Creep of a Recrystallized Aluminum SAP-Type Alloy

The creep behavior of an aluminum-aluminum oxide alloy, AT 400, fabricated by compacting an atomized aluminum powder, extruding the compact, cold working, and recrystallizing the extrusion, was investigated. This alloy has a dispersion spacing of 1 μ. Previously Ansell and Weertman[1] *determined the steady-state creep rate of a recrystallized aluminum-aluminum oxide SAP-type alloy, MD 2100, with a 0.48-μ dispersion. This alloy was found to have a steady-state creep rate of less than 10^{-8} min^{-1}, too small to be measured accurately. By employing a more sensitive method of measuring creep rate, it was possible to determine the steady-state creep rate of AT 400 alloy, which is somewhat faster than that of MD 2100, at a series of temperature and applied stresses. Above a stress of $2\mu b/\lambda$ (b = Burger's vector, λ = dispersion spacing, μ = is a shear modulus) the rate follows an equation of the type: Steady-state creep rate $K = A \exp(-Q/kT)\sigma^4/\mu^3 kT$ where A is a constant, Q is the activation energy for self-diffusion, and σ is the applied stress. At stresses lower than $2\mu b/\lambda$ the steady-state creep rate drops off sharply and is no longer in agreement with this equation. This behavior is in good agreement with that predicted by the model for dispersion strengthened alloys in the stress and temperature range investigated. The absolute value of the creep rate is, however, four orders of magnitude lower than that predicted if the normal number of active dislocation sources are assumed to be present. It is concluded that in this SAP-type alloy the usual three-dimensional dislocation network is not present. Instead, short dislocation segments extending from one dispersed particle and terminating at a neighboring particle may act as dislocation sources. With this provision the creep model proposed by Ansell and Weertman reasonably accounts for the creep behavior which was observed.*

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RECENTLY, Ansell and Weertman[1] proposed a dislocation model from which they derived creep equations to describe the steady-state creep behavior of dispersion - strengthened alloys. Following Schoeck,[2] they assumed that the rate controlling process for steady-state creep is the climb of dislocations over the dispersed particles.

In order to verify their creep model, Ansell and Weertman determined the steady-state creep behavior of an aluminum-aluminum oxide SAP-type alloy, MD-2100, in both the fine-grained as-extruded, and coarse-grained recrystallized conditions. Since the grain size was the order of the dispersion-spacing, their model was not applicable for the as-extruded alloy. It should, however, be applicable to the coarse-grained recrystallized alloy. The results they obtained were rather unexpected. No measureable steady-state creep was observed for the recrystallized MD-2100 alloy.

If their model is correct, and if the second-phase particles act solely to hinder dislocation motion, then some measureable steady-state creep would have been expected. On this basis, they postulated that, in this SAP-type alloy, the main effect of the fine dispersion was to inactivate dislocation sources rather than to hinder the movement of dislocations.

In order to understand more fully this unusual creep behavior, and to determine the validity of the model proposed,[1] the steady-state creep behavior of an aluminum-aluminum oxide recrystallized SAP-type alloy, with a somewhat coarser dispersion spacing, was investigated. The results of this study are presented in this paper.

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THEORY

The dislocation model for steady-state creep proposed by Ansell and Weertman[1] is as follows: For low stresses, where the stress is just sufficient to nucleate dislocations from a source in the matrix, up to a stress which is great enough to bow dislocations between the dispersed particles, the rate controlling process for steady-state creep is the climb of single dislocations over the second phase particles. If Frank-Read sources are present due to a continuous dislocations network, for resolved stresses in the range

$$\mu \mathbf{b}/L < \sigma < \mu \mathbf{b}/\lambda \qquad [1]$$

(μ is a shear modulus, L is the length between nodes in the dislocation network, σ is the applied stress, b is the Burgers vector of the dislocation, and λ is the dispersion spacing), the steady-state creep rate, K, for resolved stresses and strains is

$$K = \pi \sigma \mathbf{b}^3 D/2kTh \qquad [2]$$

where D is the coefficient of self-diffusion, k is Boltzmann's constant, T is the absolute temperature, and h is the height of the dispersed particle.

