NOVEL INTERPRETATION OF HIGH TEMPERATURE CREEP IN AN ODS Cu-ZrO_2 ALLOY

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The high temperature creep data for an ODS Cu-0.01ZrO_2 alloy reported by Nagorka et al. (Metall. Mater. Trans. A, *26A,* 1995, p. 873) are reinterpreted applying some microstructural data of the same authors. Two concepts of creep in this dispersion strengthened alloy are considered: (1) the true threshold stress concept and (2) the concept of thermally activated detachment of dislocations from interacting dispersed particles, specifically the model of creep presented by Rösler and Arzt – RA model (Acta Metall. Mater., *38,* 1990, p. 671). The creep data analysed cover relatively narrow interval (not exceeding three orders of magnitude) of the creep strain rates measured at two temperatures only. Nevertheless, the application of the true threshold stress concept led to the conclusion that the creep in the ODS Cu alloy is lattice-diffusion-controlled and the true stress exponent of creep rate n is close to that of creep in copper. The model of creep, controlled by thermally activated detachment of dislocations from fine interacting particles, provides perhaps an alternative but certainly far from satisfactory interpretation of creep in the alloy under consideration. The model of diffusional creep inhibited by the particles located at grain boundaries developed by Arzt, Ashby and Verrall – AAV model (Acta Metall., *31,* 1983, p. 1977) and accepted by Nagorka et al. was not found to be relevant to account for the creep behaviour of the alloy even at the creep strain rates below about 10^{-6} s^{-1}.

K e y w o r d s: ODS Cu alloys, high temperature creep, threshold-like creep behaviour, true threshold stress, thermally activated detachment

NOVÁ INTERPRETACE VYSOKOTEPLOTNÍHO CREEPU DISPERZNĚ ZPEVNĚNÉ SLITINY Cu-ZrO_2

Výsledky studia vysokoteplotního creepu mědi zpevněné disperzí částic oxidu ZrO_2 publikované Nagorkou aj. (Metall. Mater. Trans. A, *26A,* 1995, p. 873) jsou nově interpretovány za použití nezbytných mikrostrukturních dat týchž autorů. Jsou uvažovány dvě koncepce creepového chování disperzně zpevněných slitin: (1) koncepce skutečného prahového napětí a (2) koncepce tepelně aktivovaného odpoutávání dislokací od interagujících disperzních částic, jmenovitě model creepu předložený Röslerem a Arztem – RA model

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(Acta Metall. Mater., *38*, 1990, s. 671) a aplikovaný Nagorkou aj. Analýza creepových dat je poněkud omezena spolehlivostí těchto dat v důsledku úzkého intervalu (nepřekračujícího tři řády) měřených rychlostí creepu při pouhých dvou teplotách. Nicméně aplikace koncepce skutečného prahového napětí vede k závěru, že rychlost creepu v uvažované slitině je řízena mřížkovou difuzí v matrici a že skutečný napěťový exponent rychlosti creepu n je blízký hodnotě charakteristické pro čistou měď, to je 5. Model creepu řízeného tepelně aktivovaným odpoutáváním dislokací od interagujících částic vede k alternativní, i když sotva uspokojivé interpretaci creepu v uvažované slitině. Model difuzního creepu inhibitovaného částicemi situovanými na hranicích zrn předložený Arztem, Ashbym a Verrallem – AAV model (Acta Metall., *31*, 1983, s. 1977) a přijatý Nagorkou aj. nebyl shledán relevantním pro interpretaci creepového chování dané slitiny ani při rychlostech creepu nižších nežli asi 10^{-6} s^{-1}.

1. Introduction

The need of materials of desirable combination of high thermal conductivity and high temperature creep resistance have led to extensive investigations of oxide dispersion strengthened (ODS) copper alloys (e.g. Refs. [1–5]). These investigations have resulted in commercial development of copper alloys dispersion strengthened with fine γ-Al_2O_3 phase particles (e.g. Ref. [6]). The fine dispersion of alumina particles is produced by the internal oxidation of dilute copper-aluminium alloy powders [6]. The internally oxidized powder is subsequently consolidated, densified and reduced to the desired dimensions by proper deformation processing methods.

Beside Al_2O_3 particles, they are still other oxide particles potential to produce effective dispersion strengthening, specifically ZrO_2, Y_2O_3, Ce_2O_3 and Er_2O_3 [4]. Recently, the microstructure development and high temperature creep of copper dispersion strengthened with fine yttria particles as well as zirconia particles were investigated by Nagorka et al. [7, 8]. These authors preferred the splat-quenching to gas atomisation, since the former processing provides significantly higher cooling rates than the latter one. This is important, since such elements as yttrium and zirconium are practically insoluble in solid copper (which is not the case of aluminium). Besides, the internal oxidation of Cu-Y and Cu-Zr alloy ribbons produced by splat-quenching is more feasible than the internal oxidations of Cu-Y and Cu-Zr powders produced by gas atomisation. On the other hand, consolidation and densification of internally oxidized ribbons produced by splat-quenching is more complicated.

