Influence of grain size fluctuations on the ductility of superplastic magnesium alloys processed by severe plastic deformation

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Abstract

The influence of fluctuations in the grain size along the gage length on ductility is analyzed in the superplastic regime. It is demonstrated that these fluctuations produce a similar effect to that produced by variations in the initial uniformity of the sample, leading to premature necking. In order to reach superplastic elongations of 400%, fluctuations in grain size of less than 0.5% between two zones of the gage length are required. As an example, the superplastic behavior of an AZ61 alloy, processed by severe plastic deformation, SPD, with a heterogeneous microstructure, is analyzed when the grain boundary sliding mechanism controls deformation. It is found that neck formation is related to bands of fine grains that are formed during SPD processing due to the mechanism of recrystallization by rotation. Under these circumstances grain refinement is rendered unsuccessful. The present investigation emphasizes the importance of the microstructure homogeneity in developing grain refinement processing routes.

1. Introduction

In recent years most of the efforts on superplasticity studies in magnesium alloys concentrate on thermomechanical processing for the grain size refinement using methods of severe plastic deformation (SPD). Widely used methods of SPD are: Equal channel angular pressing (ECAP), [1, 2, 3, 4, 5] accumulative roll bonding (ARB) [6, 7], large strain hot rolling (LSHR), [8 9 10 11] high pressure torsion (HPT),[12] extrusion, [13 14, 15, 16 17] and other. [18 19, 20, 21, 22 23] Recently, researchers implemented processing routes combining two or more of these processes in successive steps. [24, 25, 26] These methods have been demonstrated to be successful for obtaining grain sizes of about 1 μm and deformations larger than 400%. Nevertheless, minor attention has been given to the scattering of the superplastic elongations obtained by different processing routes or even by different researchers using similar routes.

As it is well known, a low stress exponent, *n*, is needed in order to achieve large deformations by preventing tensile instabilities. A large amount of data in superplastic deformation show that a low stress exponent, close to 2, is obtained when the mechanism controlling creep is grain boundary sliding (GBS). Under such circumstances the strain rate is given by the following power law equation: [27]

$$\dot{\varepsilon} = C\left(\frac{b}{d}\right)^{p}\left(\frac{\sigma}{E}\right)^{n} D_o \exp\left(-\frac{Q}{RT}\right) \qquad (1)$$

where C is a constant, E is the elastic modulus, $D = D_o exp(-Q/RT)$ is the appropriate diffusion coefficient, Q is the activation energy, R is the gas constant, T is the absolute temperature, b is the Burgers vector, d is the grain size, and p is the grain size exponent. When GBS dominates at low stresses, $n = 2$, and $p = 2$ or 3, and $Q = Q_L$ (activation energy for lattice self-diffusion) or $Q = Q_{gb}$ (activation energy for grain boundary diffusion) respectively. By refining the grain size the strain rate increases according to equation (1) and the GBS mechanism wins in competitiveness against the slip creep mechanism, which has a higher stress exponent.

Concerning the achievable elongations, failure in tension may occur by internal cavitation or external necking. From a mechanistic point of view if two samples have both $n = 2$ (although their grain sizes may be different and therefore its respective flow stresses) the strength to develop a necking is expected to be the same, and similar elongations should be expected for both samples. Experimental observations, however, demonstrate that finer grain sizes leads to larger elongations in magnesium alloys. This is probably due to a delayed grain growth effect: finer grain sizes take more time to grow to a size which produces the transition from GBS to slip creep loosing the superplastic properties of the material. Recently, it has been pointed out that this behavior could have a limit because a decrease of elongation seems to occur for nanocrystalline materials. [28]

Moreover, there is no consensus on the type of processing route that is most favorable for large elongations. It is recognized that a considerable scattering on elongations exist when data obtained by different processing routes or by different researchers using similar routes are compared, despite of the fine microstructures generally obtained in most of these studies, for example see Refs. 25 and 26. As it is pointed out in Ref. 25 the values of n are usually low and, in most of cases, differences in tensile behavior cannot be explained in terms of differences in stress exponent. Several factors have been mentioned as responsible of this dispersion:

