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Low temperature superplasticity of hydrostatically extruded Mg-Al-Zn alloys

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Abstract. In this work, the superplastic behavior of AZ31, AZ61 and AZ80 magnesium alloys was investigated. The alloys were hydrostatically extruded at only 150 °C to get fine grained microstructures (<10 µm). Tension tests were carried out at 175, 200 and 225 °C and 10^{-2}, 10^{-3} and 10^{-4} s^{-1}. It was found that all alloys exhibited superplasticity at 200 °C, 175 °C and 225 °C for AZ31, AZ61 and AZ80 alloys, respectively. Low temperature dynamic recrystallization played an important role for generating a finer and homogeneous microstructure during testing which enhances the deformation behavior of the alloys at these temperatures.

1. Introduction

Magnesium alloys are being increasingly used in electronics, automobile and aerospace industries because of their low density, high specific strength and excellent machinability [1]. However, due to their hexagonal close-packed (HCP) structure, they generally exhibit limited ductility at low temperatures. This is because just few deformation modes like slip and twinning can be activated as the main deformation mechanisms. Recent studies have shown that ductility in magnesium alloys can be improved by reducing grain size and also superplastic behavior has been observed [2-4]. Some works have used severe plastic deformation (SPD) techniques that offer the possibility to generate ultrafine grain sizes and low temperature superplastic behavior has been reported [5-7]. However, to get such a fine grain size, a combination of a conventional thermomechanical treatment with a SPD technique is necessary. Recently, hydrostatic extrusion as an alternative thermomechanical treatment has been used successfully to produce fine grained Mg alloys [8, 9]. In this thermomechanical treatment, the billet is completely circumscribed by a pressurized liquid. The most important feature is that the hydrostatic pressure is applied to the medium leading to a homogeneous distribution of pressure over the whole surface of the billet. Thus, higher pressures than those available in the other methods can be applied. Moreover, the influence of friction is eliminated because the ram and the container do not touch the billet directly and the die is the only possible place where friction may appear. The lack of friction between billet and container also serves to reduce the extrusion force required. This allows the use of higher extrusion ratios and therefore faster production speeds [10]. It has been demonstrated that this process can be carried out at temperatures as low as 100 °C in Mg-Al-Zn alloys and fine grain microstructures have been obtained [8]. In an earlier work the intermediate temperature deformation of an AZ61 alloy processed by hydrostatic extrusion was investigated [11]. It was shown that it is possible to reach very high elongations to failure at temperatures below 200 °C. However, it would be interesting to expand the knowledge on the deformation behavior of more Mg-Al-Zn alloys processed by this method. Therefore in this work, AZ31, AZ61 and AZ80 alloys processed by hydrostatic extrusion are used to evaluate the deformation behavior at low temperatures.

2. Experimental procedure

AZ31 (2.88 wt.% Al–0.97 wt.% Zn–0.25 wt.% Mn and Mg balance), AZ61 (6.39 wt.% Al–0.93 wt.% Zn–0.20 wt.% Mn and Mg balance) and AZ80 (7.65 wt.% Al–0.48 wt.% Zn–0.27 wt.% Mn and Mg balance) magnesium alloys were selected. Billets of all the alloys were produced by gravity casting. The alloys were casted into cylindrical steel containers of 0.1 m in diameter and 0.41 m in height.

Before thermomechanical treatment, direct chill-cast billets with 0.08 m diameter were homogenized for 12 h at 350 °C then cooled down in air to room temperature.

Hydrostatic Extrusions on the alloys were carried out in an ASEA-12MN Hydrostatic press at 150 °C with an extrusion ratio of 1:28 and MoS_2 as lubricant in the die exit. The applied load was 700 MPa. The used extrusion rate was 8 m/min to produce round bars of 0.015 m in diameter. The extruded bars were water quenched after the extrusion process. Details of the hydrostatic extrusion process can be found elsewhere [8].

Tensile samples with a gauge length of 20 mm and 6 mm in diameter (according with the standard DIN 50125) were machined from the extruded bars with their longitudinal axis parallel to the extrusion direction. Tensile tests were conducted at 175, 200 and 225 °C in air using a universal testing machine (Zwick™ Z050) equipped with an electrical furnace. At each temperature three different strain rates were used 10^{-2}, 10^{-3} and 10^{-4} s^{-1}. An extensometer was attached to the samples to get accurate measurements of the strain, in addition to this; the extensometer also controlled the strain rate during tests.