At stresses great enough to pinch-off dislocation loops about the dispersed particles, Eq. [2] is no longer applicable. The rate controlling process for steady-state creep is now the climb of an array of piled-up, pinched-off dislocation loops rather than the climb of single dislocations. Therefore at stresses grater than μb/λ, steady-state creep rate predicted by the model, for resolved stresses and strains, is

$$K = 2\pi \sigma^4 \lambda^2 D/h\, \mu^3 kT \qquad [3]$$

which is valid up to stresses where

$$\sigma = (\mu k T/2\lambda \mathbf{b}^2)^{1/2} \qquad [4]$$

The density of active dislocation sources in these alloys has been implicitly assumed in the derivation of Eqs. [2] and [3] using a calculation that had been used previously for single-phase alloys.[3]

EXPERIMENTAL DETAILS

The alloy studied, designated AT 400, was fabricated by compacting and subsequently extruding 3-μ average diameter atomized aluminum powder. The alloy is therefore similar to an SAP-type alloy, R3M, whose microstructure has been reported previously.[4] The extrusion was then recrystallized by first wire drawing to a 55.6 pct reduction in area and then annealing for 2 hr at 582°C. The grain size of the recrystallized alloy was several millimeters in diameter. The microstructure of this large-grained recrystallized alloy is similar to that of the recrystallized MD 2100 alloy. Each consists of flakes of aluminum oxide, 130 Angstrom units thick and about 0.3 μ on edge, dispersed in an aluminum matrix. However, the average dispersion spacing is approximately 0.9 μ for the AT 400 alloy as compared with the 0.5-μ spacing for the MD2100 alloy.[4]

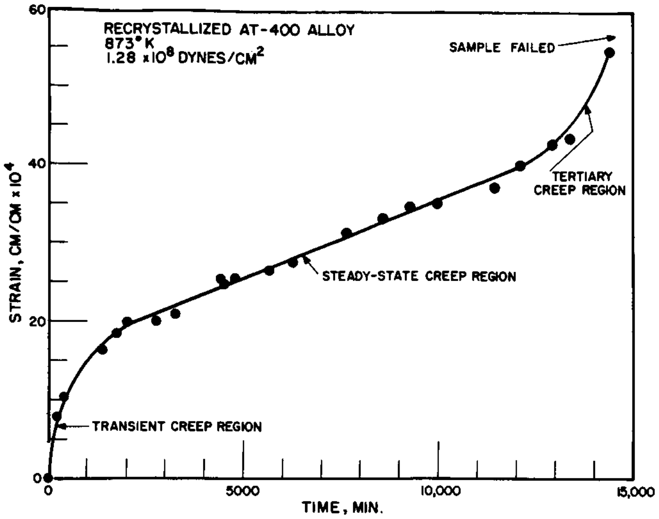

Fig. 1—Typical creep curve, recrystallized AT-400 alloy.

Creep measurements were made on wire test samples, with a 2-cm centimeter gage length, held in friction type wire grips. The test temperatures ranged between 500°and 620°C. The test temperatures were held to within 1°C for the duration of each test. All tests were run under the condition of constant loading and can be considered constant stress tests since the creep strains were small. The stresses used at each temperature ranged from a stress which was just large enough to give a measureable steady-state creep rate to the stress at which the sample fractured immediately upon loading. Creep extensions were measured with a micrometer slide cathetometer, permitting the determination of a minimum resolvable strain of 10^{-4}.

In addition to the constant temperature, constant stress creep tests, tests were made in which the temperature or stress was changed abruptly after steady-state creep had been reached, while holding the stress or temperature constant. This produced

