Creep threshold of an Mg-4Al-1Ca alloy

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Received 30 September 2008, received in revised form 6 November 2008, accepted 10 November 2008

Abstract

Creep behaviour of an advanced magnesium alloy AX41 (4 wt.% Al, 1 wt.% Ca, Mg balanced) was analysed over a wide temperature range from 343 to 673 K at stresses from 2 to 200 MPa in order to investigate the mechanisms governing the creep of this alloy. The stress dependences of the creep rate $\dot{\varepsilon}$ ranging from 10^{-9} to 10^{-3} s^{-1} were evaluated by means of compressive creep experiments with stepwise loading. The stress dependence cannot be described by a simple power-law or exponential relationship. The best description is found with the modified Garofalo's sinh relationship $\dot{\varepsilon} = A_M (\sinh [B_M (\sigma - \sigma_{th})])^n$ with a natural exponent $n = 5$. This description well reflects the apparent threshold creep behaviour of the alloy observed at high temperatures and low stresses. The parameters of this relationship support the assumption that lattice diffusion plays a decisive role in creep mechanisms under all experimental conditions. Most likely, a climb controlled creep mechanism governs at lower stresses and higher temperatures, and a glide-controlled mechanism dominates at higher stresses and lower temperatures. The threshold stress σ_{th} is connected with precipitation of a very stable $(Mg,Al)_2Ca$ phase distributed in a discontinuous skeleton at grain boundaries as well as within the grain interior. The level of the threshold stress is determined by a mechanism with a relatively low activation energy $Q \cong 20$ kJ mol^{-1}.

K e y w o r d s: magnesium alloys, creep, threshold behaviour, threshold stress concept, mechanisms of creep

1. Introduction

The creep resistance of magnesium alloys at elevated temperatures is usually increased by an addition of rare earth (RE) elements. Among the alloys of this type, the widely used magnesium based alloys QE22, ZE, AJ, etc. can be included. There is an effort to replace expensive RE elements with cheaper elements while maintaining the enhanced creep resistance. Advanced Mg-Al-Ca alloys (AX alloys) are developed as a cheaper alternative of the alloys containing expensive RE elements, namely strontium (alloys AJ). The mechanical properties of these alloys, namely their creep behaviour, are improved by precipitates of $(Mg,Al)_2Ca$ [1]. However, the effective development of this type of magnesium alloy is hindered by the lack of experimental data and detailed knowledge of mechanisms governing the creep behaviour of these alloys at elevated temperatures. In the present study, the results of creep investigation of a representative of this alloy group, i.e., the alloy AX41, are summarized. A detailed investigation of creep behaviour of this alloy in a wide range of stresses and temperatures extends knowledge of deformation mechanisms and influential factors. The results of this investigation also serve as a basis for extensive investigations of the influence of various reinforcements, namely short fibre reinforcements, on creep resistance and creep mechanisms of composites with this matrix [2, 3].

2. Experimental

Magnesium alloy AX41 with a nominal composition (in wt.%) of 4 Al, 1 Ca and balanced Mg was cast in a Zentrum für Funktionwerkstoffe in Clausthal-Zellerfeld, Germany. The alloy was annealed at 673 K for 24 h and cooled in air. The alloy microstructure is shown in Fig. 1. The average grain size, $d = 0.44$ mm, was a result of the heat treatment used. In accord

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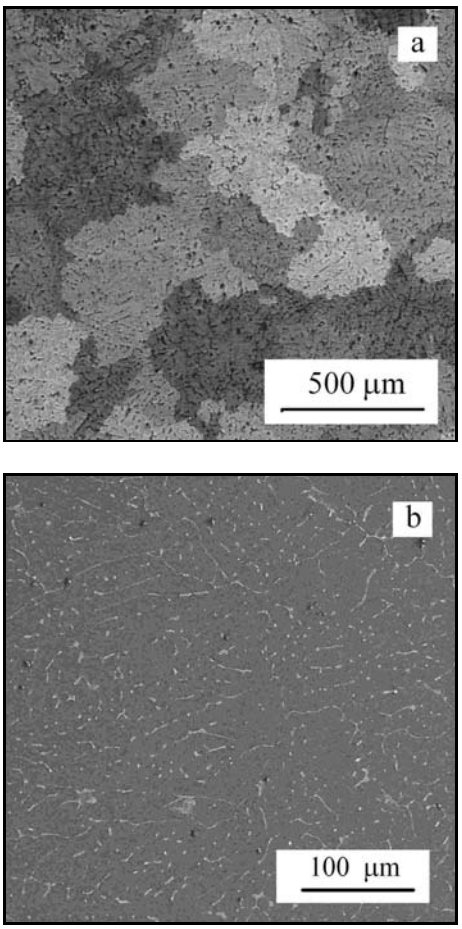

