HIGH TEMPERATURE PLASTIC DEFORMATION OF A HEAT-TREATED AZ31 MAGNESIUM ALLOY

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Abstract

High-temperature plastic deformation and dynamic recrystallization were investigated in an extruded and heat-treated AZ31alloy in the temperature range between 200 and 400°C. High-temperature straining resulted in partial dynamic recrystallization above 250°C; at 400°C recrystallization was complete and a moderate grain growth was observed.The peak flow stress dependence on temperature and strain rate was described by means of the conventional sinh equation; calculation of the activation energy for high temperature in the whole range of temperature deformation gave Q=155 kJ/mol, i.e. a value that is reasonably close to, but greater than, the activation energy for self-diffusion in Mg. When the data obtained at the lowest temperature were excluded from the calculation, the activation energy increased to 180 kJ/mol.This difference in the activation energy value can be explained by the occurrence of dynamic recrystallization in the high-temperature regime; this observation was substantially confirmed by the plots of strain-hardening rate as a function of stress that were used to identify the onset of dynamic recrystallization.

Riassunto

La formabilità ad alta temperatura della lega di magnesio AZ31 è stata studiata effettuando prove di torsione a temperature comprese fra 200 e 400°C. Le curve tensione-deformazione equivalente mostravano un andamento crescente fino ad un picco, seguito da una diminuzione della sollecitazione fino a rottura. La tensione di picco ricavata da ogni curva è stata messa in relazione con la temperatura e la velocità di deformazione ottenendo un'energia di attivazione di circa 155 kJ/mol; questo valore dell'energia di attivazione si innalzava però fino a 180 kJ/mol quando l'analisi si restringeva alle prove condotte a temperature comprese fra 250 e 400°C. L'analisi microstrutturale dimostrava che a 200 e 250°C la deformazione produceva una struttura fortemente deformata e ricca di geminati; a 250°C si osservavano inoltre finissimi grani ricristallizati lungo il bordo dei grani deformati. Innalzando la temperatura di prova a 300°C si otteneva un consistente aumento della frazione ricristallizzata e a 350°C la microstruttura era quasi completamente ricristallizzata. A 400°C si osservava una struttura equiassica completamente ricristallizzata, che aveva subito un ingrossamento del grano.

Al fine di valutare l'effetto della ricristallizzazione dinamica sul valore dell'energia di attivazione, dalle curve tensione-deformazione sono state ricavate quelle relative al coefficiente di "strain hardening"; questo tipo di analisi costitutiva ha permesso di dimostrare che in assenza di ricristallizzazione la deformazione del materiale sarebbe completamente controllata dal movimento delle dislocazioni in atmosfere di atomi di Al in soluzione solida. L'instaurarsi dei fenomeni di ricristallizzazione dinamica produceva un addolcimento del materiale e determinava quindi un aumento dell'energia apparente di attivazione del processo di deformazione plastica.

INTRODUCTION

In recent years plastic deformation of magnesium alloys has been widely investigated to overcome the problem of their inherent low ductility at low temperature due to the difficulty of the dislocation activity in non-basal slip systems. Indeed, in magnesium the critical resolved shear stress is much greater for non-basal than for basal slip at room temperature, but this difference decreases with increasing temperature [1,2]. As a result, the ductility of Mg and its alloys increases quite dramatically with increasing temperature; high-temperature forming is thus especially important either as a primary (rolling, extrusion) and as a secondary (forging) process. Albeit the vast majority of commercial Mg alloys is usually produced by casting, the AZ31 (Mg-3Al-1Zn) alloy is well suited for sheet production by rolling. Indeed, rolling is the most promising process for mass production of large bulk sheets and plates and offers the additional advantage that proper control of rolling conditions allows to produce a fine grain size due to dynamic recrystallization (DRX).

Early studies of AZ31 [3] focused on the microstructural development occurring during straining and revealed that dynamic DRX initiated at about 300°C. Later studies analysed the influence of various parameters, like texture and deformation conditions [4,5] and grain size [6,7]. DRX can also be employed to produce AZ31 sheets suitable of superplastic forming operations [8,9], even though coarse-grained materials processed in a proper window of temperature and strain rates also exhibit considerable ductility [10].

The aim of the present work was to investigate the hot formability of an AZ31 magnesium alloy in the temperature range between 200 to 400°C in terms of flow stress and microstructure of the deformed material.

