HIGH TEMPERATURE DEFORMATION OF WROUGHT Zn-CONTAINING MAGNESIUM ALLOYS

S.Spigarelli[1,a], M.El Mehtedi[1,b], D.Ciccarelli[1], M.Bamberger[2], G. Cupitò[3], Y.Rami[4]

1. Dipartimento di Meccanica, Università Politecnica delle Marche, 60131, Ancona, Italy (email: s.spigarelli@univpm.it)
2. Department of Materials Engineering, Technion-Israel Institute of Technology, 32000 Haifa, Israel
3. Centro Ricerche Fiat, 10043 Orbassano, Italy
4. Alubin Kiryat Bialik, Israel

[a] s.spigarelli@univpm.it, [b] elmehtedi@univpm.it

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Abstract. The high temperature response in torsion and creep of two extruded Mg-Zn alloys was investigated in the present study. The alloy 0 (Mg-2Zn-1Mn) was found to exhibit a lower strength than the alloy 2 (Mg-0.55Zn-0.79Mn-0.75Al-0.17Ca), even if the activation energy for creep was similar for both materials (170-180 kJ/mol). The difference in flow stress was here preliminarily attributed to the precipitation of fine Al_2Ca particles.

Introduction

Magnesium alloys possess a unique combination of light weight, high specific strength and stiffness and high recycling capability, and could replace steel, iron and, in some cases, also aluminum parts in automotive industry. Initial applications for wrought alloys are expected in form of extrusions and forgings, to be followed by sheets; extensive researches are thus required to investigate the hot working response of these materials, and, in case of alloys intended to operate at high (100°C) temperature, the creep behaviour. Only the AZ31 alloy has been extensively characterized, while very few results are available for other interesting materials, such as the Mg-Zn alloys [1,2]. The present study aims thus at investigating the high temperature properties of two Zn containing magnesium wrought alloys by combining constant strain rate (torsion) and constant load (tensile) creep experiments.

Experimental procedures

The two alloys, henceforth indicated as alloy 0 and alloy 2 were produced by Direct Chill casting and then extruded by Alubin, Israel. The chemical composition (wt.%) of the two alloys is illustrated in Table I.

Table I. Chemical composition of the investigated alloys.

Alloy	Zn	Mn	Al	Si	Fe	Cu	Ni	Ca	Mg
0	1.9	0.75	0.0031	0.0252	0.0125	0.0024	0.0012	-	bal
2	0.55	0.79	0.75	0.035	0.011	0.001	0.003	0.17	bal

Torsion tests were carried out in air on a computer-controlled torsion machine, under surface equivalent strain rates ranging from 0.05 to 5 s^{-1} and temperatures from 150 to 500°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, σ, and equivalent strain, ε, were calculated using the relationships [3]:

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

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

where N is the number of revolutions, M is the torque, m’ (strain rate sensitivity coefficient) at constant strain is $\partial \log M / \partial \log \dot{N}$, 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 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.
Specimens for creep experiments were machined from extruded rods. The two alloys were tested by constant load creep tests at 100, 125 and 150°C; some tests were carried out to fracture; in other cases the load was changed, after attaining a “secondary” regime. Elongation was continuously measured during the test. Microstructural studies were carried out by optical microscopy.

Results and Discussion

The initial microstructure of the two alloys is illustrated in Figure 1.