Fig. 1. Structure of the investigated alloy AX41: a) light and b) scanning electron microscopy.

with observations of Ninomiya et al. [1], the dominant phase $(Mg,Al)_2Ca$ precipitated at the grain boundaries as well as in the grain interior. Precipitates form a discontinuous skeleton in the grain interior with an average mesh size approximately 0.04 mm. At lower temperatures, i.e. less than approximately 473 K, precipitation of the $Mg_{17}Al_{12}$ phase can be also possible [2]. Under all experimental temperatures, the matrix can be considered as a very dilute solid solution; this was confirmed by EDX analysis. The estimated amount of remaining aluminium in the matrix did not exceed 1.5 at.% and the calcium content was less than 0.1 at.%.

For creep testing, 6 × 6 mm cross-section parallelepiped specimens, 12 mm in height, were machined. Stepwise load compressive creep tests were used in order to determine the stress dependence of the creep rate $\dot{\varepsilon}$ in an interval from 10^{-9} to 10^{-3} s^{-1} for a given temperature. After each load change, the rate $\dot{\varepsilon}$ was determined after steady state creep conditions had been re-established. This value was then assigned to the true applied stress σ corresponding to the last strain value in the step. The loads of subsequent steps were chosen randomly. The tests were conducted until the strain reached a value of $\varepsilon = 0.15$. The stress dependence of the creep rate $\dot{\varepsilon}$ at a single temperature was usually obtained from two or more tests. The results of the described stepwise procedure were verified in several cases by a comparison with results obtained from a conventional single-stress compressive test. Differences in the creep rates obtained by means of both techniques were negligible in all cases.

The tests were performed in a purified and dried argon atmosphere. An identical temperature regime was applied before each test in order to eliminate the influence of temperature on precipitation of the second phase during the test. In all tests, the sample was maintained at the testing temperature for 10 h before the test was started. The strain was measured with a sensitivity of 10^{-5}. For the stepwise stress creep experiments and evaluation of their results, special software was developed.

3. Results

The creep behaviour of the alloy was investigated at temperatures ranging from 343 to 673 K. All stress dependences of the creep rate $\dot{\varepsilon}$ are shown in Fig. 2, utilizing semi-logarithmic and double-logarithmic plots. The shapes of the dependences do not allow them to be described by simple, i.e., power-law or exponential relationships in the entire range of experimental conditions. However, the Norton power-law relation

$$\dot{\varepsilon} \propto \sigma^n \tag{1}$$

with $n \cong 5$ seems to be suitable for the description at higher temperatures and higher stresses. Interestingly, an exponential relationship

$$\dot{\varepsilon} \propto \exp[B\sigma] \tag{2}$$

appears to be convenient for the lower temperatures and all experimental stresses. At intermediate temperatures, a transition between these conditions, the power-law breakdown, see e.g. [4], is apparent on a single dependence. The power-law breakdown is usually described by Garofalo's relationship [5]

$$\dot{\varepsilon} = A(\sinh[B\sigma])^n, \tag{3}$$

where the parameters A and B depend on temperature T only and exponent n is considered to be a natural number. The temperature dependence of the parameter A is assumed to be in the form