Table I. Steady-State Creep Data AT-400 Recrystallized Alloy

Sample No.	Temp, °K	Stress, Dynes per cm²	Steady-State Creep Rate, Min⁻¹	Temp Comp Steady-State Creep Rate, Min⁻¹	Test Duration, Min
16d	893	9.75×10^7	1.64×10^{-8}	4.90×10^{-10}	60,000
16c	893	1.11×10^8	5.00×10^{-8}	1.73×10^{-9}	10,000
16a	873	9.75×10^7	7.50×10^{-9}	4.16×10^{-10}	14,348
16b	873	1.11×10^8	3.48×10^{-8}	1.94×10^{-9}	30,000
16e	873	1.20×10^8	4.24×10^{-8}	2.35×10^{-9}	9,000
15a	873	1.28×10^8	1.52×10^{-7}	8.45×10^{-9}	14,371*
14a	873	1.53×10^8	1.85×10^{-7}	1.01×10^{-8}	6,000 to 7,000*
16f	823	1.70×10^8	8.83×10^{-8}	1.87×10^{-8}	3,000 to 7,000*
4a	773	1.82×10^8	2.40×10^{-8}	2.40×10^{-8}	25,929
18a	773	2.10×10^8	4.06×10^{-8}	4.06×10^{-8}	19,885
9a	773	2.34×10^8	6.10×10^{-8}	6.10×10^{-8}	15,516
11a	773	2.60×10^8	9.20×10^{-8}	9.20×10^{-8}	9,370*
6a	773	2.92×10^8	2.53×10^{-7}	2.53×10^{-7}	1,020*

*Indicates sample failed during test.

Temperature compensated steady-state creep rate calculated using an activation energy for creep of 37,000 cal/mole and 773°K as the reference temperature.

a short period of transient creep behavior before a new steady-state creep region appeared. The steady-state creep rates under these new conditions were then measured.

RESULTS

The creep curves for each of the samples tested showed a region of steady-state creep from which the steady-state creep rate could be measured. Typical creep curves are shown in Figs. 1 and 2. Summary data from all the creep tests are listed in Table I. Some data in the table are the creep rates at several temperatures and stresses determined from a single test specimen. In these cases, the letter following the test specimen number indicates the sequence of testing conditions.

The range of stresses used for the creep testing for any one particular temperature was very limited. This was due to the narrow range of stress between the stress where the strain rate was barely measureable and the stress where the sample fractured immediately upon loading. In order to determine the effect of stress upon the steady-state creep behavior of the AT-400 alloy over a wider range of stress than a single test temperature would allow, the data from all the creep tests were adjusted to a single test temperature, 500°C., by using as a basis Eq. [3] of the creep theory for dispersion-strengthened alloys. A treatment of this type implicitly compares the experimental results with the proposed theory. This temperature-compensated creep rate was determined in the following manner:

According to Eq. [3]

Steady-state creep rate = $K = 2\pi\sigma^4\lambda^2 D_0 \exp(-Q/RT)/h\mu^3 kT$

where: $\exp(-Q/RT),\ \mu,\ T = f(T)$

$\lambda,\ D_0,\ h,\ k \neq f(T)$

Since for any particular alloy T and σ are the only independent variables treated by the creep theory, eliminating T as a variable, with 773 °K equal to the reference temperature T_θ, and T equal to the test temperature, the temperature-compensated creep rate, K^*, is

$$K^* = K(\exp(-Q/RT_\theta)/\exp(-Q/RT))(\mu_T/\mu_{T_\theta})^3 (T/T_\theta) \qquad [5]$$

or

$$K^* = K(T/T_\theta)(\mu_T/\mu_{T_\theta})^3 \exp(-Q(T-T_\theta)/RTT_\theta) \qquad [6]$$

in which the subscripts T and θ refer to the values of the properties or test conditions at test temperature and reference temperature respectively.

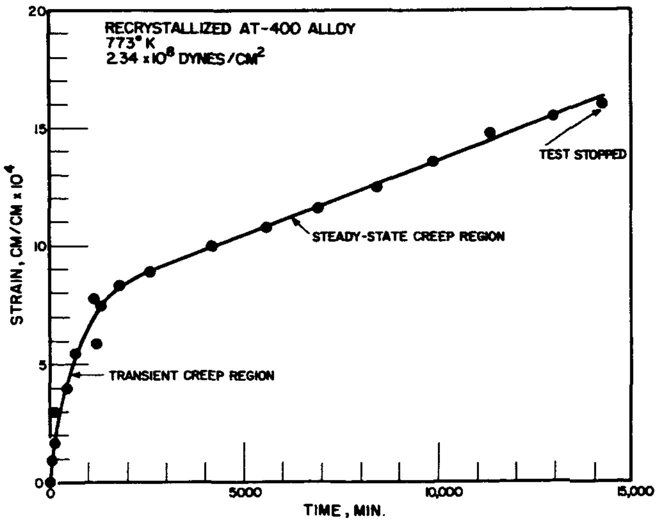

Fig. 2—Typical creep curve, recrystallized AT-400 alloy.

