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Transient Creep in High-Purity Aluminum at Ultra-Low Strain Rate and Room Temperature by Constant Stress and Changing-Stress Experiments

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Creep of high-purity aluminum (5N Al) at room temperature and ultra-low strain rate was investigated by a high sensitive helicoid-spring specimen technique under conditions of constant and changing stress. Creep deformation consists of transient creep stages, and no secondary creep stage was observed. Li's equation showed a good fit to the experimental curves.

During nominal steady-state creep, the stress exponent is equal to one regardless of initial state of specimens. However, the nominal steady-state creep rate for water quenched 5N Al is one order less than that for the static recovered specimens due to work hardening. With increase in stress, creep strengthening (the creep rate progressively decreasing in subsequent segments) was observed, which is due to different hardening remains because changing-stress creep experiment was conducted in the transient creep stage. Those phenomena of work hardening indicate creep deformation is controlled by recovery and work-hardening mechanism.

During transient creep, every decrease in stress is associated with the large and long anelastic backflow. The anelastic transient strain for stress reduction is equivalent to elastic deformation corresponding to the applied stress, while transient strain is 2.5 times greater than the equivalent elastic deformation regardless of whether stress increases or is constant. The transient effect was suggested to be due to a mix of anelastic behavior caused by the internal redistribution of stress and inelastic behavior. [doi:10.2320/matertrans.M2011175]

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1. Introduction

The creep of pure aluminum at low temperature ($T \leq 0.4T_m$) has been investigated in the last ten years by long-term[1)] and short-term uniaxal creep tests.[2,3)] The long-term creep of pure aluminum under precise constant-stress creep tests showed no occurrence of well-defined steady-state creep[1)] due to unexpected diffusion of atom at such temperatures. Recently, the short-term creep of pure aluminum[2,3)] was investigated at strain rate, $10^{-10}\,s^{-1} < \dot{\varepsilon} \leq 10^{-6}\,s^{-1}$, and creep curves were described by logarithmic equation to predict a "nominal steady-state creep rate". The results showed that the nominal steady-state creep rate was well consistent with that observed during the long-term creep, and characterized with stress exponent about 5 and very-low apparent creep activation energy (about $0.2Q_L$: Q_L, lattice diffusion energy). The creep is in the region of low-T, Q creep suggested by Matsunaga *et al.*[3)] After those studies, we attempted short-term helicoids spring creep at ultra-low strain rate, $\dot{\varepsilon} \leq 10^{-10}\,s^{-1}$, in pure aluminum with different grain sizes and impurity concentration.[4,5)] The results showed that creep may change into a new "low strain-rate region" characterized with stress exponent $n < 3$ and low activation energy of creep, $Q = 0.10$–$0.18Q_L$. However, in those study,[4,5)] an initial state of quenched 5N Al was chosen. The heat-treatment of quenching induces a high density of dislocation. Therefore, it is possible that creep behavior may have partly contributed to the high dislocation density, and this did not reflect the "true" creep behavior. On the other hand, the kinetic of creep deformation should be analyzed carefully. In addition, as an important stage of creep, transient creep behavior is still less studied in pure aluminum at low temperature.

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Contrasting to compression and tension tests, helicoid spring creep provides the highest strain sensitivity.[6)] Because of that, it has been employed to study creep of industrial parts[7–9)] at $\dot{\varepsilon} \leq 10^{-10}\,s^{-1}$. The demand for safe creep lives of such industrial parts is frequently 20 or 30 year, even longer. In that case, the creep rate can be very low, on the order of $10^{-10}\,s^{-1}$ or even lower. In this case, conventional uniaxal creep tests are less useful for measuring creep properties in such low creep rate due to very small magnitudes of strain, and very long duration of creep tests has to be produced.