The aim of the present paper is to reinterpret the creep data presented by Nagorka et al. [8] for the ODS Cu-0.01ZrO_2 alloy (0.01 is the volume fraction of zirconia particles) denoted Z9 alloy by these authors. The ODS alloy was obtained by internal oxidation of an Cu-0.32Zr alloy (concentration of Zr in at.%). Due to the proper choice of the conditions of internal oxidation (1223 K and 43.2 ks)*

* Alternatively, the alloy Cu-0.32Zr as well as the alloy Cu-0.33Y were internally oxi-

practically, all the ZrO_2 particles of 6.2 nm in mean diameter were fairly homogeneously distributed in the copper matrix of mean grain diameter of 2.4 μm. From the volume fraction of particles, i.e. 0.01, and their mean diameter, the mean interparticle spacing was estimated to 44 nm [7].

The constant load creep tests in compression were conducted at the temperatures 923 and 973 K. During any test, the load (and thus the applied stress) was increased stepwise. At any stress, the time independent strain rate – "steady state creep rate" was measured and then the stress was increased again [8].

The "steady state creep strain rates" measured by this way ranged from $\sim 8.5 \times 10^{-8}$ to $\sim 2.3 \times 10^{-5}$ s^{-1} (see later, Table 1), i.e. in the interval not fully covering three orders of magnitude. The lower bound of measured strain rates was determined by equipment resolution limits and the upper bound by material availability [8].

2. The creep data analysed

To analyse the $\dot{\varepsilon}_{\rm m} = \dot{\varepsilon}_{\rm m}(\sigma, T)$ creep data ($\dot{\varepsilon}_{\rm m}$ is the minimum creep strain rate, σ is the applied compression stress and T is the temperature) and correlate them

Table 1. ODS Cu-0.01ZrO_2, Alloy Z9. Minimum creep strain rates $\dot{\varepsilon}_{\rm m}$ corresponding to various stresses σ at temperatures 923 and 973 K. Values of $\dot{\varepsilon}_{\rm m}$ and σ read from $\dot{\varepsilon}_{\rm m}$ vs. σ relations in Fig. 1c, Ref. [8]

923 K		973 K	
σ [MPa]	$\dot{\varepsilon}_{\rm m}$ [s^{-1}]	σ [MPa]	$\dot{\varepsilon}_{\rm m}$ [s^{-1}]
43.0	8.5×10^{-8}	31.2	1.0×10^{-7}
41.1	8.9×10^{-8}	30.0	1.2×10^{-7}
45.0	1.2×10^{-7}	32.5	1.5×10^{-7}
48.0	1.6×10^{-7}	33.2	1.7×10^{-7}
45.5	2.4×10^{-7}	35.0	2.6×10^{-7}
51.5	8.0×10^{-7}	37.0	8.5×10^{-7}
49.5	1.1×10^{-6}	38.5	1.05×10^{-6}
58.0	2.6×10^{-6}	38.7	2.0×10^{-6}
62.0	8.5×10^{-6}	41.5	3.1×10^{-6}
66.0	2.7×10^{-5}	45.0	6.7×10^{-6}
		47.0	1.4×10^{-5}
		49.0	2.3×10^{-5}

dized at the temperature of 1023 K (i.e. 200 K lower) for 42.3 ks and 10.3 ks, respectively. The resulting ODS alloys were denoted Z7 and Y7. The volume fractions of the oxide particles were 0.01ZrO_2 and 0.01Y_2O_3. However, in the alloys Z7 and Y7 only the volume fractions of ~ 0.0032 of ZrO_2 and ~ 0.0014 of Y_2O_3 were present as fine dispersion of particles of about 6 nm in diameter [7].

with likely physical models of creep, the numerical values of $\dot{\varepsilon}_m$ corresponding to various applied stresses and the temperatures under consideration (923 and 973 K) must be available. In Table 1, such numerical values read from $\dot{\varepsilon}_m$ vs. σ relations in Fig. 1c of the paper by Nagorka et al. [8] are listed. In Fig. 1, the values of $\dot{\varepsilon}_m$ are plotted against applied stress for both temperatures, i.e. 923 and 973 K. In double logarithmic co-ordinates, the $\dot{\varepsilon}_m$ vs. σ relations for both temperatures under consideration are linear in the relatively narrow interval of applied stresses. (In Fig. 1, R_c means the correlation coefficient). From the $\dot{\varepsilon}_m = \dot{\varepsilon}_m(\sigma, T)$ relations, the apparent stress exponent m_c and the apparent activation energy Q_c defined as

$$m_c = \left(\frac{\partial \ln \dot{\varepsilon}_m}{\partial \ln \sigma}\right)_T \text{ and } Q_c = \left[\frac{\partial \ln \dot{\varepsilon}_m}{\partial(-1/RT)}\right]_\sigma, \tag{1}$$

respectively, were determined and are listed in Table 2 (in the definition equation for Q_c, R means the gas constant). In Table 2 the apparent activation energy for $\sigma = 45$ MPa corrected for the temperature dependence of shear modulus G, Q_c^{corr} is

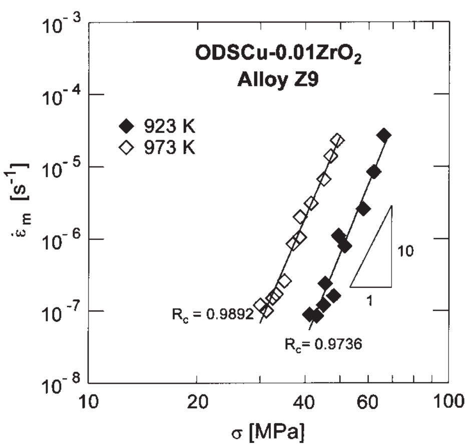

Fig. 1. Relations between minimum creep strain rates $\dot{\varepsilon}_m$ and applied stresses σ for temperatures 923 and 973 K in double logarithmic co-ordinates. R_c means the correlation coefficient. Data after Nagorka et al. [8] (see Table 1).

