DETERMINATION OF CONSTITUTIVE PARAMETERS FOR SELF-SIMILAR INDENTATION CREEP AND ITS APPLICATIONS

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ABSTRACT

Indentation creep experiments and finite element calculations were carried out to establish a reliable and systematic way to estimate characteristic creep parameters during indentation creep tests. Finite-element computations confirmed that the indentation creep strain field can be treated as self-similar during a constant-load indentation creep test beyond the initial transient period. Self-similar indentation creep can be readily described using an established constitutive equation from which the power-law creep exponent, *n*, the activation energy for creep, Q_c, and the critical stress, $\bar{\sigma}_c$, where there is an *n*-value transition, can be extracted. Samples made from an Al-5.3mol%Mg solid-solution alloy were carefully tested at temperatures ranging from 546 to 590 K. Analysis of indentation creep curves indicates that the *n*-value changes distinctively from 4.9 to 3.0 below a critical stress level $\bar{\sigma}_c$ ($\bar{\sigma} \cong p/3$, where *p* is the average indentation pressure or hardness) as indentation creep proceeds. In the stress range H ($n = 4.9$), the obtained activation energy for creep is $Q_c = 146$ kJ/mol, which agrees well with the value for the lattice diffusion of pure Aluminum, 144 kJ/mol. The Q_c-value in the stress range M ($n = 3.0$) is evaluated to be 137 kJ/mol, which is close to the mutual diffusion of this alloy, 130 kJ/mol. The critical stress level $\bar{\sigma}_c$ for the *n*-value transition significantly decreases as temperature increases, while the corresponding indentation strain rate $\dot{\varepsilon}_c$ increases slightly with increasing temperature. The findings suggest that the $\bar{\sigma}_c$-value corresponds to the critical stress for breakaway of dislocations from their solute atmosphere. The results are in good agreement with those obtained from conventional uniaxial creep tests in the dislocation creep regime. It is demonstrated that indentation creep testing technique is a powerful tool to extract mechanical properties including the creep characteristics at high temperatures.

1 INTRODUCTION

Considerable efforts and significant progresses were made in using instrumented indentation to extract room temperature elastic as well as elasto-plastic properties (e.g. [1-5]). Various studies were also carried out on indentation creep [6-18]. In this study, carefully designed indentation creep experiments together with finite element computations have been performed in order to establish a robust and systematic method to extract creep properties during indentation creep tests.

2 CONSTITUTIVE EQUATION FOR SELF-SIMILAR INDENTATION CREEP

For a constant-load indentation creep test, the stress or strain field under the indenter is highly non-uniform and closely related to the plastic deformation history. Another concern is whether the initial transient response (not self-similar) will result in the loss of self-similarity throughout the entire indentation creep test. To address these issues, recent experimental and computational studies [13, 14, 18, 19] showed that, after a short initial transient period, the plastic region under a sharp indenter extends while maintaining its geometrical self-similarity as creep indentation proceeds, that is the contours of time-dependent plastic strain change only in scale, and not in shape. Then, a constitutive equation for self-similar indentation creep is given by [18, 19]

$$\dot{\varepsilon}_{\mathrm{in}} = \frac{\dot{u}}{u} = A \left(\frac{\bar{\sigma}}{E}\right)^n \exp\left(-\frac{Q_{\mathrm{c}}}{RT}\right), \qquad (1)$$

where $\dot{\varepsilon}_{\mathrm{in}}$ is the indentation strain rate, $\dot{u}$ and u are the indenter velocity and the indenter displacement respectively, A is a material constant, $\bar{\sigma}$ is the average equivalent (Von Mises) stress in the plastic zone just below the indenter ($\bar{\sigma} \cong p/3$, p is the average indentation pressure or hardness), E is the Young's modulus at test temperature, R is the gas constant, T is the test temperature. Consequently, the stress exponent for creep, n, is given by

$$n = \partial \ln \dot{\varepsilon}_{\mathrm{in}} / \partial \ln (\bar{\sigma} / E) \ . \qquad (2)$$

The activation energy for creep, Q_{c}, is obtained by

$$Q_{\mathrm{c}} = -R\left(\frac{\partial(\ln K)}{\partial(1/T)}\right), \qquad (3)$$

where $K = \dot{\varepsilon}_{\mathrm{in}} \, (E/\bar{\sigma})^n$. Detailed development of eqn (1) can be found in [18].