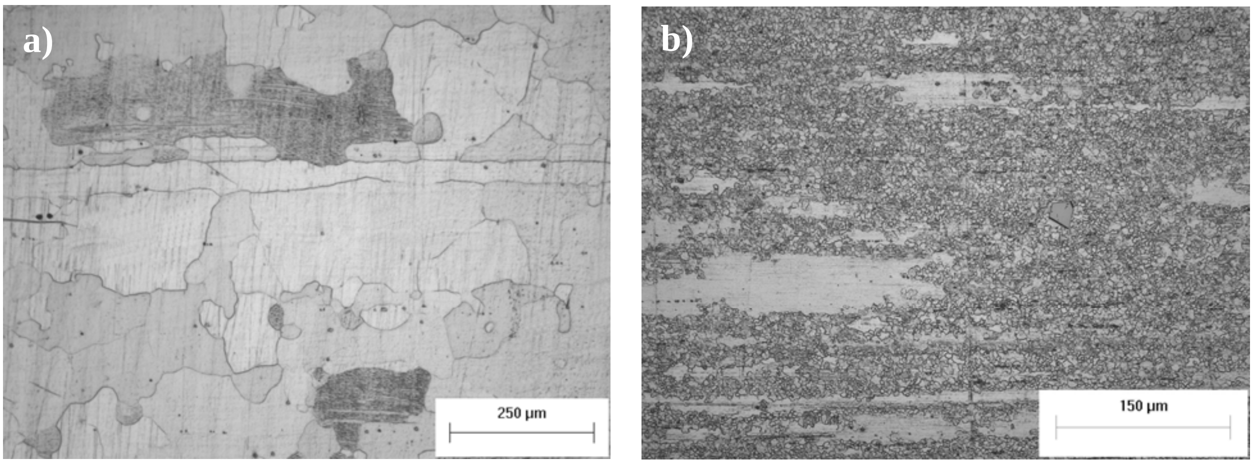


Figure 1. Microstructure of the as-extruded alloys; a) alloy 0; b) alloy 2.

The Figure 2 shows typical examples of the equivalent stress vs equivalent strain curves obtained in torsion for both alloys. Although the shape of the curve is similar, the higher strength of the alloy 2 is readily apparent. The values of the peak flow stress as a function of the testing strain rate are plotted in Figure 3. For the sake of simplicity, the peak flow stress (σ) dependence on strain rate ($\dot{\varepsilon}$) and temperature was expressed by the simple Norton equation, i.e.

$$\dot{\varepsilon} = A\,\sigma^n \exp(-Q/RT) \qquad (3)$$