EXPERIMENTAL PROCEDURE

The extruded AZ31 (Mg-3Al-1Zn) alloy investigated in the present study was heat-treated at 500°C for 2 h in order to achieve a fully equiaxed grain structure. Specimens for torsion testing, with gauge radius, R, and length, L, 5 and 20 mm respectively, were machined from extruded rods. Torsion tests were carried out in air on a computer-controlled torsion machine following a matrix of surface equivalent strain rates: $5x10^{-2}$, $5x10^{-1}$, 5 s^{-1} and temperatures from 100 to 400°C. Specimens were heated 1°C/s by induction coil and maintained 5 minutes to stabilize them at the testing temperature before torsion. Temperature was measured by means of thermocouple in contact with the gauge section. Tests have been performed in air and specimens just after the fracture were rapidly quenched with water jets to avoid microstructure modifications during slow cooling.

The von Mises equivalent stress, s, and equivalent strain, e, were calculated using the relationships:

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

$$\varepsilon = \frac{2\pi N R}{\sqrt{3}\,L} \qquad (2)$$

where N is the number of revolutions, M is the torque, m' (strain rate sensitivity coefficient) at constant strain is $\frac{\partial \log M}{\partial \log \dot{N}}$, and n' (strain hardening coefficient) at constant strain rate is $\frac{\partial \log M}{\partial \log \dot{N}}$. At the peak stress, clearly n'=0.

To reveal the microstructure, longitudinal sections at the periphery of the sample gauge length, i.e. in the point where the strain and strain rates assumed the values calculated by eqns. 1-2, were observed by means of optical microscopy.

RESULTS

The microstructure of the alloy after extrusion and heat treatment at 500°C for 2 h, is illustrated in Figure 1. Extrusion produced a bimodal distribution of very fine recrystallized and large elongated grains (Figure 1a); after heat treatment, a fully homogeneous equiaxed microstructure was produced (Figure 1b).

The equivalent stress vs. equivalent strain flow curves of the alloy at different deformation temperatures and strain rates are reported in Figure 2. Typically, the flow stress increases to a maximum and then decreases to final rupture. The strain to fracture is much greater than ε=1 only at the highest temperature and under the lowest strain rate. The temperature and strain rate dependence of the peak stress are shown in Figure 3. The experimental data are well described by the usual equation:

$$\dot{\varepsilon} = A[\sinh(\alpha\sigma)]^n \exp(-Q/RT) \qquad (1)$$

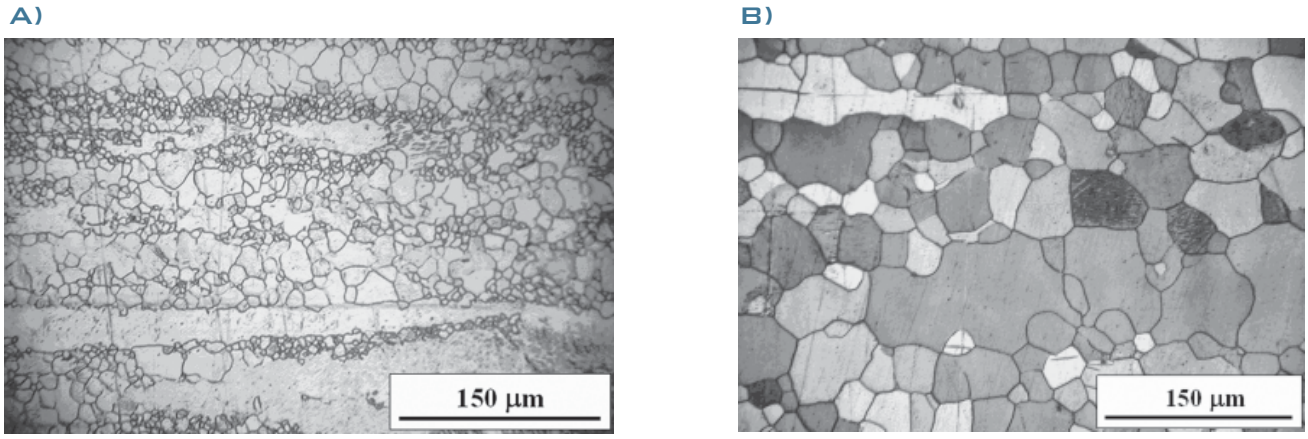

Fig. 1: Optical micrographs of the as-extruded (a) and heat-treated (500°C-2 h) alloy (b).