1) Differences in resistance to damage. [16, 29] In contrast to aluminum alloys, cavitation of SPD processed Mg-alloys has not been extensively analyzed. The volume fraction of cavities developed in an extruded AZ31 (d = 2.9 μm) is lower than 0.025 (at $\varepsilon \approx$ 1.5, T close to 300°C, and strain rates between 10^{-3} and 10^{-2} s^{-1}) having a maximum elongation of 800%. On the other hand, in spite of the presence of high volume fraction of precipitates $Mg_{17}Al_{12}$, Mussi *et al.* found very low cavity volume fraction 0.0141 (at ε = 1.4, T = 250°C, $\dot{\varepsilon} = 10^{-3}$ s^{-1}) in an 8-passes ECAPed AZ91 (d = 0.5 μm) alloy with a

maximum elongations of 500%. Strikingly, the AZ31 alloy shows simultaneously larger cavity volume fraction and larger elongations than AZ91 for similar conditions. Moreover, Mussi et al. show that no significant coalescence between cavities occurs in the fracture zone which support the conclusion that fracture is associated with necking rather than with catastrophic cavity coalescence. Lee *et al.* conclude that cavitation in the AZ31 alloy is much less severe than that observed in Al base alloys.

2) Texture differences in samples processed by different routes. There is a controversy on the literature about the texture effect on GBS. [30] However, in our recent work on texture effects on superplasticity of an AM60 alloy, [31] it was shown that there are not texture effects in the superplastic regime with $n = 2$.

3) Under some processing conditions samples with a non-equilibrium grain boundaries exhibit lower elongations than the alloy with equilibrium grain boundaries after an annealing treatment. [32]

4) Differences in thermal stability of the grain size. There could be an indirect effect of texture on grain stability; some microstructures with different types of misorientation distributions could have different thermal stability leading to larger elongations in the more stable microstructure.

5) Differences in the dimensions of the testing samples. [33] This is an important factor when comparing data from different authors. Tensile samples with smaller gage length and high radius of curvature at the heads usually yield larger nominal elongations.

6) Heterogeneous grain sizes. It is generally recognized that the use of increasing number of ECAP passes, or combinations of different processings methods, increase homogeneity and grain refinement with, subsequently, increasing tensile elongations. However, there are reports on bimodal microstructures showing similar enhanced superplastic behavior. [25,18] For example, the excellent superplastic behavior of the ZK60

alloy of Ref. 25 has been attributed by Figueiredo and Langdon to the contribution of an appreciable volume fraction of very fine grains with $d \approx 0.4$ μm. Recently, Blandin and Dendievel [34] analyzed the effect of grain size distributions on the degree of homogeneity of deformation taking into account the contributions of GBS and dislocation creep along bands crossing a simulated microstructure. These authors calculate the effective strain rate sensitivity of these bands. From the dispersion in sensitivity these authors estimate the tendency to develop tensile instabilities for a given microstructure. They expect some localization of deformation in the case of a microstructure with a significant agglomerate of large grains .

In the present work it is demonstrated that minor fluctuations in the grain size along the sample length produces a similar effect to that caused by variations in the initial uniformity of the specimen leading to premature necking. This important factor is generally underestimated in superplasticity studies. This kind of heterogeneous microstructure is typical of SPD magnesium alloys and it depends strongly on the geometry of deformation, temperature, initial texture of the material, number of passes, etc. As discussed below, the development of a heterogeneous microstructure in magnesium alloys is related to their particular recrystallization mechanism. As an example, the deformation of an AZ61 alloy processed by LSHR, with a heterogeneous microstructure, is analyzed during superplastic deformation.

2. Experimental

The alloy used for this study was AZ61 (6%Al-1%Zn), provided by Magnesium Elektron in the form of a sheet, 3 mm in thickness. The alloy was received in the condition AZ61-O (rolled and annealed). Samples in this conditions were subsequently processed by LSHR at 400ºC using three passes with, respectively, 10%, 30% and 60% thickness reductions with a re-heating of 10 min at 400ºC between passes.

The processed AZ61 alloy was compared with other AZ61 alloy processed by rolling as reported in previous investigations. [8, 9] This material was received as-extruded in the form of a sheet, 10 mm in thickness, with an initial grain size of 54 μm, and processed by LSHR consisting of three passes of reductions of 20, 35, and 55% at 375°C, with a re-heating of 10 min at 375ºC between passes. In both cases, rolling was carried out in a Carl-Wezer rolling mill, furnished with 13 cm diameter rolls rotating at 52 rpm.

Metallographic preparation included grinding with increasingly finer SiC papers, mechanical polishing with 6 μm and 1 μm diamond paste, and a chemical etching step with a solution of 0.5 g of picric acid, 0.5 ml of acetic acid, 1 ml of distilled water and 25 ml of ethanol in order to reveal grain boundaries. The grain size was measured by the linear intercept method.

