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Experimental and computational study on the load-jump tests of Al–Mg solid–solution alloy using instrumented indentation technique

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Abstract

Constant-load tests and load-jump tests were carried out using a microindenter in Al–5.3 mol% Mg solid–solution alloy. When the representative stress $\bar{\sigma}$ beneath the indenter decreases to a critical stress level, $\bar{\sigma}_c \cong 61$ MPa, during indentation creep at 573 K, the stress exponent n for creep changes from 5 ($\bar{\sigma} > \bar{\sigma}_c$) to 3 ($\bar{\sigma} < \bar{\sigma}_c$). With load-jump tests in the stress range of $n = 3$, instantaneous plastic deformation (IPD) is initiated at stresses of $\bar{\sigma} > 54$ MPa or more. When IPD does not occur, the n value obtained from load-jump tests agrees fairly well with that of constant-load tests. The experimental results are supported by finite-element simulations on load-jump tests. In order to evaluate the n value robustly by the load-jump test, the test should be conducted under a loading condition at which only elastic deformation takes place or no IPD occurs.

Keywords: Aluminum–magnesium alloy; Self-similar indenter; Indentation; Creep; Instantaneous plastic deformation; Stress exponent

1. Introduction

In general, advanced materials being produced daily in research and development are often available only in minute quantities or as small-volume specimens or structures. For this reason, it has been desired to establish a material testing method that can provide accurate mechanical properties at high temperatures from a specimen as small as a rice grain. We recently developed a microindenter which can be used under high-temperature test conditions [1], and carried out constant-load indentation creep experiments for Al–Mg solid–solution alloy [2,3]. The stress exponent and activation energy for creep obtained are in good agreement with those obtained from conventional uniaxial creep tests [4,5]. The creep rate-controlling process of this alloy changes from the glide motion ($n \cong 3$, the range A) to the climb motion of dislocations ($n \cong 5$, the range M) depending on stress and creep rate.

In the present study, load-jump tests of an Al–Mg solid–solution alloy, together with finite-element computations, were carried out to examine the following three aspects:

(i) the physical meaning of the critical stress level at which instantaneous plastic deformation (IPD) in load-jump tests is initiated;
(ii) comparison of indentation creep curve (ICC) right after load jumping and that for the corresponding constant-load test;
(iii) evaluation of stress exponent using the data obtained from ICC right before and after load jumping.

On the basis of the above results, we will demonstrate that in order to evaluate the stress exponent robustly by the load-jump test, the test should be conducted under a loading condition at which only elastic deformation occurs or no IPD occurs.

2. Experimental and computational procedure

An ingot of Al–5.3 mol% Mg alloy was cut into approximately 5 mm × 10 mm × 5 mm pieces and carefully shaped into rectangular parallelepipeds using special jigs and emery paper. After annealed in Ar gas atmosphere, specimens were electropolished to remove the surface layer to up to about 40 μm in thickness and immediately placed in the testing machine.

Indentation creep tests were carried out at a temperature of 573 K ($0.63T_m$, T_m: the liquidus temperature) using a

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microindenter manufactured by ULVAC-RIKO in Japan [1]. The indenter tip was made of diamond and conical in shape (the included half-apex angle: 68°). With load-jump tests, the load was abruptly changed in the indentation creep test. The time required for load jump was within 0.1 s. The constant-load tests and the load-jump tests were computationally analyzed using a commercial finite-element (FE) program (ABAQUS). A two-dimensional and axisymmetric FE-model was constructed to simulate the indentation response of elasto-plastic solids. An indenter tip was assumed to be rigid. More details of the computational model setup can be found elsewhere [2].