On the other hand, the helicoid spring specimen technique is not free of its own limitations. The problems include the following: (1) complex strain/stress state. Although the strain/stress state in a helicoid spring is very complex, it is just normal strain and torsion strain contributing to creep deformation. Strains in other directions do not contribute creep deformation, and just influence on laternal contraction of helicoid coils. The distribution and change of strain were analyzed by finite element methods (FEM).[10)] The results showed the maximum of torsion strain was in the region of inside of surface and normal strain could be neglected under the condition of change of coil pitch spacing less than 5 mm. Those results are consistent with theory of helicoid sping.[11,12)] Because of that, the surface shear stress and strain are used to evaluate creep at low strain. It do be noted the neglect of normal strain contributing to creep may induce a slight less evaluation of creep strain; (2) the non-uniform distribution of stress across the specimen coil cross-section. The stress and strain increase from the center to the surface, where these values are at a maximum. Therefore, a redistribution of stress will occur when creep behavior is non-viscous.[13,14)] For non-viscous creep controlled by single creep mechanism, stress redistribution do not influence the creep strain rate and stress exponent, and just influence the threshold stress.[13)] Therefore, the helicoid spring creep tests

can be attempted to study creep controlled by single creep mechanism at ultra-low strain rate.

To better understand creep at $\dot{\varepsilon} \leq 10^{-10}\,\mathrm{s}^{-1}$, especially transient creep behavior, we performed helicoid spring creep tests under conditions of constant and changing stress in quenched high-purity aluminum (5N Al)[4,5] located in air within one year at room temperature ($T = 0.32T_\mathrm{m}$). For the specimen, dislocation density can be expected to be lower due to static recovery. First, the kinetics of creep deformation was studied, and creep curves were analyzed by the use of constitutive creep equation. Next, the nominal steady-state creep was studied by comparing with the results of helicoid spring creep tests in quenched 5N Al.[4,5] Finally, the magnitude and kinetic of a transient creep process was analyzed.

2. Experimental

The test materials were wire materials with diameter $d = 1.6\,\mathrm{mm}$ of 99.999% (5N) Al (NIPPON LIGHT METAL COMPANY, LTD).

The helicoid spring specimen technique[4,5,10] was used for creep testing at 298 K ($0.32T_\mathrm{m}$) and 0.18–4.20 MPa. The technique was modified to allow changes in stress. Elongation data were measured on personal computers at 1 s intervals at a stress in change. The coil-pitch spacing of helicoid samples was measured using a light-emitting diode with a resolution of 0.5 μm, which corresponds to a strain resolution of 8.4×10^{-8}. Helicoid spring specimens with a mean coil diameter $D = 18.8\,\mathrm{mm}$ were prepared by winding wires on threaded stainless steel bolts. The bolts with wound wires were quenched from 673 K. After quenching, the helicoid spring specimen was taken off from stainless bolts very carefully using liquid lubricant to easily take off and obtain an equal interval, δ and an equal diameter, D of the coils. The helicoid specimens were cleaned using acetone in order to minimize surface effects of liquid lubricant on the creep behavior. After that, the specimen was located in air within one year. In this case, the initial mobile dislocation density should be lower due to static recovery. The average grain size is $d_\mathrm{g} = 120\,\mu\mathrm{m}$. The effect of static recovery was evaluated by measuring the hardness change of water quenched specimen and the specimen located in air within one year. The changes of hardness were measured using Vickers method subjected to a load of 0.5 kg.

The following equations[11,12] are used to calculate the mean surface shear stress, τ, and the surface shear strain, γ, assuming pure torsion of the helicoid spring specimen:

$$\tau = \frac{8PD}{\pi d^3}, \tag{1}$$

$$\gamma = \frac{\delta d}{\pi D^2}, \tag{2}$$

where P is the average load, D is the coil diameter (18.8 mm), d is the wire diameter and δ is the average rate of deflection. The change of δ during creep deformation is between 2.5 mm to 3.5 mm. Since the stress and strain in the helicoid spring are essentially shear values, they can be transformed to the equivalent tensile quantities using tensile stress $\sigma = \sqrt{3}\tau$ and tensile strain $\varepsilon = \gamma/\sqrt{3}$.