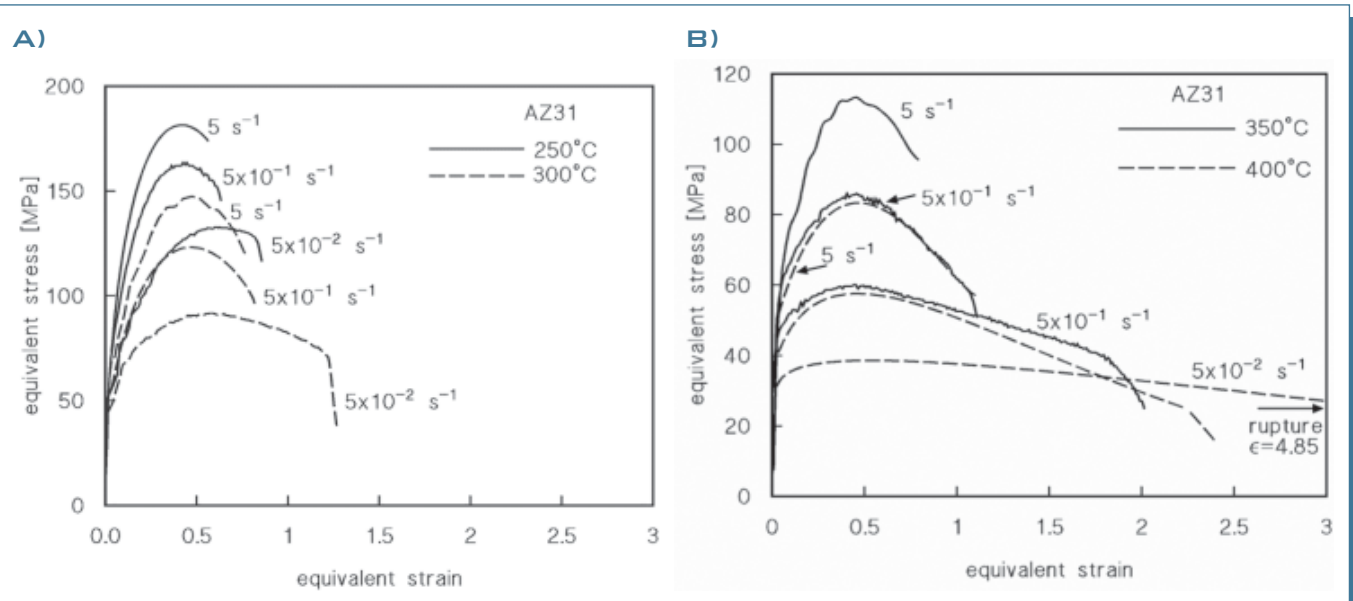

Fig. 2: Equivalent stress vs equivalent strain curves between 250 and 400°C.

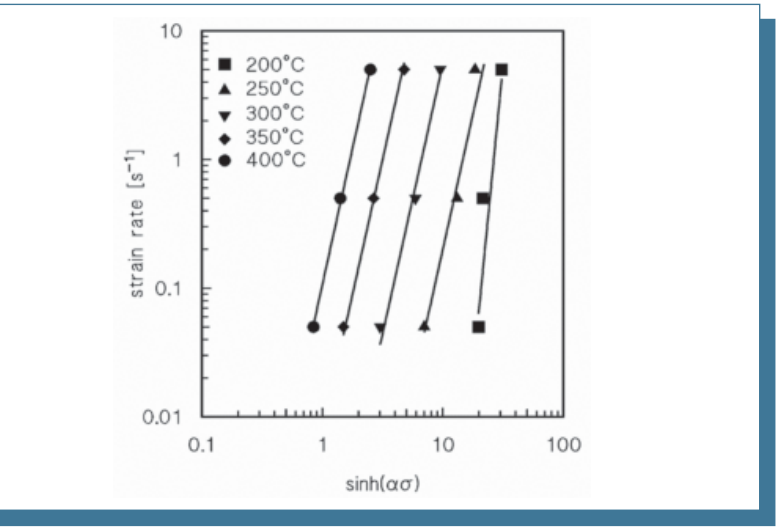

Fig. 3: Constitutive analysis of the strain rate vs peak stress dependence at constant temperature.