Flat tensile coupons of 15 mm gage length, and radius 3 mm were cut out of the as-received and processed materials. The mechanical behaviour of these materials were measured by means of uniaxial tensile tests performed at a constant strain rate of 2×10^{-4} s^{-1} in an electromechanical Servosis testing machine at 250^{o}C. Additionally, in order to determine the stress exponents, strain rate change tensile tests were also carried as described elsewhere. [31, 35]

3. Results and Discussion

3.1 Analysis of the neck growth.

In the following, we first present an analysis of the effect of fluctuations in cross-sectional area on elongation of the work of Avery and Stuart [36, 37.] This analysis consists in the study of neck growth under the assumptions that n is constant and grain growth is negligible during deformation. Subsequently, this analysis is extended to analyze the formation of a necking zone due to an heterogeneous grain size along the gauge length.

In a simple form, equation (1) can be rewritten as:

$$\dot{\varepsilon} \quad = \quad k\,\sigma^{n} \tag{2}$$

Simultaneously, the strain rate is given by:

$$\dot{\varepsilon} \quad = \quad \frac{1}{L}\frac{dL}{dt} = \quad -\frac{1}{A}\frac{dA}{dt} \tag{3}$$

From equations (2) and (3) and taking into account that the stress is $\sigma = F/A$:

$$\frac{dA}{dt} \quad = \quad -A^{1-n}\,k\,F^{n} \tag{4}$$

Integrating over the time interval from zero to t corresponding to a given strain:

$$\frac{1}{k}\int_{A_o}^{A} A^{n-1}\,dA \quad = \quad -\int_{0}^{t} F^{n}\,dt$$

(5)

The right hand of this equation does not depend on the position along the specimen or on the grain size in a given position. The following two situations can be evaluated by means of equation (5):

1) At $t = 0$ a small neck of cross sectional area αA_o exist, and at time t it will have the area βA. This situation is depicted in Fig. 1*a*. The evolution of the cross sectional areas is

given by the expression:

$$\int_{A_o}^{A} A^{n-1} dA \quad = \quad \int_{\alpha A_o}^{\beta A} A^{n-1} dA$$

(6)

leading to:

$$\frac{A}{A_o} \quad = \quad \left(\frac{1-\alpha^n}{1-\beta^n} \right)^{1/n} \tag{7}$$

2) At $t = 0$ there are two zones of equal cross sectional area A_o with different grain sizes d_1 and $d_2 < d_1$. In this case, two parameters are needed to be defined for each zone:

$$k_1 \quad = \quad CD(b/d_1)^p \qquad \text{and} \qquad k_2 \quad = \quad CD(b/d_2)^p \tag{8}$$

This situation is depicted in Fig 1*b*. Furthermore, at time *t* the zone with fine grain size will have the area βA. The evolution of the cross sectional areas are related according to:

$$\frac{1}{k_1} \int_{A_o}^{A} A^{n-1} dA \quad = \quad \frac{1}{k_2} \int_{A_o}^{\beta A} A^{n-1} dA \tag{9}$$

leading to:

$$\frac{A}{A_o} \quad = \quad \left(\frac{1-(d_2/d_1)^p}{1-(d_2/d_1)^p \, \beta^n} \right)^{1/n}$$

(10)

On the other hand, the relation A/A_o of equations (7) and (10) is related to the percentage elongation by:

$$e\% \;=\; \left(\frac{A_o}{A}-1\right)\times 100$$

(11)

With the help of equations (7), (10) and (11) it is possible to predict the percentage elongation for a 10% variation in the cross sectional area ($\beta = 0.9$, assumed as critical for fracture initiation) for a given value of α or d_2/d_1 respectively to both cases of Fig. 1.

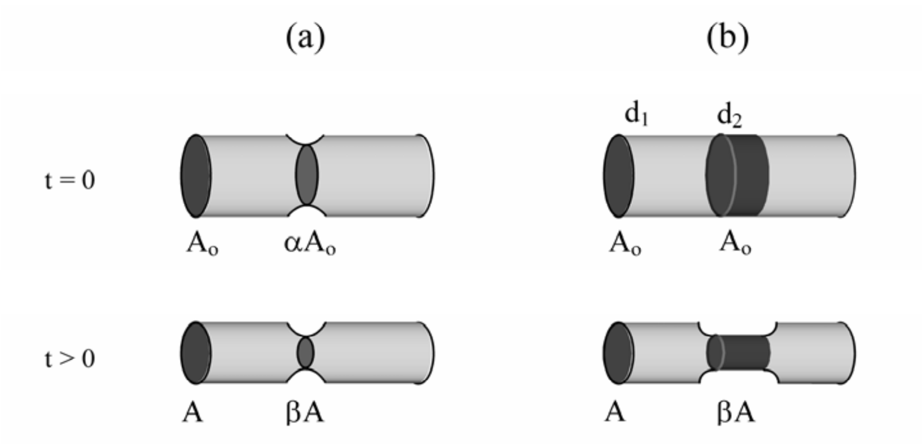

Figure 1 Analysis of neck growth in two cases: (a) small neck of initial cross-sectional area αAo and (b) two zones of equal cross-sectional area Ao with different grain sizes d_1 and $d_2 < d_1$.

