Rate-Controlling Process of Power Law Creep at High Temperatures in Powder Metallurgy Tungsten Wires

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Creep tests and scanning electron microscopy (SEM) observation of grain boundary cavitation are made to decide the most likely mechanism which rate-controls the power law creep at high temperatures in powder metallurgy tungsten wires. The modified Monkman-Grant relation holds for the overall time, the strain to fracture and the minimum creep rate during creep. The activation energy for steady state creep is obtained to be about 130 kJ/mol which is much less than the energies for surface, dislocation, grain boundary and lattice diffusion of tungsten. The value does not approach the activation energy for lattice diffusion as the mechanism of dislocations climb even if the effect of grain boundary cavitation on creep rates is taken into account. It is concluded that the small activation energy for steady state creep in doped wire results from a combination of the effect of grain boundary cavitation on creep rates and the deformation of the constraining surroundings which proceeds near the surface of wire.

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I. Introduction

A small amount of Al, K and Si are doped in the powder-metallurgical manufacturing process of tungsten ingots to get a high-temperature creep resistance. The doping elements develop many arrays of small bubbles along the wire axis during fabrication and thereby provide some benefits termed "bubble effects". Two main effects to prevent a lamp filament from sagging should be mentioned. First, a number of bubbles existing on grain boundaries play a role in controlling the morphology of secondary recrystallized grains⁽¹⁾. They produce an elongated and interlocked grain structure by impeding the transverse grain growth across the wire axis⁽²⁾. The elongated grain structure reduces creep strains resulting from the grain boundary itself such as grain boundary diffusion and sliding. On the other hand, the interlocked grain structure will suppress the rotative grain boundary sliding due to torsional stresses imposed on coiled wires under the influence of gravity⁽³⁾⁽⁴⁾. Second, the bubbles existing inside a grain interact with dislocations and may strengthen the matrix in three ways⁽⁵⁾: (i) When bubbles are cut by dislocations, work has to be done because the surface of the bubbles increases. (ii) The equilibrium shape of bubbles is often polyhedral because it must give the lowest value of the total interfacial free energy for fixed volume of the bubbles⁽⁶⁾. If the total interfacial energy is not minimum, the bubbles are non-equilibrium, their shape being irregular⁽⁷⁾. The dislocation then moves into the misfit-stress field around the non-equilibrium

bubbles. (iii) Dislocations are attracted by both equilibrium and non-equilibrium bubbles because they annihilate a part of the dislocation stress field. The last strengthening mechanism is considered as a most effective one in blocking the climb of dislocations at high temperatures⁽⁸⁾.

It is also reported that some cavities grow from the largest potassium bubbles (larger than 0.08 μm) initially present on the grain boundaries and their growth may be enhanced by grain boundary sliding⁽⁹⁾. Accordingly, the bubble should take part in the deformation at high temperatures in various ways; the rate-controlling mechanism in the region of power law creep may be one or a combination of the processes such as diffusion controlled cavity growth, climb of dislocations, thermally assisted breakaway of dislocations from bubbles, grain boundary sliding, short-circuit diffusion along dislocations and grain boundaries, diffusion along the surface of cavities and wires. The purpose of this paper is to decide the most likely mechanism which rate-controls power law creep at high temperatures in powder metallurgy tungsten wires.

II. Experimental Procedure

Materials used in the present work were doped and non-doped tungsten wires of which the purity was above 99.96% and the diameter was 0.13 mm. The concentrations of residual doping elements of Al, K and Si were <0.001, <0.007 and 0.001 ± 0.0003 in mass %, respectively. All of the heating was done by applying direct current to the wire specimens in a vacuum of 10^{-4} Pa. Secondary recrystallization was completed by annealing for 300 s at 2373 K after heating at the heating rate of