where A and a 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. A best-fitting procedure gave α=0.02 MPa^{-1} and n=4.2 for temperatures between 250 and 400°C; at the lowest temperature (200°C), the stress exponent was far larger, exceeding n=9. The activation energy, calculated by plotting sinh (αs) as a function of 1/T, was Q=155 kJ/mol when all the experimental data points were considered. 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). Nevertheless, when the data points at 200°C were excluded from the analysis, a better

linearity was obtained; this calculation gave Q=180 kJ/mol (Figure 4), a value substantially greater than that of self-diffusion of solute atoms. The plot in Figure 4 also includes tension test results on a similar AZ31 alloy under a strain rate of $1.3x10^{-4}$ s^{-1} produced by other authors [11]. Also in this case the activation energy was close to 180 kJ/mol.

Figure 5 plots the Zener-Hollomon parameter Z

$$Z = A\,[\sinh(\alpha s)]^n = \dot{\varepsilon}\exp(Q/RT) \qquad (2)$$

as a function of peak stress, with Q=180 kJ/mol. As expected, all the data obtained between 250 and 400°C lie on the same curve. By contrast, low-temperature and high-strain rate data identify a different regime.

The microstructure of the alloy after torsion at 200 and 250°C and a strain rate of 5 s^{-1} is shown in Figure 6. Straining at 200°C resulted in deformed grains heavily decorated with twins, a ususal behaviour in Mg alloys tested at these low temperatures [12]; raising the testing temperature to 250°C did not alter the microstructure dramatically, although thin colonies of very fine grains appear at boundaries of heavily deformed grains. The microstructure of the sample strained at 5 s^{-1} at 300 and 350°C, shown in Figure 7, consisted of a bimodal sized grains. Large, elongated grains representing the un-recrystallized portion of the microstructure were separated by layers of fine, equiaxed, dynamically recrystallized grains. DRX was almost completed at 350°C. A further increase in straining temperature to 400°C resulted in DRX producing a fully equiaxed microstructure in which grains underwent moderate growth (Figure 8). Optical microscopy of the samples deformed at the same temperature under the strain rate of 0.05 s^{-1} revealed only minor differences in recrystallized microstructure; at 300 and 350°C, for example, the grain size was slightly more homogeneous after deformation at 5 s^{-1} than at 0.05 s^{-1}, while at 400°C, deforming under the lowest strain rate led to a more pronounced grain growth.

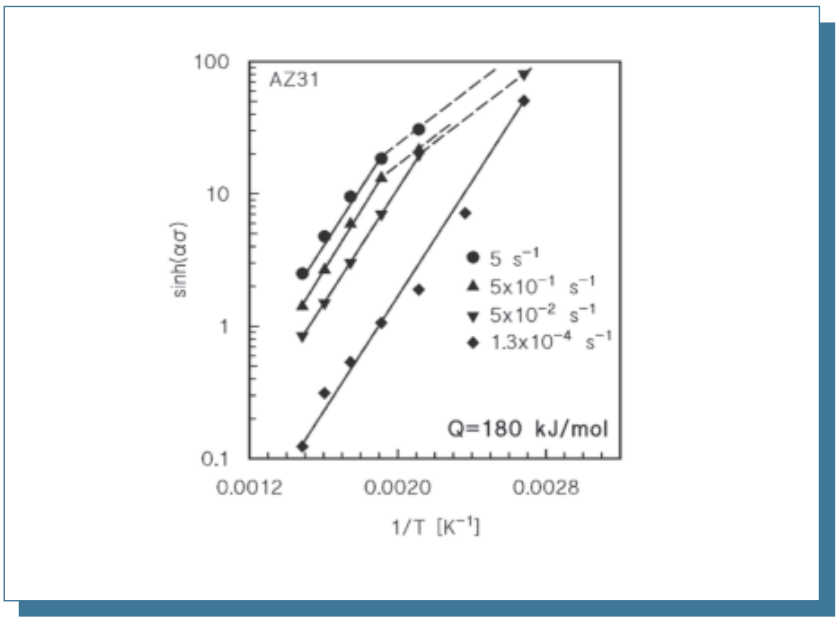

Fig. 4: Calculation of the activation energy for high-temperature deformation; data from ref.[11] are included for comparison.