For metallographic inspection, samples were mechanically grinded with SiC paper grit 800, 1000, 1500 and 2000. For final polishing oxide polishing suspension (OPS) of 0.05 µm was used. The polished samples were chemically etched with a solution of picric acid (150 ml of ethanol, 40 ml distilled water, 6.5 ml acetic acid, 3-4 g picric acid)

3. Results and discussion

3.1 Microstructure

The microstructures of the extruded alloys are presented in Fig. 1 (where the extrusion direction lies horizontal). For the AZ31 alloy, a rather inhomogeneous microstructure is revealed (see Fig. 1a). It is composed by large elongated grains resulting from the original cast microstructure. The elongated grains are surrounded by new fine and recrystallized grains. The average grain size of the recrystallized grains is about 2.5 µm. Fig. 1b presents the microstructure of AZ61. It appears to be more homogeneous and well recrystallized. However, some coarse grains are still visible in the extrusion direction. The average grain size is 5 µm. In the picture, dark bands are observed where a high concentration of a secondary phase $Mg_{17}Al_{12}$ can be found. Fig. 1c depicts the microstructure of AZ80 alloy. The observed microstructure is much more homogeneous than the previous two alloys. For this alloy the etching was more complicated due to high aluminum content which resulted in a very high concentration of secondary phase $Mg_{17}Al_{12}$ that makes easy the over etching. This secondary phase is mainly located on the grain boundaries. Nevertheless, particles are also observed within the grains. The average grain size is 8 µm.

It should be noted that by increasing the aluminum content in alloys AZ61 and AZ80 a general improvement of the microstructure with respect to the homogeneity is observed. This is in agreement with results reported by Swiostek et al. [12] for hydrostatically Mg-Al-Zn alloys extruded at 200 and 300 °C. On one hand, the improvement in the microstructure could be a result of and enhanced recrystallization behavior during dynamic thermo-mechanical treatment with increasing the aluminum content. On the other hand, the solidus temperature lowers with increasing aluminum content and, therefore, it also could influence the recrystallization behavior leading to a more homogeneous and well recrystallized microstructure.

