in superplastic regime was interpreted in terms of the microstructural features developed during ECAP processing. The observation of the fracture surface and the outer surface of the tensile specimens revealed unusually deep pores, presumably from pull-out of particles at an early stage of the tensile test, which elongated during the test. The ductility and load-bearing capacity of the ligaments were shown to be extremely high, terminating with typical ductile rupture. Secondary fibres separated by smaller-size precipitates had the same high level of ductility. It is expected that elimination of large particles by heat treatment would increase ductility even further. The study of specific microstructural features of the specimens tested revealed the influence of texture produced by ECAP processing on deformation and fracture of the material in the superplastic mode.

References

[1] T. Mukai, M. Yamanoi, H. Watanabe, K. Hiqashi: Scripta Materialia 45:1 (2001), p. 89

[2] H.K. Lin, J.C. Huang, T.G. Langdon: Materials Science and Engineering A 402:1-2 (2005), p. 250

[3] S.R. Agnew, G.M. Stoica, L.J. Chen, T.M. Lillo, J. Macheret, P.K. Liaw, in: *Ultrafine Grained Materials II*, *TMS Annual Meeting,* edited by Y.T. Zhu et al, Publication of The Minerals, Metals & Materials Society, (2002), p. 643

[4] R. Lapovok, P.F. Thomson, R. Cottam, Y. Estrin: *Materials Science & Engineering A* 410-411 (2005), p. 390

[5] R. Lapovok, Y. Estrin, M.V. Popov, T.G. Langdon: *Advanced Engineering Materials* 10:5 (2008), p. 429

[6] R. Lapovok, Y. Estrin, M.V. Popov, S. Rundell, T. Williams: *Journal of Materials Science* (2008), in press.

[7] R. Lapovok: *Journal of Materials Science*, 40:2 (2005), p. 341

[8] S.R. Agnew, T.M. Lillo, J. Macheret, G.M. Stoica, L. Chen, Y. Lu, D. Fielden, P.K.Liaw, in: *Magnesium Technology,* edited by J. Hryn, Publication of The Minerals, Metals & Materials Society, (2001), p. 243

[9] Y. Estrin, S.B. Yi, H.-G. Brokmeier, Z. Zúberová, S.C. Yoon, H.S. Kim, R.J. Hellmig: Journal of Materials Research, 99:1 (2008) p.50

[10] L.E. Miller, G.C. Smith: *Journal Iron Steel Institute London*, 208 (1970), p. 998