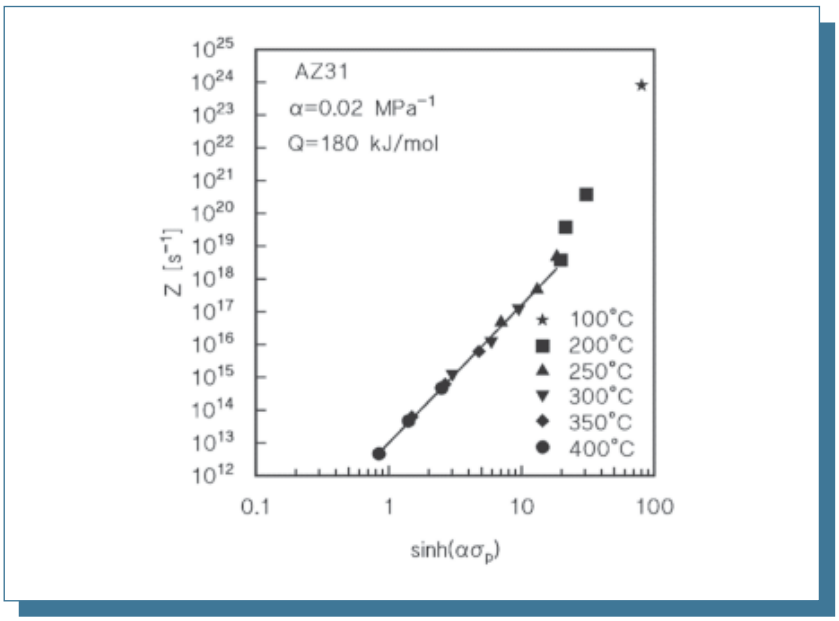

Fig. 5: Zener Hollomon parameter as a function of peak stress; data obtained at low temperature and high stran rate clearly identify a different regime..

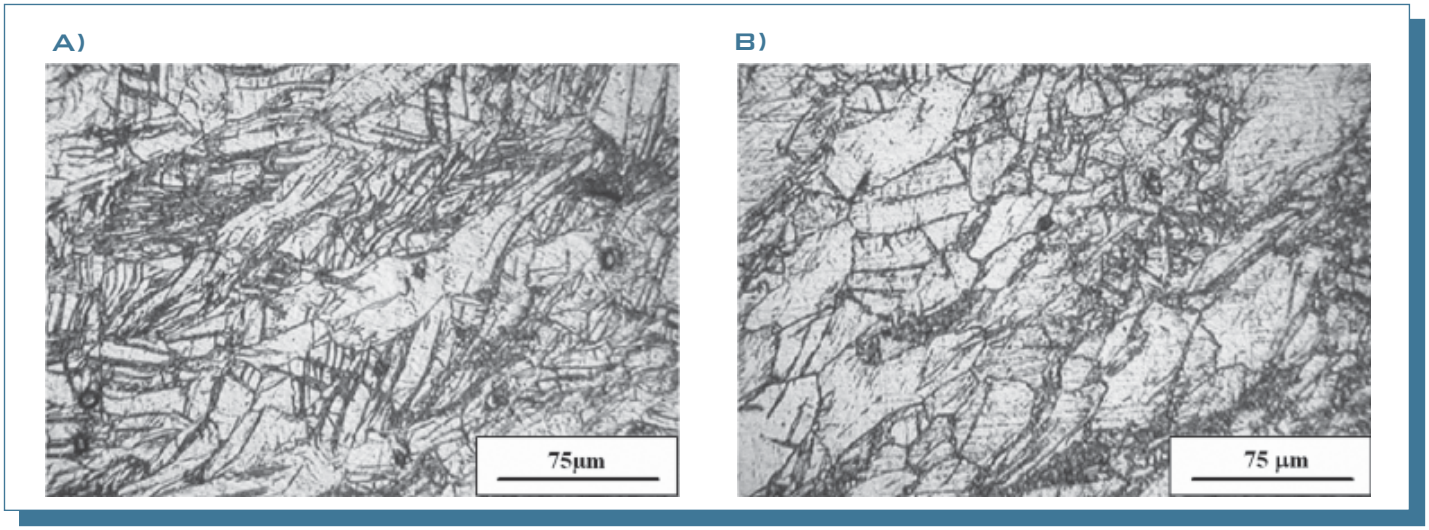

Fig. 6: Microstructure of the samples deformed at 5 s^{-1}: a) 200°C; b) 250°C.