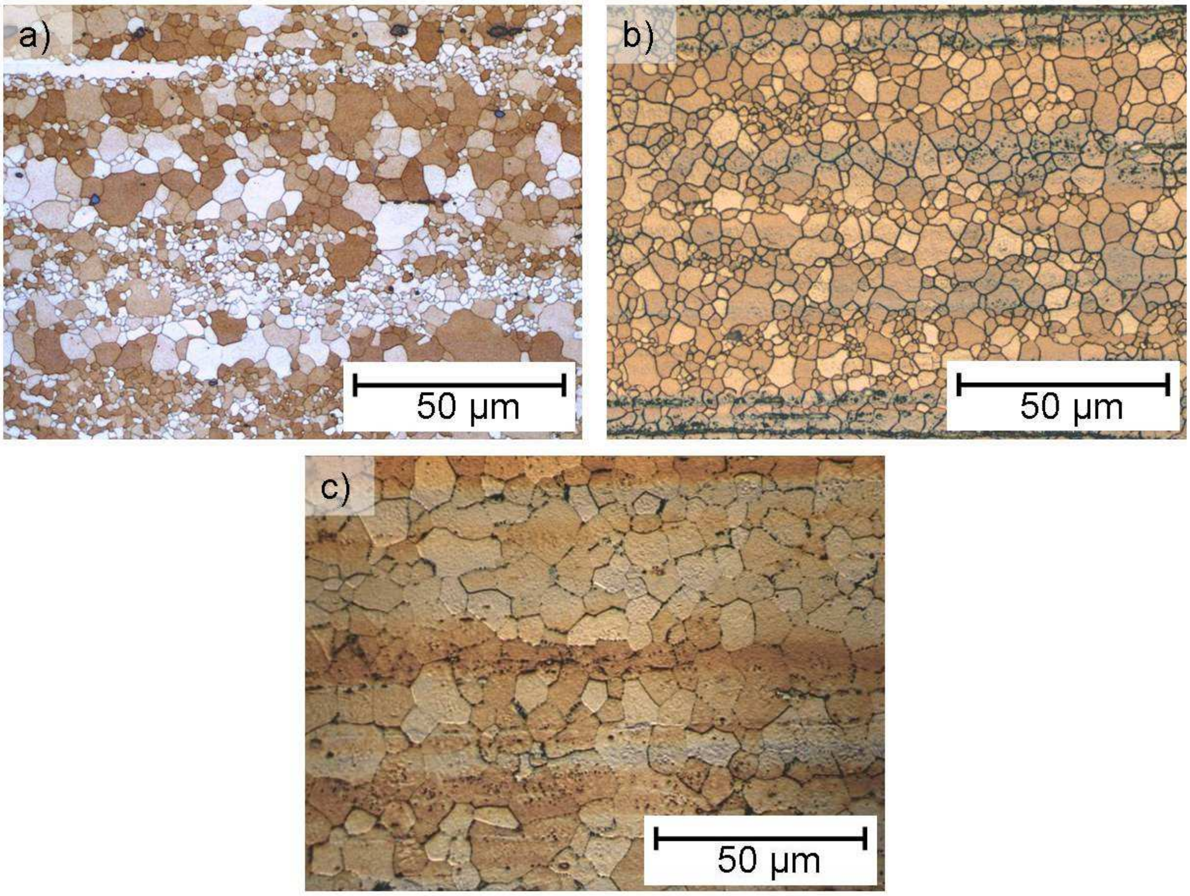

Fig. 1. Microstructure of hydrostatically extruded alloys a) AZ31, b) AZ61 and c) AZ80.

3.2 Tension tests

Exemplarily, Fig. 2 shows the true stress- true strain curves of all alloys tested at 225 °C and 10^{-3} s^{-1}. In general from the curves, it can be seen that when the flow stress reaches the peak stress, it is followed by work softening in all alloys. This behavior is much more pronounced in the alloys with the higher aluminum content. Such a reduction in the flow stress suggests that dynamic recrystallization (DRX) took place. It is widely known that for a metal which presents DRX, initially the flow stress increases with strain due to being dominated by strain hardening, and as DRX takes place upon a critical strain, the flow stress begins to decrease after it reaches certain peak value. When equilibrium is reached between softening due to DRX and strain hardening, the curves drop to a steady-state region [13, 14].

In a previous work it was demonstrated that DRX played and important role during the tensile deformation of the AZ61 alloy tested at the same temperatures [11]. It has been pointed out by Tan et al. [15] that work softening during the first stages of deformation (approximately 0.5 true strain) can be attributed to DRX. From Fig. 2 it is worth to note that the slope of the work softening increases as the aluminum content increases. In addition to this, the AZ61 and AZ80 alloys show a similar behavior in which both reach very high tensile elongations at this temperature and strain rate.

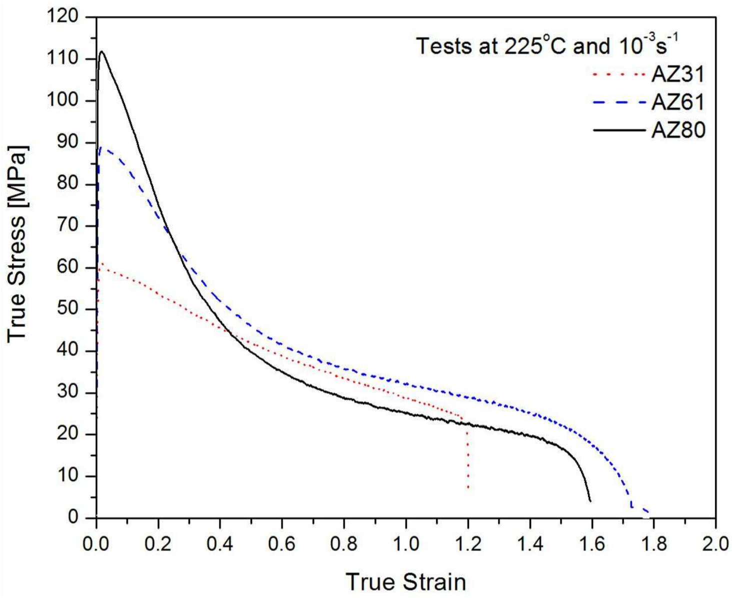

Fig. 2. True Stress-True Strain curves for all alloys tested at 225 °C and 10^{-3} s^{-1}