In order to reduce the steady-state creep rate at temperatures other than 500°C to a temperature compensated creep rate, it is necessary to know both the temperature dependence of the shear modulus of aluminum, and the value of the activation energy, Q, for steady-state creep.

The temperature dependence of the shear modulus was determined from the data of Sutton.[5] Sutton reported the elastic constants of aluminum over the temperature range 315° to 773°K. The shear modulus, μ, was calculated from these data using the equation, $\mu = \sqrt{1/2\, C_{44}(C_{11} - C_{12})}$, where C_{ij} are the usual elastic constants. These data were then extrapolated linearly to 893°K.

The creep theory assumes that the activation energy for steady-state creep is the same as that for self-diffusion. The activation energy for steady-state creep was therefore assumed to be 37,000 cal per

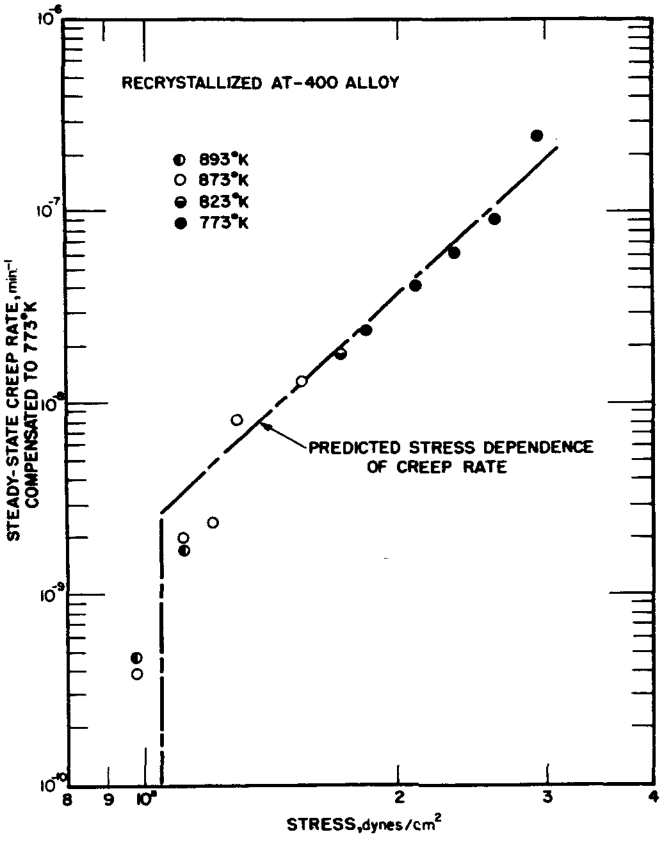

Fig. 3—Logarithm of the temperature-compensated steady-state creep rate of recrystallized AT-400 alloy plotted vs the applied stress.

mole, in accordance with indirect estimates of the activation energy for self-diffusion in aluminum.[6,7] The calculated temperature compensated creep rates for all the steady-state creep data are shown in column 5 of Table I.

This method of compensating the creep rates obtained at different test temperatures to a single temperature, in this case 500 °C., made it possible to determine the effect of stress on the creep rate of this alloy over a reasonably large stress range. The logarithm of the temperature-compensated steady-state creep rate is shown plotted vs the logarithm of the applied stress in Fig. 3. In this same figure, a line was drawn whose slope corresponds to a fourth power stress dependence of the steady-state creep rate. The position of the fourth power line was arbitrarily taken to fit the data.

The reasonable fit of the data with the fourth power line indicates that, for the stress region where according to the dislocation theory for creep piled-up dislocation loops are present, the steady-state creep rate follows the fourth power stress dependence predicted in agreement with Eq. [3]. At lower stresses however, the steady-state creep rate does not follow the stress dependence predicted by the theory, as indicated by Eq. [2], but falls off sharply with decreasing stress. Therefore a line was arbitrarily drawn in Fig. 3 to correspond to an infinite stress dependence of the steady-state creep rate at a stress equal to $2\mu b/\lambda$.

