

COMPARATIVE STUDY OF HIGH TEMPERATURE WORKABILITY OF ZM21 AND AZ31 MAGNESIUM ALLOYS

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Abstract

High temperature regime, 300-450°C for Mg-Al-Zn alloys, is currently used in primary processing, such as rolling and extrusion, as well as for secondary operation like forging. The knowledge of temperature and strain rate proper combination (processing window) as well as the microstructure evolution occurring during hot deformation clarifies the relationships between forming variables and final properties of components. Numerous data on AZ31 and few other Mg-Al alloys, produced by laboratory testing, are available in the scientific and technical literature. The ZM21, Mg-2Zn-1Mn, by contrast, is characterized by absolute lack of scientific data. In the alloy the addition of manganese, by suppressing the formation of beta phase, increases the solidus temperature that results in the larger processing window than in AZ31. The benefit requires extensive analysis aimed at optimizing the deformation variables that affect the microstructure refinement under dynamic and static recrystallization. The high-temperature plastic deformation and the microstructure evolution of the ZM21 were thus investigated in the temperature range between 200 and 500°C and results were analysed and compared with those of a conventional heat-treated AZ31.

Riassunto

Il regime delle alte temperature 300-450°C, è comunemente usato per le operazioni primarie di deformazione plastica, come la laminazione e l'estrusione, come pure per quelle secondarie come la forgiatura o lo stampaggio delle leghe Mg-Al-Zn. La conoscenza della combinazione adatta temperatura-velocità di deformazione (finestra di processo) e della evoluzione della microstruttura fa luce sulle relazioni tra variabili di deformazione e proprietà dei componenti. Numerosi dati, ottenuti con prove di compressione o torsione, sono disponibili sulla lega AZ31 e su pochi altre leghe della classe Mg-Al. Sulla lega ZM21 (Mg-2Zn-1Mn) non sono invece disponibili dati scientifici. Nella lega l'aggiunta del Mn sopprime la formazione della fase beta ed aumenta la temperatura del solidus, producendo una finestra di processo più ampia di quella dell'AZ31. Questi aspetti richiedono analisi approfondite sono rivolte all'ottimizzazione delle variabili di deformazione che poi governano l'affinamento della microstruttura per effetto della ricristallizzazione sia dinamica che statica. La deformazione plastica ad alta temperatura e l'evoluzione della microstruttura della lega ZM21 sono state analizzate nell'intervallo di temperatura 200-500°C e i risultati sono confrontati con quelli di una lega convenzionale trattata termicamente.

1. INTRODUCTION

Lightweight constructions are becoming more and more important for the automotive industries due to increase of fuel prices and legislative requirements like CAFÉ in the US, and the directive or the control of CO₂ emissions in the EU [1,2]. Magnesium alloys for their unique combination of light weight, high specific strength and stiffness and high recycling capability are candidate to replace steel, iron and, in some cases, also aluminum parts. The growing interest in magnesium alloys is expected to increase in the next years [3]. The use of components manufactured via die-, sand-, mould- and rheo-casting is fairly well introduced in a wide number of applications which are ranging from steering wheels, instrumental panels, gear box housing and even to hybrid engine blocks. The scenario for the automotive industry is that the use of magnesium alloys for many components, mainly as castings, is further expanding in the short term, whereas the application in body and chassis components, including sheet and extrusions, is anticipated in the medium term [4].

A driving force for the introduction of wrought alloys is their property enhancement, since casting alloys are affected by porosity, which would be of particular importance for structural and crash-relevant parts. Initial applications for this category are expected for extrusions and forgings, to be followed by sheets. These requirements may address researches towards the modification of known alloys as well as the development of new ones aiming at improving both mechanical performances, also by ageing (T6), and high temperature stability. The growing interest in use of magnesium alloys is expected to increase in the next years, and to be sustained by research [5]. From recent magnesium alloys conferences [6], the following items stand out as limiting or retarding factors for the use of wrought alloys: 1) poor cold forming properties; 2) limited number of wrought alloys and lack of processing data. As far as the item 1) is concerned, it is important to note that magnesium alloys are difficult to work at low

temperature (<200°C); moreover, due to hcp structure and inactiveness of non basal slip systems, deformation is accommodated by twinning [7]. Many authors have investigated the magnesium and its alloys slip systems and their temperature dependence. The results indicate that the alloys have poor ductility and shape formability near ambient temperature [7,8] and that at room temperature critical resolved shear stress (CRSS) for basal a dislocation glide is approximately two orders of magnitude smaller than that for pyramidal dislocation glide. This gives rise to preferential basal a dislocation glide and to high anisotropic deformation behavior. However, since the basal a dislocation glide cannot provide five independent slip systems, other deformation mechanisms are necessary. A new technique that may be considered a breaking with conventional rolling technology, based on high speed heavy rolling, has been proposed by Utsunomiya et al. [9]. The AZ31 showed excellent workability when processed with high-speed rolling; at 200°C and 2000m/min, the sheet rolled by 44% in one pass, had equiaxed structure with grains size 2.2 microns.