The results from the tension tests at all temperatures and strain rates for all alloys are plotted in Fig. 3. It can be seen that the maximum elongation near 400% for AZ31 alloy is achieved at 225 °C and $10^{-4}s^{-1}$. In general for AZ31 alloy, when the strain rate is increased further, the total elongation decreases monotonically at all temperatures (see Fig 3a). This performance fits well with the results reported by Lee et al. [16] for warm extruded sheets (processing temperature 300 °C) of an AZ31 alloy tested in the low strain rates of $6x10^{-4}s^{-1}$ and temperatures around 225 °C. In that work, a fine and homogeneous microstructure was obtained with an average grain size of 3 µm. Such a grain size is quite comparable to the one obtained by means of HE in this investigation for the AZ31 alloy. It is worth to mention that the extrusion rate used in that work to get such a fine and homogeneous microstructure was rather low of 0.001 s^{-1}, meanwhile, the AZ31 alloy processed by hydrostatic extrusion in this work allowed much higher extrusion rates of 0.130 s^{-1} and it shows similar superplastic behavior. In the case of AZ61 alloy, the superplastic results are presented in Fig. 3b. Very high elongations to failure are observed at intermediate strain rates at 200 and 225 °C. Interestingly, if the strain rate is decreased to $10^{-4}s^{-1}$, the alloy shows lower elongations at 200 and 225 °C. On the other hand, at 175 °C the elongation to failure increased monotonically when the strain rate is reduced. At this temperature and strain rate of $10^{-4}s^{-1}$, the AZ61 alloy shows excellent superplastic behavior, reaching a total elongation to failure of 520%. Kim et al.[17] worked with AZ61 sheets processed by hot rolling. In that work the results obtained are comparable with the elongations to failure recorded in this work. However, the thermomechanical treatment was performed at substantially higher temperature of 375 °C. That temperature is more than twice the temperature used in HE in this investigation. The resulting microstructure from that rolling was homogeneous with an average grain size of 8.7 µm. Nevertheless, the necessary temperature to achieve such elongations was considerable high. The tension tests were performed at 400 °C where the maximum elongations to failure of about 550 % laid in the range of 10^{-4} to 10^{-3} s^{-1}. Conversely, in this work an elongation to failure of 520 % was only at 175 °C in the low strain rate.

The AZ80 alloy shows a similar behavior compared to AZ31 alloy when the material is tested at 175 and 200 °C, when the strain rate is decreased the elongation to fracture increases monotonically, however, the material is able only to reach a strain about 160% at 200 °C and 10^{-4} s^{-1} (see Fig. 3c). Conversely, at 225 °C a similar behavior to the presented by the AZ61 alloy is found; at intermediate strain rate, the material shows excellent plasticity reaching elongations near 400%. This ability of reaching such a high elongations is also decreased in the lowest strain rate where the material only reaches 220%. The reduction in elongation to failure in the AZ61 and

AZ80 alloys can be related to the coalescence of precipitates of the secondary phase $Mg_{17}Al_{12}$ that induced cavitation. In an earlier work, it was shown how cavities affect the performance of the AZ61 alloy when tested in tension at intermediate temperatures in the low strain rate [11]. Since the AZ80 alloy contains a higher Al content than the AZ61, the appearance of more precipitates affect even more the elongation to failure when the material is tested at 10^{-4} s^{-1}.