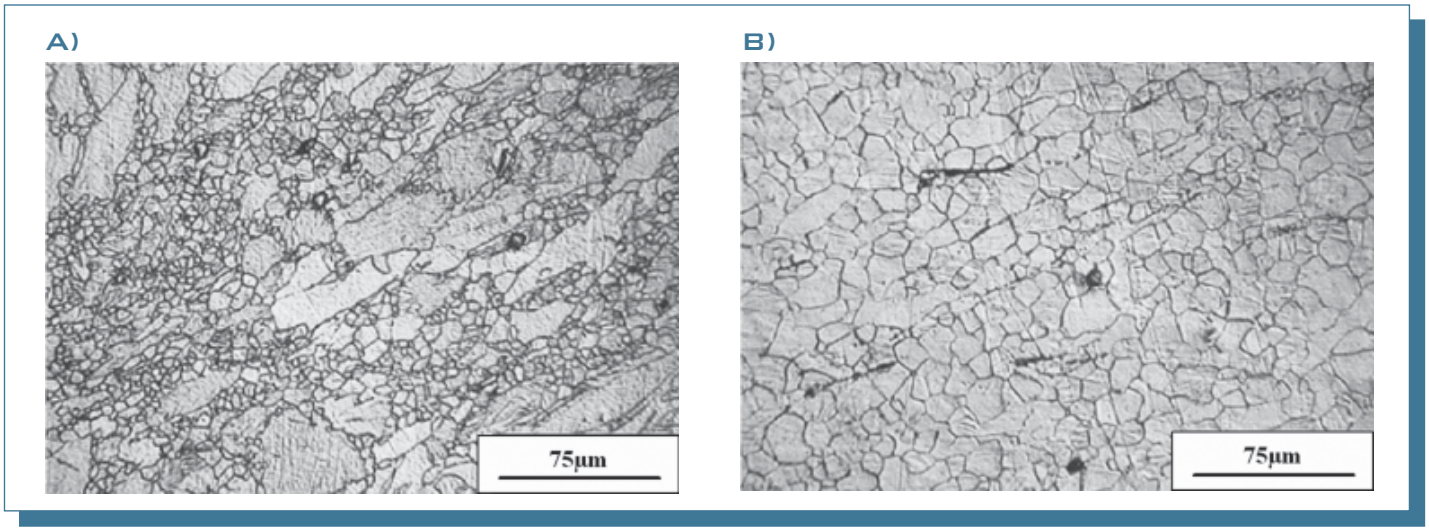

Fig. 7: Microstructure of the samples deformed at 5 s^{-1}: a) 300°C; b) 350°C.

DISCUSSION

The correlation between DRX mechanisms and deformation conditions in a Mg alloy deformed at high temperature has recently been investigated [13]. Galiyev and co-workers analysed three different regimes:

1) low-temperature regime, below 200°C, where twinning accommodates plastic strain; since this combination only partly meets the compatibility constraints at grain boundaries, compatibility stresses may locally exceed the critical resolved shear stress for non-basal slip. This regime is also characterized by a low value of the activation energy, connected with the need for non-conservative motion of dislocations driven by pipe-diffusion at low temperature;
2) intermediate temperature regime (200-250°C) where dislocation glide is assisted by cross-slip; the activation energy in this regime is close to 92 kJ/mol. The onset of continuous DRX was recorded in this regime;
3) high-temperature regime (>250°C), where the activation energy increases to a value close to the activation energy for self-diffusion; dislocation climb was thus proposed to provide the conditions allowing the DRX occurence.

The high-temperature regime was also investigated in the AZ31 by Watanabe and co-workers [10], albeit under lower strain rates (10^{-3}-10^{-5} s^{-1}). The authors described the peak stress dependence on strain rate by means of equation in the form:

$$\dot{\varepsilon} \propto (\sigma - \sigma_0)^n \exp(-Q/RT) \quad (4)$$

where n=3, Q is close to the activation energy for self-diffusion, and σ_0 represents a threshold stress (of unspecified nature). The authors do not mention the occurrence of recrystallization. On the other hand, Barnett [4] postulated the DRX role to increase the activation energy of hot-working in this regime. The onset of dynamic recrystallization in hot-strained AZ31 was

extensively investigated by Barnett et al. [6], who drew the strain vs. Z (with Q=135 kJ/mol) map illustrated in Figure 9. The map identifies different regions of strain, strain rate and temperature that correspond to complete, partial or no recrystallization of the deformed structure. The solid symbols represent the experimental condition used in this study (strain is equivalent to strain to fracture); the map clearly indicates that the lowest the initial grain size, the easiest the onset and completion of DRX. The only significant discrepancy between the results of microstructural analysis and the map in Figure 9 is that according to the latter, partial DRX at 200°C should occur in alloy with initial grain size similar of the one of alloy tested in the present instance.

