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Steady-state creep characteristics of Ni–22 at % Cu alloy at high and intermediate temperature

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The creep deformation characteristics of Ni–22 at % Cu solid solution alloy have been investigated in the temperature range of 595 to 1015 K (0.36 to 0.64 T_m) and under constant stress condition of 10 to 392 MPa. Using the effective diffusivity, $D_{eff} = f_L D_L + f_P D_P$, the dependence of steady-state creep rate, $\dot{\epsilon}_s$, on the stress has been analysed and found to be explained with the equation

$$\dot{\epsilon}_s = \alpha \frac{E\mathbf{b}}{kT}\left[D_L + 8.5\left(\frac{\sigma}{E}\right)^{1.5} D_P\right]\left(\frac{\sigma}{E}\right)^3$$

where α is a numerical constant, E is the Young's modulus, **b** is Burger's vector, D_L and D_P are the diffusivities of lattice and pipe diffusion, respectively. From this equation it is predicted that at the high temperature and low stress, the contribution for the creep deformation of lattice diffusion becomes predominant to give the stress exponent value of 3. On the other hand at lower temperature and higher stress, pipe diffusion has a strong influence on the deformation to make the exponent value 4.5. The measured creep deformation activation energy is found to be dependent on the temperature and stress applied. At high temperature (above 0.7 T_m) the activation energy for the deformation approaches to the value of lattice diffusion and at low temperature (below 0.4 T_m) the creep deformation energy becomes similar to that for the pipe diffusion. Under the same temperature condition the activation energy is observed to be decreasing with the applied stress.

1. Introduction

Since for most crystalline materials the values of the activation energies and the activation volumes of high temperature creep deformation are identical with those of self diffusion, there has been encouragement to develop the tenable theories of creep deformation in terms of the lattice self diffusion. However, for low temperature creep deformation, many suspected phenomena were supposed to explain the reported low activation energies for creep, i.e. 40 to 80% of the lattice self diffusion values.

Because there is no significant difference in creep behaviour between high ($\geqslant$ 0.6 to about 0.7 T_m) temperatures many attempts have been made to predict the creep behaviour at the inter-

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mediate temperatures using the data obtained at the high temperatures. Namely, the effective diffusivity concept [1] was introduced based on the assumption that the creep deformation at both high and intermediate temperatures is controlled by the same mechanism, diffusion, but only the diffusion path changes from a lattice to a dislocation core.

After Barret *et al.* [2] reported that the creep deformation of copper is controlled by pipe diffusion in the temperature range of 0.4 to 0.64 T_m, Robinson and Sherby, in 1969, had some success in explaining lower creep activation energy at the intermediate temperature than that of self diffusion by simply introducing the effective diffusivity term into the existing theory. However, in their adapted effective diffusivity concept, they assumed that the dislocation density remained constant with stress.

In 1975, after Weertman [4] announced at a symposium held in the memory of J. E. Dorn, the fact that all the existing theories predict a third power stress dependence of the creep rate in the absence of *ad hoc* assumptions and do not agree well with experimental results of pure metals, an attempt to explain larger stress exponent values by incorporating the effect of pipe diffusion was made by Evans *et al.* [5]. These investigators insist that their model provides good agreement with the experimental observation for a number of common materials. Their theoretical equation has the same form as Nabarro's creep equation [6] obtained using the idea of "short circuit diffusion". Recently, considering motion of dislocation, curvature of dislocation and local force, Spingarn *et al.* [7] also derived an equation for creep similar to that of Evans and Knowles [8]. Such models contain the experimental fact that the stress exponent must be larger by a factor of two when the pipe diffusion process predominates over the lattice diffusion.

For Cu–Ni alloys, Brebec *et al.* [9] confirmed that the activation energy for creep was different from that for self diffusion. This experimental evidence shows that the creep deformation mechanism of Cu–Ni alloys may not be controlled by the lattice self diffusion process only. The purpose of this study is to investigate the creep behaviour of the Ni–Cu alloy at high and intermediate temperatures for the further interpretation of the role of "short circuit diffusion" on the creep deformation mechanism.

