

Analysis of the creep response of a fine-grained magnesium-rare earths alloy produced by rapid solidification

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Abstract

The creep response of a experimental Mg-Zn-Ca-Ce-La alloy produced by rapid solidification was investigated at temperatures ranging from 498 to 548K, under stresses from 7 to 50MPa. The creep curves exhibited a short primary region, during which the strain rate decreases, followed by a short minimum creep rate range, and by a very long tertiary stage. The analysis of the creep rate dependence on applied stress at different strain suggested that the creep mechanisms was cross slip on non-basal plane, or alternatively glide of dislocation on pyramidal slip system controlled by nucleation of kink motion.

Riassunto

È stato analizzato il comportamento a creep di una lega Mg-Zn-Ca-Ce-La prodotta per solidificazione rapida. Le prove di creep a carico costante sono state condotte alle temperature di 498, 523 e 548K, sotto sollecitazioni variabili da 7 a 50MPa. La forma della curva di creep presentava un breve primario, durante il quale la velocità di deformazione diminuiva velocemente con la deformazione, seguita da un altrettanto breve intervallo durante il quale la velocità di deformazione rimaneva approssimativamente costante, e quindi da un terziario estremamente lungo.

Lo studio della dipendenza della velocità di deformazione, misurata a diverse deformazioni, in funzione della sollecitazione applicata, ha suggerito che i meccanismi di creep operanti dovrebbero essere lo scorrimento incrociato (cross slip) delle dislocazioni su piani non-basali, o alternativamente lo scorrimento (glide) delle dislocazioni sui piani piramidali.

INTRODUCTION

The creep response of Mg and its alloys has been studied extensively in the past [1-11]; in general the steady state rate ($\dot{\epsilon}_s$) was related to the applied stress and temperature by means of the conventional power law equation $\dot{\epsilon}_s \propto \sigma^n \exp(-Q/RT)$, where σ is the applied stress, R and T are the gas constant and the absolute temperature, while the stress exponent n and the apparent activation energy for creep, Q , are parameters of material. In one of these studies, Tegart [1] found that in the temperature range of 450 and 550

K, $n=5.5$ and $Q=117$ kJ/mol, while at higher temperatures ($T>750$ K) $n=4.0$ and $Q=218$ kJ/mol. Other authors [2] identified a high strain rate regime, where $n=8$ and $Q=360$ kJ/mol, and a low strain rate regime, where $n=4.5$ and $Q=105$ kJ/mol. The change in creep behaviour was attributed to a transition from climb-controlled creep to cross-slip from basal to non-basal planes as a dominating mechanisms. Another extensive study on pure Mg was carried out by Vagarali and Langdon [4], that showed that at temperatures lower

than 650 K the values $n=5.2$ and $Q=135$ kJ/mol were clear indication that creep is climb-controlled. Only at higher temperatures cross-slip of dislocation from basal to non-basal planes became the rate-controlling mechanism. More recently, Shi and Northwood [5] tested a pure Mg at 473 K, obtaining $n=5.6$. Other interesting studies were carried out on a composite Mg+MgO [6], on the Mg-0.8%Al solid solution alloy [7] and on the AZ91 (Mg-Al-Zn-Mn) [8-10]. Alloying Mg with rare earth is reported to have beneficial influence on both mechanical properties and corrosion re-

sistance [11-12]; a high strength Mg-Zn-Zr alloy produced by powder metallurgy and rapid solidification, for example, can be alloyed with rare earth, yielding a refined microstructure and improved corrosion properties [11]. Since the creep behaviour of fine-grained Mg alloys produced by rapid solidification need to be further evaluated, a Mg-Zn-Ca-Ce-La alloy of this family was studied in the temperature range between 473-523 K under constant stress [13-14]. This paper reports the results of a new series of constant load tests carried out on the same alloy.