The second problem is the limited number of alloys. While magnesium cast products have achieved a break-through into industrial lightweight applications, the development is now focusing on wrought products, mainly belonging to the Mg-Al family. This leads to a broader variety of shapes as well as to improved material properties of magnesium but faces limited knowledge on the processability as well as a limited number of commercial wrought alloys. In the Conference "Magnesium Technology 2006" [6], the Wrought Alloys sections had 18 papers dealing with the AZ31; the AZ61, AZ80, AZ90 and AM50 had each two papers. While the technology for processing magnesium alloys is available today, the industrial use is not well established because of the technical and economical limitations of both the direct, and indirect, extrusion processes. In the framework of the 6 EC research program, the major European car producers compared the behaviour of AZ31 with ZM21 and two modified alloys. The ZM21 showed very high performances in hydrostatic extrusion [10]. On the other hand, there is a lack of basic data on the influence of processing parameters and alloy compositions during high-temperature deformation. This indicates a high demand for research and development along the complete chain of production of magnesium structural components. The hydrostatic extrusion process, for example, could be used to broaden the processing window for magnesium alloys and to pave the way for the manufacturing of lightweight, cost effective, magnesium structures. An objective is also to conduct research on suitable alloy compositions, to obtain a better understanding of the process variables for the production of magnesium structural components with improved formability and performance of the alloy. In the case of ZM21, higher extrusion speeds of hollow profiles, up to 120 m/min, were possible [10]. The present study aims at investigating the hot formability of an extruded ZM21 alloy and comparing its behaviour with the one previously observed in extruded conventional AZ31.

EXPERIMENTAL

The AZ31 experimental result data belong to the authors' wide database obtained in the last 3 years by testing a batch of AZ31 with different initial microstructure: ingot, extruded, heat treated, overaged. These investigations aimed at clarifying the microstructure features that significantly affect the high temperature deformation of the alloy; the data were partly published [11,12] and used to identify the processing window (strain rate and temperature conditions) that is successfully used for industrial forming operations. Warm forming experiments were also carried out to assess the AZ31 sheet formability [13]. The present data were obtained by testing the specimens machined from rolled AZ31 heat treated at 385°C for 14 h, at the Pohang University.

The ZM21 alloy was produced and extruded by Alubin, Israel. The specimens for torsion testing, with gauge radius R, and length L, 5 and 10 mm respectively, were machined from extruded rods. Torsion tests were carried out in air on a computer-controlled torsion machine, under equivalent strain rates ranging from 10^{-3} to 5 s^{-1} and temperatures from 200°C to 400°C. Specimens were heated 1°C/s by induction coils and maintained 5 minutes before torsion to stabilize the testing temperature that was measured by thermocouple in contact with the gauge section. Specimens just after the fracture were rapidly quenched with water jets to avoid microstructure

modifications during slow cooling.

The von Mises equivalent stress, σ , and equivalent strain, ε , were calculated using the relationships:

$$\sigma = \frac{\sqrt{3} M}{2 \pi R^3} (3 + m' + n') \quad 1)$$

$$\varepsilon = \frac{2 \pi N R}{\sqrt{3} L} \quad 2)$$

where N is the number of revolutions, M is the torque, m' (strain rate sensitivity coefficient) at constant strain is $\partial \log M / \partial \log \dot{N}$, and n' (strain hardening coefficient) at constant strain rate is $\partial \log M / \partial \log N$; at the peak stress, clearly $n'=0$. To reveal the microstructure, longitudinal sections at the periphery of the sample gauge, i.e. in the point where the strain and strain rates assumed the values calculated by equations 1) and 2), were investigated by means of optical microscopy.

RESULTS AND DISCUSSION

The initial microstructure of the alloys before testing is displayed in Figure 1. The heat-treated AZ31 exhibited an equiaxed and fine microstructure. The microstructure of the extruded ZM21 is composed by duplex grain size, the small and the large grains were 20 and 60 μm respectively. Figures 2 and 3 show typical equivalent stress vs. equivalent strain curves obtained by testing at 200 and 400°C the rolled and heat treated AZ31 and the extruded ZM21, respectively. The flow curves shape of the rolled AZ31 does not differ appreciably from that observed in the cast [11,12]. At

200°C, under all strain rates, the flow curves of both alloys exhibit well defined σ_y , ~60 MPa for AZ31 and ~20 MPa for ZM21, a continuous increase of work hardening up to σ_{\max} , which is larger in AZ31, followed by fracture that in ZM21 occurs at larger strain. At 400°C, AZ31 the flow curve, after yielding and very limited work-hardening, gets a steady state region, typical behaviour of dynamic recovery (DRV), up to fracture. The ZM21 flow curves display yielding