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880 K/s. The transverse grain size d_t after secondary recrystallization was measured to be 40 and 15 μm , giving the grain-aspect ratio of 10 and 2 for doped and non-doped wires, respectively. Simple tension and creep tests were then carried out using an Instron type tensile testing machine at 1873–2673 K. While the specimens with a gage length of 40 mm were deformed at the nominal strain rate of $3.3 \times 10^{-4} \text{ s}^{-1}$ in tensile tests, the loads imposed on the specimens with a gage length of 50 mm were kept constant by an automatic load controller in creep tests. The constant power supply was specially designed to control the current so that the temperature of the specimens remained constant during tensile deformation. The fractured specimens were observed using scanning electron microscopy (SEM) to examine the fracture mode and the grain boundary sliding. To get more information about grain boundary cavitation, some of the specimens were mounted in a resin after the interruption of creep tests at the middle of the steady state creep and polished for observation of the cavities on a longitudinal flat plane. The shape and the size distribution of the cavities were then measured mainly in the doped wires after etching for a few seconds with a solution of $30 \text{ gK}_3\text{Fe(CN)}_6 + 2 \text{ gNaOH} + 100 \text{ cm}^3 \text{ H}_2\text{O}$. Details of the procedure are described elsewhere⁽¹⁰⁾.

III. Experimental Results

Typical true stress-true strain curves of both materials are shown in Fig. 1. The flow stresses of the doped wire are almost equal to twice those of the non-doped wire at the proportional limits where the macroscopic deformation starts. The difference is thought to result from blocking the climb of dislocations by bubbles. The higher work-hardening rates result in the small total elongations for non-doped wires. However, these small total elongations may not represent the true ones because Joule heating causes a specimen wire to melt down as soon as plastic instability or necking starts. Typical creep curves at 2673 K are given in Fig. 2. Creep strains of doped wire are an order of magnitude less than those of non-doped wire, and the decelerating (primary) stage appears in the

Fig. 1 True stress-true strain curves as a function of temperature for doped and non-doped tungsten wires heated to 2373 K at the rate of 880 K/s and annealed for 300 s.

Fig. 2 Typical creep curves as a function of stress for (a) doped and (b) non-doped tungsten wires crept at 2673 K after they were heated to 2373 K at the rate of 880 K/s and annealed for 300 s.

low stress range. This implies that the exhaustion of thermally activated deformation events occurs slowly during the primary stage of doped wire⁽¹¹⁾. Namely the increase in internal stress during creep at the primary stage proceeds more slowly in doped than in non-doped wire. This idea can be easily checked by different work-hardening rates of Fig. 1. The minimum creep rates $\dot{\epsilon}_m$ obtained from the creep curves are plotted in Figs. 3 and 4 against stress normalized by the respective shear modulus⁽¹²⁾. The data for doped wire were located in the region of power law creep in the deformation mechanism maps except for those with a slope of one at 2673 K suggesting the occurrence of diffusional creep⁽¹⁰⁾. The creep rates of non-doped wire are approximately two orders of magnitude larger than those of doped wire. The stress exponent n ranges from 3 to 5 and 2 to 5 for doped and non-doped wires, respectively. It is seen from the figures that the deformation of the former wire obeys the natural creep law with $n=3$ over a wide range, while n of the latter wire changes continuously. The drastic increases of n at the high stress levels, viz. the breakdown of power law creep, may originate in the enhanced diffusion due to the excess vacancies produced during the cutting of dislocations⁽¹⁰⁾⁽¹³⁾. Failures of both wires occur with slight necking as shown in Fig. 5, and their fractographies reveal primarily intergranular fracture. It should be also noticed that the creep of non-doped wire is strongly affected by

Fig. 3 Logarithmic plot of the minimum creep rate against the modulus corrected stress for doped wire rapidly heated to 2373 K and annealed for 300 s.

Fig. 4 Logarithmic plot of the minimum creep rate against the modulus corrected stress for non-doped wire rapidly heated to 2373 K and annealed for 300 s.

grain boundary sliding.