2. Experimental procedure

The alloy was prepared by melting electrolytic nickel and copper of purity not lower than 99.9%. The slabs were homogenized for 12 h at 1173 K and cold-rolled into sheets of 1 mm thickness. Tensile specimens with a gauge length of 25 mm and a width of 4 mm were machined and subsequently annealed at 1073 K for 2 h in vacuum to give an average grain size of about 0.025 mm. The chemical composition of the alloy is Cu: 21.5 at %, Ni: 78.2 at %, Mn: 0.189 at %, Fe: 0.027 at %, Pb: 0.002 at %, Co: 0.144 at %.

The creep tests were conducted using a creep machine equipped with an Andrade–Chalmers constant stress arm. All creep tests were conducted in air. The extension of the specimen was measured by a Schaevitz model HR1000 LVDT with an accuracy of 0.5×10^{-6} m. The steady-state creep rate was measured from the slope of the secondary or steady-state region. The test temperature ranged from 595 K (0.36 T_m) to 1050 K (0.64 T_m) and the stress levels ranged from 10 to 392 MPa.

3. Experimental results and discussion

3.1. Creep curves and transient creep behaviour

All the creep curves obtained from low to high stress tests (Fig. 1) show the typical form of that for the class II type alloys. In Fig. 2, the schematic drawing of the stress change tests is illustrated. Regardless of the value of stress and temperature conducted, the transient behaviour of the alloy was similar in that normal primary creep was observed with increasing stress. Specimens were also unloaded after they had undergone creep at a certain strain in the steady-state and left for 3 h at the test temperature to see of there is any possibility of the formation of an atmosphere around the dislocations. On reloading the specimen to the original stress level, a normal transient primary creep deformation was observed again in both regions of the different stress exponent, i.e. $n = 3$ for low stress and high temperature and $n = 4.5$ for high stress and low temperature conditions (see Fig. 3; this will be discussed later).

Considering the above mentioned experimental results, one may think that the dislocation glide process may not be the controlling mechanism but that the diffusion assisted dislocation climb process may be the possible controlling mechanism but that the diffusion assisted dislocation