$$A \propto \exp\left[-\frac{Q_G}{RT}\right], \tag{4}$$

where Q_G represents the activation energy and R is

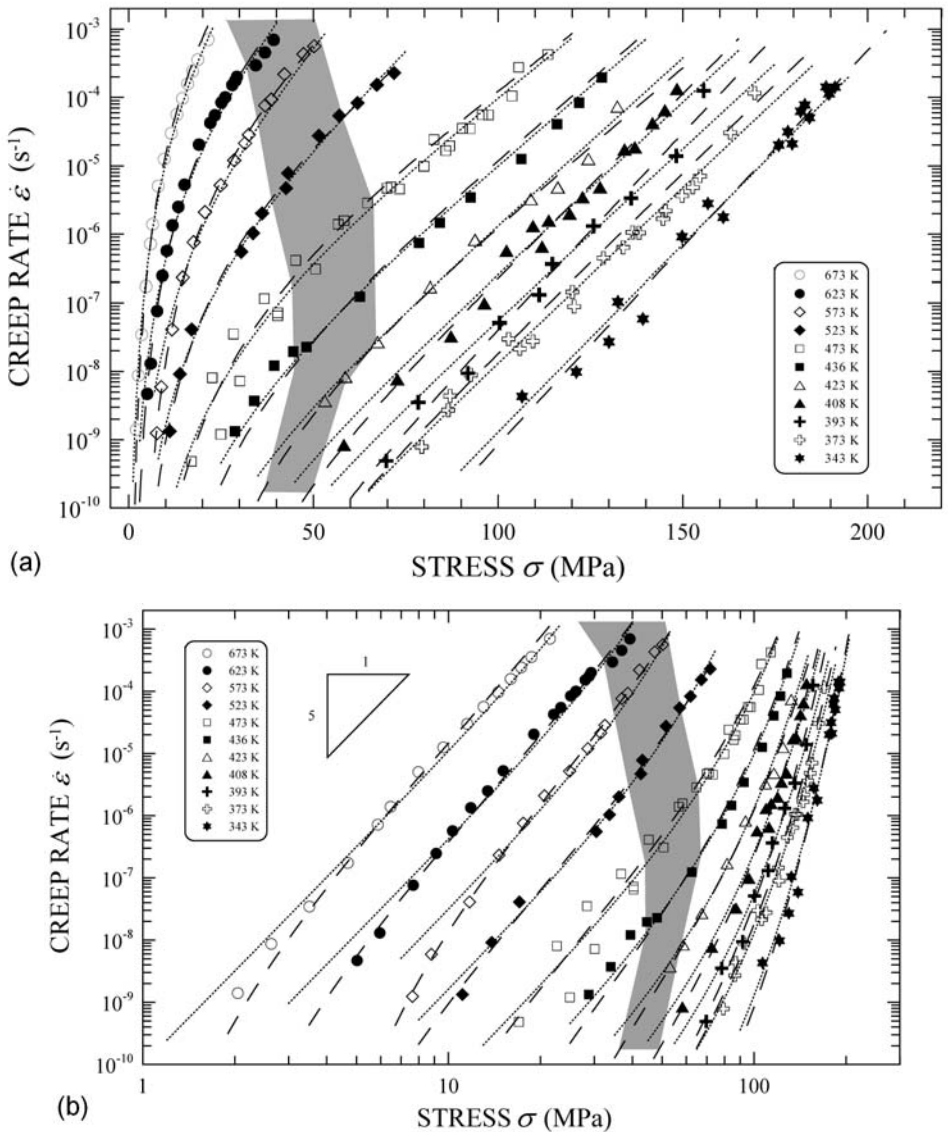

Fig. 2. Stress dependences of creep rate in a) semi-logarithmic and b) double-logarithmic plots.

the gas constant. For values of $B\sigma \leq 0.8$, the sinh function is approximately equal to its argument and, therefore, Eq. (3) can be well approximated by a power-law relationship

$$\dot{\varepsilon} = A\left(\sinh\left[B\sigma\right]\right)^n \cong AB^n\sigma^n. \tag{5}$$

On the other hand, for $B\sigma \geq 1.2$, the sinh function can be well described by an exponential relationship with the argument $nB\sigma$, i.e.

$$\dot{\varepsilon} = A\left(\sinh\left[B\sigma\right]\right)^n \cong \frac{A}{2}\exp\left[nB\sigma\right]. \tag{6}$$