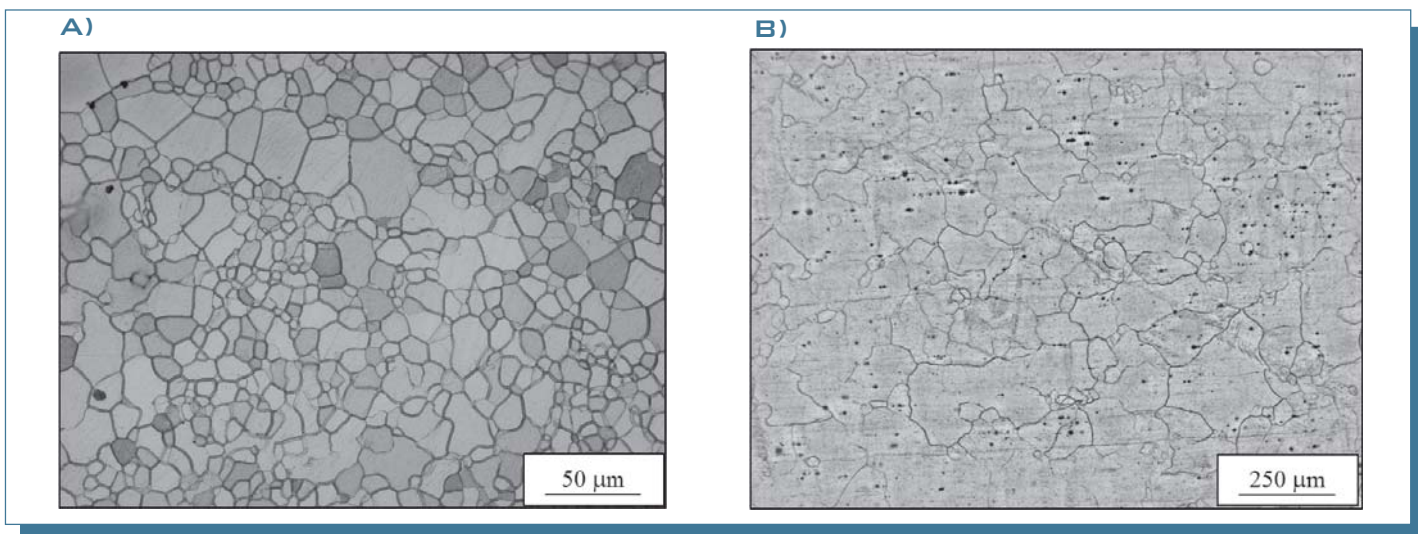


Fig. 1: Microstructure of the rolled and heat-treated AZ31 (a) and of the extruded ZM21 (b).

and rapid hardening up to σ peaks located at $\epsilon \sim 0.7$, irrespective of the different strain rates, followed by a continuous softening towards the fracture that occurs at ϵ larger than in AZ31; at 400°C and strain rate 0.05 s⁻¹ the maximum strain value is 2.5. The flow curve exhibiting a peak followed by softening in stationary state is indicative of dynamic recrystallization (DRX) that nucleates in proximity of the peak producing new stable grains whose size controls the softening and the σ of stationary state.

In the present instance, the absence of stationary state and the decrease of σ up to fracture may be indicative of three processes promoting the continuous softening: (i) the onset of DRX around the peak, producing new small grains, (ii) the fast new-grain growth at the critical size for (iii) crack nucleation and growth.

A significant decrease in the strain to failure in torsion can be observed (Figure 4), in particular in the high-temperature region; an additional difference is the observation of a well defined yielding in ZM21, (Figure 3) i.e. of the presence of a short strain range without load increase, an effect