As reported previously, large elongations are not just a function of n but, in an important degree, of the initial uniformity of the sample. Figure 2*a* shows that typical mechanical tolerances of $\alpha = 0.99$ gives elongations larger than 200% with $n = 2$.

On the other hand, Figure 2*b* shows that the uniformity in grain size along the sample has a similar importance on obtaining large elongations. Elongations larger than 400% are reached under the condition $1 > d_2/d_1 > 0.995$. This quite restrictive condition originates in the strong dependence of the strain rate on the grain size as given in equation (1).

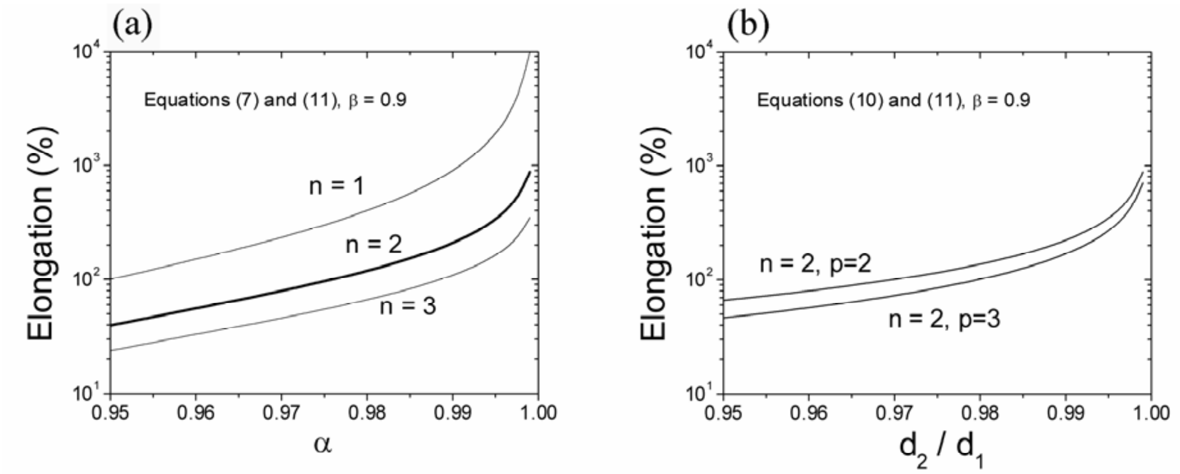

Figure 2 Elongation is plotted as function of a initial area ratio α and (b) grain size ratio d_2/d_1 for various values of *n* and *p* when neck develops to final ratio $\beta = 0.9$

3.2 Microstructural characterization of the AZ61 alloy.

The microstructure of the as-received material is formed by coarse equiaxed grains of 45 μm in size (Fig. 3*a*). A basal texture predominates that is characteristic of hot rolled Mg alloys. [8,10,11] After large strain rolling a partially recrystallized microstructure develops, Fig 3*b*. After a short thermal treatment of 15 min at 250°C a bimodal microstructure develops which is stable at this temperature up to times of 1 hour (Fig. 3*c*). However, the recrystallized microstructure shows traces of the deformation bands produced during rolling containing grains of about 2-3 μm in diameter, which cross the thickness of the sample and zones of larger grain size, of about 7 μm in size that remain from the original grains, Fig. 3*d*. This kind of heterogeneous microstructure is typical of LSHR magnesium alloys and it depends strongly on deformation geometry, rolling temperature, initial texture of the material, etc.

The development of a heterogeneous microstructure during high temperature deformation of magnesium is related to the recrystallization mechanisms in which new grains are formed at the old grain boundaries by a mechanism denominated “rotational recrystallization” leading to wider bands of fine grains as deformation increases. [8, 38, 39]

The SPD processing tends to form bands of fine grains in the shear directions. A bimodal banded microstructure is also observed in magnesium alloys after few ECAP passes as it is shown in Figs. 2 and 3 of Ref. 40. As it can be seen the alloying content strongly affects the homogeneity of the ECAPed samples, higher concentrations of aluminum produces an increase of the localization of deformation and recrystallization on thinner bands and very fine grain sizes inside these bands. The AZ31 alloy is the most homogeneous of the AZ series under SPD processing, although the AZ31 alloy also shows larger grain sizes than others of the AZ series because its lower grain stability. Even if the direction of the deformation is changed during processing, as occurs during ECAP by route Bc, there could be some heterogeneity of grain size due to the last ECAP pass.