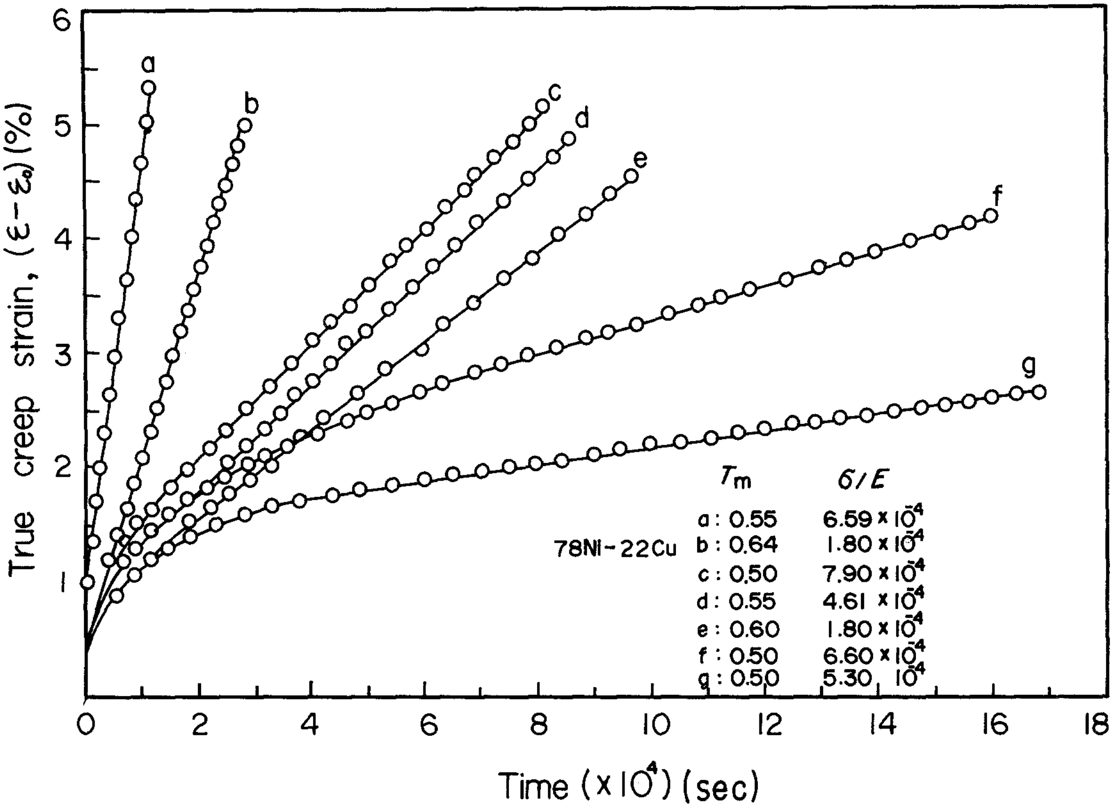

Figure 1 Typical creep curves of 78Ni–22Cu alloy.

climb process may be the possible controlling mechanism for deformation. On the basis of this thought, the diffusion assisted process will be considered throughout this paper.

3.2. Diffusivity and development of creep rate equation

The effective diffusivity concept was suggested by Hart [1] originally to explain anomalies in low temperature diffusion experiments. He expressed the virtual effective diffusivity as follows:

$$D_{\mathrm{eff}} = f_{\mathrm{L}} D_{\mathrm{L}} + f_{\mathrm{P}} D_{\mathrm{p}} \qquad (1)$$

where f_{L}, D_{L} and f_{P}, D_{P} are the atomic fraction and diffusivity related to the lattice and pipe diffusion, respectively. The fraction f_{P} is assumed [10] to be equal to $(n/N)\rho$, where n is the number of atoms in a dislocation core of atomic length, N is the number of atoms per unit area and ρ is the dislocation density.

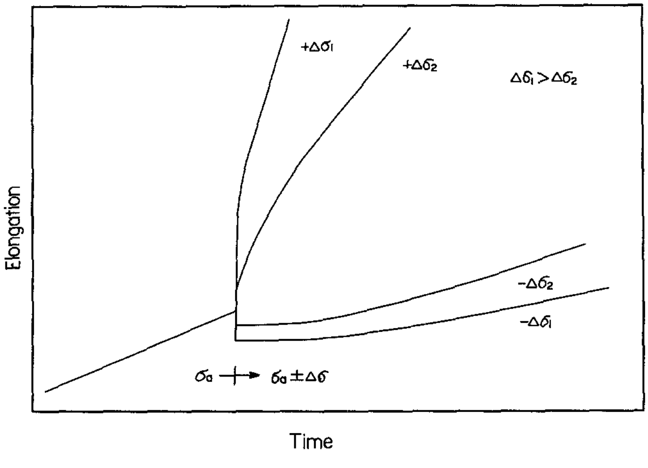

Figure 2 Schematic drawing of the transient creep behaviour upon small stress change.

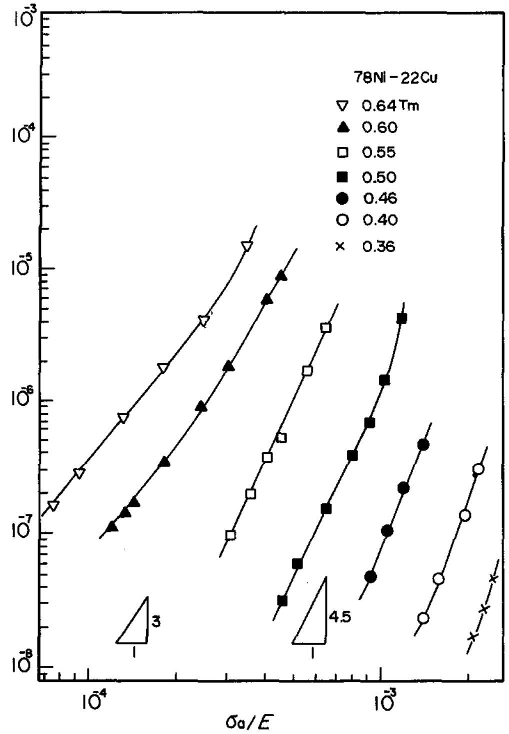

Figure 3 Steady-state creep rate of 78Ni–22Cu alloy as function of modulus corrected stress.