Figure 6 shows some cavities grown on a grain boundary facet in a crept wire. A void cluster is formed along the boundary in the figure, the shape of each void being polyhedral for doped wires. The void diameter $2r$, the center-to-center void spacing $2l$, the cluster spacing $2L$ (see Fig. 11), the grain size d_i and the grain boundary area fraction of voids $f(=r^2/l^2)$ were measured from the micrographs with the total number of voids of about 1000. Figure 7 represents the stress and the temperature dependence of $2r$ and f . The three levels of stress correspond approximately to the modulus corrected stresses of $\log(\sigma/G) = -3.60, -3.44$ and -3.36 , respectively. It is clear that $2r$ and f increases as the temperature increases and the stress decreases. While $2l$ remains almost constant to be $5 \pm 0.5 \mu\text{m}$ independent of stress and temperature, $2L$ and d_i depend on temperature. The temperature dependence of $2L$ and d_i is shown in Fig. 8. The fact that $2L/d_i = 1.0\text{--}1.5$ indicates that all grain boundaries are not cavitated.

(a) 46 MPa

(b) 35 MPa

Fig. 5 SEM photographs of fractured surface for (a) doped and (b) non-doped wires crept under each stress at 2273 K after the completion of secondary recrystallization.

IV. Discussion

1. Modified Monkman-Grant relation

Dobeš and Milička⁽¹⁴⁾ suggested the modified Monkman-Grant relation which gives the relation between the overall time t_f and the strain ϵ_f to fracture and $\dot{\epsilon}_m$ in creep tests as follows:

$$\epsilon_f/t_f = K_0 \dot{\epsilon}_m^{m_0}, \quad (1)$$

where m_0 and K_0 are constants which are close to unity and independent of temperature, respectively. If ϵ_f is assumed constant, eq. (1) reduces to the original Monkman-Grant⁽¹⁵⁾ relation. Figure 9 shows that the experimental data points agrees well with eq. (1).

The time to fracture t_f is defined as the time for a cavity to grow from some initial radius r_i to the limiting radius

Fig. 6 A cluster of voids grown on the grain boundary of doped wire crept for 7.2 ks under a stress of 30 MPa at a temperature of 2073 K.

Fig. 7 Stress and temperature dependence of the void diameter $2r$ and the area fraction $f[(r/l)^2]$.

l , where $2l$ is cavity spacing. With the radial cavity growth rate $\dot{r}$, t_f is expressed by⁽¹⁶⁾

$$t_f = \int_{r_i}^r \frac{dr}{\dot{r}(r, \sigma)} = \int_{r_i}^r F(r, \sigma) dr, \quad (2)$$

where $\dot{r}$ or F is a function of cavity radius r and stress σ . Rice⁽¹⁷⁾ showed that the function $F(r)$ can be split into the sum of two terms

$$F(r) = F_1(r) + F_2(r). \quad (3)$$

Equations (2) and (3) give

$$t_f = (t_f)_1 + (t_f)_2. \quad (4)$$

Finally, Rice⁽¹⁷⁾ assumed two mechanisms determining

Fig. 8 Stress and temperature dependence of the void clusters spacing $2L$ and the transverse grain size d_t .

Fig. 9 Modified Monkman-Grant relation for tungsten wires crept at several test temperatures after the completion of secondary recrystallization.

the overall time to fracture; one is based on the Hull-Rimmer⁽¹⁸⁾ cavity growth mechanism by grain boundary diffusion and the other is based on the deformation of the constraining surroundings⁽¹⁶⁾, viz. power law creep in the matrix around the cavities which controls the cavity growth. He obtained the times, $(t_f)_1$ and $(t_f)_2$, corresponding to these two mechanisms, respectively, as

$$(t_f)_1 = \frac{16hl^3kT}{315D_B\delta_B\Omega\sigma} \left[1 - \frac{105}{16} f_i^{3/2} \ln(1/f_i) - \frac{1}{8} f_i^{3/2} \left(63f_i^{1/2} - \frac{175}{4} - \frac{45}{4} f_i \right) \right], \quad (5)$$