MATERIAL AND EXPERIMENTAL PROCEDURES

The alloy was produced by Hydro Norsk Corp., Porsgrunn, Norway, by planar-flow casting and extrusion and had the following chemical composition (wt.%): 3 Zn, 1 Ca, 2.5MM (MM being Ca, Ce, La), Mg bal. The extrusion temperature was 585 K, and the ram speed was $0.05 \mu\text{m s}^{-1}$. The microstructure of the alloy after extrusion consisted of extremely

fine grains (0.5 mm) and of a dispersion of particles (Mg_2Ca , Mg_7Zn_3 , $\text{Mg}_3(\text{Ce,La})$). Creep samples of 5 mm in diameter and 25 mm in gauge length were machined from the extruded rods, parallel to the extrusion direction. Uniaxial constant-load creep tests were performed in air at the temperatures of 498, 523 and 548 K, the initial applied stress ranging from 7 to 50 MPa. Maximum engineering strain obtained without rupture or necking was $e=120\%$.

RESULTS AND DISCUSSION

Figure 1 shows a typical strain vs time and strain rate vs time creep curve; a very short "normal" primary region during which the strain rate sharply decreases is followed by a short minimum creep-rate range, and a very extended tertiary region. The instantaneous creep-rate as a function of strain at 523 K is shown in fig.2. Constant-stress creep tests carried out on the same alloy under similar experimental conditions, indeed show that below 10 MPa the creep curves are characterized by a minimum in creep-rate at strains less than 0.01, while in the intermediate stress regime (14-85MPa) the creep-rate decreases relatively quickly with strain up to $\epsilon=0.03-0.04$, and then continues to decrease approximately linearly with strain [13-14].

Constitutive equations for creep in hcp metals

At room temperature Mg, like other hcp metals, can be deformed by dislocation motion on basal planes; thus, the low ductility typical of these materials is the consequence of the low number of active slip systems. At $T>473$ K, additional non basal (pyramidal or prismatic) planes begin to operate,

giving a substantial contribution to deformation and hence to the ductility [15]. A roughly linear dependence of the activation energy for creep on the reciprocal of the applied stress in alpha-Zirconium [3,16] suggested that at sufficiently low stresses creep is controlled by glide of dislocations in pyramidal slip systems; in this context Gilman [17] proposed a model based on the basic assumption that dislocation velocity is controlled by nucleation of kink motion; accordingly, the velocity of dislocation can be expressed as:

$$v \propto \exp\left[-\frac{\Delta H^*}{RT}\right] \exp\left[-\frac{\eta^*}{\sigma^* RT}\right] \quad (1)$$

where σ^* is the effective stress, ΔH^* is the activation enthalpy for kink formation, and η^* is a constant. Since $\dot{\epsilon} \propto \rho_m v$, where ρ_m is the moving dislocation density on the respective non basal plane,

$$\dot{\epsilon} \propto \rho_m \exp\left[-\frac{\Delta H^*}{RT}\right] \exp\left[-\frac{\eta^*}{\sigma^* RT}\right] \quad (2a)$$

The Gilman's equation is formally similar to a earlier relationship suggested by Friedel [18]; according to his model, the cross slip of dislocations, gliding on the basal plane, to pyramidal or prismatic planes can lead to an equation in the form

$$\dot{\epsilon} \propto \rho_m (\sigma^*)^2 \exp\left[-\frac{\Delta H^*}{RT}\right] \exp\left[-\frac{\eta^*}{\sigma^* RT}\right] \quad (2b)$$

where ΔH^* is the enthalpy of constriction, η^* is a parameter that depends on the line energy of a dislocation and on the stacking fault energy, and ρ_m is the moving dislocation density on basal planes.