Table 2. ODS Cu-0.01ZrO$_2$, Alloy Z9. Values of the apparent stress exponent m_c, the apparent activation energy Q_c, the activation energy corrected for the temperature dependence of shear modulus, Q_c^{corr}. Values of the true threshold stress σ_{TH}, estimated accepting the true stress exponent $n = 5$, and the values of the ratio of the true threshold stress to the shear modulus, σ_{TH}/G

T [K]	m_c	Q_c [kJ·mol^{-1}]	Q_c^{corr} [kJ·mol^{-1}]*	σ_{TH} [MPa]	σ_{TH}/G
923	12.3	573	526	31.5	9.95×10^{-4}
973	11.3		523	21.9	7.10×10^{-4}

* $Q_c^{corr} = Q_c + m_c R(T^2/G)(dG/dT)$

also shown. The equation expressing Q_c^{corr} is given in the footnote of Table 2. The values of m_c as well as the value of Q_c^{corr} are close to those presented by Nagorka et al. [8] in their Fig. 1c.

The values of the mean ZrO_2 particle diameter (d_p) as well as the mean inter-particle spacing (λ) were mentioned in Section 1 of the present paper.

3. Analysis and discussion

3.1 Interpretation of creep in terms of the true threshold stress

From Table 2 it follows that the apparent stress exponent of creep strain rate in ODS Cu alloy is much higher than the value of the true stress exponent of creep strain rate in pure copper (reported in [9] as 4.8) and the apparent activation energy Q_c^{corr} much higher than the activation enthalpy of lattice self-diffusion in copper (reported in [9] as 197.0 $kJ \cdot mol^{-1}$). The anomalously high values of m_c and Q_c strongly suggest threshold-*like* creep behaviour.

The *true* threshold creep behaviour is characterized by apparent stress exponent m_c increasing with decreasing applied stress. The true threshold stress σ_{TH} (which is applied-stress-independent by definition), then represents the applied stress below which the creep does not take place at all or, at least, does not take place by the same mechanism as above it.

Usually, the true threshold stress is estimated using the well known linear extrapolation method and assuming a proper value of the true stress exponent n (e.g. [10, 11]). In the present analysis it is quite natural to consider $n = 5$, i.e. the value of n close to that reported for pure copper [9]. In Fig. 2, values of $\dot{\varepsilon}_m^{1/n}$, where $n = 5$, i.e. $\dot{\varepsilon}_m^{1/5}$, are plotted against applied stress σ in double linear co-ordinates. It can be seen that the data points can be satisfactorily fitted by straight lines. Extrapolating these straight lines to $\dot{\varepsilon}_m = 0$, values of the true threshold stress σ_{TH} are obtained; they are given in the Figure as well as in Table 2. In this table, also the values of the σ_{TH}/G ratio (G is the shear modulus of copper [9]) are given. As expected, this ratio is lower at the higher testing temperature; apparently the true threshold stress decreases with increasing temperature more strongly than the shear modulus. Such a true threshold stress behaviour is commonly observed [11, 12].

In Fig. 3, the normalized minimum creep strain rates $\dot{\varepsilon}_m b^2/D_L$ are plotted against normalized effective stresses $(\sigma - \sigma_{TH})/G$; b is the length of the Burgers vector in copper and D_L is the coefficient of lattice self-diffusion in copper [9]. The data points can be fitted by a single straight line; the slope of this line is only slightly smaller than 5, namely 4.63, i.e. the above assumed value of the true stress exponent n. The fact that the data points can be fitted by a single straight line suggests the lattice self-diffusion in the alloy matrix as a creep strain rate controlling process.

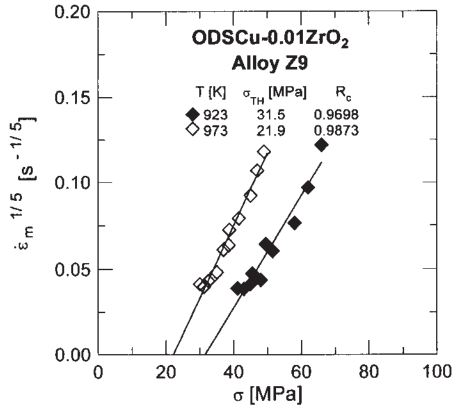

Fig. 2. Relations between $\dot{\varepsilon}_{\mathrm{m}}^{1/n}$ vs. σ in double linear co-ordinates. The true stress exponent of creep strain rate, n, is taken equal to 5 (cf. Ref. [9]).