$$(t_f)_2 = \frac{4hl}{3q\dot{\epsilon}d_f} (1 - f_i^{3/2}), \quad (6)$$

where $f_i = (r_i^2/l^2)$ is the grain boundary area fraction of voids with some initial radius r_i , $\dot{\epsilon}$ the creep rate, D_B the

grain boundary diffusion coefficient, δ_B the grain boundary width taking part in diffusion, Ω the atomic volume, d_f the grain boundary facet diameter, q a dimensionless factor which is of the order of unity, k the Boltzmann constant, T the temperature. h is a function of θ given by

$$h(\theta) = [1/(1 + \cos \theta) - \cos \theta/2]/\sin \theta, \quad (7)$$

where θ is the angle between the tangent to the cavity surface at the cavity tip and the grain boundary plane. Typically, eq. (7) gives $h \approx 0.6$ ⁽¹⁶⁾. It is also known that if the cavitated facets are well isolated from one another, then $q = 0.9$ for power law creep without grain boundary sliding; however, q might increase to 1.8 for freely sliding boundaries⁽¹⁶⁾. The present results give $d_f (\approx d_t) = 45 \mu\text{m}$, $l = 2.5 \mu\text{m}$ under conditions of $T = 2273 \text{ K}$, $\sigma = 43 \text{ MPa}$ and $\dot{\epsilon} = 10^{-6} \text{ s}^{-1}$. Therefore, we can get the ratio of $(t_f)_2$ to $(t_f)_1$ using $h \approx 0.6$, $q = 0.9$, $r_i = 0.04 \mu\text{m}$ ⁽⁹⁾, $D_B = 10^{-10} \text{ m}^2 \text{ s}^{-1}$ estimated from sintering experiments⁽¹⁹⁾⁽²⁰⁾, $\Omega = 1.6 \times 10^{-29} \text{ m}^3$ ⁽²¹⁾ and $\delta_B = 2b$ ⁽¹⁶⁾ with the Burgers vector of $b = 2.74 \times 10^{-10} \text{ m}$ under these conditions. The ratio was calculated to be about 0.01.

Pavinich and Raj⁽²²⁾ reported that the following equation can be obtained when power law creep is the dominant mechanism of cavity growth during intergranular failure;

$$\epsilon_f \approx 0.23l/d, \quad (8)$$

where d is the grain size. As the contribution of grain boundary diffusion to the cavity growth is almost negligible as mentioned above, eqs. (6) and (8) predict the modified Monkman-Grant equation (1) with $m_0 = 1$ since $f_i \ll 1$, $d_f \approx d$ and h does not strongly depend on θ ⁽¹⁶⁾. The results of Fig. 9 suggest the deformation of the constraining surroundings controlled by power law creep.

2. Constrained cavity growth

The Arrhenius plot of $\dot{\epsilon}_m$ is given in Fig. 10. It is evident from Figs. 3, 4 and 10 that the apparent activation energies for creep Q_a of both materials were about 130 kJ/mol independent of stress. These values are much less than the activation energies of tungsten by different controlling mechanism such as for surface 270–330⁽²³⁾⁽²⁴⁾, dislocation core 380, grain boundary 390 and lattice diffusion 640⁽²⁰⁾ kJ/mol, respectively. Moreover, the Figs. 6 to 8 imply that the grain boundary cavitation has an important effect on creep, although it does not occur uniformly through the grain boundaries. Therefore, it is most probable that the deformation during creep is motivated by the constrained cavity growth (the growth of non-uniformly distributed voids)⁽²⁵⁾ on grain boundaries.

The model⁽²⁵⁾ of the constrained cavity growth is schematically illustrated in Fig. 11. Three simple mechanisms which limit void growth may be possible on the grain boundary between the grains of A and B: (a) surface diffusion at the interface of the voids, (b) grain boundary diffusion and (c) power law creep due to dislocations climb in the vicinity of the grain boundary. The growth rate of these voids is determined by the deformation rate of the surrounding cage of shaded grains.

Fig. 10 Arrhenius plot to get the activation energy for creep at the stresses of $\log(\sigma/G) = -3.6$ in Figs. 3 and 4.