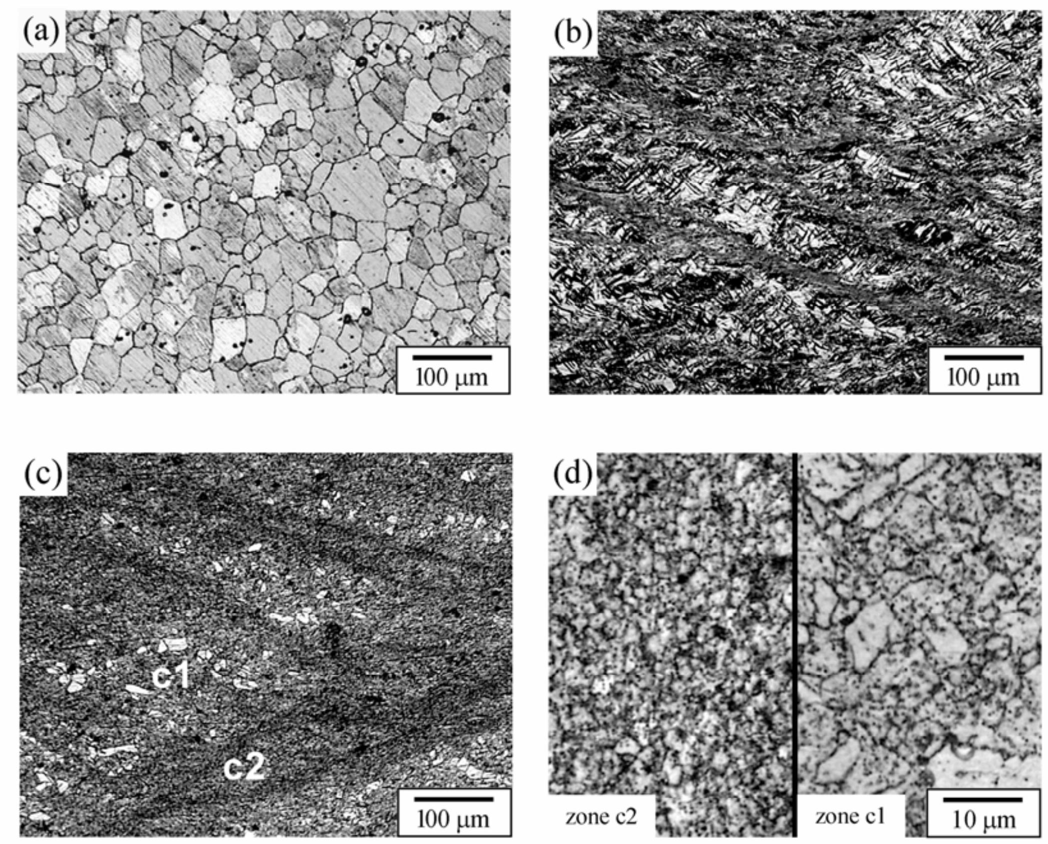

Figure 3 Microstructure of AZ61 alloy: (a) as received material; (b) after LSHR; (c) after LSHR plus thermal treatment of 15 min at 250 ^{o}C; (d) in zones c1 and c2 in Fig. 3c

Moreover, the grain size distribution obtained by severe rolling could be affected by deformation geometry. For example, in Refs. 8 and 9 the extruded plate of AZ61, 10 mm in thickness, was processed using the same rolling mill and a similar rolling temperature of 375^{o}C. The initial texture corresponds also to a basal fiber. The LSHR consist of three passes of reductions of 20, 35, and 55% also similar to those used in the present work. As can be seen in Fig. 5*a* of Ref. 8 or Fig. 1*b* of Ref. 9 the resulting microstructure is still heterogeneous but much less than in the case of our 3 mm sheet of Fig. 3*c* of this work. The zones with smaller grains appear to be less concentrated in bands and the smaller recrystallized grains have a size of approximately 5 μm in diameter. These differences probably originate in differences of initial sample thickness affecting the geometry of deformation during rolling as it was reported recently. [41]