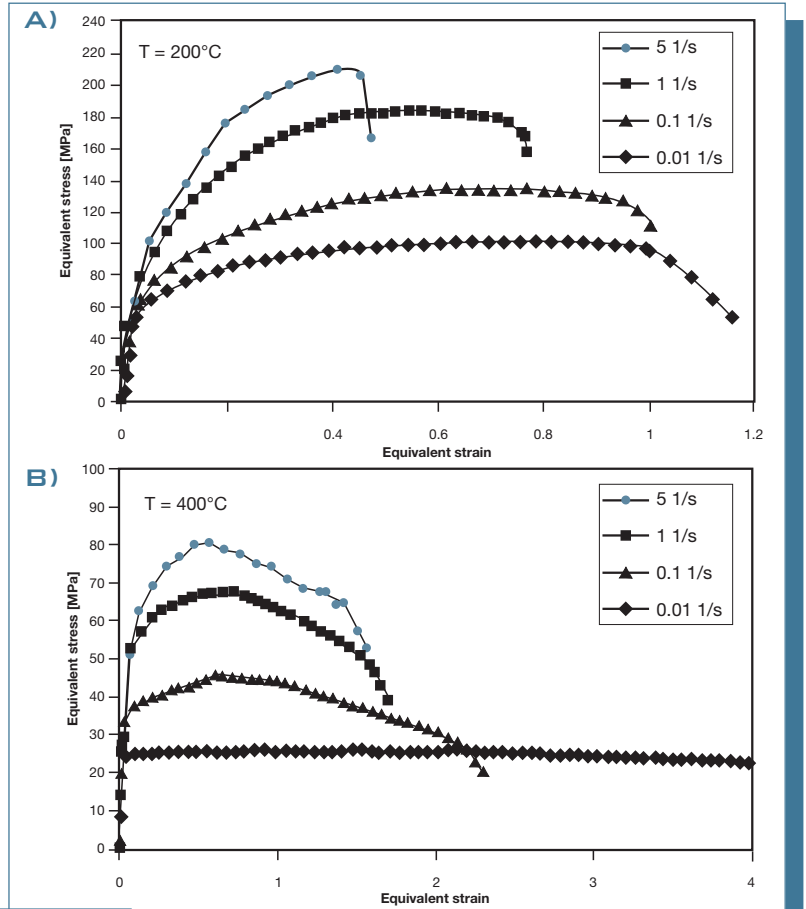


Fig. 2: Equivalent stress vs. equivalent strain curves obtained in torsion for AZ31 at 200 (a) and 400°C (b).

probably due to a sudden increase in the number of dislocations unpinned from their solute atmosphere (either Zn or Mn).

The temperature and strain rate dependence of the peak stress are shown in Figure 5. The experimental data are well described by the equation:

$$\dot{\epsilon} = A [\sinh(\alpha\sigma)]^n \exp(-Q/RT) \quad (3)$$

where A and α are material parameters, n is a constant, R is the gas constant, T is the absolute temperature, and Q is the apparent activation energy for high-temperature deformation process. In the AZ31 a best-fitting procedure gave $\alpha = 0.017 \text{ MPa}^{-1}$ and $n = 4.4$ for temperatures between 200°C and 400°C. The activation energy, calculated by plotting $\sinh(\alpha\sigma)$ as a function of $1/T$, was $Q = 140 \text{ kJ/mol}$. The value is relatively close to the one of self-diffusion of Mg (135 kJ/mol) or for diffusion of Al in Mg (143 kJ/mol). In the case of the ZM21, α was close to 0.049 MPa^{-1} , and n ranged between 2.6 and 4.4 (average $n = 3.3$). The activation energy for high temperature deformation was close to 200 kJ/mol, higher than in the case of AZ31.

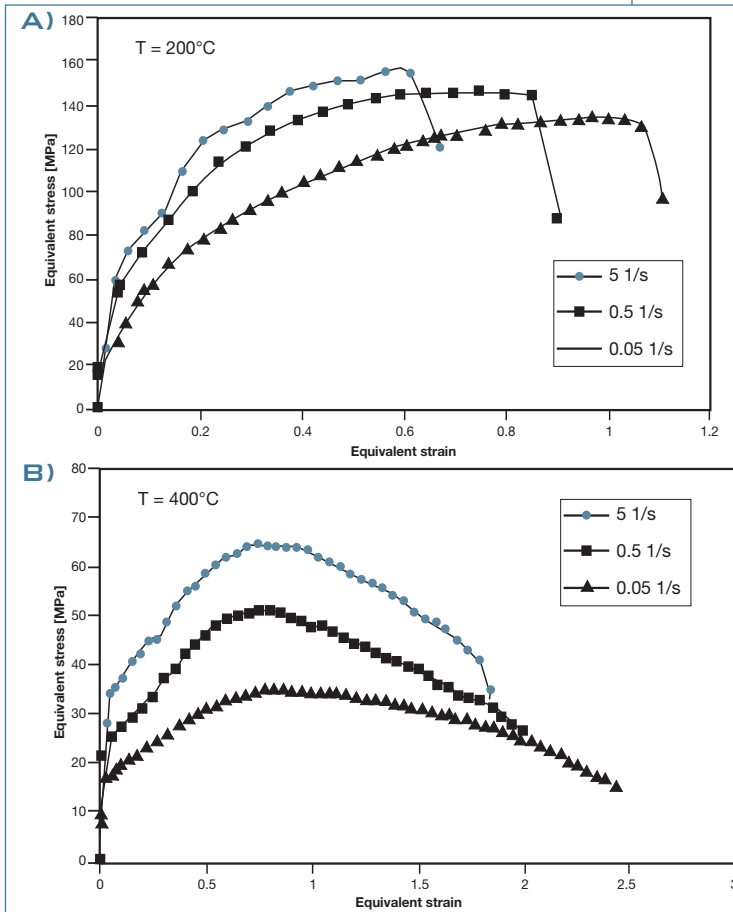


Fig. 3: Equivalent stress vs. equivalent strain curves obtained in torsion for ZM21 at 200 (a) and 400°C (b).