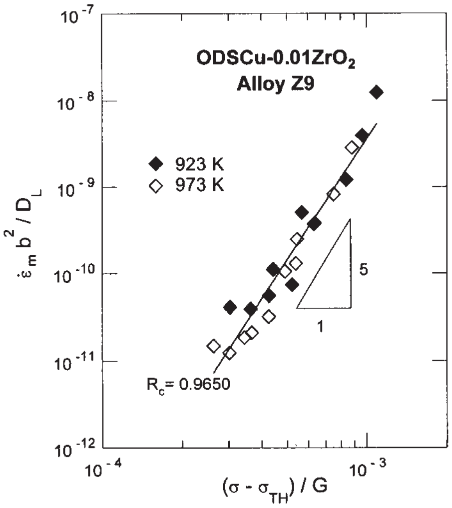

Fig. 3. The normalized minimum creep strain rates $\dot{\varepsilon}_{\mathrm{m}} b^2/D_{\mathrm{L}}$ plotted against normalized effective stresses $(\sigma - \sigma_{\mathrm{TH}})/G$ in double logarithmic co-ordinates. The data points can be fitted by a single straight line, the slope of this line is close to the value of the true stress exponent $n = 5$.

With respect to the narrow range of measured minimum creep strain rates as well as the scatter of $\dot{\varepsilon}_{\mathrm{m}}(\sigma, T)$ data, the result of the above analysis is certainly satisfactory: the creep in the alloy under consideration can be interpreted in terms of the true threshold stress concept.

An identification of the origin of the true threshold stress is straightforward: the threshold stress is due to athermal detachment of dislocations from fine ZrO_2 particles. Arzt and Wilkinson [13] and Arzt and Rösler [14] expressed the stress σ_{d} necessary to detach a dislocation from an interacting particle as

$$\sigma_{\mathrm{d}} = \sigma_{\mathrm{OB}}\sqrt{1 - k_{\mathrm{R}}^2}, \tag{2}$$

where σ_{OB} is the Orowan bowing stress and k_{R} is the relaxation factor that characterizes the diffusional relaxation of dislocation line energy at the particle-matrix interface. The value of k_{R} generally varies from 0 (total line energy relaxation) to 1 (no line energy relaxation). From Eq. (2) it follows that unless the factor k_{R} increases with increasing temperature the detachment stress σ_{d} scales the shear modulus G, since [15]

$$\sigma_{\mathrm{OB}} = \frac{0.84MGb}{\lambda - d_{\mathrm{p}}}, \tag{3}$$

where M is the Taylor factor; λ and d_{p} were defined above. Consequently, since the true threshold stress σ_{TH} decreases with increasing temperature more strongly than the shear modulus G, it cannot be directly identified with the detachment stress σ_{d} as expressed by Eq. (2). However, this is not of a primary importance in the present analysis (see e.g. [16–18]).

Accepting the true threshold stress concept and taking into account the result presented in Fig. 3, the temperature and applied stress dependence of the minimum creep strain rate can be expressed as (see e.g. [10, 11])

$$\frac{\dot{\varepsilon}_{\mathrm{m}} b^2}{D_{\mathrm{L}}} = A \left(\frac{\sigma - \sigma_{\mathrm{TH}}}{G} \right)^n, \tag{4}$$

where A is a dimensionless constant. The potentiality of this equation to describe adequately the true threshold creep behaviour of dispersion strengthened alloys is supported by numerous results, specifically the results obtained investigating creep of an Al-8.5Fe-1.3V-1.7Si alloy at temperatures ranging from 673 to 723 K and a broad interval of applied stresses (measured minimum creep strain rates covering six orders of magnitude [19]).

In Fig. 4, relations between the normalized minimum creep strain rate $\dot{\varepsilon}_{\mathrm{m}} b^2/D_{\mathrm{L}}$ and normalized applied stress σ/G are shown in double logarithmic co-ordinates. The data points for 923 K and 973 K do not fit a single straight line. Within the scatter limits the relations are linear. At any value of σ/G the difference between $\dot{\varepsilon}_{\mathrm{m}} b^2/D_{\mathrm{L}}$ for 973 K and $\dot{\varepsilon}_{\mathrm{m}} b^2/D_{\mathrm{L}}$ for 923 K can be accounted for either by the contribution of temperature dependence of the true threshold stress to the apparent activation energy of creep, or by the activation energy of thermally activated detachment of dislocations from the interacting ZrO_2 phase particles (see Section 3.2), depending on whether the true threshold stress concept or the RA creep model is relevant.

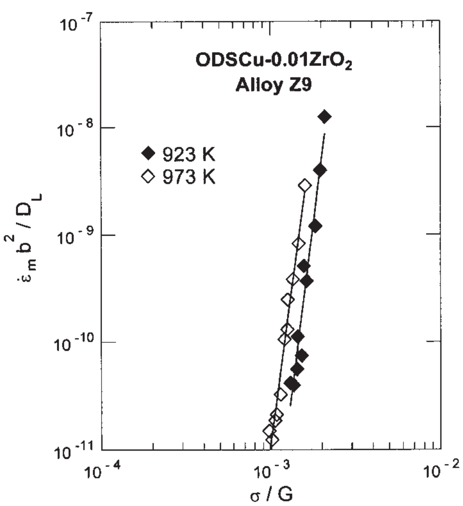

Fig. 4. Relations between the normalized creep strain rates $\dot{\varepsilon}_{\mathrm{m}} b^2/D_{\mathrm{L}}$ and the normalized applied stresses σ/G in double logarithmic co-ordinates.