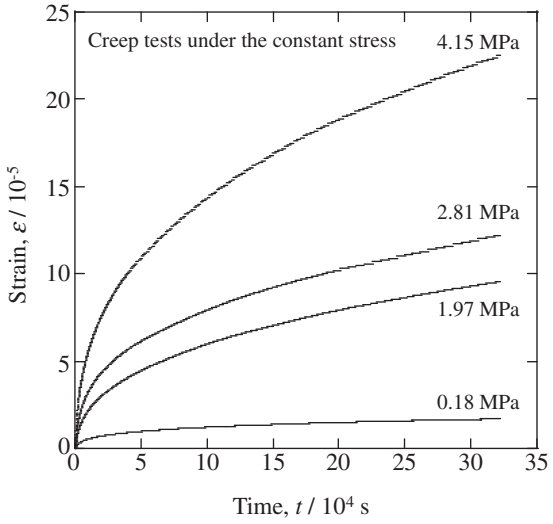


Fig. 1 Creep curves at 298 K obtained by creep tests under constant stress.

3. Results and Discussion

3.1 Creep curves

The creep curves of strain against time recorded under constant stress are shown in Fig. 1. All of the curves exhibit a transient stage which the creep rate decreases with time. To analyze the kinetic of creep process and identify whether the steady-state creep commences, creep strain rate is plotted against time in Fig. 2(a) and strain in Fig. 2(b) on a log-log plot. Creep deformation of the pure aluminum consists of primary creep stages, and no steady-state creep stage is observed.

The strain versus time creep curve recorded in the changing-stress creep experiment is shown in Fig. 3(a), where the elastic strain has been removed. Segments of the creep curve are denoted by the sequential number of precedent changes in stress k. Each increase in stress is followed by a pronounced transient effect that resembles the transient stage of the constant-stress creep curve. Under a decrease in stress, backward creep flow was seen in all cases. The backflow is seen in more detail in inset, which shows part of the creep curve with a change in stress from 4.08 to 5.03 MPa and back, corresponding to a change in k from 8 to 9, and then to 10.

The strain rate versus time curve in a log-log scale is shown in Fig. 3(b). This figure shows the following: (1) no apparent steady-state creep stage regardless of increase and decrease of stress is observed even for a long period of 1.3×10^6 s; (2) the transient responses recorded after increase and decrease of stress are different. For an increase of stress, the creep rate decreases with increase of testing time due to work hardening mechanism. For a decrease of stress, the creep rate increases with increase of testing time to the maximum at B. Following the maximum, the creep rate decreases. The results indicate that anelastic creep recovery firstly occurs, and then work hardening starts under a decrease of stress. The duration of the elastic creep recovery is long, $\sim 2.3 \times 10^5$ s.

Figure 4 shows strain versus time (a) and strain rate versus time (b) in a log-log scale individual segments ($k = 1$, 3 and