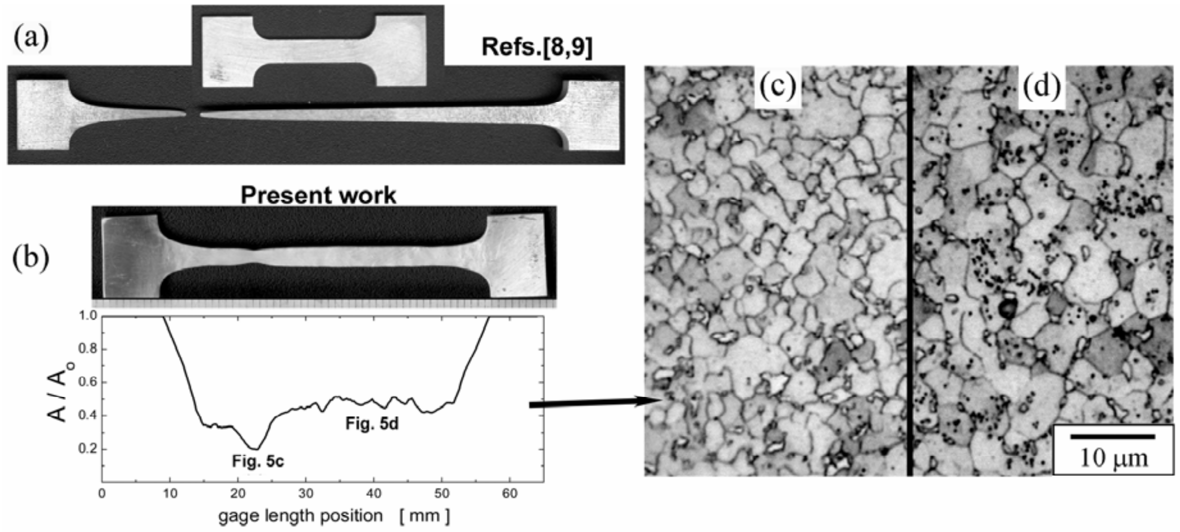

Figure 4: (a) tensile samples of AZ61 alloy processed in Refs. 8 and 9,(b) tensile sample of AZ61 alloy (present work) and variation in transversal area along gauge length, (c) and (d) microstructure in necking zone and in rest of sample respectively, as pointed out in Fig. 4b

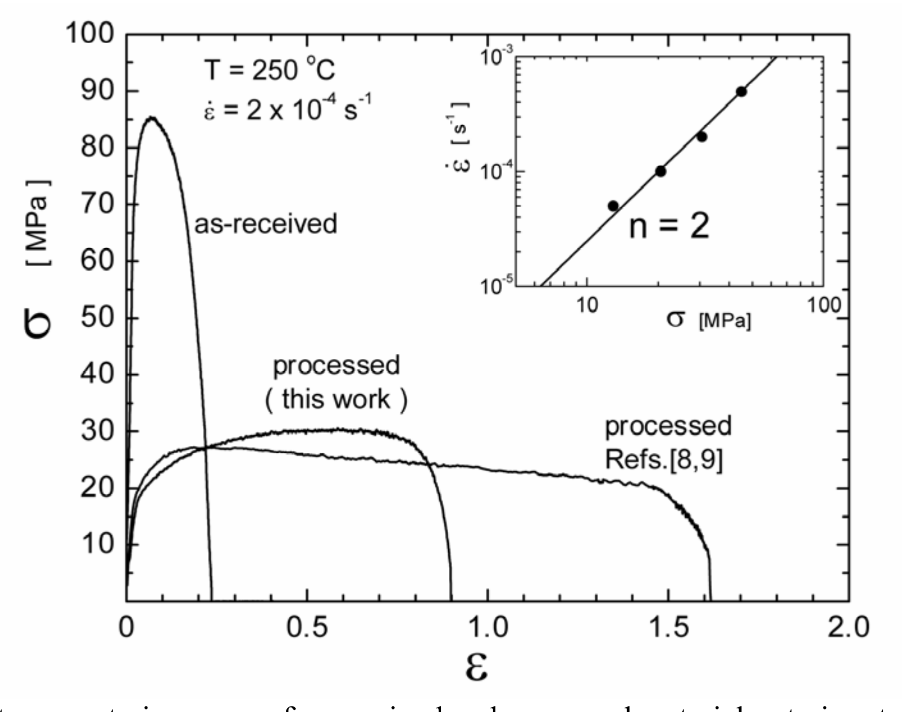

Figure 5 Stress v. strain curves of as received and processed materials: strain rate–stress data is given in insert

3.3 Mechanical behavior of the processed AZ61 alloy

Samples of processed AZ61 were deformed in tension at a strain rate of 2 x 10^{-4} s^{-1} and 250^{o}C. Figure 5 shows the stress vs. strain curves of the as-received and the processed materials. As it can be seen, the processing allows obtaining deformations close to 100% but considerably lower than expected for this fine microstructure. Additionally, a stress exponent of two was measured using the strain rate change test, insert of Fig. 5.