Combining definition equation for Q_c, Eq. (1), with Eq. (4) the following expression is obtained

$$Q_\mathrm{c} = \Delta H_\mathrm{L} - \frac{nRT^2}{G}\left(\frac{G}{\sigma - \sigma_\mathrm{TH}}\frac{\mathrm{d}\sigma_\mathrm{TH}}{\mathrm{d}T} + \frac{n-1}{n}\frac{\mathrm{d}G}{\mathrm{d}T}\right), \tag{5}$$

where ΔH_L is the activation enthalpy of lattice self-diffusion in copper, i.e. 197.0 $\mathrm{kJ\cdot mol^{-1}}$ [9]. Thus, the contribution of temperature dependence of σ_TH to Q_c, denoted $\Delta Q_\mathrm{c}^\mathrm{th}$, is expressed as

$$\Delta Q_\mathrm{c}^\mathrm{th} = -\frac{nRT^2}{\sigma - \sigma_\mathrm{TH}}\frac{\mathrm{d}\sigma_\mathrm{TH}}{\mathrm{d}T}. \tag{6}$$

At $\sigma/G = 1.5 \times 10^{-3}$, $\Delta Q_\mathrm{c}^\mathrm{th} = 318\ \mathrm{kJ\cdot mol^{-1}}$. The contribution $-[(nRT^2/G)(n-1)/n]\mathrm{d}G/\mathrm{d}T$ amounts to $\sim 16\ \mathrm{kJ\cdot mol^{-1}}$ and thus $Q_\mathrm{c}^\mathrm{corr} = 197.0 + 318 + 16 = 531\ \mathrm{kJ\cdot mol^{-1}}$ in excellent agreement with the value of Q_c following from the $\dot{\varepsilon}_\mathrm{m}(\sigma, T)$ creep data, Fig. 1, and corrected for the temperature dependence of shear modulus, i.e. $Q_\mathrm{c}^\mathrm{corr} \sim 525\ \mathrm{kJ\cdot mol^{-1}}$, Table 2.

3.2 Interpretation of creep in terms of the Rösler-Arzt (RA) model

Because of the narrow interval of measured minimum creep strain rates the possibility that the creep strain rate in the alloy under consideration is controlled by thermally activated detachment of dislocations from fine ZrO_2 particles cannot be a priori excluded. Nagorka et al. [8] suggested the thermally activated detachment of dislocations from fine ZrO_2 particles as the creep strain rate controlling process at creep strain rates above about $10^{-6}\ \mathrm{s^{-1}}$ (see Fig. 6c in Ref. [8]). However, these authors satisfied themselves with an estimation of the value of relaxation factor k_R applying Eqs. (A6) and (A7) [8] developed by Rösler and Arzt [20] – RA model. Thus, their only argument for the thermally activated detachment mechanism consists in a (temperature independent) value of the relaxation factor $k_\mathrm{R} = 0.80$, i.e. the value, which seems reasonable.

According to the RA model [20], the temperature and applied stress dependence of the minimum creep strain rate can be expressed as

$$\frac{\dot{\varepsilon}_\mathrm{m} b^2}{D_\mathrm{L}} = C \exp\left\{-\frac{Gb^2 d_\mathrm{p}\left[(1-k_\mathrm{R})(1-\sigma/\sigma_\mathrm{d})\right]^{3/2}}{2kT}\right\}, \tag{7}$$

where

$$C = 6\lambda b\rho \tag{8}$$

is the dimensionless structure factor, ρ is the density of mobile dislocations. Assuming the mobile dislocation density $\rho = 10^{14}$ m^{-2}, the factor C calculated from the structure data, Eq. (8), and further denoted C_s is equal to 6.75×10^{-3}.

To correlate the $\dot{\varepsilon}_\mathrm{m}(\sigma, T)$ creep data (Table 1) with predictions of the RA model, the value of the relaxation factor k_R obtained by Nagorka et al. [8] will be accepted. For details of the correlation procedure see e.g. Ref. [16].

If the relaxation factor is temperature independent, the values of the detachment stress σ_d at various testing temperatures and a constant applied stress σ can be estimated solving the equation [16]

$$\left(\frac{\sigma}{\sigma_\mathrm{d}}\right)^3 - \left(\frac{\sigma}{\sigma_\mathrm{d}}\right)^2 = -\frac{K(T)^2}{(1-k_\mathrm{R})^3}, \tag{9}$$