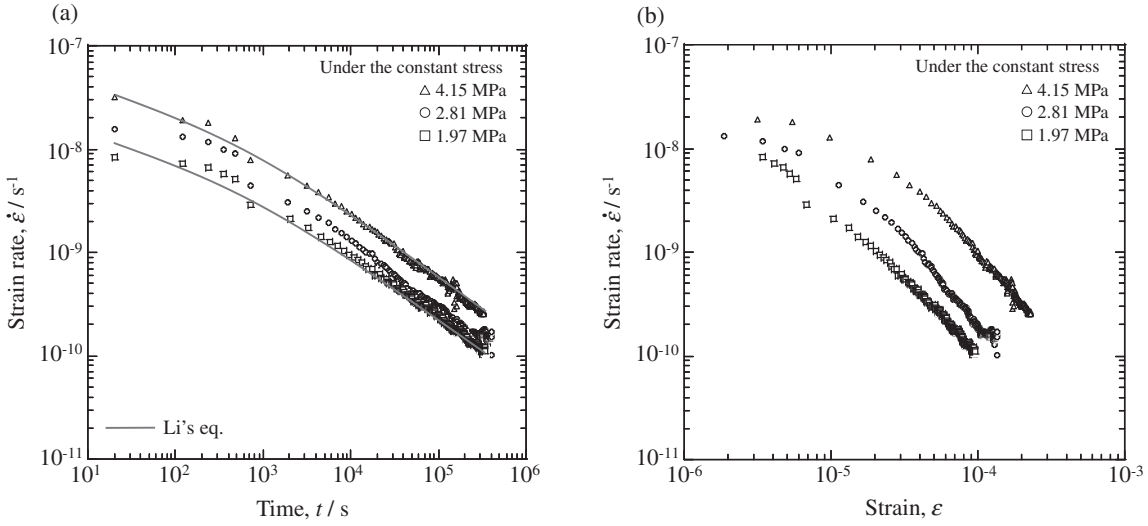


Fig. 2 Strain rate versus time curves (a) and strain (b) on a log-log scale at 298 K under constant stress.

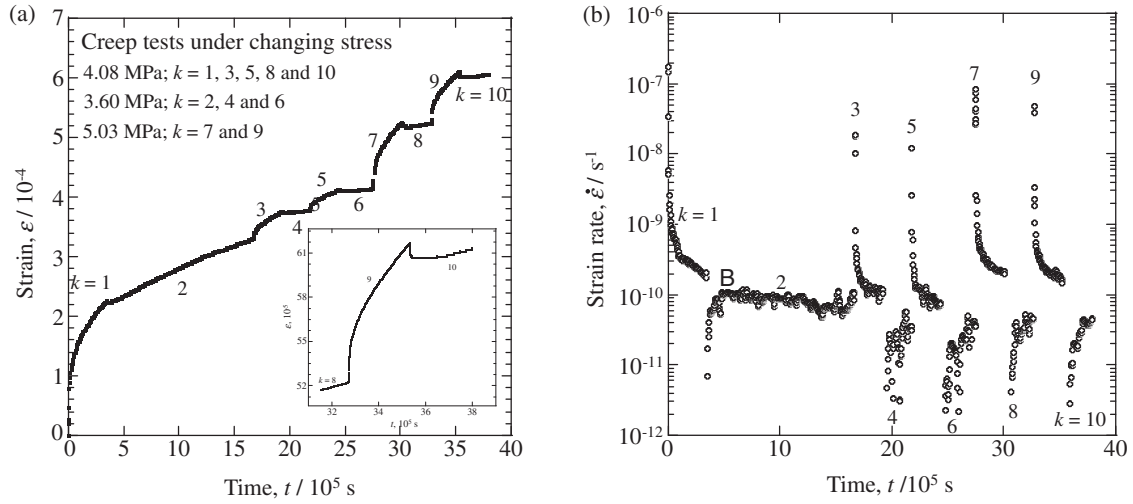


Fig. 3 Creep curves for the changing-stress experiment at 298 K. Individual segments are marked by a sequential number k. (a) Strain versus time curves and (b) Strain rate versus time curves. Mark "B" indicates the maximum of anelastic strain rates for a decrease of stress.