DISCUSSION

The present study of the AT-400 recrystallized alloy shows that the steady-state creep data observed may be described by the theoretical treatment of Ansell and Weertman if the activation energy for steady-state creep is assumed to be the same as that which has been previously estimated for self-diffusion in aluminum. This assumption for the activation energy implies that the climb of dislocations over the second-phase particles is the rate controlling process for steady-state creep in this alloy. At this point it is now relevant to compare the steady-state creep data with the Eqs. [2] and [3] predicted by their model.

At the low stresses, in the range given by Eq. [1], the rate-controlling process for steady-state creep was proposed to be the climb of single dislocations over the dispersed particles. The steady-state creep rate in this stress region should be a first-power function of the applied stress as shown in Eq. [2]. From Fig. 3, apparently this is not the case. In this stress region the steady-state creep rate decreases rapidly with decreasing stress, and for stresses just a little lower than $2\mu b/\lambda$ the steady-state creep rate falls off essentially to zero. At these stresses, the applied stress is probably too low to nucleate dislocations from the sources present in the alloy. This would explain why at these stresses, the steady-state creep rate falls off essentially to zero.

At stresses greater than $2\mu b/\lambda$ dislocations are nucleated from some type of sources in the alloy. The rate-controlling process for steady-state creep is now the climb of piled-up arrays of pinched-off dislocation loops over the oxide particles, and Eq. [3] should be applicable. It has been shown previously that the stress and temperature dependence of the steady-state creep rate of the AT-400 alloy follows Eq. [3]. The absolute values of the observed creep rate, however, are much lower than those predicted. Correcting Eq. [3] for unresolved stresses and strains, *i.e.* dividing the right-hand side of the equation by $16 \times \sqrt{2}$, the predicted rate is approximately 4 orders of magnitude faster than the observed one. Since the stress and temperature dependence of the creep rate, and also the stress range where this behavior is observed follows Eq. [3], it appears that the model used to describe the creep behavior is correct, except that the assumed density of active dislocation sources (dislocation sources which are generating new dislocations during steady-state creep) is much too high. The model used for the calculation of the density of these sources was one which holds well for single-phase metals. The conclusion must, therefore, be drawn that the density of active dislocation sources in the AT-400 alloy is approximately 10^{-4} that of a single-phase metal. It is proposed that this difference is largely due to the character of the dislocation sources present, specifically that there is no continuous dislocation network present in this alloy. Perhaps, it is absent due to interactions of dislocations with the oxide flakes as proposed previously by Ansell;[8] The stress at which dislocations are nucleated in this alloy corresponds to Frank-Read sources whose lengths are the average spacing between oxide flakes dispersed in the alloy, in agreement with the suggestion by Ansell and Weertman, that the dislocation sources present are short dislocation segments extending from one oxide flake to another.

CONCLUSIONS

1) The rate controlling process for steady-state creep in this aluminum-aluminum oxide SAP-type alloy is the climb of dislocations over the dispersed aluminum oxide flakes.

2) The density of active dislocation sources in the AT-400 alloy is abnormally small as compared with the density of active dislocation sources in a single-phase metal.

3) The creep theory proposed by Ansell and Weertman for the steady-state creep of a dispersion strengthened alloy appears to apply to the AT-400 alloy, except for their assumption as to the density of active dislocation sources present.

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REFERENCES

[1] G. S. Ansell and J. Weertman: *Trans. Met. Soc. AIME*, 1959, vol. 215, p. 838.
[2] G. Schoeck: *Creep and Recovery*, ASM, Cleveland, p. 199, 1957.
[3] J. Weertman: *J. Appl. Phys.*, 1957, vol. 28, p. 362.
[4] F. V. Lenel, G. S. Ansell, and E. C. Nelson: *AIME Trans.*, 1957, vol. 209, p. 117.
[5] J. Weertman: *J. Appl. Phys.*, 1956, vol. 27, p. 832.
[6] Nochtrieb, Weil, Catalano, and Lawson, *J. Chem. Phys.*, 1952, vol. 20, p. 1189.
[7] G. S. Ansell: *Trans. Met. Soc. AIME*, 1959, vol. 215, p. 294.