where A and n should be material parameters, R is the gas constant and Q is the activation energy for high temperature deformation.
Figure 4 shows the minimum strain rate dependence on applied stress as obtained by creep testing. Also in this case the simple Equation 3 was used to model the experimental data. A comparative analysis of Figures 3 and 4 suggests few simple conclusions: i. in both torsion and tensile creep the stress exponent was found to decrease with increasing temperature; ii. The stress exponent observed in the creep, i.e. in a low-strain rate regime, is lower than that obtained by torsion, under higher strain rates.

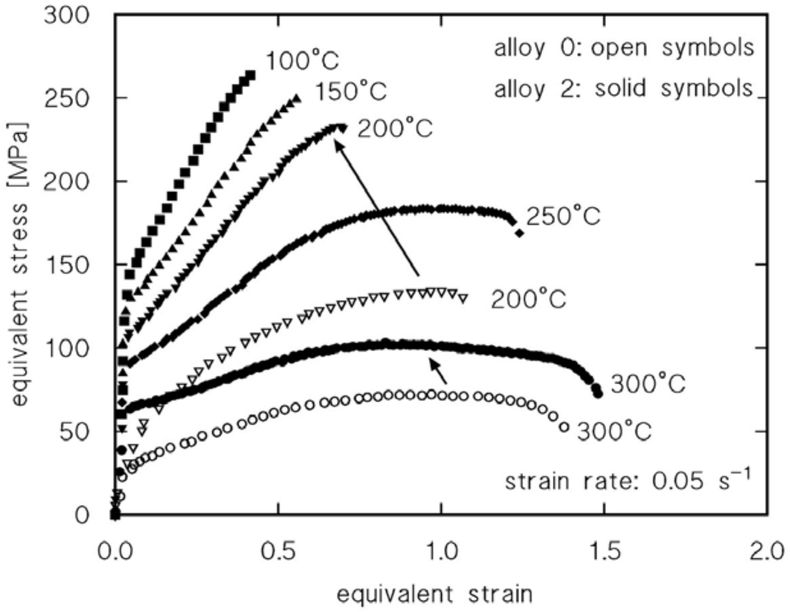
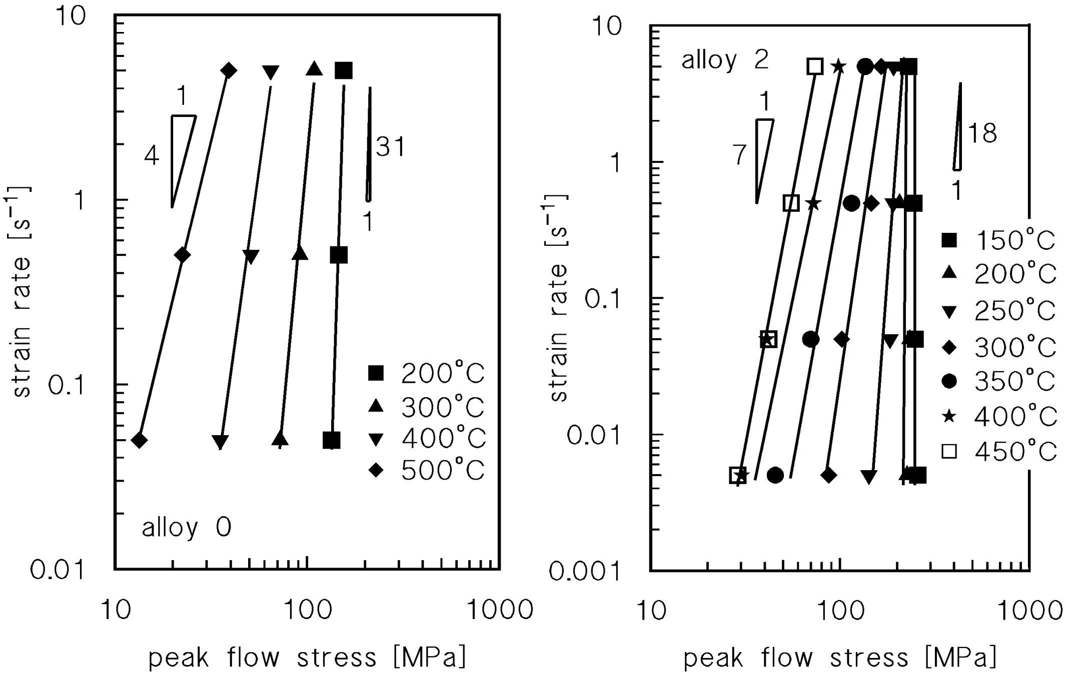