For Ni–Cu alloys, the steady-state dislocation density is experimentally observed to be a function of the applied stress as [11, 12]

$$\rho = \rho_0(\sigma/E)^{n'}. \qquad (2)$$

Jones and Sellars [11] reported $n' = 1.7$ for Ni–10Cu alloy and, Hong and Nam [12] experimentally measured n' to be 1.44 for Ni–30Cu alloy in the same stress and temperature conditions as conducted in this investigation. On the basis of the above mentioned reports, if we assume that n' for this work is about 1.5, the effective diffusivity in Equation 1 may be expressed as,

$$D_{\mathrm{eff}} = f_{\mathrm{L}}D_{\mathrm{L}} + \beta(\sigma/E)^{1.5}D_{\mathrm{P}} \qquad (3)$$

where f_{L} is known to be about unity and β is a numerical constant which is expressed as $(n/N)\rho_0$. To get the value of D_{eff} as a function of temperature and stress one has to know those values of D_{L} and D_{P} for various temperatures. For a binary solid solution, the appropriate diffusion coefficient for climb-controlled creep is shown by Chin *et al.* [13] as

$$D_{\mathrm{cl}} = \frac{D_1^*D_2^*}{D_1^*X_2 + D_2^*X_1} \qquad (4)$$

where D^* and X are the radioactive tracer diffusion coefficient and mole fraction, respectively. To solve Equation 4, the radioactive diffusivities of Ni^{63} and Cu^{64}, are used. These values were obtained experimentally by Monma *et al.* [14] for Cu–Ni alloys at the temperature range of 0.78 to about $0.9\,T_{\mathrm{m}}$. For this work these high temperature values are extrapolated to the test temperatures. Since this diffusion coefficient for climb may be considered for the lattice diffusion coefficient we replace D_{cl} for D_{L}, and the calculated values of D_{cl} for various temperatures are shown in Table I.

In order to calculate the value of pipe diffusivity of the alloy, it is necessary to know the detailed analysis of the pipe diffusion mechanism contributing to dislocation climb, namely, the diffusion of solute and solvent atoms along the dislocation core and the interaction of solute atoms with vacancies. However, no detailed mechanism for this has yet been developed. Therefore, we have used the results obtained by Kaganovsky and Wang-hai [15]. The diffusivity data were obtained from the surface diffusion of Ni–Cu solid solution alloys and are also shown in Table I.

As previously mentioned in Section 1, Weertman [4] proposed the following equation for the climb controlled creep at high temperatures

$$\dot{\epsilon} = \alpha\frac{E\mathbf{b}}{kT}D_{\mathrm{L}}(\sigma/E)^3. \qquad (5)$$

TABLE I Calculated diffusion data ($\rho = 3 \times 10^{10}$)

Temperature (K)	Tracer diffusivity		Climb diffusivity D_{cl}	Pipe diffusivity D_{P}	Effective diffusivity D_{eff} (ρ = const)
	D^*_{Ni}	D^*_{Cu}			
598	8.83×10^{-24}	1.63×10^{-22}	1.12×10^{-23}	3.63×10^{-17}	5.38×10^{-21}
661	1.94×10^{-21}	2.09×10^{-20}	2.42×10^{-21}	1.34×10^{-15}	2.00×10^{-19}
744	5.83×10^{-19}	3.55×10^{-18}	7.14×10^{-19}	6.11×10^{-14}	9.75×10^{-18}
826	5.31×10^{-17}	2.06×10^{-16}	6.35×10^{-17}	1.25×10^{-12}	2.49×10^{-16}
909	2.23×10^{-15}	5.98×10^{-15}	2.59×10^{-15}	1.53×10^{-11}	4.85×10^{-15}
992	5.02×10^{-14}	9.85×10^{-14}	5.63×10^{-14}	1.23×10^{-10}	7.45×10^{-14}
1050	3.30×10^{-13}	5.37×10^{-13}	3.61×10^{-13}	4.33×10^{-10}	4.25×10^{-13}

As this lattice diffusion controlled high temperature creep model predicts a third power stress dependence of the creep rate and cannot account for the creep behaviour of the intermediate temperature range ($< 0.6\,T_m$), Equation 3 will be substituted into the Equation 5 to give

$$\dot{\varepsilon} = \alpha \frac{E\mathbf{b}}{kT} [D_L + \beta(\sigma/E)^{1.5} D_P](\sigma/E)^3 \qquad (6)$$

This is similar to the result of Spingarn *et al.* [7] but Equation 6 implies that $n = 4.5$ when pipe diffusion controls creep and $n = 3$ if lattice diffusion predominates the creep formation.