where $K(T) = 4kTm_\mathrm{c}/3Gb^2d_\mathrm{p}$. The values of the detachment stress σ_d for an applied stress $\sigma = 45$ MPa are estimated to ~ 221 MPa for 923 K as well as 973 K. Using this value of σ_d and $k_\mathrm{R} = 0.80$, the experimental $\dot{\varepsilon}_\mathrm{m}(\sigma, T)$ creep data plotted in $\dot{\varepsilon}_\mathrm{m}b^2/D_\mathrm{L}$ vs. σ/G co-ordinates are compared with the model equation, Eq. (7), in Fig. 5. From the Figure it may seem that the prediction of the RA model is in satisfactory agreement with the experimental $\dot{\varepsilon}_\mathrm{m}(\sigma, T)$ creep data. However, the differences of the values of the structure factor C following from the fitting procedure and the value of this factor calculated from the structure data, Eq. (6), i.e. $C = C_\mathrm{s}$ is significant. In fact, while $C_\mathrm{s} = 6.75 \times 10^{-3}$, the values of C are equal to $\sim 6.7 \times 10^3$ at 923 K and $\sim 6.2 \times 10^3$ at 973 K at $\sigma = 45$ MPa. Thus the values of C following from the fitting procedure are in average by a factor of $\sim 10^6$ greater than the value of C_s calculated from the structure data, Eq. (8).

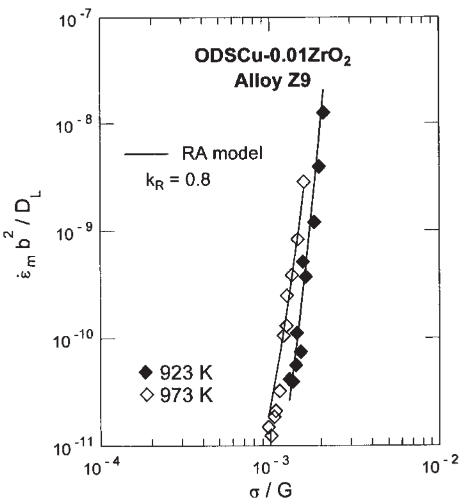

Fig. 5. Comparison of the Rösler-Arzt model with the experimental creep data. The relaxation factor k_R independent of temperature and equal to 0.80 is accepted.

Alternatively, the activation energy of detachment which is expressed as (cf. Eq. (7)).

$$Q_\mathrm{d} = \frac{1}{2}Gb^2d_\mathrm{p}\left[(1-k_\mathrm{R})\left(1-\frac{\sigma}{\sigma_\mathrm{d}}\right)\right]^{3/2} \tag{10}$$

can be calculated and compared with the activation energy estimated from the $\dot{\varepsilon}_m b^2/D_L$, σ/G creep data presented in Fig. 4 (cf. Section 2). The energy Q_d calculated by means of Eq. (10) for the applied stress of 45 MPa amounts in average to 243 kJ·mol^{-1}. This value of Q_d should be close to the energy estimated from the difference of $\dot{\varepsilon}_m b^2/D_L$ for 973 K and 923 K at $\sigma/G = 1.5 \times 10^{-3}$, i.e. 318 kJ ·mol^{-1}. However, the difference of this value and that of the activation energy Q_d amounts to 75 kJ·mol^{-1}. This difference is significant and suggests that the prediction of the RA model is less likely than the prediction based on the concept of the true threshold stress (see Section 3.1), especially if the difference of the structure factor C_s calculated from the structure data, Eq. (8), and this factor following from the above fitting procedure, C (Fig. 5), is taken into consideration. Thus, the conclusion of Nagorka et al. [8] that the creep strain rate in their Z9 alloy at the creep strain rates above 10^{-6} s^{-1} is controlled by thermally activated detachment of dislocations from fine ZrO_2 particles can be hardly accepted.

4. Additional remarks

1. The test technique used by Nagorka et al. [8] to measure strain rates at various loads (and/or various applied stresses) cannot fully substitute the constant stress creep test technique and measurement of the minimum (and/or steady state) creep strain rates. The authors did not present any example of their strain--time relations. Thus, it is hardly possible to appreciate the scatter of the $\dot{\varepsilon}(\sigma, T)$ data points presented in their Fig. 1c (cf. Fig. 1 in the present paper). With the strain rates close to the lower bound determined by the equipment resolution limit, i.e. the strain rates close to 10^{-7} s^{-1}, a large scatter is associated. Perhaps, it was just this scatter that made Nagorka et al. [8] to suggest that the creep at the strain rates below $\sim 10^{-6}$ s^{-1} is due to the diffusional creep mechanism inhibited by the fine ZrO_2 phase particles located at grain boundaries and to accept the Arzt-Ashby-Verrall (AAV) model [21] (Fig. 6) (see also Refs. [18, 22]). The present authors believe that it is hardly justified to divide the narrow strain rates interval (not fully covering three orders of magnitude) into

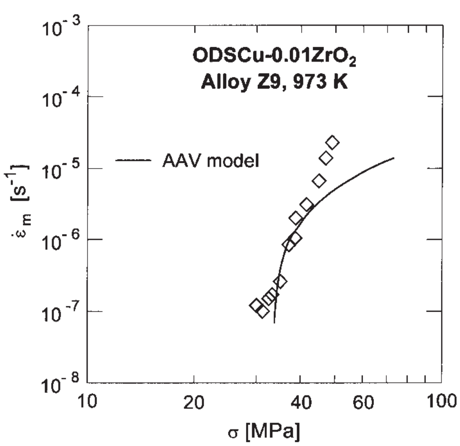

Fig. 6. The $\dot{\varepsilon}_m(\sigma, T)$ creep data presented in the double logarithmic $\dot{\varepsilon}_m$ vs. σ co-ordinates compared with the prediction of the Arzt-Ashby-Verrall (AAV) model of diffusional creep inhibited by fine particles located at grain boundaries (after Nagorka et al. [8]).