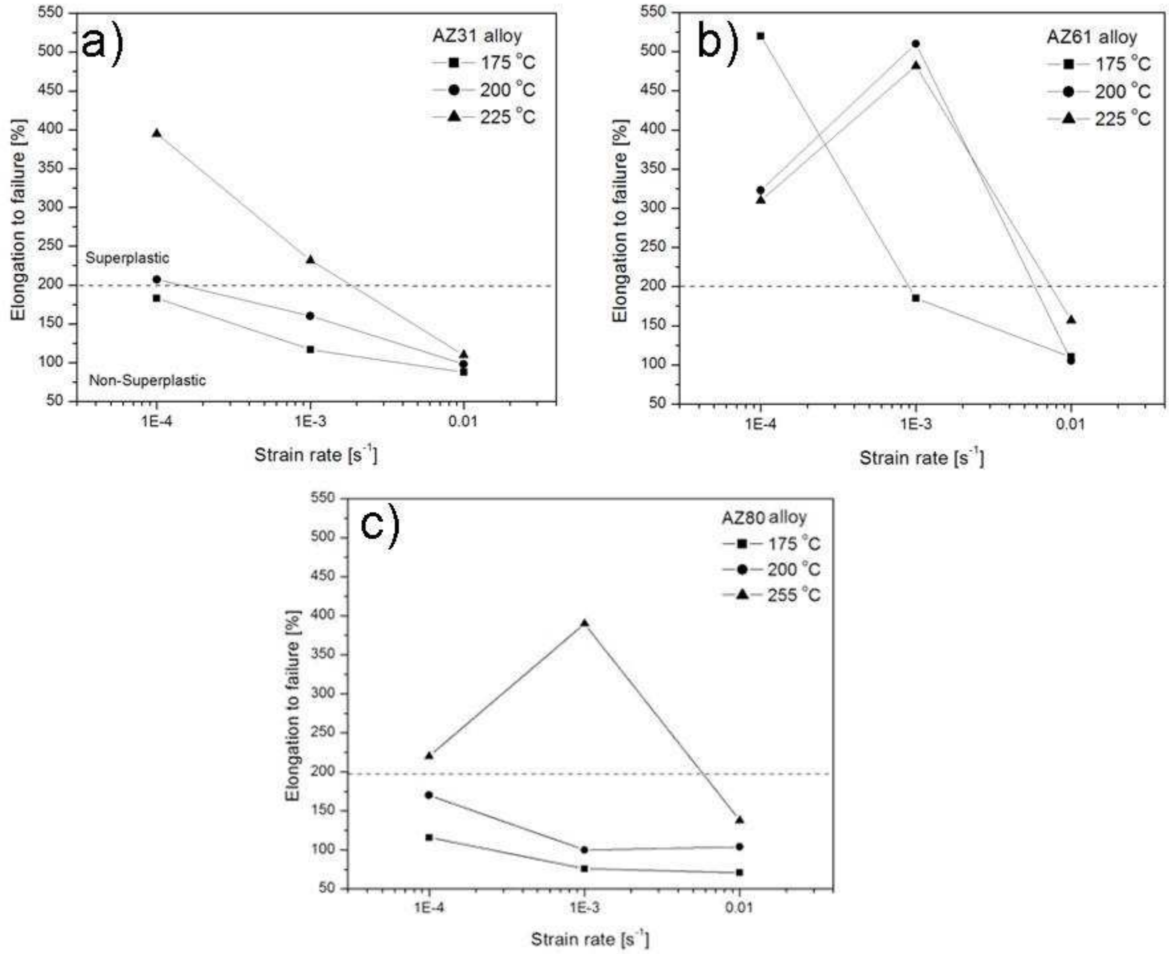

Fig. 3. Elongation to failure vs. strain rate for a) AZ31, b) AZ61 and c) AZ80 alloys tested at 175 °C, 200 °C, and 225 °C.

In Fig. 4 the deformed samples from mechanical tests are presented. Only two samples that were able to reach superplastic behavior are shown for each alloy. For these samples, two important characteristics are revealed; first for the AZ31 alloy tested at 225 °C and 10^{-3} s^{-1}, a localized neck develops and leads to failure of the material (See Fig. 4a). However, for this alloy, the neck is less pronounced in the sample tested at 225 °C and 10^{-4} s^{-1}. Second for the AZ61 alloy, a diffuse neck rather than localized is observed in almost all samples (See Fig. 4b). This behavior is also shown by AZ80 in Fig. 4c. It is important to note that in most of the cases where no evidence of macroscopic necking within the gauge lengths a true superplastic condition could be present [18]. That condition should be intrinsically related with a characteristic deformation mechanism of grain boundary sliding (GBS). It has been demonstrated by means of strain rate change tests that GBS is an active mechanism at these low temperatures and it yields to reach very high elongations in the case of the AZ61 alloy [11]. This mechanism could also be activated during tensile deformation of the AZ80 alloy in the conditions presented since similar characteristics of the samples and similar behavior in elongation to failure is observed when the alloy is subjected to tension at 225 °C and 10^{-3} s^{-1}.

Fig. 4. Deformed samples in tension tested at different strain rates and temperatures for a) AZ31, b) AZ61 and c) AZ80.

In Fig. 5, the final grain sizes of all alloys after deformation at different temperatures and strain rates are plotted (just for comparison the as-extruded grain size is indicated by a dashed line). For AZ31 alloy when the temperature increases or the strain rate decreases, the average grain size increases monotonically (see Fig. 5a). This behavior is followed by the AZ61 alloy only at 225 °C. Interestingly, at 175 and 200 °C at 10^{-2} s^{-1}, the final grain size is coarser than at 225 °C. It has been pointed out that at these conditions an incomplete recrystallization behavior is observed, in which not all the grains are able to recrystallize completely generating a bimodal microstructure with deformed grains and newly recrystallized grains with grain size of approximately 1 μm [11]. The same is found for the AZ80 alloy. It is interesting to note that at 225 °C and 10^{-3} s^{-1}, the condition in which the maximum elongation was reached, the grain size is slightly finer than the two previous alloys. This strengthens the hypothesis that recrystallization behavior is affected by the aluminum content. It is known that most industrial alloys contain more than one phase; the microstructure frequently is composed by a matrix phase and dispersed second-phase particles. If these particles are present during deformation, they will affect the microstructure development [19]. Thus, the presence of a high amount of particles of a secondary phase $Mg_{17}Al_{12}$ in AZ61 and AZ80 alloys could increase the driving pressure for recrystallization leading it to a finer microstructure after deformation.