3.3. Stress dependence of creep rate

In Fig. 3, the steady-state creep rates obtained under the various temperature and stress combination are plotted against the normalized stress with Young's modulus. The temperature dependence of Young's modulus for Ni–Cu alloys is reported by Orlov and Fedotov [16], Parlov *et al.* [17] and Iguchi and Udagawa [18]. These papers give common results such as Young's modulus decreases almost linearly with increasing temperature. The Young's modulus obtained from the above results are used for the normalization of the stress for this work. As shown in Fig. 3, in the power law region, the stress exponent increases with increasing stress and decreasing temperature.

The increase in the stress exponent in the power law range is predicted from Equation 6 and the value is readily obtained from the expression,

$$n = \frac{\partial \ln \dot{\varepsilon}}{\partial \ln (\sigma/E)} = \frac{1.5\beta(\sigma/E)^{1.5} D_P}{D_L + \beta(\sigma/E)^{1.5} D_P} + 3. \qquad (7)$$

Equation 7 shows that the value of n will be 3 when the stress is small and as the stress increases the first term on the right hand side of Equation 7 will become larger to give the maximum value of n as equal to 4.5.

The creep rates in Fig. 3 were normalized with the effective diffusivity in which the dislocation density is assumed to be constant. From a best fit method for superposition of all the data in Fig. 3, dislocation density in the effective diffusivity was calculated to be $3 \times 10^{14}\ \mathrm{m}^{-2}$, a value which is in good agreement with the experimental result of Hong and Nam [12]. In Fig. 4, these normalized creep rates are plotted against the normalized stress with Young's modulus. Interestingly enough, this master plot shows that the exponent gradually increases from 3 to 4.5 as the stress increases.

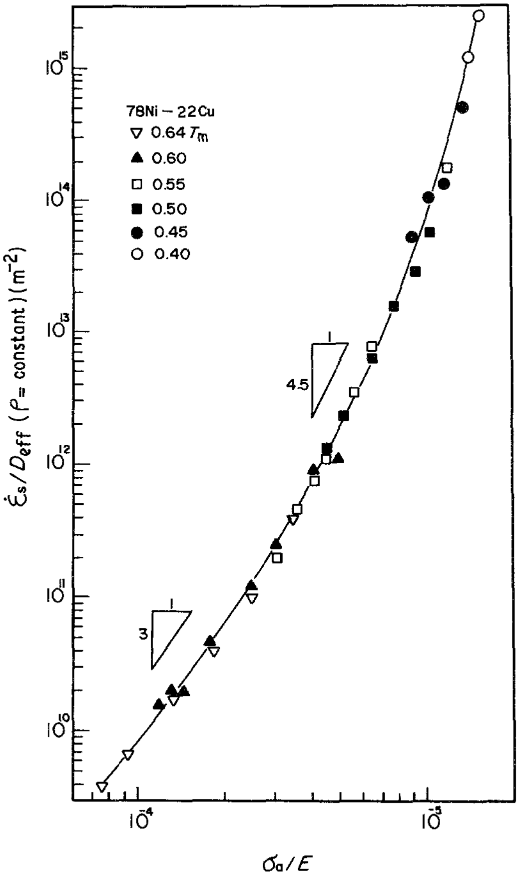

Figure 4 Steady-state creep rate over effective diffusivity predicted by assuming that the dislocation density remains constant against stress over Young's modulus for 78Ni–22Cu alloy.

There could be two different ways to interpret the change in the stress exponent. One possibility originates from the increasing influence of pipe diffusivity as the dislocation density increases with increasing stress and decreasing temperature. The other is the change of the deformation mechanism from dislocation glide due to the formation of the atmosphere under low stress to dislocation climb with increasing stress. However, as previously mentioned, the latter is excluded on the basis of the results of the stress change test and intermittent stress test. Therefore, the contribution of the pipe diffusion process will be considered.