3. Constitutive equation for indentation creep

When a conical indenter is pressed into the surface of a hot specimen, the plastic region below the indenter extends while maintaining its geometrical self-similarity as indentation creep proceeds. In this case, the indentation strain rate $\dot{\varepsilon}$ is defined as $\dot{u}/u$ [6], where u is the indenter displacement and $\dot{u}$ is the indenter velocity. The average equivalent stress or the representative stress $\bar{\sigma}$ in the plastic zone that governs the indenter velocity (hereafter, the zone is called the control volume) can be approximately written as $\bar{\sigma} = p/3 \propto F/u^2$. Here, F is the indentation load and p is the indentation load divided by the projected contact area of an impression. When the material's creep behavior obeys the well-known power law, a constitutive equation for the steady-state indentation creep can be given as [1,2]

$$\dot{\varepsilon} = A\left(\frac{\bar{\sigma}}{E}\right)^n \exp\left(-\frac{Q}{RT}\right), \tag{1}$$

where A is a material constant, Q is the activation energy for creep, R is the gas constant and T is the test temperature. Assuming that the microstructure S in the control volume is maintained during the indentation creep owing to sufficient dynamic recovery, the stress exponent n for creep is given by

$$n = \left[\frac{\partial \ln \dot{\varepsilon}}{\partial \ln(\bar{\sigma}/E)}\right]_{T,S}. \tag{2}$$

For load-jump tests, the measured values of $\bar{\sigma}$ and $\dot{\varepsilon}$ right before and after abrupt increase of load are denoted as $\bar{\sigma}_1$, $\dot{\varepsilon}_1$, and $\bar{\sigma}_2$, $\dot{\varepsilon}_2$, respectively. If the microstructure is unchanged during load jumping, the stress exponent n can then be obtained by

$$n = \left[\frac{\ln(\dot{\varepsilon}_2/\dot{\varepsilon}_1)}{\ln(\bar{\sigma}_2/\bar{\sigma}_1)}\right]_{T,S}. \tag{3}$$

4. Results and discussion

4.1. Indentation creep curve

Fig. 1 shows the ICCs (thin lines) obtained by constant-load tests with 0.39, 0.43 and 0.59 N. The indenter displacement rapidly increases within a short time, and then the indenter slowly penetrates the material via creep. The thick lines are for the load-jump tests, in which the loads were abruptly increased from 0.39 to 0.43 or 0.59 N at $t = 400$ s. The ICCs (thick lines) gradually approach the corresponding ICCs (thin lines) obtained by the constant-load test. The insets show that the indenter displacement instantaneously increases upon abrupt increase in load.

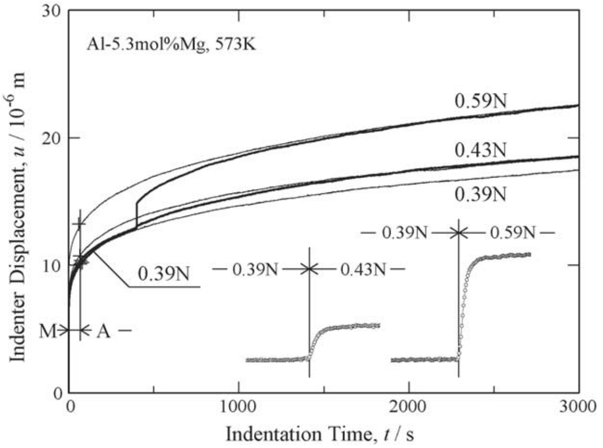

Fig. 1. Indentation creep curves. In the insets, the ordinate for load increase from 0.39 to 0.43 N is enlarged by 1.5 times that for load increase to 0.59 N.

Fig. 2 shows the experimentally measured indentation strain rate $\dot{\varepsilon}$ versus normalized average equivalent stress obtained by the constant-load test ($F = 0.39$ N), both on logarithmic scales. The experimental data lie on two straight lines having different slopes, except for the initial transient stage right after loading and the range beyond the measurement limit of the displacement sensor. This linear relationship indicates that the indented material's behavior follows the power-law creep. From Eq. (2), the slope of the straight line allows us to determine the stress exponent. The stress exponent n changes distinctly from 4.9 (the range M) to 3.0 (the range A), below a critical stress level $\bar{\sigma}_c$, as indentation creep proceeds. The $\bar{\sigma}_c$ value is estimated experimentally to be $\bar{\sigma}_c = 61 \pm 6$ MPa on average. These results are in fairly good agreement with those of conventional uniaxial creep tests [4,5]. Therefore, our result suggests that the creep rate-controlling process changes from the climb motion of dislocations ($n \cong 5$) to the glide motion of dislocations ($n \cong 3$) which drag their solute atmosphere below the critical stress level $\bar{\sigma}_c$.