The onset of DRX precedes the peak stress; the start point is identified at the inflection of the $\theta-\sigma$ curves (where $\theta=\delta\sigma/\delta\varepsilon$) [13]. The Figure 10 represents $\theta-\sigma$ curves for the investigated AZ31 and two extrapolation segments of saturation stress σ^*. The saturation stress, representing a hypothetical value of the flow stress "in steady state" in the absence of DRX, is calculated by extrapolating at $\theta=0$ the value of the linear portion of strain-hardening rate curve. Indeed, in the presence of DRX the strain-hardening curve falls to 0, the point of inflection being the critical stress for DRX. The θ-σ curves can thus be used to calculate approximately the σ^* values between 250 and 400°C (although the difference between peak stress and saturation stress is not negligible only above 300°C); the constitutive analysis of the saturation stress dependence on strain rate (Figure 11) gave Q=143 kJ/mol and n=3, i.e., in the absence of DRX, the values of the stress exponent and of the activation energy are consistent with viscous glide of dislocation as the rate-controlling mechanism. The high value of the activation energy observed in this study (Q=180 kJ/mol) should thus be mainly related to the occurrence of DRX.

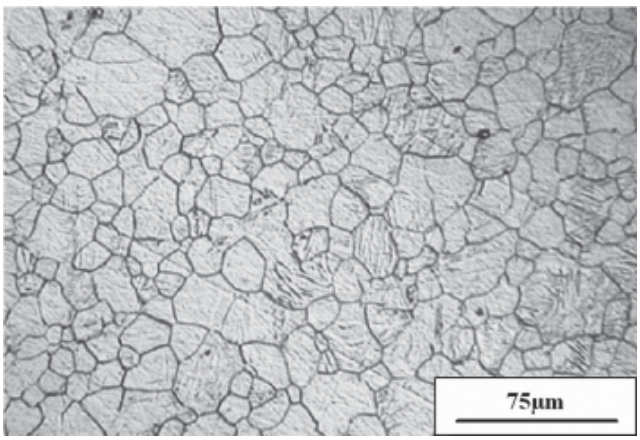

Fig. 8: Microstructure of the specimens deformed at 5 s^{-1}- 400°C.

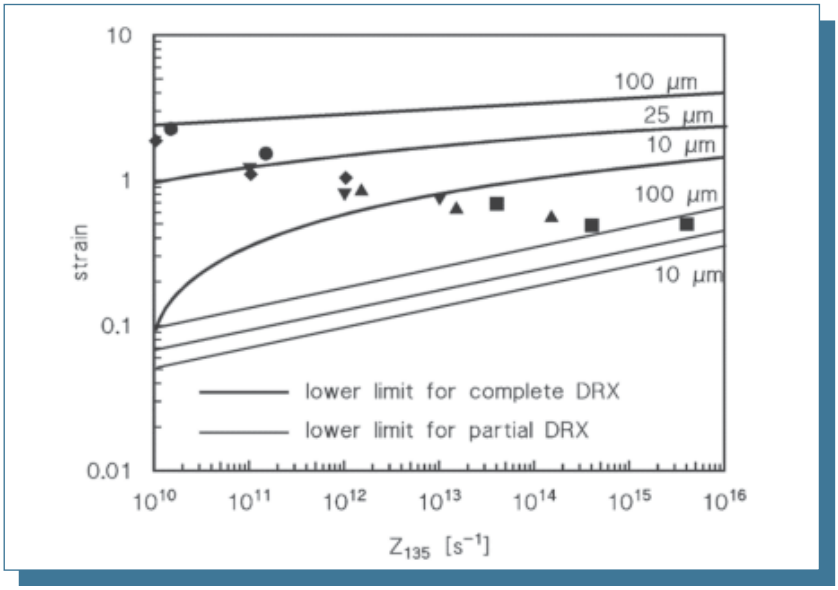

Fig. 9: Zener-Hollomon parameter vs. strain map identifying boundaries for partial and complete DRX in AZ31 with different initial grain size; data of the present study are reported on the map (symbols as in Fig.3).