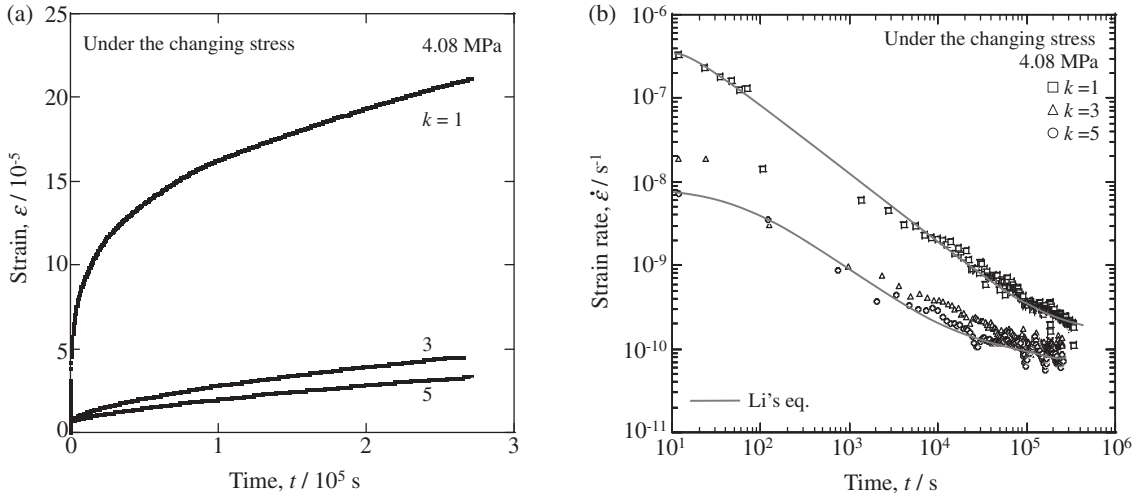


Fig. 4 Individual segments (k = 1, 3 and 5) of creep curve recorded in the initial stress, 4.08 MPa and after a change to 4.08 MPa. (a) Strain versus time. (b) Strain rate versus time on a log-log scale.

5) of creep curve recorded in the initial stress, 4.08 MPa and after a change to 4.08 MPa. With changes in stress, the strain and strain rate progressively decreases in subsequent segments.

To describe the experimental creep curves under constant stress and increase in stress, constitutive creep equations for the transient and steady-state creep stages should be used. We selected Li's equation[15] to describe creep curves of pure aluminum, which was used to fit creep curves at low temperature.[16] The equation is based on the multiplication and exhaustion (immobilization) of dislocations in the transient creep stage:[15]

$$\varepsilon = \varepsilon_0 + \dot{\varepsilon}_{n\text{-}s} t + \frac{\dot{\varepsilon}_s}{k_t} \ln\left[1 + \frac{\dot{\varepsilon}_i - \dot{\varepsilon}_{n\text{-}s}}{\dot{\varepsilon}_{n\text{-}s}} (1 - e^{-k_1 t})\right] \quad (3)$$

where ε is the strain, ε_0 is the instantaneous strain, $\dot{\varepsilon}_{n\text{-}s}$ is the nominal steady-state creep rate, $\dot{\varepsilon}_i$ is the initial strain rate, t is the creep time, k_1 is the dislocation multiplication rate constant and ε_p and β_p are parameters characterizing the primary creep region.

The results of a regression analysis are shown in Fig. 2(a) for creep under constant stress and in Fig. 4(b) for creep under increase of stress. The Li's equation very well duplicates the creep rate versus time curve. Therefore, Li's equation can be used to describe creep in this study.

For creep under the constant stress and increase stress, the nominal steady-state creep rate is described by the parameter $\dot{\varepsilon}_{n\text{-}s}$ while the transient creep can be characterized by the transient strain ε_t, for which the relation $\varepsilon_t = \dot{\varepsilon}_{n\text{-}s} t_p \ln(\dot{\varepsilon}_i/\dot{\varepsilon}_{n\text{-}s})$ holds. We do not consider the $\dot{\varepsilon}_{n\text{-}s}$ on decrease of stress, because most of observed creep deformation is still in the stage of anelastic backflow. The total anelastic-strain ε_a is identical to the strain derived directly at the end of each segment of the creep curve in Fig. 3(a) that is corresponding to the maximum value at B in Fig. 3(b).