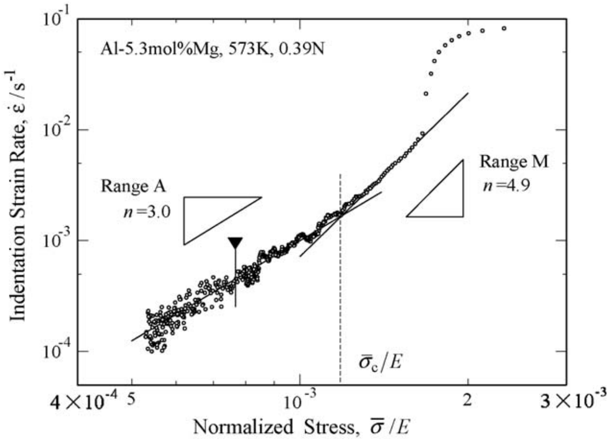

Fig. 2. Logarithmic plots of indentation strain rate versus average equivalent stress normalized by Young's modulus. The symbol (▼) indicates the normalized stress level at which load-jump test was carried out.

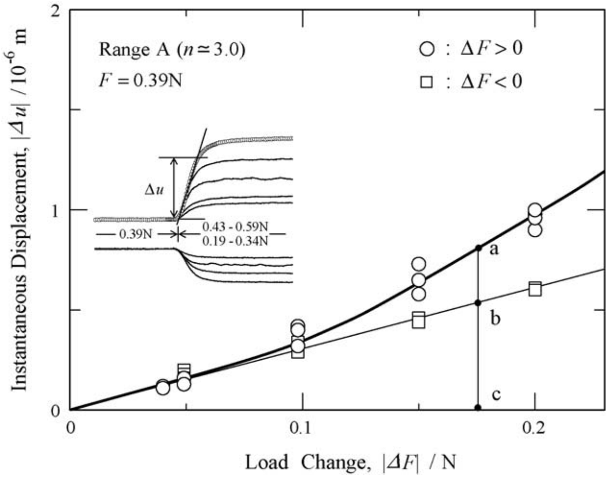

Fig. 3. Dependence of instantaneous displacement on load change. a–b, Instantaneous plastic deformation of specimen; b–c, elastic deformation of testing machine and specimen.

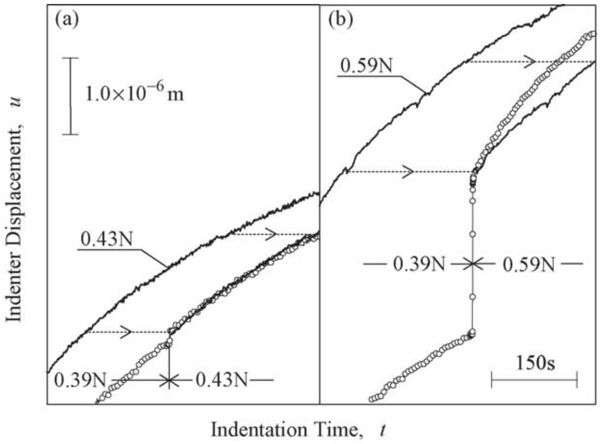

Fig. 4. Magnified view of indentation creep curves. The solid line shows results of constant-load test, while the small circles exhibit the results of load-jump test. Part of the solid line is shifted to compare the two results.

The finding implies that the $\bar{\sigma}_c$ value is close to the critical stress level for the breakaway of dislocations from their solute atmosphere.