In Equation 6, it is shown that the stress exponent will always be 3 if the proper value of the dislocation density at a certain stress is used. In other words, when the steady-state creep rates are normalized with the effective diffusivity term in Equation 6, one obtains

$$\frac{\dot{\epsilon}}{D_{\mathrm{L}} + \beta(\sigma/E)^{1.5} D_{\mathrm{P}}} = \frac{\dot{\epsilon}}{D_{\mathrm{eff}}[\rho = \rho(\sigma)]} = \alpha \frac{E\mathbf{b}}{kT}(\sigma/E)^{3}. \tag{8}$$

This equation implies that if the steady-state creep rates are normalized with the effective diffusivities, in which the dislocation density is not a constant but a function of the stress, and plotted against the applied stress normalized with the temperature-compensated Young's modulus, then the stress exponent value of the master plot has to be 3 in the low power region.

To see the feasibility of the Equation 8 one has to know the values of $\beta = (n/N)\rho_0, \rho = \rho_0(\sigma/E)^{1.5}$ and α. Assuming the experimental result of Hong and Nam [12] for the dislocation density (mentioned previously) of Ni–30 Cu is effective for this work, the stress dependence of the dislocation density is expressed as $\rho(\mathrm{m}^{-2}) = 2.88 \times 10^{18}(\sigma/E)^{1.5}$.

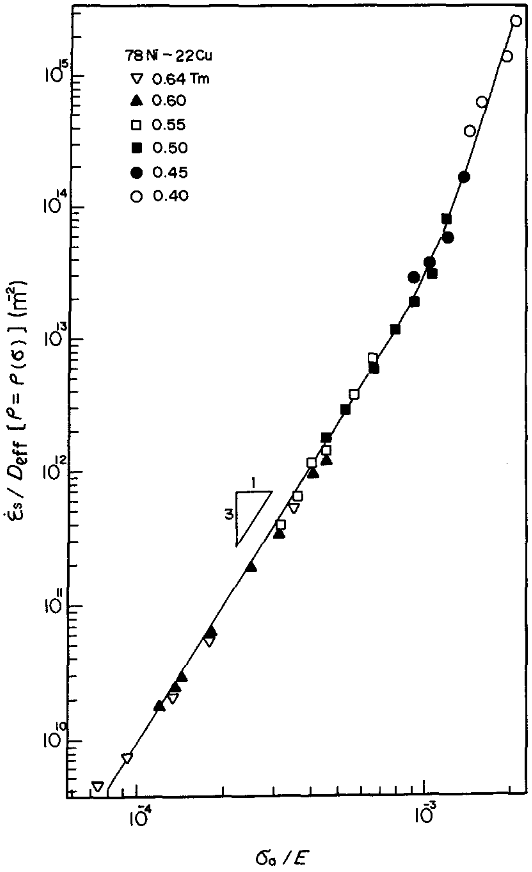

Figure 5 Steady-state creep rate over effective diffusivity predicted by taking account of the stress dependence of the dislocation density against stress over Young's modulus for 78Ni–22Cu alloy.

For a good fit of the data to a single master curve in Fig. 5, the values $\alpha = 28$ and $\beta = 8.5$ were obtained. From the values of $\rho_0 = 2.88 \times 10^{18}$ and $\beta = 8.5$, one calculates $n = 60$ ($N = 2.03 \times 10^{19}\ \mathrm{m}^{-2}$ for Ni–22 at % Cu). This value is rather higher than the number of 10 for n chosen by Shewmon [10]. But his study was for the case of static diffusion. As mentioned by Robinson and Sherby [3], there is a possibility that for the case of moving dislocations n may be higher than 10. These authors calculated $n = 40$ to about 400 for a reasonable value of dislocation density. It can be said that the rather high value of n in this study may be due to the effect of dynamic dislocation behaviour. Also, the value of 28 for α agrees with the result of Spingarn *et al.* [7]. From the above results, it is suggested that Equation 8 is valid for the steady-state creep deformation.

The result shown in Fig. 5 supports the fact that, in the absence of *ad hoc* assumptions, all the existing theories based on lattice diffusion predict a third power law stress dependence which was brought to public notice by Weertman [4] and reconfirmed by Poirier [19]. The result also suggested that the creep deformation mechanism of this alloy is explained in terms of the localized climb of edge dislocations, which can occur by lattice or core diffusion.