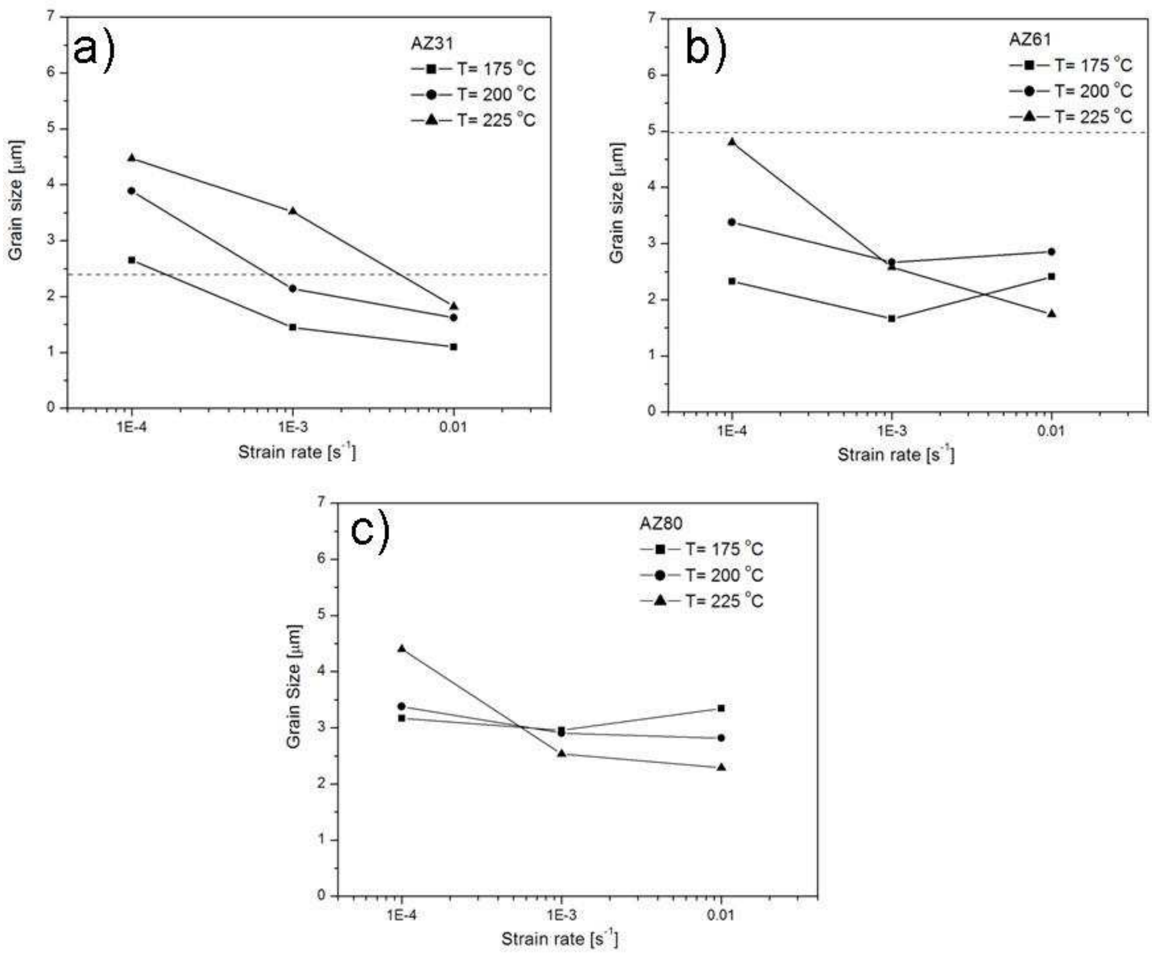

Fig. 5. Average grain size after deformation at different temperatures as a function of the strain rate a) for AZ31, b) for AZ61 and c) AZ80 alloy.

Exemplarily, the microstructures of samples deformed at 225 °C and 10^{-3} s^{-1} are presented in Fig. 6 for all alloys. Basically for AZ31 alloy, it can be seen a completely different microstructure after deformation compared to the as-extruded one. All the elongated grains disappeared during deformation and a homogeneous microstructure is found (See Fig. 6 a). In the case of AZ61 alloy a slightly finer microstructure compared to AZ31 alloy is presented (See Fig. 6b). It is interesting to note that the grains appear to be more equiaxed compared to the AZ31 alloy. In the same picture, very small particles (indicated by the arrows) were identified as precipitates of the secondary phase $Mg_{17}Al_{12}$ located mainly on the grain boundaries. For the AZ80 alloy a comparable microstructure to the AZ61 is found (See Fig. 6c). In this alloy, it is also evident the presence of particles of $Mg_{17}Al_{12}$ located on grain boundaries and also inside grains. The differences in grain sizes and morphology of the grains compared to the AZ31 alloy could be the reason why the last two alloys were able to reach such a high elongations to failure at these specific parameters. Moreover, precipitation could play an important role. It is well known that for maintaining a fine grain size during superplastic deformation, the presence of a second phase or particles at the grain boundaries are required [20]. In a separate work it has been pointed out that the size and distribution of precipitates play an important role during deformation of the AZ31 and AZ61 not only keeping a homogeneous microstructure, they could also be the reason for reducing and changing the texture distribution [21]. It is known that for materials with hexagonal close-packed structure (HCP) a low texture can improve their ductility [4, 22]. Thus, the combined effects of the grain morphology generated by DRX, the necessary amount of precipitates along grain boundaries and the reduction of the texture could enhance the superplastic behavior of the AZ61 and AZ80 alloy at 225 °C and 10^{-3} s^{-1}.