3.2 Nominal steady-state creep

The dependence of the nominal steady-state creep rate $\dot{\varepsilon}_{n\text{-}s}$ obtained by Li's equation on a constant and increase of stress normalized is summarized in Fig. 5. At high stress, constant-stress data obtained in uniaxial creep tests were taken from Ref. 3). At low stress, the data in quenched 5N Al obtained in helicoids spring creep tests marked by solid round were taken from Ref. 4, 5). The data marked by hollow shapes were obtained in quenched 5N Al located in air within one year by helicoid-spring creep tests. The stress exponent is equal to one that is consistent with our earlier studies.[4,5] However, the nominal steady-state creep rate for quenched specimens is one order less than that for quenched specimens located in air within one year. The creep behavior was in term of the effect work (cold) hardening on creep. The hardness for 5N Al after quenching was about 18 HV, whereas it is about 13 HV for the specimens located in air within one year.

With increase in stress, the creep rate for the first segment corresponds well with the constant-stress results, while it progressively decreases in subsequent segments. Thus, increase in stress induces creep strengthening, which may be due to different hardening remains because changing-stress creep experiment was conducted in the transient creep stage. Those phenomena of work hardening indicate creep deformation is controlled by recovery and work-hardening mechanism.

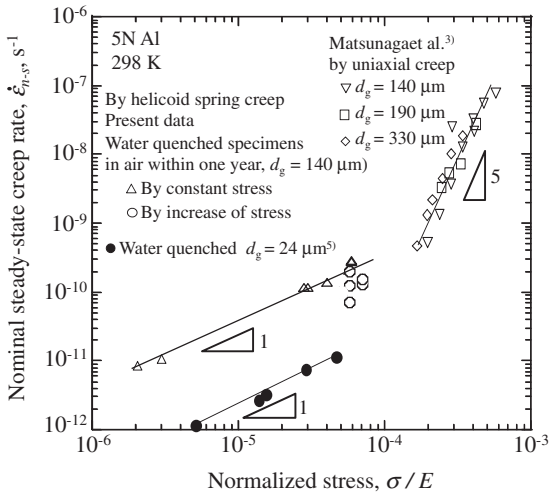


Fig. 5 Dependence of the nominal steady-state creep rate on the normalized stress, compared with the results for constant-stress by uniaxial creep tests[3] and helicoid-spring creep tests.[5]

Table 1 $\Delta\sigma^-/\Delta\varepsilon^-$ calculated from decrease in stress.

Stress change, $\Delta\sigma^-$ (MPa)	Loading histrory, k	Instantaneous strain, ε^-_{ins}	$\Delta\sigma^-/\varepsilon^-_{ins}$ (GPa)
0.48⁻	1 → 2	1.5×10^{-5}	32
	3 → 4	1.4×10^{-5}	34
	5 → 6	1.1×10^{-5}	44
0.95⁻	7 → 8	3×10^{-5}	32
	9 → 10	3.1×10^{-5}	31

3.3 Transient creep

For each stress reduction, the instantaneous strain should reflect elastic contraction. Thus, the value calculated from $\Delta\sigma^-/\Delta\varepsilon^-$ in Table 1 should correspond to the elastic modulus of aluminum. However, the average value of 35 GPa is smaller than the elastic strain, 70 GPa in pure aluminum at $T = 0.32\,T_m$. This result reflects the presence of anelastic strain during creep deformation. Anelasticity causes backward flow of creep deformation after a decrease in stress.

This anelastic backflow should be due to the internal redistribution of stress,[17,18] which assumes some weaker and stronger sites in solid, and the instantaneous strain after a decrease in stress should probably be attributed to the internal redistribution of stress. The measured instantaneous strain ε_i is equal to $\varepsilon_e + \varepsilon_a$ (anelastic strain). The value of 35 GPa is half of 70 GPa. Thus, anelastic strain is equal to elastic strain. In addition, the ratio of $|\varepsilon_a|/\varepsilon_e$ is equal to one shown in Fig. 6(a), which is consistent with the above assumption.