4.2. Load-jump test

The load-jump tests are carried out at the stress level of the range A ($\bar{\sigma} < \bar{\sigma}_c$, $n \cong 3$), as shown in Fig. 1. Fig. 3 shows the instantaneous displacement $|\Delta u|$ as a function of load change $|\Delta F|$. It can be seen that in the case of load decrease (negative jump), the instantaneous displacement values (□) lie on a straight line. The slope of the straight line is approximately consistent with the compliance of the mechanical system consisting of the testing machine and the specimen [3]. Therefore, the instantaneous displacement obtained upon load decrease is caused by elastic recovery of the mechanical system. With the abrupt increase of load, the instantaneous displacement (○) for $\Delta F \leq 0.10$ N is in very good agreement with the data for the abrupt decrease of load. This fact indicates that the instantaneous displacement here is caused by elastic deformation. In contrast, the instantaneous displacement (○) for $\Delta F \geq 0.10$–0.15 N lies above the straight line having the slope corresponding to the compliance of the mechanical system, indicating that instantaneous plastic deformation (IPD) occurs in the specimen right after load increase. Assuming $u \cong 12.5 \times 10^{-6}$ m and $F = 0.49$–0.54 N, the critical stress level at which IPD occurs is $\bar{\sigma} = 54 - 60$ MPa. These values are close to the critical stress level in Fig. 2, i.e., $\bar{\sigma}_c = 61$ MPa. This finding suggests that IPD is initiated in the specimen when the representative stress $\bar{\sigma}$ is abruptly increased from the range A ($\bar{\sigma} < \bar{\sigma}_c$) to the range M ($\bar{\sigma} > \bar{\sigma}_c$) [7].

Fig. 4 shows the ICCs in the range A for the constant-load test (solid line) and those for abruptly increased load during indentation (designated by small circles). Fig. 4(a) shows the ICC (small circles) when the load is abruptly increased from 0.39 to 0.43 N within the range A, where only elastic displacement occurs in the mechanical system. In the figure, part of the ICC (solid line) is shifted so that it overlaps with the indenter displacement right after the abrupt increase of load (small circles). One lies upon the other completely. The result shows that the relationship between $\bar{\sigma}$ and $\dot{\varepsilon}$ is independent of the loading path. The finding suggests that the creep rate-controlling process is unchanged before and after load jumping. Fig. 4(b) shows the ICC (small circles) for when the load is abruptly increased from 0.39 to 0.59 N corresponding to the range M, where IPD takes place in the specimen. It is found that the two lines do not overlap and the small circles lie above the solid line. The deformation behavior can be considered as follows. Immediately after the abrupt increase of load, the representative stress in the control volume is temporarily higher than the critical stress level $\bar{\sigma}_c$, and then dislocation multiplication becomes more active. Along with this, the indentation strain rate $\dot{\varepsilon}(= \dot{u}/u)$ right after load increase becomes higher than that obtained with the same load in the constant-load test.

4.3. Evaluation of stress exponent

It is possible to evaluate the stress exponent from Eq. (3) using $\bar{\sigma}$ and $\dot{\varepsilon}$ immediately before and after the abrupt increase of load. In Fig. 4(a), $\bar{\sigma}_1 = 41.2$ MPa and $\dot{\varepsilon}_1 = 6.47 \times 10^{-4}\,\mathrm{s}^{-1}$ right before load increase, while $\bar{\sigma}_2 = 45.5$ MPa and $\dot{\varepsilon}_2 = 8.96 \times 10^{-4}\,\mathrm{s}^{-1}$ right after load increase. It is clear that all the values lie within the range A of Fig. 2. Using Eq. (3), the stress exponent n is calculated as 3.3. This value agrees fairly well with $n = 3.0$ in the range A obtained by the constant-load test. On the other hand, in Fig. 4(b), $\bar{\sigma}_1 = 38.2$ MPa, $\dot{\varepsilon}_1 = 3.72 \times 10^{-4}\,\mathrm{s}^{-1}$, $\bar{\sigma}_2 = 56.1$ MPa and $\dot{\varepsilon}_2 = 7.76 \times 10^{-3}\,\mathrm{s}^{-1}$. It should be noted that the values of $\bar{\sigma}_2$ and $\dot{\varepsilon}_2$ lie in the range M of Fig. 2. In this case, $n = 7.9$, which markedly deviates from the n value obtained by the constant-load test. This discrepancy can be explained as follows: In the case of (a), only elastic deformation occurs in the specimen upon the abrupt increase of load, so that the microstructure in the control volume only negligibly changes. Therefore, the n value obtained by the load-jump test agrees with