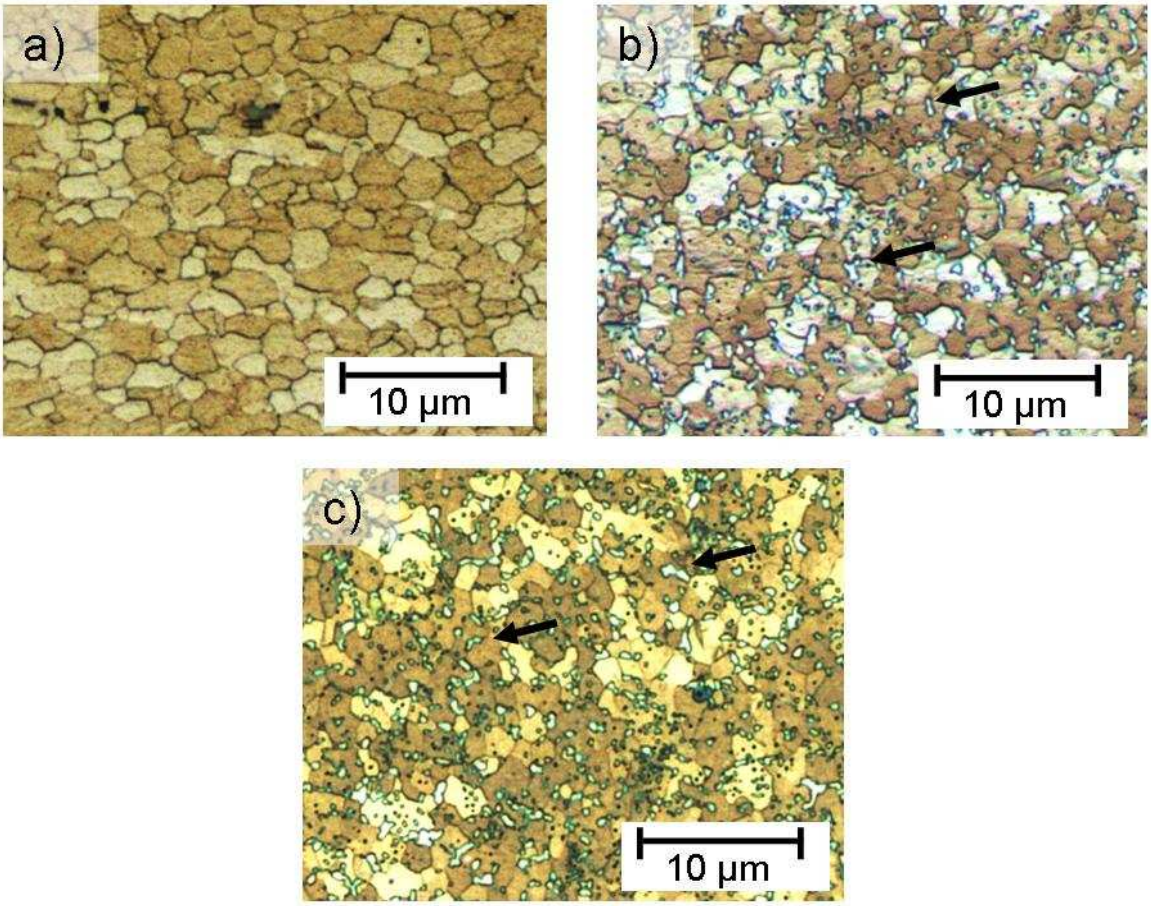

Fig. 6. Microstructures of samples deformed up to fracture at 225 °C and 10^{-3} s^{-1} for a) AZ31, b) AZ61 and c) AZ80 alloy.