3 EXPERIMENTAL PROCEDURES

Ingots of Al-5.3mol%Mg alloy were homogenized by keeping in an argon gas for 86.4 ks at 773 K ($0.85T_{\mathrm{m}}$, T_{m}: the absolute melting point). They were cut into approximately 10 mm × 5 mm × 5 mm pieces with a metal saw, and carefully shaped into parallelepipeds using special jigs and emery papers. All specimens were subjected to annealing in an argon gas for 3.6 ks at 773 K. After that, they were electropolished and acid cleaned to remove the surface layer up to ~40 μm in thickness, and immediately placed in the testing machine. Before the indentation tests, the diamond indenter tip is brought close to the sample surface. Both the sample and the diamond tip are maintained at the desired test temperature for about one hour. The temperature is measured in the vicinity of the sample. The indentation tester was built in collaboration with ULVAC-RIKO in Japan [13]. The indenter tip is conical in shape up to a height of 120 μm (the included half apex angle $\theta = 68^{\mathrm{o}}$). Indentation creep tests for Al-5.3mol%Mg solid-solution alloy were carried out under the conditions such that dynamic recrystallization does not occur. The test conditions applied are listed as follows.

- Indentation load: 0.39 N
- Total indentation time: 1200 s
- Test temperatures: 546 K–590 K (Temperature variation was within ±1 K)

4 RESULTS AND DISCUSSIONS

Indentation creep experiments were conducted at four different test temperatures 546 K, 564 K, 573 K and 590 K ($0.60\,T_{\mathrm{m}}$–$0.65T_{\mathrm{m}}$). Fig. 1 shows indentation creep curves of Al-5.3mol%Mg

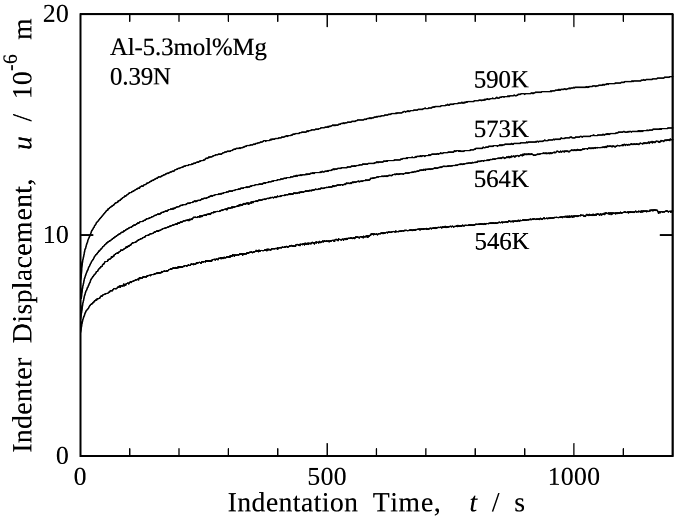

Figure 1. Experimentally measured indentation creep curves at different test temperatures.

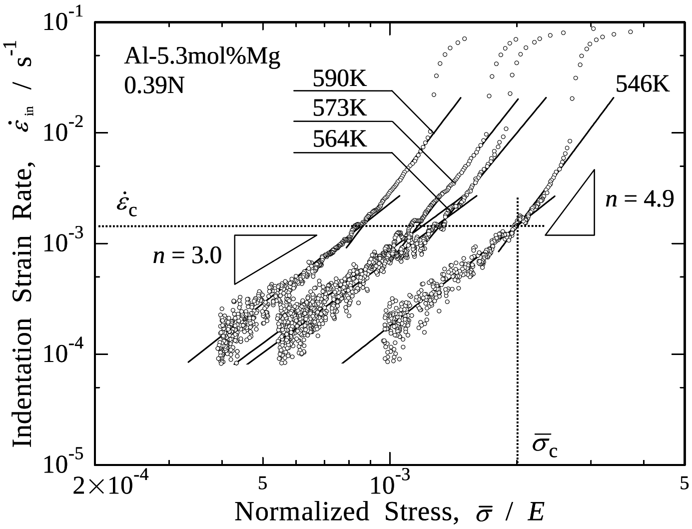

Figure 2. Experimentally measured indentation strain rate versus normalized average equivalent stress at temperatures ranging from 546 to 590 K, both on logarithmic scales.