The stress exponent points toward grain boundary sliding as the controlling mechanism. In contrast, the AZ61 alloy processed in Refs. 8 and 9, also shown in Fig 5, with a more homogenous microstructure, yields elongations of about 400% at the same testing conditions.

Figure 4*a* and 4*b* shows tensile samples of AZ61 alloy processed in Refs. 8 and 9 and in the present work respectively. Figure 4*a* shows a uniform deformation with the characteristic diffuse necking, this result is attributed to the homogeneity of the microstructure. Conversely, Fig. 4*b* shows considerable non-uniformity or undulations along the gage length; the variation in the transversal area along the gauge length is also shown (the test of Fig. 4*b* sample was interrupted once the necking was formed, previously to the fracture). Necking was often observed in more than one place along the gauge length of the sample. The microstructure of the sample shown in Fig. 4*b* presents a finer grain size in the necking zone, Fig. 4*c*, than in the rest of the sample, Fig. 4*d*. Moreover, there is practically no cavitation in the necking zone indicating that its origin cannot be attributed to localized damage caused during processing.

Another aspect to consider is the effect on ductility of grain growth during deformation and the role of bi-modal grain size distributions. As it can be seen by comparison of Fig. 3*d* and Fig. 4*c*-*d* some grain growth occurs during deformation. It is well known that enhanced grain growth occurs during superplasticity and it may occur that, if deformation is larger in the zones of fine grain size, the grain growth stimulated in this zones acts as a strain hardening, through equation (1), avoiding the progress of the tensile instability. It is a matter of speculation, however, to estimate the influence of this mechanism on elongation. In the present case, it is found that grain growth in the fine grain zones is insufficient to produce such effects.

On the other hand, it has been shown in Ref. 25 that bimodal microstructures yield excellent superplastic properties. It is contentious that two scale lengths should be considered regarding the grain structure: a microscale where few grains are considered in small regions and a large scale that comprises the entire gauge length of the sample. Heterogeneities in the microscale, for instance in the form of a bimodal distribution, should not affect the ductility whereas heterogeneities in the large scale, for instance in the form of grain size gradients, should have a detrimental effect on ductility.

4. Conclusions

It has been shown that grain size gradients in the form of bands of fine grains across the sample increase the tensile instabilities. This impedes the attainment of large elongations, despite of the high strain rate sensitivity of these samples. In the present work, it is demonstrated that minor fluctuations in the grain size along the sample length produces a similar effect to that of the variations in initial uniformity of the sample, leading to premature necking. The mathematical analysis shows that a very restrictive condition of grain size uniformity rules the growth of this kind of neck. Elongations larger than 400% are reached under the condition that the fluctuations in grain size are less than 0.5% between two zones of the gauge length. Additionally, the superplastic properties of an AZ61 alloy processed by SPD with a heterogeneous microstructure are analyzed at strain rates when grain boundary sliding controls deformation. It is found that the formation of necking is related to bands of fine grains in the processed material. Under these circumstances grain refinement processing is rendered unsuccessful. The present investigation emphasizes the importance of the evaluation of the microstructure homogeneity in developing grain refinement processing routes.

Acknowledgments

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References

1. M. Mabuchi, K. Ameyama, H. Iwasaki and K. Higashi: *Acta Mater.*, 1999, **47**,2047-2057.
2. B. Q. Han and T .G. Langdon: *Mater. Sci. Eng. A*, 2005, **410-411**, 435-438.
3. M. Furui, C Xu, T. Aida, M. Inoue, H. Anada and T. G. Langdon: *Mater. Sci. Eng. A*, 2005, **410-411**, 439-442.
4. H. K. Lin, J. C. Huang and T. G. Langdon: *Mater. Sci. Eng. A*, 2005, **402**, 250-257.
5. K. Mathis, J. Gbieza and N. H. Nam: *J. Alloys and Compounds*, 2005, **394**, 194-199.
6. M. T. Perez-Prado, J. A. del Valle and O. A. Ruano: *Scr. Mater.*, 2004, **51**, 1093-1097.
7. J. A. del Valle, M. T. Perez-Prado and O. A. Ruano: *Mater. Sci. Eng. A*, 2005, **410,** 353-357.
8. J. A. del Valle, M. T. Perez-Prado and O. A. Ruano: *Mater. Sci. Eng. A*, 2003, **355,** 68-78.
9. M. T. Perez-Prado, J. A. del Valle and O. A. Ruano: *Mater. Sci. Forum*, 2004, **447-448**, 221-226.
10. J. A. del Valle, M. T. Pérez-Prado, J. R. Bartolomé, F. Peñalba and O. A. Ruano: *Mater. Trans.*, 2003, **44**, 2625-2630.
11. M. T Pérez-Prado, J. A. del Valle, J. M. Contreras and O. A. Ruano: *Scr. Mater.*, 2004, **50**, 661-665.