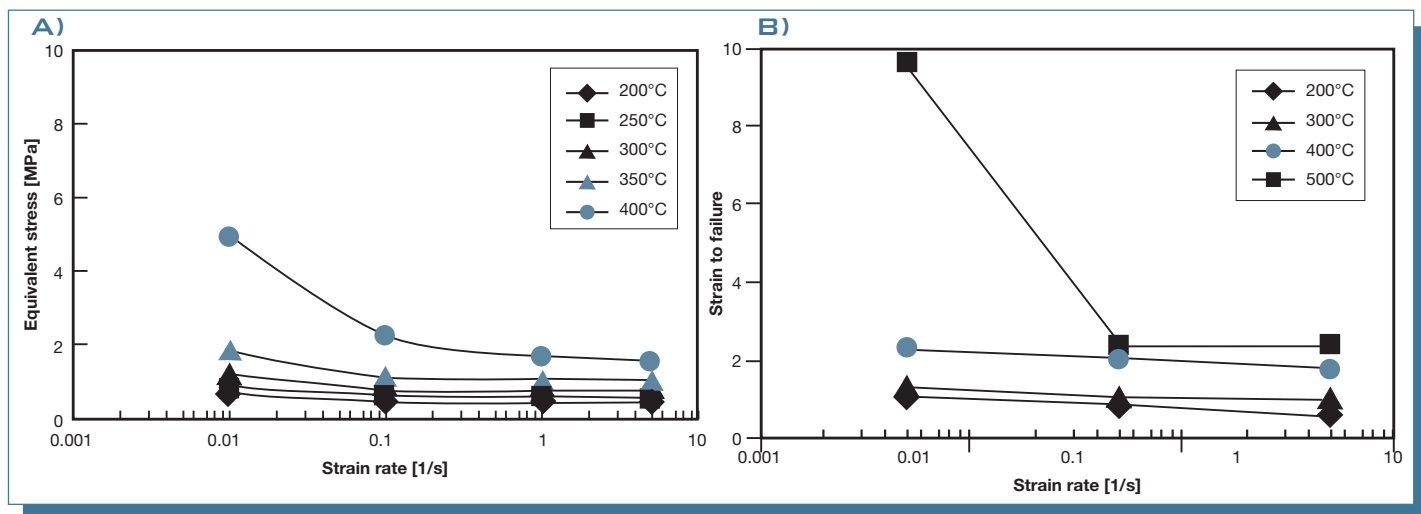


Fig. 4: Equivalent strain to failure for AZ31 (a) and ZM21 (b).

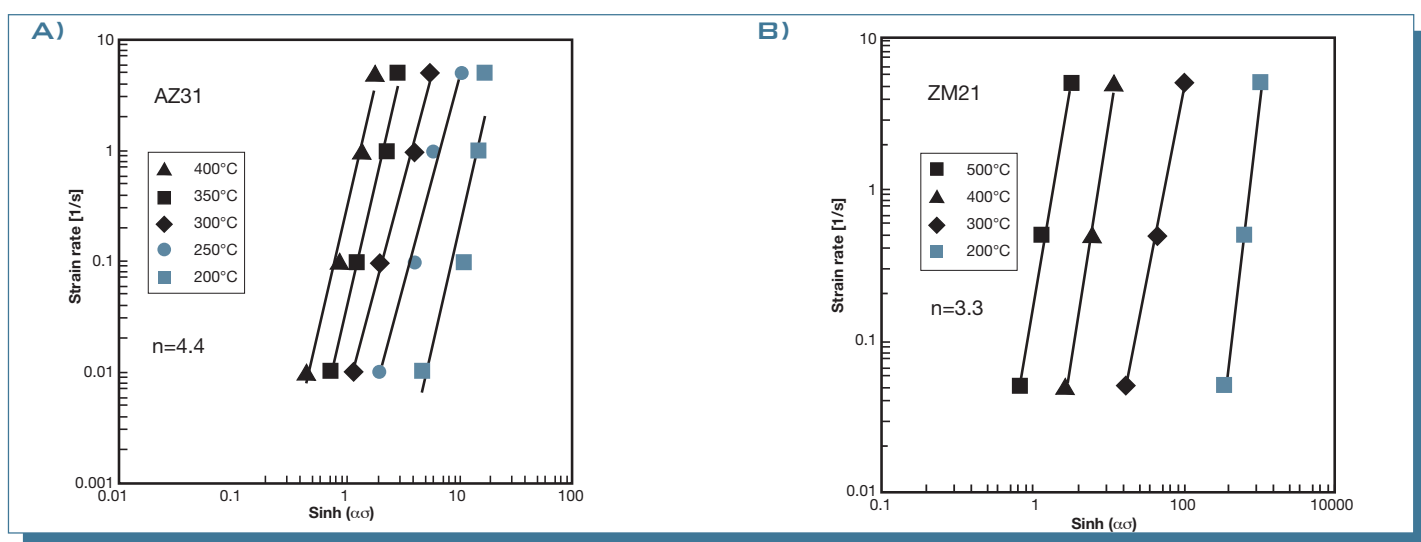


Fig. 5: Peak flow stress dependence on applied stress as a function of temperature for AZ31 (a) and ZM21 (b).