3.4. Temperature and stress dependence of activation energy

The activation energies for steady-state creep were calculated from the plot of the ln $\dot{\epsilon}$ against $(1/T)$ at constant σ/E. Using Equation 6, one can write the following equation,

$$Q_{\mathrm{eff}} = -\frac{R\,\mathrm{d}\ln\dot{\epsilon}}{\mathrm{d}(1/T)}\bigg|_{\sigma/E} = -\frac{R\,\mathrm{d}\ln D_{\mathrm{eff}}}{\mathrm{d}(1/T)} \tag{9}$$

where Q_{eff} is the effective activation energy. The activation energies for a climb-controlled process were found by using the formula,

$$D_{\mathrm{L}} = D_{\mathrm{cl}} = D_{\mathrm{cl}}^{0} \exp\left(-\frac{Q_{\mathrm{cl}}}{RT}\right) \tag{10}$$

where D_{cl}^{0} is a constant and Q_{cl} is the activation energy for climb-controlled creep. From Equations 4 and 10, D_{cl}^{0} and Q_{cl} values were found to be $0.003\ \mathrm{m}^2\,\mathrm{sec}^{-1}$ and $280\ \mathrm{kJ\,mol}^{-1}$, respectively.

Substituting Equation 10 into Equation 3, differentiating with respect to $(1/T)$, and letting $T = T(T_{\mathrm{m}}/T_{\mathrm{m}})$ leads to the following equation,

$$Q_{\text{eff}} = \frac{D_{\text{cl}}^{0} Q_{\text{cl}} \exp\left[-\dfrac{Q_{\text{cl}}}{RT_{\text{m}}(T/T_{\text{m}})}\right] + 8.5 D_{\text{P}}^{0} Q_{\text{P}} \exp\left[-\dfrac{Q_{\text{P}}}{RT_{\text{m}}(T/T_{\text{m}})}\right] [(\sigma/E)^{1.5}]}{D_{\text{cl}}^{0} \exp\left[\dfrac{Q_{\text{cl}}}{-RT_{\text{m}}(T/T_{\text{m}})}\right] + 8.5 D_{\text{P}}^{0} \exp\left[-\dfrac{Q_{\text{P}}}{RT_{\text{m}}(T/T_{\text{m}})}\right] [(\sigma/E)^{1.5}]}. \tag{11}$$

In Fig. 6, the dark lines indicate the variation of the activation energy predicted from Equation 11 as a function of the homologous temperature at different magnitudes of σ/E. Below $0.4\,T_{\text{m}}$, Q_{eff} is about equal to the activation energy for pipe diffusion and above $0.7\,T_{\text{m}}$, Q_{eff} is about equal to the activation energy for lattice diffusion. In the temperature range of 0.4 to about $0.7\,T_{\text{m}}$, Q_{eff} is seen to be dependent on σ/E. As σ/E is increased, i.e. dislocation density is increased, the pipe diffusion term becomes predominant at a given homologous temperature and Q_{eff} decreases towards the activation energy for pipe diffusion. The experimentally determined activation energies for steady-state creep are also given in the same figure. As can be seen the prediction of Equation 11 agrees well with the experimental data.

A plot of Q_{eff} against σ/E at a given homologous temperature can be calculated from Equation 11 and the results of such calculations are shown in Fig. 7. At low stresses, the pipe diffusion term becomes negligible with increasing temperature and Q_{eff} becomes the activation energy for lattice diffusion. Even at low stresses, the pipe diffusion term is important in the low temperature range. At high stresses, the pipe diffusion term becomes dominant and Q_{eff} should be equal to the activation energy for pipe diffusion. The comparison of experimentally measured activation energy with that predicted from Equation 11 shows a tendency that the activation energy for steady state-creep is decreasing towards the activation energy for pipe diffusion with the combined effect of increasing stress and decreasing temperature.