solid-solution alloy at different temperatures. When a diamond conical indenter is pressed into the hot material, the indenter displacement rapidly increases within a short time and then slowly penetrates the material by creeping. The indentation displacement u increases with increasing test temperature at the same total time t. The indenter displacement also increases with the passage of indentation time, and it would finally reach a saturation value. The indenter displacement for 1200 s at 590 K is about 1.5 times that at 546 K, and the final saturation displacement clearly increases with a rising temperature.

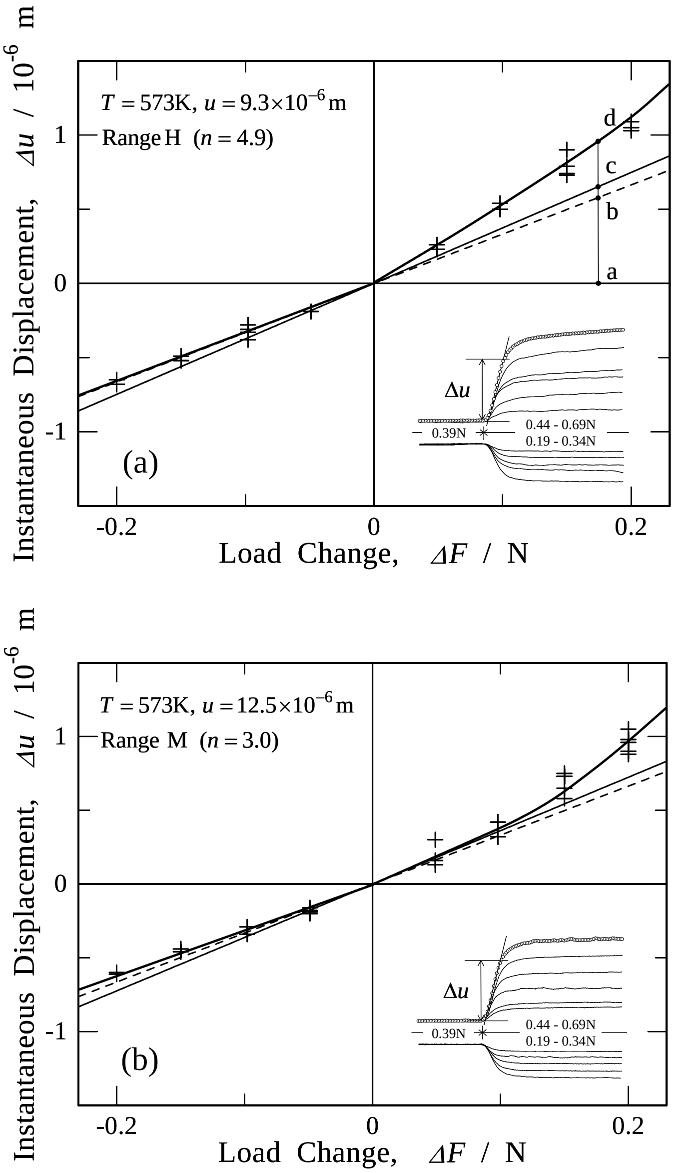

Figure 3. Relationship of instantaneous displacement against load change: (a) in the range H, and (b) in the range M. See text for details.