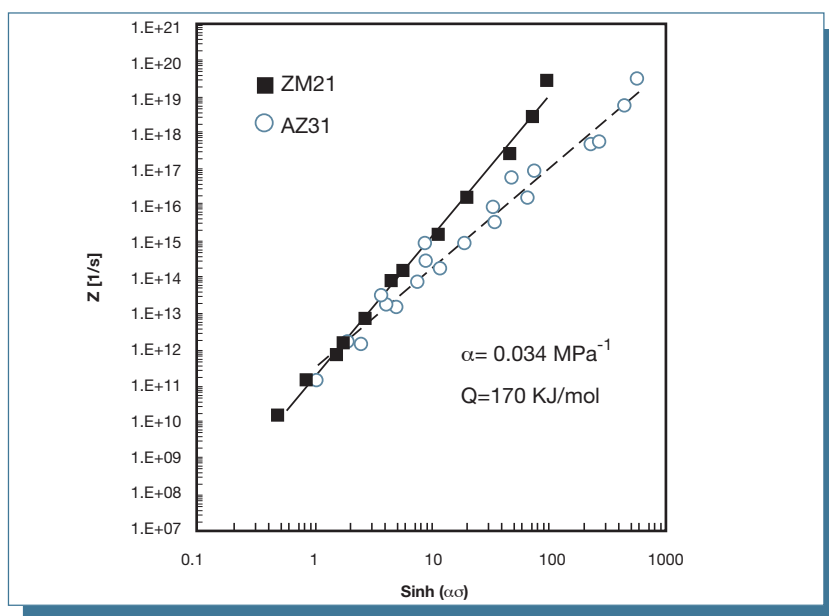


Fig. 6: Zener-Hollomon parameter as a function of peak-flow stress for AZ31 and ZM21.

Figure 6 plots the Zener-Hollomon parameter, Z , defined as

$$Z = \dot{\epsilon} \exp(Q/RT) = [\sinh(\alpha\sigma)]^n \quad 4)$$

as a function of peak stress, with $Q = 170 \text{ kJ/mol}$ and $\alpha = 0.034 \text{ MPa}^{-1}$ (mean values of those obtained experimentally for AZ31 and ZM21). Analysis of Figure 6 clearly demonstrates that the peak flow stresses are higher in the AZ31 than in the ZM21. Figure 7 shows typical microstructure of the AZ31 quenched after torsion testing. At 200°C , and a strain rate of 10^{-2} s^{-1} , the structure is mostly composed by slightly elongated grains (Figure 7a), with very fine recrystallized grains on several grain boundaries. At 300°C , under the same strain rate, the recrystallized fraction is substantially higher than at the lower temperature, and the average size of the recrystallized grains is larger (Figure