The results of an attempt to describe the experimental dependences by the original Garofalo's relationship, Eq. (3), have been published in more detail elsewhere [6]. Optimal parameters of the equation were obtained by non-linear regression under criterion of the least squares of relative deviations. Natural values of the exponent n from 3 to 6 were tested in fitting. The value $n = 5$ was statistically optimal. The applicability of this original relationship with the exponent $n = 5$ is documented by the drawn dotted curves in Figs. 2a,b. A relatively large discrepancy between experimental and fitted values appears at low stresses and higher temperatures. Under these conditions, a bend of the data from the power-law dependence to lower creep rates is typical, see Fig. 2b. Such a bend in the dependence is usually attributed to the threshold stress creep behaviour (threshold curvature). In order to take into account this behaviour, a description by a modified Garofalo's relationship has been suggested, with a threshold stress $\sigma_{\mathrm{th}}(T)$ as the "opposite" stress, i.e., the relationship

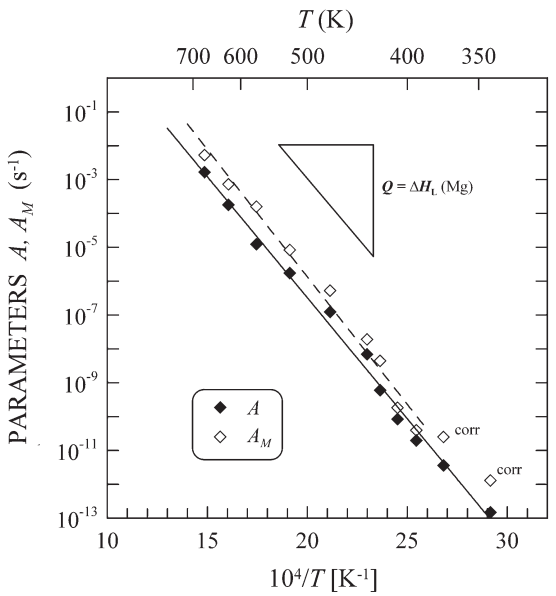

Fig. 3. Temperature dependence of the parameters A and A_{M}.

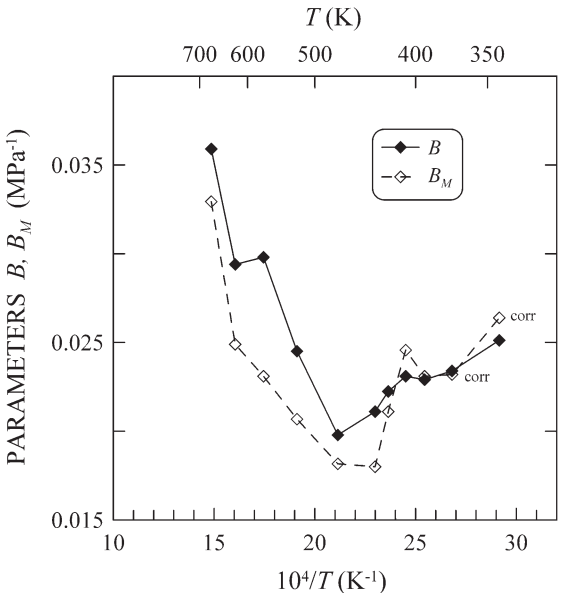

Fig. 4. Temperature dependence of the parameters B and B_{M}.

$$\dot{\varepsilon} = A_{\mathrm{M}} \left(\sinh\left[B_{\mathrm{M}} \left(\sigma - \sigma_{\mathrm{th}}\right)\right]\right)^{n} \tag{7}$$

with A_{M}, B_{M} and $\sigma_{\mathrm{th}}(T)$ depending on temperature only. The results of data fitting based on this relationship are plotted in Figs. 2a,b by drawn dashed lines. The exponent $n = 5$ was again optimal from a statistical point of view. Apparently, this description is quite convenient at higher temperatures and lower stresses; the plotted curves imitate the threshold bends much better. Contrary to this, no substantial differences were observed in fitting the dependences at the lowest temperatures. Due to high stresses and high values of the argument $B_{\mathrm{M}}(\sigma - \sigma_{\mathrm{th}})$, Eq. (7) can be replaced by an exponential relationship,