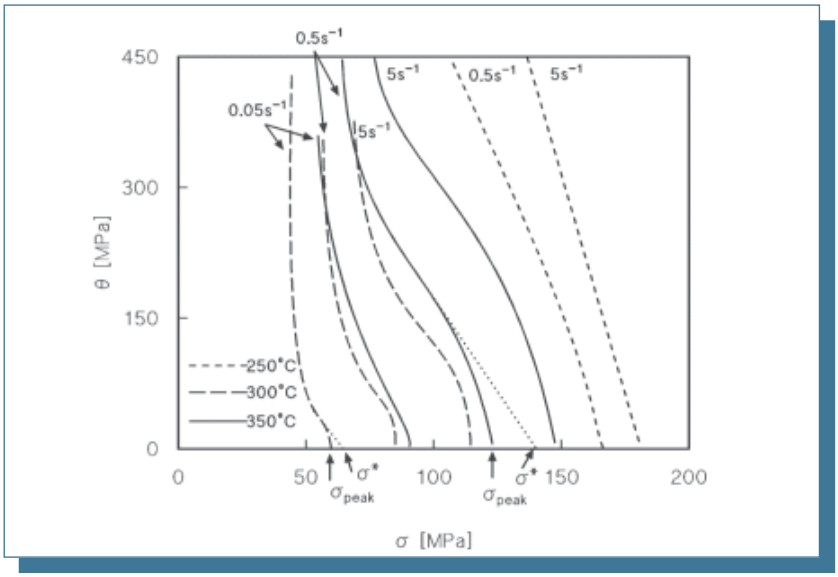

Fig. 10: Strain hardening rate curves as a function of the applied stress; two examples of calculation of the saturation stress are shown.

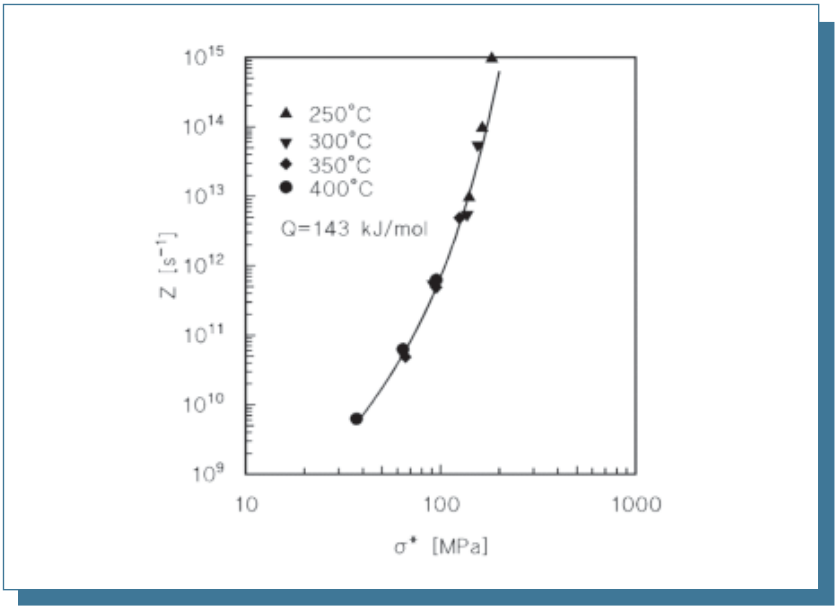

Fig. 11: Zener-Hollomon plot (Q=143 kJ/mol) as a function of saturation stress as calculated in Figure 10.

CONCLUSIONS

High-temperature formability of an AZ31 alloy was investigated by torsion testing between 200 and 400°C. Constitutive analysis of the experimental data at temperatures above 250°C gave a high value of activation energy for deformation (180 kJ/mol). Microstructural analysis demonstrated that straining at these temperatures resulted in the onset of dynamic recrystallization whose extension increased with temperature going to completion at 350°C. Straining at 400°C resulted in complete DRX and grain growth. The high value of the activation energy was related to the occurrence of DRX.

AKNOWLEDGMENTS.

The Italian authors are indebted to the Ministry of University for the funds assigned in the frame of PRIN 2005-06.

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