12. G. Sakai, K. Nakamura, Z. Horita and T. G. Langdon: *Mater. Sci. Eng. A*, 2005, **406**, 268–273.
13. T. Mukai, T. G. Nieh, H. Iwasaki and K. Higashi: *Mater. Sci. Technol.*, 1988, **14**, 32-35.
14. N.V. Ravi Kumar, J. J. Blandin, C. Desrayaud, F. Montheillet and M. Suery: *Mater. Sci. Eng. A*, 2003, **359**, 150-157.
15. H. Watanabe, T. Mukai, K. Ishikawa, M. Mabuchi and K. Higashi: *Mater. Sci. Eng. A*, 2001, **307**, 119-128.
16. C. J. Lee and J. C. Huang: *Acta Mater.*, 2004, **52**, 3111-3122.
17 T. Mukai, H. Watanabe and K. Higashi: Mater. Sci. Technol.,2000, 16, 1314–1319.
18. A. Galiyev and R. Kaibyshev, *Scr. Mater.*, 2004, **51**, 89-93.
19. H. Q. Sun, Y. N. Shi, M. X. Zhang and K. Lu: *Acta Mater.*, 2007, **55**, 975–982.
20. Q. Gou, H. G. Yan, Z. H. Chen and H. Zhang: *Mater. Charact.*, 2007, **58,** 162-167.
21. Q. Yang and A. K. Gosh: Acta Mater., 2006, **54**, 5147-5158.
22. F. Y. Hung, C. C. Shih, L. H. Chen and T. S. Lui: *J. Alloys and Compd.*, 2006, **428**, 106-114.
23. M. M. Attallah and H.G. Salem: *Mater. Sci. Technol.*, 2004, **20**,1370-1376.
24. Y. Miyahara, K. Matsubara, Z. Horita and T. G. Langdon: *Metall. Mater. Trans. A*, 2005, 36, 1705-1711.
25. R. Lapovok, P. F. Thomson, R. Cottam and Y. Estrin: *Mater. Sci. Eng. A*, 2005, **410-411**, 390-393.
26. R. B. Figueiredo and T.G. Langdon: *Mater. Sci. Eng. A*, 2006, **430**, 151-156.
27. O. A Ruano and O. D. Sherby: *Revue Phys. Appl.*, 1988, **23**, 625-637.
28. A. V. Sergueeva, N. A. Mara, R. Z. Valiev and A. K. Mukherjee: *Mater. Sci. Eng. A*, 2005, **410-411**, 413-416.
29. A. Mussi, J. J. Blandin, L. Salvo and E. F. Rauch: *Acta Mater.*, 2006, **54**, 3801-3809.
30. K. A. Padmanabhan and K. Lücke: *Z. Metallkd.*, 1986, **77**, 765-770.
31. J. A. del Valle and O. A. Ruano: *Acta Mater.*, 2007, **55**, 455-499.
32. M. Mabuchi, K. Ameyama, H. Iwasaki and K. Higashi: *Acta Mater.*, 1999, **47**, 2047-2057.
33. H. Watanabe, F. Ono and K Higashi: *Mat. Sci. Forum*, 2004, **447-448**, 189-196.
34. J. J. Blandin and R. Dendievel: *Acta Mater.*, 2000, **48**, 1541-1549.
35. J. A. del Valle, M. T. Perez-Prado and O. A. Ruano: *Metall. Mater. Trans. A*, 2005, **36**, 1427-1438.
36. D. H. Avery and J. M. Stuart, 14th Sagamore Army 1427-1438.Materials Research Conf., Raquette Lake, NY, 1967.
37. J. W. Edington, K. N. Melton and C. P. Cutler: *Prog. Mater. Sci.*, 1976, **21**, 61.
38. S. E. Ion, F. J. Humphreys and S. H. White: *Acta Metall.*, 1982, **30**, 1909-1919.
39. Y. Liu and X. Wu: *Metall. Mater. Trans A.*, 2006, **37**, 7-17.
40. M. T. Perez-Prado, J. A. del Valle and O. A. Ruano: *Mat. Lett.*, 2005, **59**, 3299-3303.
41. M. T. Perez-Prado, J. A. del Valle and O. A. Ruano: *Scr. Mater.*, 2004, **50**, 667-671.