intervals above and below the strain rate of about 10^{-6} s^{-1}. This present authors' belief is supported by the results of correlations presented in Fig. 3, which illustrates that the concept of athermal detachment of dislocations from fine ZrO_2 phase particles provides good result. This is why the diffusional creep inhibited by fine particles at grain boundaries at the creep strain rates below $\sim 10^{-6}$ s^{-1} is rejected by the present authors. In fact, Fig. 6, in which the $\dot{\varepsilon}_{\mathrm{m}}(\sigma)$ creep data are compared with the prediction of AAV model, hardly supports the interpretation of Nagorka et al. [8] of the creep behaviour below about 10^{-6} s^{-1}.

2. Nevertheless, the principal difficulty of an unambiguous interpretation of the experimental $\dot{\varepsilon}_{\mathrm{m}}(\sigma, T)$ creep data presented by Nagorka et al. [8] consists in a very narrow interval of measured minimum creep strain rates (and/or very narrow interval of the applied stresses) and, of course, in the fact that the minimum creep strain rates were measured at two temperatures only, namely at homologous temperatures T/T_{m} (T_{m} is the melting temperature of copper in K) of 0.68 and 0.72. In creep of ODS Al-Al_2O_3 alloy the true threshold creep behaviour was proved [23] at temperatures at least up to 723 K, i.e. up to the homologous temperature of 0.77. From this comparison the present authors suggest that the homologous temperature of 0.72 is too low for the thermally activated detachment of dislocations from the interacting ZrO_2 phase particles in the ODS Cu-0.01ZrO_2 alloy can be reasonably expected. Therefore, they prefer the concept of athermal detachment of dislocations from dispersed particles (the true threshold stress concept) to the concept of thermally activated detachment of dislocations from the particles (the RA model of creep) at the external conditions of temperatures and applied stresses under consideration.

3. It is worth pointing out that, recently, Broyles et al. [5] published the experimental $\dot{\varepsilon}_{\mathrm{m}}(\sigma, T)$ creep data for a commercial GlidCop Al-15, i.e. ODS Cu--0.007Al_2O_3 alloy, the number 0.007 means the volume fraction of alumina particles (the mean particle diameter was $\sim$ 10 nm), from which the data for 774 K and 977 K are of special interest. At these temperatures, the measured creep strain rates covered five orders of magnitude. The relations between $\dot{\varepsilon}_{\mathrm{m}}$ and σ for these temperatures suggest the true threshold creep behaviour: the apparent stress exponent m_{c} (Eq. (1)) obviously increases with decreasing applied stress at least at creep strain rates below 10^{-6} s^{-1} (see Fig. 2 in Ref. [5]). Nevertheless, the authors [5] preferred to interpret their $\dot{\varepsilon}_{\mathrm{m}}(\sigma, T)$ creep data in terms of the thermally activated detachment of dislocations from fine alumina particles. They estimated the relaxation factor k_{R} to $\sim$ 0.90. This relatively high value of k_{R} the authors accounted for by their observation that the particle/matrix interface is partially coherent in ODS Cu-Al_2O_3 alloys.

4. Very recently, Rösler and Bäcker [24] analysed possible creep strengthening of metallic alloys at high temperatures due to the dispersion strengthening and the whisker (or short fibre) reinforcement. Dispersion strengthening is caused by

an attractive dislocation/particle interaction. In conjunction with reinforcement strengthening the authors have discussed the strengthening by load transfer and generation of geometrically necessary dislocations, and relaxation of load transfer by diffusion causing weakening of the composite. Modeling the creep behaviour of materials strengthened by combination of dispersion and reinforcement the authors presented a steady state creep equation assuming that deformation of the matrix is controlled by thermally activated dislocation detachment and reinforcement strengthening is due to the load transfer mechanism. The authors' theoretical analysis predicts that the synergistic interaction between fine particles and whiskers (short fibres) leads to superior high temperature creep strength. Just copper is the metal most suitable for the above "dual scale particle strengthening".

The aim of this last remark is to encourage experimental investigations of ODS copper reinforced with proper whiskers or short fibres. Along with Rösler and Bäcker [24], it should be pointed out that "dual scale particle strengthened copper is not only a nice model system. There are also a number of potential applications…".

5. Summary and conclusions

In the present paper the experimental $\dot{\varepsilon}_{\mathrm{m}}(\sigma, T)$ creep data presented by Nagorka et al. for an ODS Cu-0.01ZrO$_2$ – Z9 alloy, are reinterpreted alternatively in terms of (i) true threshold stress concept, i.e. the concept of *athermal* detachment of dislocations from fine interacting particles, and (ii) concept of *thermally* activated detachment of dislocations from such particles. The main conclusions can be expressed as follows:

1. Considering the true threshold stress concept, the creep strain rate in the alloy under consideration was found to be lattice self-diffusion controlled and the true stress exponent of minimum creep strain rate to be close to that for pure copper.