Fig. 2 shows the experimentally measured indentation strain rate $\dot{\varepsilon}_{\mathrm{in}}$ versus normalized average equivalent stress $\bar{\sigma}/E$ at temperatures ranging from 546 to 590 K, both on logarithmic scales. $\bar{\sigma}_{\mathrm{c}}$ is the critical stress level for stress exponent transition and $\dot{\varepsilon}_{\mathrm{c}}$ is the corresponding indentation strain rate. The experimental data lie on two straight lines with different slopes at each test temperature, except for the initial transition stage right after loading and the range beyond the measurement limit of the liner variable-differential transformer, LVDT. Analysis of indentation creep curves shown in Fig. 2 indicates that the *n*-value changes distinctively from 4.9 to 3.0 below a critical stress level $\bar{\sigma}_c$ as indentation creep proceeds. In the stress range H ($n = 4.9$, this value is equivalent to that of pure metals), the obtained activation energy for creep is $Q_c = 146$ kJ/mol, which agrees well with the value for the lattice diffusion of pure Aluminum, 144 kJ/mol. The Q_c-value in the stress range M ($n = 3.0$) is evaluated to be 137 kJ/mol, which is close to the mutual diffusion of this alloy, 130 kJ/mol. The critical stress level $\bar{\sigma}_c$ for the *n*-value transition significantly decreases as temperature increases, while the corresponding indentation strain rate $\dot{\varepsilon}_c$ increases slightly with increasing temperature. The findings suggest that the $\bar{\sigma}_c$-value corresponds to the critical stress for breakaway of dislocations from their solute atmosphere [20].

Fig. 3(a) shows the relationship of instantaneous indentation displacement against load change in the range H. The time for load change is within less than 0.1s. A positive Δu denotes indenter displacement in the direction of load and a negative Δu represents indentation displacement in the reverse direction. Also, a positive ΔF means an increase in load and a negative value means a decrease in load. The solid line represents the total compliance including the machine and the specimen. The dashed line represents the machine compliance consisting of the frame and the conical diamond indenter. $\overline{\mathrm{ab}}$ indicates the elastic deformation of the microindenter, $\overline{\mathrm{bc}}$ the elastic deformation of specimen, and $\overline{\mathrm{cd}}$ the instantaneous plastic deformation of specimen. In the negative ΔF region, the data lie on the straight line. This shows that the instantaneous displacement Δu is due to the elastic deformation of the microindenter and the specimen. On the other hand, all the data in the positive ΔF region are away from the straight line. This finding exhibits that instantaneous plastic deformation occurs even when load is slightly increased, that is, dislocations can quickly move to the next obstacles. The inset shows the magnified part of load change in the range H during indentation creep. Fig. 3(b) shows the relationship of instantaneous indentation displacement against load change in the range M. In the negative ΔF region, the data lie on the straight line. On the other hand, it appears that the data of the positive ΔF region lie on the straight line with a load increase not exceeding $\Delta F = 0.1$ N. This finding exhibits that when $\Delta F < 0.1$ N only elastic deformation occurs right after load change and moving dislocations drag their solute atmosphere. When the total load $F + \Delta F$ is 0.39 N + (0.10–0.15 N), the corresponding stress $\bar{\sigma}$ is evaluated to be 54–60 MPa. The values are close to the critical stress level for *n*-value transition ($\bar{\sigma}_c / E = 1.12 \times 10^{-3}$, thus $\bar{\sigma}_c = 65\,\mathrm{MPa}$) at 573 K in Fig. 2.

The above experimental results can be understood such that the creep in the high-stress regime when $\bar{\sigma} > \bar{\sigma}_c$ is rate-controlled by some recovery process dependent on the climb motion of dislocations. On the other hand, the creep in the low-stress regime when $\bar{\sigma} < \bar{\sigma}_c$ is rate-controlled by viscous glide of dislocations which drag solute cloud. These results are in good agreement with those obtained from conventional uniaxial creep tests in the dislocation creep regime [17, 18].

5 CONCLUSIONS

Indentation creep experiments (including load change experiments) and numerical simulations were carried out in order to establish a robust and systematic method to extract creep properties during indentation creep tests. Finite element computations confirmed that, for a power law creep material, the indentation creep strain field is self-similar under a constant load, except during short transient periods. The stress exponent and the activation energy for creep can be accurately extracted, and these results are consistent with those obtained from conventional uniaxial creep tests in the dislocation creep regime.

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