Fig. 7 indicates that the activation energy depends on the stress term at a given temperature. This means that the activation energy for steady-state creep can not be expressed as a function of temperature only. Some workers [20] reported that the activation energies were independent on the stress term under certain experimental conditions. This could be true only for a narrow stress range in which one type of diffusion is predominant. On the other hand, many investigators reported that the apparent activation energy is dependent on the applied stress by introducing the concept of internal stress. As states above, it is suggested that the influence of applied stress on the activation energy should be explained by the concept of effective diffusivity in a diffusion-controlled flow. The good correlation with the experimental data and the curves predicted from equation 11 shows the validity of an effective diffusivity for creep.

4. Conclusions

1. The behaviour of a transient creep test shows

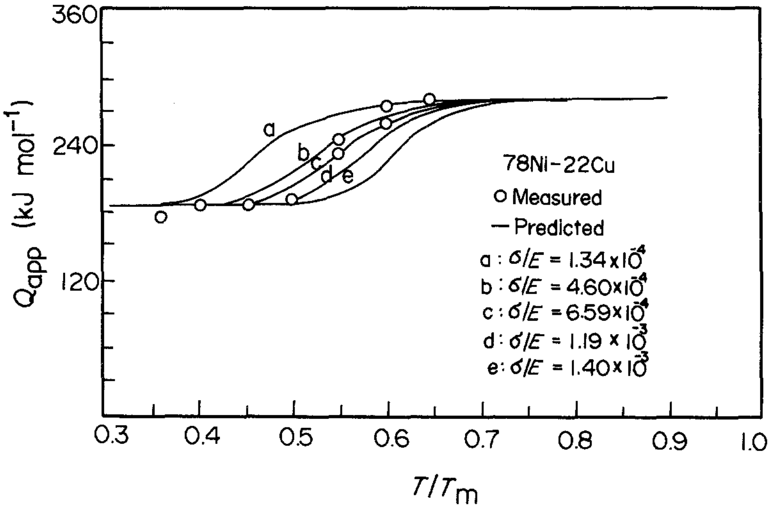

Figure 6 Comparison of the measured activation energy with the calculated of 78Ni–22Cu alloy at various σ/E as a function of homologous temperature.

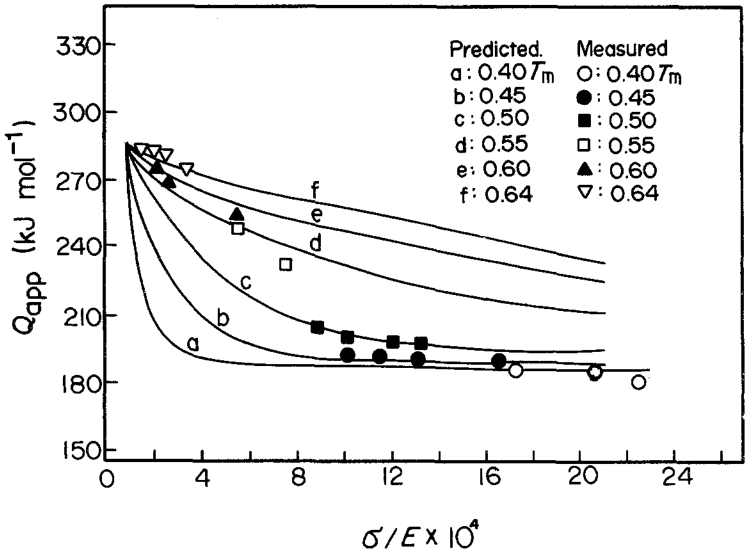

Figure 7 Stress over Young's modulus dependence of apparent activation energy.

no indication of the solute atmosphere formation around dislocations in Ni–22 at % Cu alloy. This implies a dislocation dragging process may not be occurring in the creep deformation process under the test conditions used.

2. Using the concept of effective diffusivity, the value of the stress exponent in the low power range is observed to be 3 if the lattice diffusion assisted dislocation climb controls the creep deformation process while the value of the exponent becomes 4.5 if diffusion assisted localized dislocation climb controls the time-dependent plastic deformation mechanism.

3. The measured and the calculated activation energies, on the basis of effective diffusivity, were almost identical and show stress and temperature dependence. As stress increases and temperature decreases the activation energy decreases from the lattice diffusion value to that of the pipe diffusion process.

4. The creep deformation controlling mechanism of the alloy is a diffusion controlled process in which the climb of edge dislocations can occur either through lattice diffusion or dislocation pipe diffusion.

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