2. Considering the concept of thermally activated detachment, the creep behaviour of the alloy was tolerably correlated with the Rösler-Arzt model accepting the relaxation factor $k_{\mathrm{R}} = 0.80$ that follows from the $\dot{\varepsilon}_{\mathrm{m}}(\sigma, T)$ creep data. The difference between the structure factor obtained from the fitting procedure and that calculated using the structure data amounts to 6 orders of magnitude. The activation energy of detachment of dislocations from interacting particles Q_{d} was estimated to 243 kJ$\cdot$mol^{-1}, i.e. to the value by 75 kJ$\cdot$mol^{-1} lower than that following from the experimental $\dot{\varepsilon}_{\mathrm{m}}(\sigma, T)$ creep data. These differences seem to disqualify the validity of this model in the whole interval of creep strain rates as measured at temperatures 923 and 973 K.

3. Since the $\dot{\varepsilon}_{\mathrm{m}}(\sigma, T)$ creep data in the whole region of measured creep strain rates can be well interpreted in terms of the true threshold creep behaviour, the interpretation of these data for $\dot{\varepsilon}_{\mathrm{m}} \leq 10^{-6}$ s^{-1} in terms of the Arzt-Ashby-Verrall

model of diffusional creep inhibited by fine ZrO_2 phase particles located at grain boundaries was rejected.

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REFERENCES

[1] KOSKI, J. A.—BOYD, R. D.—KEMPKA, S. M.—ROMIG, A. D.—SMITH, M. F.—WATSON, R. D.—WHITLEY, J. B.: J. Nucl. Mater., *121*, 1984, p. 309.
[2] ROSENWASSER, S. N.—STEVENSON, R. D.—LISTVINSKI, G.—VRABLE, D. L.—McGREGOR, J. E.—NIR, N.: J. Nucl. Mater., *122–123*, 1984, p. 1107.
[3] ENGLISH, C. A.—MAZEY, D. L.: Nuclear Energy, *29*, 1990, p. 67.
[4] NAGORKA, M. S.—LEVI, C. G.—LUKAS, G. E.—RIDDER, S. D.: Mater. Sci. Eng., *A142*, 1991, p. 277.
[5] BROYLES, S. E.—ANDERSON, K. R.—GROZA, J. R.—GIBELING, J. C.: Metall. Mater. Trans. A, *27A*, 1996, p. 1217.
[6] ROBLES, J.—ANDERSON, K. R.—GROZA, J. R.—GIBELING, J. C.: Metall. Mater. Trans. A, *25A*, 1994, p. 2235.
[7] NAGORKA, M. S.—LEVI, C. G.—LUKAS, G. E.: Metall. Mater. Trans. A, *26A*, 1995, p. 859.
[8] NAGORKA, M. S.—LUKAS, G. E.—LEVI, C. G.: Metall. Mater. Trans. A, *26A*, 1995, p. 873.
[9] FROST, H. J.—ASHBY, M. F.: Deformation Mechanism Maps—The Plasticity and Creep of Metals and Ceramics. Oxford, Pergamon Press 1982, p. 21.
[10] ČADEK, J.—OIKAWA, J.—ŠUSTEK, V.: Mater. Sci. Eng., *A190*, 1995, p. 9.
[11] ČADEK, J.—KUCHAŘOVÁ, K.—MILIČKA, K.—ZHU, S. J.: Kovove Mater., *37*, 1999, p. 213.
[12] ČADEK, J.—KUCHAŘOVÁ, K.—ZHU, S. J.: Kovove Mater., *38*, 2000, p. 130.
[13] ARZT, E.—WILKINSON, D. S.: Acta Metall., *34*, 1986, p. 1893.
[14] ARZT, E.—RÖSLER, J.: Acta Metall., *36*, 1988, p. 1053.
[15] KOCKS, U. F.: Philos. Mag., *13*, 1966, p. 541.
[16] ČADEK, J.—KUCHAŘOVÁ, K.—ZHU, S. J.: Mater. Sci. Eng., *A283*, 2000, p. 172.
[17] ČADEK, J.—KUCHAŘOVÁ, K.—ZHU, S. J.: Kovove Mater., *39*, 2001, p. 77.
[18] ČADEK, J.—KUCHAŘOVÁ, K.—ZHU, S. J.: Kovove Mater., *39*, 2001, p. 221.
[19] ZHU, S. J.—KUCHAŘOVÁ, K.—ČADEK, J.: Metall. Mater. Trans. A, *31A*, 2000, p. 2229.
[20] RÖSLER, J.—ARZT, E.: Acta Metall. Mater., *38*, 1990, p. 671.
[21] ARZT, E.—ASHBY, M. F.—VERRALL, R. A.: Acta Metall., *31*, 1983, p. 1977.
[22] KUCHAŘOVÁ, K.—ČADEK, J.—ZHU, S. J.: Kovove Mater., *40*, 2002, p. 69.
[23] ČADEK, J.—ZHU, S. J.—MILIČKA, K.: Mater. Sci. Eng., *A252*, 1998, p. 1.
[24] RÖSLER, J.—BÄCKER, M.: Acta Mater., *48*, 2000, p. 3553.

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