$$\dot{\varepsilon} = A_{\mathrm{M}} \left(\sinh\left[B_{\mathrm{M}} \left(\sigma - \sigma_{\mathrm{th}}\right)\right]\right)^{5} \cong$$
$$\cong \left(A_{\mathrm{M}}/2\right) \exp\left[5B_{\mathrm{M}} \left(\sigma - \sigma_{\mathrm{th}}\right)\right] = A' \exp\left[5B_{\mathrm{M}}\sigma\right], \tag{8}$$

where the term $\exp\left[-5B\sigma_{\mathrm{th}}\right]$ is included in the factor A' and cannot be reliably extracted by any fitting procedure. In order to also consider the threshold behaviour at the lowest temperatures, relevant values of $\sigma_{\mathrm{th}}(T)$ were extrapolated from the data for higher temperatures (see next chapter).

The dependences of the parameters A and A_{M} on inverse temperature are plotted in Fig. 3. It can be seen that both these dependences are well described by Eq. (4). An assumption of temperature dependence of parameters A and A_{M} in the form $A \propto \exp\left[-Q_{\mathrm{G}}/RT\right]$ and $A \propto \exp\left[-Q_{\mathrm{GM}}/RT\right]$ gives activation energies $Q_{\mathrm{G}} = 136.8$ and $Q_{\mathrm{GM}} = 144.1$ kJ mol^{-1}. Both these values are very close to the activation enthalpy of lattice diffusion $\Delta\ H_{\mathrm{L}} = 135$ kJ mol^{-1} in pure magnesium [7].

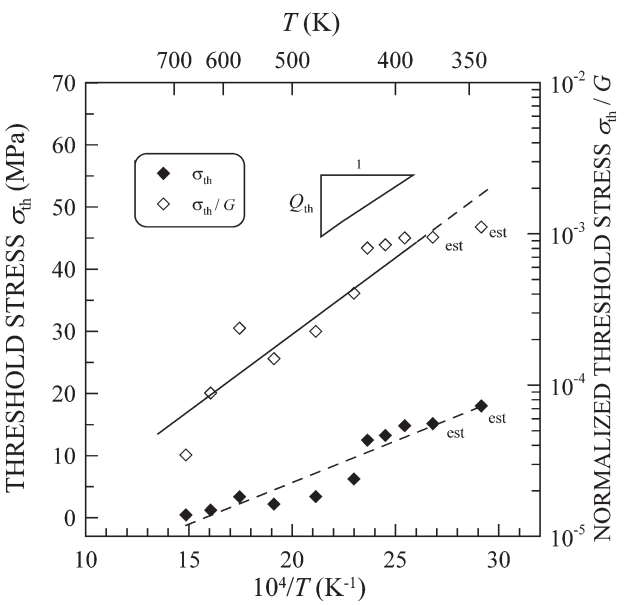

Fig. 5. Temperature dependences of the threshold stress $\sigma_{\mathrm{th}}(T)$ and its values normalized to the shear modulus G.

The temperature dependences of the parameters B and B_{M} are plotted in Fig. 4. Neither dependence is monotonic. The minimum values of both parameters correspond to temperature $T \approx 450$ K.

Dependence of the threshold stress $\sigma_{\mathrm{th}}(T)$ on temperature is illustrated in Fig. 5. The stress $\sigma_{\mathrm{th}}(T)$ increases with decreasing temperature from 0.81 MPa

at 673 K to 18.6 MPa at 393 K. Values of $\sigma_{\mathrm{th}}(T)$ for lower temperatures could not be determined by fitting because exponential parts of the dependence $\dot{\varepsilon} = \dot{\varepsilon}(\sigma)$ correspond to these experimental conditions. Therefore, the values of $\sigma_{\mathrm{th}}(T)$ for the lowest temperatures were simply estimated by a linear extrapolation from values for higher temperatures. Extrapolated values were then taken as input constant values in the fitting procedure.