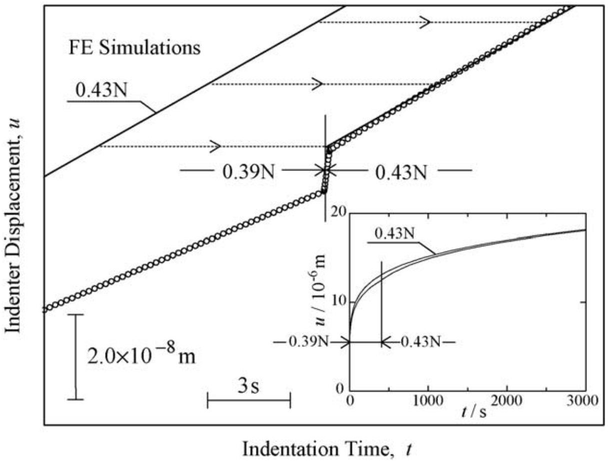

Fig. 5. Magnified view of indentation creep curves obtained by FE computations. The inset shows the entire indentation creep curves.

that obtained by the constant-load test. In contrast, in the case of (b), IPD is initiated in the specimen and then the microstructure may change greatly during load increase. As a result, the n value calculated from Eq. (3) does not agree with that obtained by the constant-load test, using Eq. (2).

Fig. 5 shows ICCs obtained by FE computations. The results were calculated with Young's modulus $E = 58.0$ GPa and Poisson's ratio $\nu = 0.345$. Each finite element was deformed in accordance with power-law creep ($\dot{\varepsilon} = B\sigma^n$; where the stress exponent $n = 3.0$ and the creep constant $B = 1.33 \times 10^{-9}\,\mathrm{MPa}^{-3}\,\mathrm{s}^{-1}$). The constant-load test was simulated with $F = 0.43$ N (solid line). In the load-jump test (small circles), load was abruptly changed from 0.39 to 0.43 N at $t = 400$ s. For the abrupt increase of load, the computational model was designed such that only elastic deformation takes place or no IPD occurs. As shown in the inset, the ICC obtained from the load-jump test gradually approaches that obtained by the corresponding constant-load test. This tendency is similar to that of the experimental data (Fig. 1). In the figure, part of the ICC (solid line) is shifted so that it overlaps with the indenter displacement right after the abrupt increase of load (small circles). One lies upon the other completely. According to the FE computations, for $\bar{\sigma}_1 = 43.4$ MPa, $\dot{\varepsilon}_1 = 4.52 \times 10^{-4}\,\mathrm{s}^{-1}$, and for $\bar{\sigma}_2 = 47.9$ MPa, $\dot{\varepsilon}_2 = 6.38 \times 10^{-4}\,\mathrm{s}^{-1}$. The n value obtained by substituting these values into Eq. (3) is 3.5. These findings correspond well with the experimental observations shown in Fig. 4(a).

5. Conclusions

Indentation creep tests of Al–5.3 mol% Mg solid–solution alloy were carried out at 573 K. The key results of this study can be summarized as follows:

(i) The stress level at which IPD is initiated in load-jump tests is close to the critical stress level $\bar{\sigma}_c$ when the stress exponent n changes from 4.9 to 3.0 as indentation creep proceeds.
(ii) When no IPD occurs in load-jump tests, the relationship between the representative stress in the control volume and indentation strain rate is independent of the loading path. In this case, the n value obtained from load-jump tests agrees fairly well with the result of constant-load tests. The experimental results are supported by finite-element simulations on load-jump tests.
(iii) When the representative stress in the control volume is higher than the critical stress level $\bar{\sigma}_c$, IPD is initiated in the specimen. As a result, the indentation strain rate right after load increase becomes higher than that obtained with the same load in the constant-load test.
(iv) In order to evaluate the stress exponent robustly by the load-jump test, the test should be carried out under a loading condition at which no IPD occurs.

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