Conclusions

Hydrostatic extrusion carried out at 150 ºC and high extrusion rate of 8 m/min was an effective thermomechanical treatment for refining the microstructure of the magnesium alloys AZ31, AZ61 and AZ80 all under 10 µm.
During extrusion, the effect of Al content is directly related to the recrystallization process. This can be seen for the well recrystallized microstructure with equiaxed grains with increasing the Al content.
All alloys showed superplastic behavior at low temperature: 200 °C for AZ31, 175 °C for AZ61 and 225 °C for AZ80. Interestingly, the AZ61 alloy exhibited low temperature superplasticity at only 175 °C. At this temperature the elongation to failure recorded was about 520 % tested at a strain rate of 10^{-4} s^{-1}. The AZ61 alloy reached the maximum elongation of all alloys.
As a result of low temperature dynamic recrystallization, significant work softening was observed during the tests. The microstructure produced by DRX was equiaxed and homogeneous that was retained up to the end of the tests. This could enhance GBS as the predominant mechanism to reach the high elongations recorded.
There is a processing window where complete recrystallization took place and where the maximum elongations were reached:
For AZ31 alloy: T= 200 – 225 ºC, strain rate from $1x10^{-4}$ to $1x10^{-3}$ s^{-1}, true strain from 1.1 to 1.6
For the AZ61 alloy: T= 200 – 225 ºC, strain rate from $1x10^{-4}$ to $1x10^{-3}$ s^{-1}, true strain from 1.4 to 1.8
For the AZ80 alloy: T= 225 ºC, strain rate from $1x10^{-4}$ to $1x10^{-3}$ s^{-1}, true strain from 1.2 to 1.6

References

[1] M.Y. Zheng, K. Wu, M. Liang, S. Kamados, Y. Kojima, Mater. Sci. Eng. A 327 (2004) 66

[2] P. Panicker, A.H. Chokshi, R. K. Mishra, R. Verma and P.E. Krajewski, Acta Mater. 57(2009) 3683-3693

[3] W.J. Kim, J.D. Park, U.S. Yoon, J. Alloys and Compounds, 464 (2008) 197-204

[4] H. Watanabe, M. Fukusumi, Mater. Sci. Eng. A 477 (2008) 153-161

[5] W.J. Kim, M.J. Kim and J.Y. Wang, Mater. Sci. Eng. A 527 (2009) 322-327

[6] M. Mabuchi, K. Ameyama, H. Iwasaky and K. Higashi, Acta Mater. 47 (1999) 2047-2057

[7] H. Watanabe, T Mukai, K. Ishikawa, M. Mabuchi and K. Higashi, Mater. Sci. Eng. A 307(2001) 119-128

[8] J. Swiosteck, J. Goken, D. Letzig and K.U. Kainer, Mater. Sci. Eng. A 424 (2006) 223-229

[9] J. Goken, J. Swiosteck, D. Letzig and K.U. Kainer, Mater. Sci. Forum. 482 (2005) 387-390

[10] E. Meza Garcia, Extrusion of Magnesium-Zn based alloys, Edit. Suedwestdeutscher Verlag fuer Hochschulschriften (2011)

[11] J. Victoria-Hernandez, D. Hernandez-Silva, S.B. Yi, D. Letzig and J. Bohlen, Mater. Sci. Eng. A 530 (2011) 411-417

[12] J. Swiostek, J. Bohlen, D. Letzig and K.U. Kainer, Mater. Sci. Forum, vol. 488-489 (2005) 491-494]

[13] J. Liu, Z. Cui and C. Li, Comput. Mater. Sci. (2007), doi:10.1016/j.commatsci.2007.04.024,

[14] A. Galiyev, R. Kaibyshev and G. Gottstein, Acta mater. 49 (2001) 1199–1207

[15] J.C. Tan, M.J. Tan, Mater. Sci. Eng. A339 (2003) 81-89

[16] C.J. Lee, J.C. Huang, Acta Mater. 52 (2004) 3111–3122

[17] W. J. Kim, S. W. Chung, C. S. Chung and D. Kum, Acta Mater. 49 (2001) 3337–3345

[18] T.G. Langdon, Met. Sci. 16 (1982) 175–183

[19] F.J. Humphreys, M. Hatherly, Recrystallization and related annealing phenomena, second edition, Elsevier, 2004, pp. 48, 49

[20] T.G. Nieh, J. Wadsworth and O.D. Sherby, Superplasticity in metals and ceramics, Cambridge University Press, 1997, p. 23

[21] J. Victoria-Hernandez, D. Hernandez-Silva, S.B. Yi, D. Letzig and J. Bohlen, to be submitted

[22] J. Bohlen, M. R. Nürnberg, J. W. Senn, D. Letzig, S. R. Agnew, Acta Mater. 55 (2007) 2101-2112