Figure 2. Comparison between the equivalent stress vs equivalent strain obtained by testing the two alloys under a strain rate of 0.05 s^{-1}.

Both the above mentioned conclusion were largely expected, since equation 3 is known to be fairly inaccurate in describing the material behaviour above a certain strain rate, due to the "power law breakdown" typical of high-strain rate testing. By contrast, the value of the activation energy for high temperature deformation was found to be reasonably constant in the case of alloy 0, and close to 170 kJ/mol, either in creep as in torsion. As a result, all the experimental data, irrespective of the experimental technique, for this alloy collapse on a single curve when expressed in the form of Zener-Hollomon parameter:

$$Z = \dot{\varepsilon} \exp(Q/RT) \tag{4}$$

(Figure 5). The situation is more complex in the case of the alloy 2; in the creep regime, the activation energy was found to be reasonably constant and close to 180 kJ/mol; these experimental data are plotted in form of Zener-Hollomon parameter in Figure 5b, where, for the sake of comparison with Fig.5a, a value of Q=170 kJ/mol was used.



a) b)

Figure 3. Peak flow equivalent stress as a function of the testing strain rate (torsion testing) for the two investigated alloys.

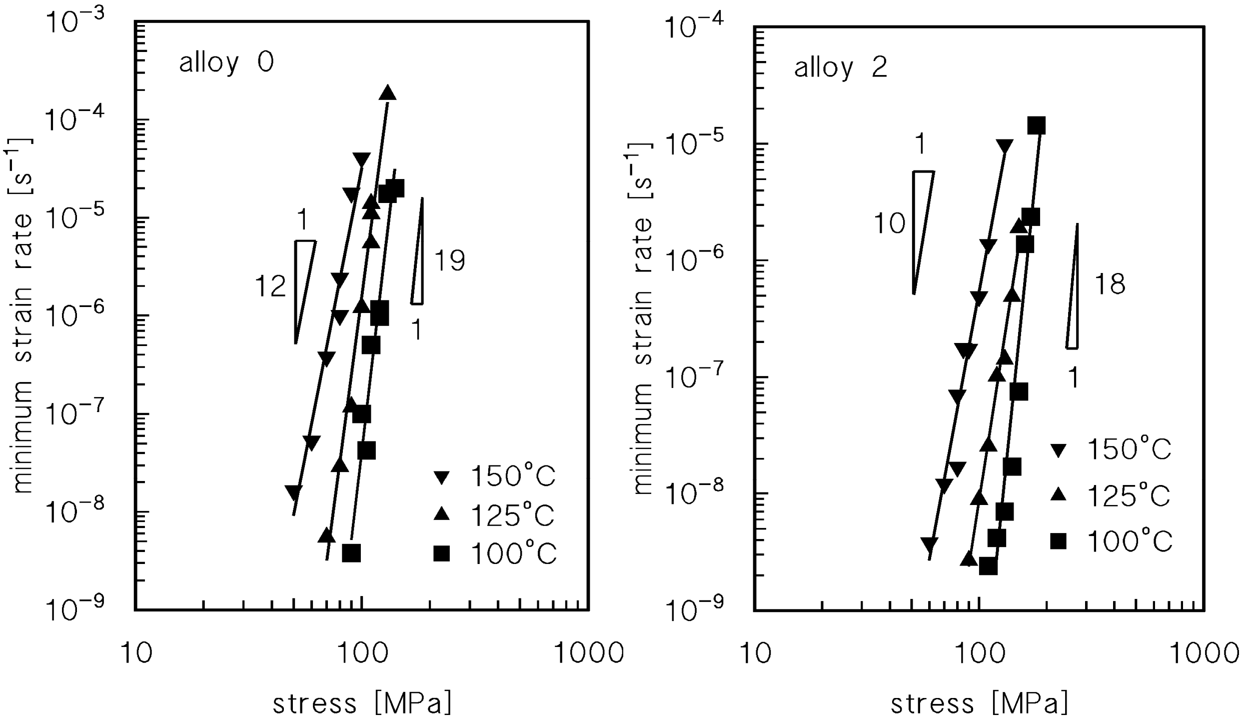
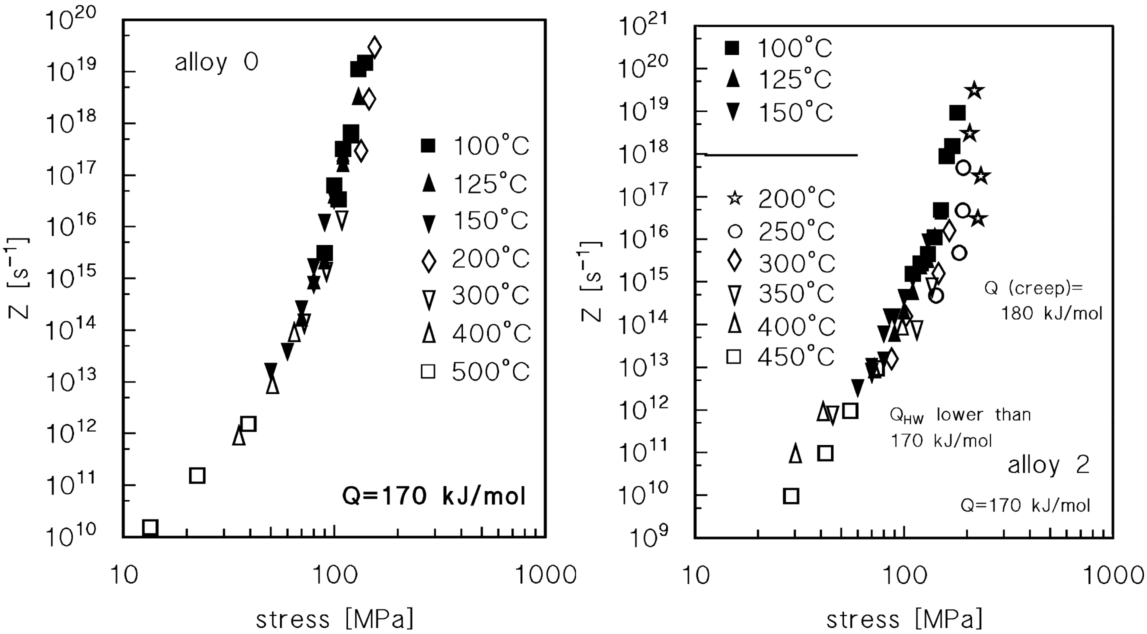


Figure 4. Minimum creep rate as a function of applied stress for the two alloys.

In the wide range of temperature investigated in torsion, the situation is different; in an intermediate strain-rate region, the superimposition of the data obtained at different temperature confirms that Q is close to 170 kJ/mol; by contrast, in the low-strain-rate regime, the Q value giving the overlapping of data for different testing temperatures is lower.



a) b)

Figure 5. Zener-Hollomon parameter as a function of stress for both tensile creep and torsion data. The same value of the activation energy (Q=170 kJ/mol) was used.