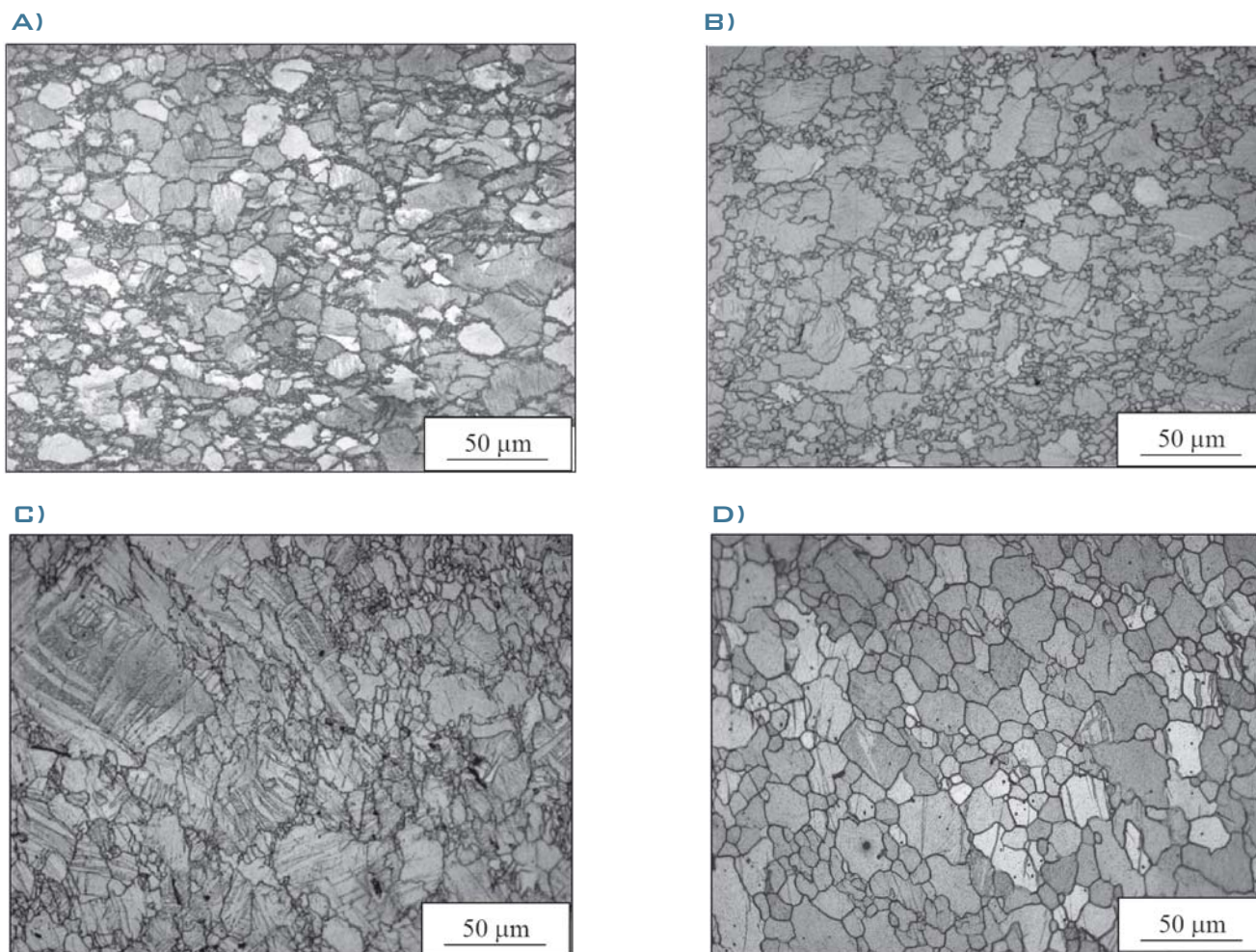


Fig. 7: Microstructure of the AZ31 tested at: 200°C- 10^{-2} s^{-1} (a) 300°C- 10^{-2} s^{-1} (b) and 400°C- 1 s^{-1} (c); (d) 400°C- 10^{-3} s^{-1} .

7b). Fully recrystallization and localised grain growth at 400°C, in the high strain rate range, resulted in the presence of some large grains heavily deformed by twinning, albeit a relatively large fraction of the structure is composed by fine equiaxed grains. Under lower strain rate, the structure is composed by a homogeneous distribution of relatively coarse equiaxed grains (Figure 7d).

The mechanism of dynamic recrystallization (DRX) has been analysed by McQueen and Konopleva [15]; twinning in fact takes place before the other mechanisms, except basal glide that occurs in favourably oriented grains. At low strains, twinning favourably reorients grains for slip. As deformation reaches a sufficiently high value, DRX starts where high misorientations have been created by accumulation of dislocations, i.e. where slip has occurred on several slip system (near grain boundaries and twins). The new fine grains form a mantle (or a “necklace”) along grain boundaries

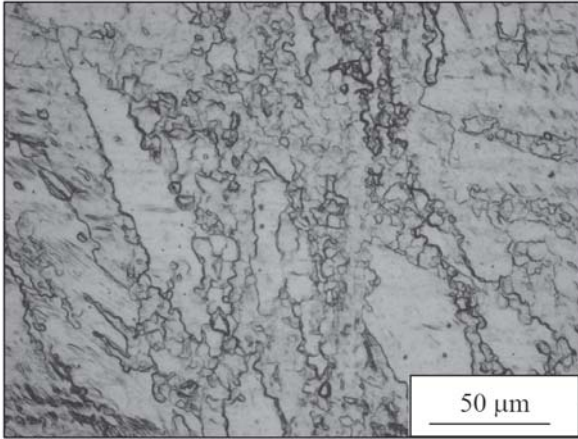
and deform more easily than the grain core, thus repeatedly undergoing recrystallization [12].

Figure 8 shows typical micrographs of the microstructure of ZM21 tested in torsion. The structure, even at 300°C, is largely unrecrystallized, with elongated grains; at 400°C, the grains are mostly equiaxed, but elongated structures still persist. The strong decrease in flow stress after the peak should be attributed to the late onset (at strain close to $\epsilon=0.7-0.8$) of recrystallization and coarsening phenomena, that in this alloy exhibit quite different kinetics than in the case of the AZ31. Only at the highest temperature (500°C), the microstructure underwent complete recrystallization and grain growth, a process that resulted in the presence of coarse (50 μm) grains.

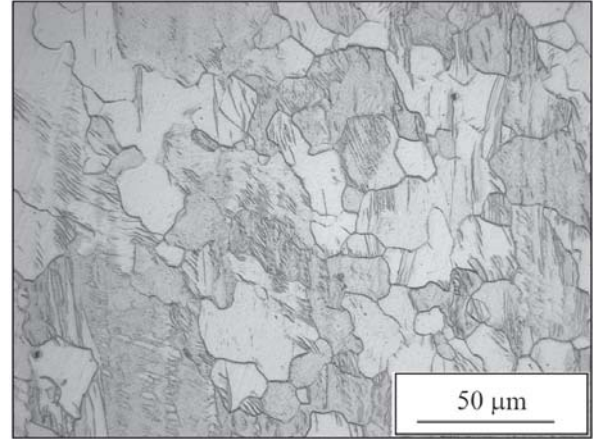
The above analysis gives some interesting preliminary indications on the high-temperature formability of the extruded ZM21 alloy. In particular:

1. the intrinsic ductility, i.e. the equivalent failure strain in torsion, is lower in ZM21 than in AZ31;
2. the fraction of recrystallized structure, at all the investigated temperatures, is lower in ZM21 than in AZ31;
3. the maximum equivalent stress, i.e. the working load, is higher in AZ31 than in ZM21; yielding has been observed in ZM21, thus suggesting a solute strengthening role played by either Zn or Mn;

A)



B)



C)

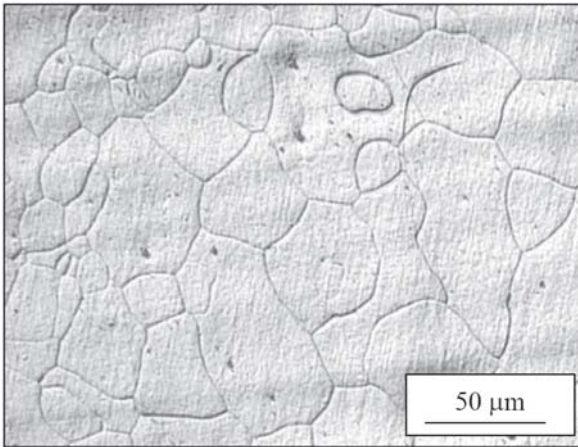


Fig. 8: Microstructure of the ZM21 tested at: 300°C-5 s⁻¹ (a) 400°C- 5x10⁻² s⁻¹ (b) and 500°C- 5 s⁻¹ (c).

4. the different composition of the ZM21 enables the use of higher working temperature, up to 500°C, i.e. well above the upper limit allowed for AZ31. On the other hand, even though strain to failure is relatively high at 500°C, the microstructure undergoes extensive grain

growth, that precludes the fine grain size that has been observed to be a prerequisite for optimum sheet formability.

CONCLUSIONS

The high temperature workability of the extruded ZM21 alloy has been investigated by torsion tests between 200 and 500°C. Both mechanical and microstructural results were compared with those observed in a rolled AZ31 alloy; the comparison indicated that the strain to fracture in torsion were significantly lower in ZM21 than in AZ31, even though in general in the former alloy the ductility exceeded the minimum allowable value of $\epsilon=1$. These differences in mechanical response were attributed to a lower tendency to dynamic recrystallization; while in rolled AZ31 early dynamic recrystallization was observed at temperatures as low as 200°C, microstructural observations clearly suggested that in the extruded ZM21 the lower limit for DRX was shifted toward significantly higher temperatures.

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REFERENCES

- 1) CAFÉ, <http://www.nhtsa.dot.gov>
- 2) Communication from the Commission to the Council and European Parliament: Implementing the Community strategy to reduce CO₂ emissions from cars. Third annual report (reporting year 2001), COM (2002) 693.
- 3) EU Directive on the End of Life of Vehicles, 2000/53/EC.
- 4) S. Schumann, Mat. Sci. Forum 488-489 (2005), 1-8.
- 5) Ya Zhang, X. Zeng, C. Lu, W. Ding, Y. Zhu, Mat. Sci. Forum, 488-489, (2005), 123.
- 6) Magnesium Technology 2006, A.A. Luo et al, eds. TMS, 2006.
- 7) F.J. Humphreys, M. Hatherly: Recrystallization and related annealing phenomena. Pergamon Press, Oxford, 1996.
- 8) J. Koike, Mat. Sci. Forum, 419-422 (2003), 199.
- 9) H. Utsunomiya, T. Sakai, S. Minamiguchi, H. Koh, Magnesium Technology 2006, A.A. Luo et al. eds. TMS, 2006, 201.
- 10) J. Bohlen, J. Swoistek, W.H. Sillekens, P.J. Vet, D. Letzig, K.U. Kainer, Magnesium Technology 2005, TMS, 2005, 241.
- 11) S. Spigarelli, M. El Mehtedi, E. Evangelista, J. Kaneko, Metall. Sci. Techn. 23 (2005) 11-17.
- 12) S. Spigarelli, M. El Mehtedi, M. Cabibbo, E. Evangelista, J. Kaneko, A. Jäger, V. Gartnerova, Mater. Sci. Eng. A 462 (2007), 197.
- 13) A. Forcellese, M. El Mehtedi, M. Simoncini, S. Spigarelli, Key Engineering Materials 344 (2007) 31.
- 14) S. Spigarelli, E. Evangelista, M. El Mehtedi, L. Balloni, Mechanical And Microstructural Aspects Of High Temperature Formability Of AZ31 Sheets, Proceeding 10 ESAFORM 2007, Zaragoza, Spain, 1305.
- 15) H.J. McQueen, E.V. Konopleva, in Magnesium Technology 2001, J. Hryn ed., TMS, 2001, p. 227.