In eqns.1 and 2, the effective stress could be given as $\sigma^* = \sigma - \sigma_i$, where σ_i is an internal stress whose magnitude depends on dislocation configuration [19]. The change in internal stress with strain was measured for a number of metals and alloys by means of the conventional strain-transient and stress-transient tests [20-23]. In most of these studies the internal stress was found to increase with strain during primary creep, while during secondary creep (including alpha-Zr tested between 773 and 1023 K) the ratio $\alpha = \sigma_i / \sigma$ was constant [24] in many materials. Thus, the strain rate dependence on applied stress of eqn.2 can be rewritten as

$$\dot{\epsilon} = A(1-\alpha)^2 \sigma^2 \exp\left[-\frac{\Delta H^*}{RT}\right] \exp\left[-\frac{\eta}{\sigma RT}\right] \quad (3)$$

where, for the sake of simplicity, A is held constant (i.e. any stress dependence of the moving dislocation density is neglected), and

$$\eta = \frac{\eta^*}{1-\alpha} \quad (4)$$

where $\alpha(\epsilon, T)$ is a function of strain and temperature.

Strain rate dependence on stress and temperature in the Mg-Zn-Ca-Ce-La alloy

The analysis of eqn.3 revealed that, at given strain and temperature value, the stress-sensitivity parameter $m = \left. \frac{\partial \log \dot{\epsilon}}{\partial \log \sigma} \right|_{T=\text{const.}}$ decreases when the applied stress increases. This is clearly the case of the Mg-Zn-Ca-Ce-La alloy investigated under constant load in the present study and under constant stress in [13] (fig.3). A good fit of the experimental constant-load data is obtained by taking $\Delta H=132$ kJ/mol as in pure Mg [3], while η increases, at a given strain, when temperature decreases. Interpolation of the experimental strain rate vs stress data at different strain at 523 K gives the measure of the η variation with strain (fig.4). The figure clearly reveals that η (and consequently σ_i) sharply increases in the early stage of the test, i.e. during the "normal" primary creep, while, for $\epsilon > 0.05$, the increase in η with strain becomes almost linear. It follows from fig.4 that the variation of η with strain obeys an empirical relationship in the form:

$$\alpha = \alpha_0 [1 - \exp(-c\epsilon)] + b\epsilon \quad (5)$$

provided that internal stress is ϵ at $\epsilon=0$ (i.e. $\alpha=0$ at $\epsilon=0$). In order to simplify calculation, c and b were considered to be constant, like η^* , while α_0 was found to depend on tem-

perature. Calculation gave $\eta^*=136.7 \pm 10$ kJ MPa/mol, $b=0.196$, $c=230$ and $\alpha_0 = 0.39, 0.198$ and 0.02 at 498, 523 and 548 K respectively (fig.5-6).

Even if the calculated variation of internal stress during the test is somewhat speculative (because the stress dependence of moving dislocation density was neglected), it is nevertheless consistent with the curve obtained by testing pure Al under constant stress [19]. In this case the magnitude of α in Al as measured by stress reduction at 573 K (initial stress 4.74 MPa) was higher than in the present alloy ($\alpha \approx 0.4$ at $\epsilon=0.05$, $\alpha \approx 0.6$ at $\epsilon=0.20$).

Equations 3-5 can be used to predict the shape of the creep curve within the range of stress and temperature investigated in the present study. Fig.7 shows the results of these calculation for constant-load creep tests carried out at 523 K; here $\eta^*=137$ kJ MPa/mol, $b = 0.196$, $c = 230$, $\alpha_p = 0.197$, $\Delta H = 132$ kJ/mol and $\sigma = \sigma_0 \exp(\epsilon)$, where σ_0 is the initial applied stress. The same creep equation can be used also to predict the shape of the creep curve under constant stress; an example of such calculation is given in Fig.8 ($T=498$ K, $\eta^*=137$ kJ MPa/mol, $b = 0.196$, $c = 230$, $\alpha_p = 0.39$, $\Delta H=132$ kJ/mol and $\sigma=\sigma_0$). Figure 9 compares experimental data and calculated curves at different temperatures and 20 MPa; since α_p markedly decreases with increasing temperature, the "normal" primary region practically disappears at 548 K, except under very low stresses (<10 MPa). Thus, the above analysis confirms that eqn.3 can be used to obtain a reliable simulation of the creep curve in the stress range between 10 and 85 MPa, while it fails in describing the very low stress creep behaviour.