4. Discussion

The stress dependences of the creep rate $\dot{\varepsilon}$ of the investigated alloy can be well-described using Garofalo's relationship, Eq. (3) [6]. A better description of these dependences yielded a modified version of this relationship, Eq. (7). For both these descriptions, the natural exponent $n = 5$ appeared to be optimal. The similarity and nature of these descriptions, as well as identical exponents n and close values of the parameters A, B, A_{M} and B_{M} appearing in both Eqs. (3) and (7), allow a discussion of the physical basis of the creep behaviour to a great extent simultaneously; different conclusions appear in an interpretation of the stress variable. Using Eq. (7), one considers the lowered stress $(\sigma - \sigma_{\mathrm{th}})$ to be the decisive stress for the creep rate.

The description of the stress dependences according to Garofalo's original sinh relationship, Eq. (3), is usually ascribed to the transition of major mechanisms operating at low and higher stresses (power-law breakdown); see [4] for a review. The mechanism dominating at lower stresses is described by the Norton power-law relationship with the stress exponent n and, at higher stresses, the major mechanism is described by an exponential law. In order to specify these mechanisms, their activation parameters, i.e. the activation energy, stress exponent and derived activation volumes (areas) should be evaluated and taken into account.

Both activation energies Q and Q_{M} obtained from the temperature dependence of parameters A and A_{M}, respectively, are very close to the activation enthalpy of lattice diffusion in pure magnesium. Most likely, there is no specific diffusion data for the investigated alloy AX41. However, it can be expected that the enthalpy of lattice diffusion ΔH_{L} in this alloy does not differ substantially from that in pure magnesium because the matrix is a very dilute solid solution with a Mg basis. Under this assumption, the values of the obtained energies Q and Q_{M} support an expectation that the creep behaviour of the alloy is controlled by lattice diffusion processes under all experimental conditions, i.e., in both stress ranges with power-law and exponential descriptions.

The stress exponent $n = 5$, corresponding to the low stress and higher temperature region, Eq. (3a), is the typical value usually assigned to M (metal) creep behaviour [8]. With respect to the fact that the matrix does not differ substantially from pure magnesium, the metal creep behaviour of the alloy seems to be understandable under these testing conditions. As a rule, a mechanism connected with dislocation climb is considered to govern the creep straining for such a value of the stress exponent and under the controlling role of lattice diffusion, see e.g. [9] for a review. Such a mechanism is relevant to the motion of edge dislocations or edge segments. Contrary to this, the exponential relationship, which is applicable at high stresses and low temperatures, can be explained by an activity of a dislocation glide mechanism controlled by lattice diffusion which results from the values of the activation energies Q and Q_{M}. The glide of jogged screw dislocations [10] or screw segments seems to be the most probable mechanism from commonly considered models of dislocation motion of such a feature.

The apparent activation volume v is evaluated from the experimental stress dependences of the creep rate $\dot{\varepsilon}$ according to the equation

$$v = kT \left(\frac{\partial \ln \dot{\varepsilon}}{\partial \sigma} \right)_T , \tag{9}$$

where k is the Boltzmann constant. The approximate values of the apparent activation volumes for the mechanism described by power-law relationships are

$$v_{\mathrm{l}} = v_{\mathrm{l,M}} = 5kT/\sigma \tag{10}$$

and for mechanisms described by an exponential relationship

$$v_{\mathrm{h}} = 5kTB \quad \text{and} \quad v_{\mathrm{h,M}} = 5kTB_{\mathrm{M}}; \tag{11}$$

indices l and h mark the low and high stress regions, respectively. Within the range of limiting experimental conditions (approximately $\sigma \cong 2$ MPa at 673 K and $\sigma \cong 200$ MPa at 343 K), the values of the apparent activation volume decrease from 700 to $5b^3$ and from 46 to $19b^3$ for climb and glide controlled mechanisms, respectively. An alternative consideration, that the stress reduced by the threshold stress is considered to be the decisive stress variable, gives $v_{\mathrm{l,M}} = 5kT/(\sigma - \sigma_{\mathrm{th}})$. As the threshold stress σ_{th} is only a small part of the experimental applied stresses at a given temperature, the apparent activation volumes do not differ substantially from values corresponding to the applied stress as the decisive variable.