In contrast, the ratio of transient strain to calculated elastic strain increases to about 2.5 after an increase in stress and under constant stress shown in Fig. 6(b). This indicates that an inelastic mode of transient creep also contributes to transient creep behavior. This inelastic model may be related to the grain boundary.[5]

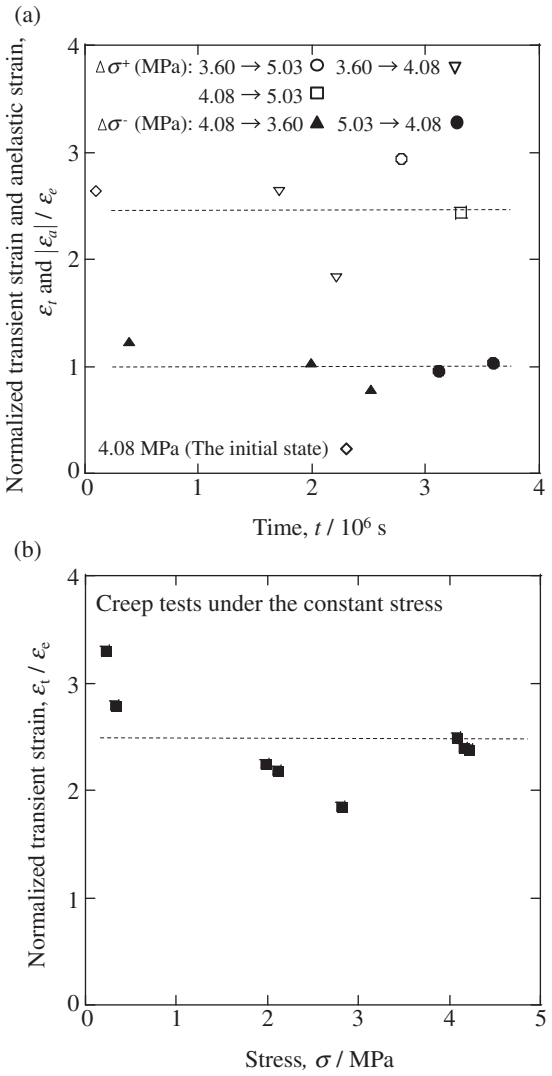


Fig. 6 The normalized transient strain ε_t and anelastic strain ε_a plotted against the time of the corresponding change in stress (a); and the normalized transient strain plotted against stress in constant-stress creep (b).

Based on the above discussion, the transient creep in 5N Al at low strain-rate and room temperature should be due to a mix of anelastic behavior caused by the internal redistribution of stress and inelastic behavior that may be related to the grain boundary.

4. Conclusion

Creep of water quenched high-purity aluminum (5N Al) located in air within one year at room temperature (298 K) and ultra-low strain rate ($\leq 10^{-10}\,s^{-1}$) was investigated by the helicoid spring specimen technique under conditions of constant and changing stress and comparing with creep behavior in water quenched 5N Al.[4,5] Creep deformation consists of transient creep stages, and no secondary creep stage was observed. A preliminary investigation suggested that the Li's equation could accurately reproduce the experimental creep curves.

Under the nominal steady-state creep, the stress exponent is equal to one regardless of initial state of specimens. However, the nominal steady-state creep rate for water quenched 5N Al is one order less than that for quenched specimens located in air within one year due to work (cold) hardening. The hardness for 5N Al after quenching was about 18 HV, whereas it is about 13 HV for the specimens located in air within one year. With increase in stress, creep strengthening (the creep rate progressively decreasing in subsequent segments) was observed, which is due to different hardening remains because changing-stress creep experiment was conducted in the transient creep stage. Those phenomena of work hardening provided evidences of creep controlled by recovery and hardening mechanism.

Under transient creep, every decrease in stress is associated with large and long anelastic backflow. The anelastic strain is equivalent to elastic deformation corresponding to the applied stress, while it is 2.5 times greater than the equivalent elastic deformation under increase of stress and constant stress. The transient effect was suggested to be due to a mix of anelastic behavior caused by the internal redistribution of stress and inelastic behavior.

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