Process controlling the creep rate

The experimental data obtained in the present study cover a limited range of stress and temperature values; nevertheless, once the data obtained under very low stress are excluded from calculation, the model of eqn.3 can be successfully used to plot the strain rate vs stress dependence. Therefore creep strain rate could be controlled either by dislocation slip on non basal slip systems, dependent on nucleation of dislocation kink motion, or by dislocation slip in the basal planes, dependent on cross slip in non-basal plane [3]. A detailed comparison between the physical implications of the two above presented models was carried out by Vagarali and Langdon [4]; the calculation of the activation energy for creep $Q = \left. \frac{\partial \ln \dot{\epsilon}}{\partial (-1/RT)} \right|_{\sigma=\text{const.}}$ showed that in pure Mg, tested between 723 and 800 K,

$$Q = \left(140 \pm 10 + \frac{295}{\sigma} \right) \quad [\text{kJ/mol}] \quad (6a)$$

while for Mg-0.8%Al at the same temperature and in the high stress regime [7]

$$Q = \left(160 \pm 10 + \frac{225}{\sigma} \right) \quad [\text{kJ/mol}] \quad (6b)$$

Since the value of 140-160 kJ/mol is much higher than the estimated values of 20-40 kJ/mol associated with kink formation in metals [4,25], it was concluded that the Gilman's model is not rate controlling in this case. To achieve a greater consistency between results and model, the authors suggest that in this range of temperature the Friedel mechanism of cross-slip controls the creep process. A major discrepancy between pure Mg and Mg-Zn-Ca-Ce-La creep behaviour can be easily found when comparing the different temperature regimes in which the Friedel mechanism seems to operate; as previously observed, the peculiar stress dependence of the apparent activation energy of eqn.6 was experimentally observed only in the temperature range between 700-823 K in pure Mg, and between 473 and 548 K in the Mg-Zn-Ca-Ce-La alloy. This result indicates that the dislocation climb-controlled creep that is operative in pure Mg between 473 and 700 K under stresses ranging from 5 to 50 MPa, is rate-controlling only in the high stress regime in the present alloy. At stresses above 100 MPa creep becomes lattice-diffusion controlled and can be described by means of the conventional power law equation:

$$\dot{\epsilon} = A' \left(\frac{b}{d} \right)^p \sigma^n \exp \left[- \frac{\Delta H'}{RT} \right] \quad (7)$$

where A' is a constant, d is the grain size, p is a constant [4], $n=6-7$ and $\Delta H'=135$ kJ/mol [14]. Provided that the slowest operating mechanism is rate controlling, Sustek et al [14] suggested a possible explanation of the transition from cross-

slip controlled creep in the intermediate range of stress to recovery-controlled creep at high stresses. At intermediate stress, recovery is sufficiently fast to partially remove the effect of strain hardening (although the continuous decrease of creep rate in constant stress clearly indicates that strain hardening predominate on recovery), so that cross-slip is the rate limiting mechanism. When the strain rate is high, on the other hand, the movement of dislocation is sufficient fast and recovery is the slowest (and rate controlling) mechanism.

CONCLUSIONS

The fine grained Mg-Zn-Ca-Ce-La exhibits a complex strain rate dependence on stress and temperature. At temperatures between 473 and 548 K, under stresses ranging from 10 to 90 MPa, the main rate-controlling mechanism appears to be glide on basal plane controlled by cross slip on non-basal plane. This behaviour substantially contrasts with the one observed in pure Mg, where cross-slip of dislocations from the basal to prismatic planes becomes operative as rate-controlling mechanism only at higher temperatures (600-750 K).

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