The description of the creep behaviour by the modified Garofalo equation, Eq. (5), allows estimation of the width of the transition region between two assumed dominant mechanisms. As Eq. (5) can

be well substituted for $B_{\mathrm{M}}(\sigma - \sigma_{\mathrm{th}}) \leq 0.8$ and $B_{\mathrm{M}}(\sigma - \sigma_{\mathrm{th}}) \geq 1.2$ by the power-law and the exponential function, respectively, one can assume the dominant role of the relevant mechanisms at these conditions.

The transition region of the creep rate corresponding to the stresses and temperatures between these two conditions is illustrated in Fig. 2 by shadowing. It is apparent that the transition region occurs in a relatively narrow stress range and that margin stresses of the region increase only slightly with decreasing temperature. It is also noteworthy that at higher creep rates, i.e. $\dot{\varepsilon} \geq 10^{-5}\,\mathrm{s}^{-1}$, the transition corresponds to temperatures higher than approximately 500 K. Based on some mechanical testing procedures using such strain rates, changes of strain mechanisms at these temperatures connected with increased activity of non-basal slip of dislocations were deduced (see, e.g., [11]).

Above analysis of the experimental data has shown that an application of the threshold stress concept yields a better description of the stress dependence of the creep rate $\dot{\varepsilon}$. The idea of a phenomenological description of creep behaviour of two- or multiphase alloys by means of a certain formal stress opposed to the applied stress was suggested four decades ago by Lagneborg [12]. A comprehensive review of the back, threshold and internal stresses in creep is given e.g. in [9]. Recently, a great effort has been devoted to explaining relationships between the values of the threshold stresses and phenomena realized in multiphase structures during the creep strain, namely in MMCs [13, 14].

The structure of the investigated alloy consists of hard and stable precipitates distributed in the matrix in a discontinuous skeleton. It is quite appropriate to assume that the influence of such a structure on creep can be described by a threshold stress concept. Until now, no simple models have been suggested for the estimation of the threshold stress produced by such a distribution. However, certain conclusions can be deduced from the temperature dependence of the threshold stress σ_{th}. Mohamed, Park and Lavernija [15] assumed that the stress σ_{th} is governed by a thermally activated process and, therefore, temperature dependence of the stress σ_{th} has a form

$$\frac{\sigma_{\mathrm{th}}}{\mathrm{G}} \propto \exp\left[\frac{Q_{\mathrm{th}}}{RT}\right]. \qquad (12)$$

Q_{th} is an energy, which appears to be associated with the process of overcoming the obstacles of mobile dislocations. Data from Fig. 5 clearly support the suggestion of the determining role of thermal activation. The activation energy $Q_{\mathrm{th}} \approx 20\,\mathrm{kJ\,mol}^{-1}$ results from the slope of the dependence of the normalized threshold stress on inverse temperature in Fig. 5. No mechanism can be reliably assigned to this relatively low energy. On the other hand, a relatively low energy Q_{th} was also obtained from the temperature dependences of the threshold stresses in some metal matrix composites on aluminium or magnesium bases [14].

5. Conclusions

The following conclusions can be drawn from the investigation of creep behaviour of the AX41 alloy at temperatures ranging from 343 to 673 K:

– The creep behaviour of the alloy AX41 can be well explained by the threshold stress concept.

– The stress dependences of the minimum creep rate $\dot{\varepsilon}$ can be well described by a modified Garofalo sinh relationship, Eq. (5).

– The parameters appearing in this relationship can be obtained and physically interpreted.

– Lattice diffusion controls creep of the investigated AX41 alloy in the entire experimental range.

– A combined climb-slip dislocation mechanism dominates at low stresses and higher temperatures, while a glide mechanism governs at lower temperatures and higher stresses. The transition region, in which activities of both mechanisms are comparable, is narrow.

– The threshold stress increases with decreasing temperature. Its value is governed by a mechanism with low activation energy.

Acknowledgements

The author thanks Prof. P. Lukáč for valuable discussions. The work was supported by Project 106/06/1354 of the Grant Agency of the Czech Republic.

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