The analysis of Figures 2 and 4 clearly shows that the alloy 2 exhibits a strength notably higher than that of the alloy 0, and one can wonder the reason for this enhanced creep strength. The solubility curves for Al and Zn in binary Mg alloys show that a 2% Zn content, in the temperature range for creep testing considered in the present study, results in a limited precipitation of secondary phases. By contrast, Zn and Al contents as low as 0.5-1%, should not lead to any significant precipitation in binary alloys. The above analysis does not necessarily corresponds to the real microstructural evolution in ternary and quaternary materials, but in any case is can be reasonably inferred that the precipitation of Mg-Al and/or Mg-Zn secondary phases in alloy 2 is, at most, very limited, and cannot be responsible for high creep strength. Both Al and Zn should thus only play a solid solution strengthening effect that cannot be accounted for the highest strength of alloy 2.

Vogel and co-workers [4] analysed the creep response of the ZA85 (Mg-8%Zn-5%Al) and found that this alloy exhibited a better creep resistance than the AZ91; the interesting point was that creep strength was further enhanced by Ca (0.3 and 0.9%). Even the minimum creep rate observed in the ZA85-0.3Ca was substantially lower than that of the base alloy; on this basis, the authors concluded that the presence of Ca led to the segregation of this element at the matrice/precipitates interfaces, an effect that was described by introducing a threshold stress, calculated without taking into account microstructural dishomogeneities, that increased with Ca content. On the other hand, the alloys investigated by Vogel and co-workers were in the as-cast condition, and as a consequence the distribution of the alloying elements was highly dishomogeneous. As a matter of fact, the authors demonstrated that the Zn and Al contents were 2 and 1% respectively in grain interior, while the same elements were present in higher concentrations (5 and 3%) in the grain boundary zones, where all the Ca segregated during high temperature exposure. In the light of the alloying elements segregation, even a mere addition of 0.3% Ca, as long as these dishomogeneities persist, results in a substantially higher local concentration in the grain boundary zones. A recent paper [5] clearly suggested that this kind of microstructure should be modelled as a composite, composed by soft (the grain interior) and hard (the grain boundary region) zones, each characterized by a different value of the threshold stress. A similar situation was observed by other authors [6] in the case of small Ca addition in an AZ91 alloy. As usual in these cast alloys, the microstructure was composed by soft α-dendrites, surrounded by a divorced eutectic rich in Al, decorated by a multitude β-$Mg_{17}Al_{12}$ particles. Again, all Ca segregated in the intergranular zone; this effect was accompanied

by a marked reduction in grain size, a refining effect attributed by the authors to a significant role of Ca in controlling the growth of the primary α-dendrites.
Small Ca additions (0.3-0.6%) were found to significantly increase the creep response of the die-cast ZA104 alloy [7]; Zhang and co-workers, as could be reasonable expected, found that the microstructure consisted in a network of precipitates surrounding the Mg-dendrites. The X-ray mapping indicated that the highest Ca concentration was in the secondary phases; two intermetallic phases (τ'_1 and τ'_2) were indeed identified in the grain boundary region: both were similar to the [$Mg_{32}(Al,Zn)_{49}$] (τ), but had different Zn/Al ration and Ca content, even if the latter was harder and contained more Ca. The differences found in the creep response of the investigated alloys were thus attributed to the presence of the τ'_2 phase, more extensively observed in the material containing 0.6% Ca. In any case, again the segregation in the interdendritic zones led to local concentration of Ca by far higher than the nominal content, either 0.3 or 0.6%.
Both the alloys considered in the present study contains Zn (2% and 0.6% respectively) and Mn (0.8-1%); in one case, the material also contains also 0.75% Al and traces amounts (0.17%) of Ca. In both cases the alloys were extruded, and as a consequence exhibited a recrystallized microstructure composed by equiaxed grains. While a certain amount of precipitation of Zn-containing phases could be expected in the Mg-2Zn-1Mn alloy, the same, as above mentioned, cannot be said for the Mg-Zn-Al-Mn-Ca alloy; on the other hand, a comparison with the calculated phase diagram for Mg-Al-Ca alloys reported in [8] seems to suggest that limited precipitation should result in at least a modest presence of Al_2Ca at room temperature.

Conclusions

The high temperature deformation of two Mg alloys was investigated by constant strain rate torsion and constant load creep testing. The analysis of the peak flow stress dependence on minimum strain rate and temperature demonstrated that the material with Al, Zn and Mn contents lower that 1% was substantially higher than that of the Mg-2Zn-1Mn alloy. The only viable conclusion about the higher strength of the former alloy was the effect of minor (<0.2%) addition of Ca that should precipitate in form of Al_2Ca particles.

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