This model is equal to the one of Raj and Ghosh⁽²⁶⁾ if the surrounding cage is restricted in the grains of C and D. Therefore, various couplings of (a)–(c) with power law creep in the surrounding cage are considered as the elementary rate-controlling mechanism of apparent power law creep. However, we do not need consider the possibility of mechanism (a); the shape of voids grown by surface diffusion should be irregular “crack-like” as reported in non-doped wire⁽⁷⁾, but the experimentally observed voids were regular and polyhedral. The coupling mechanism of (b) gives⁽²⁵⁾

$$\dot{\epsilon} = \frac{8\phi}{3 \ln(1/f)} \left(\frac{l}{d} \right) \left(\frac{\sigma_c}{\sigma_0} \right) \dot{\epsilon}_0, \quad (9)$$

where

$$\dot{\epsilon}_0 = A \frac{D G b}{k T}, \quad (10)$$

$$\frac{\sigma_c}{\sigma_0} = \frac{\frac{1}{(1-d/2L)^n} \left(\frac{\sigma}{\sigma_0} \right)^n}{\left[\frac{1}{(1-d/2L)^n} - 1 \right] \left(\frac{\sigma}{\sigma_0} \right)^{n-1} + \frac{8l\phi}{3d \ln(1/f)}}, \quad (11)$$

$$\phi = \frac{2D_B \delta_B \Omega \sigma_0}{k T l^3 \dot{\epsilon}_0}, \quad (12)$$

$$D = D_0 \exp(-Q_c/kT), \quad (13)$$

and A is a constant given by the empirical equation of $A \approx 1/1025^{2.7}$, G the shear modulus, σ_0 the creep constant satisfied with $\sigma_0 = \sigma(\dot{\epsilon}/\dot{\epsilon}_0)^{-1/n} \approx G/1025$ ⁽²⁷⁾, D the diffusion coefficient, D_0 the frequency factor, n and Q_c the stress exponent and the activation energy, respectively, for creep without voids. If the deformation mechanism is power law creep, D is equal to the lattice diffusion coefficient D_L and the following equation must be satisfied.

$$\left[\frac{1}{(1-d/2L)^n} - 1 \right] \left(\frac{\sigma}{\sigma_0} \right)^{n-1} \ll \frac{8l\phi}{3d \ln(1/f)}. \quad (14)$$

Fig. 11 Constrained cavity growth model taken from Ref. (25).

Equation (14) was examined using $n=7^{(28)}$, $D_L=10^{-17} \text{ m}^2 \text{ s}^{-1(20)}$, $2L=60 \mu\text{m}$, $G=118040 \text{ MPa}^{(12)}$ in addition to the parameters given above under conditions of $T=2273 \text{ K}$ and $\sigma=43 \text{ MPa}$. It was proved that eq. (14) fails to hold because the left-hand term is quite large compared with the right-hand. Accordingly, the mechanism of (b) must be given up.

The mechanism of (c) is schematically illustrated on the right-hand side in Fig. 11. A void can grow by the power law creep of the surrounding matrix. The shaded slab containing the void in the figure extends at a rate determined by the net section stress $\sigma/(1-f)$, while the rest of the cylinder (unshaded) extends at a rate determined by σ alone as follows:

$$\dot{\epsilon} = \dot{\epsilon}_0 \left(\frac{\sigma}{\sigma_0}\right)^n. \quad (15)$$

The shaded slab dilates, causing the holes to grow. The application of the continuity equation concerning the volume of matter flowing out of the void leads to the expression for the strain rate⁽²⁵⁾:

$$\dot{\epsilon} = \left\{ 1 + \frac{2r}{d} \beta \left[\frac{1}{(1-f)^n} - 1 \right] \right\} \left(\frac{\sigma}{\sigma_0}\right)^n \dot{\epsilon}_0, \quad (16)$$

where β is a constant (~ 0.6). When σ_u and σ_c are used as the mean stresses in undamaged (C and D grains) and damaged (A and B grains) regions, σ of eqs. (15) and (16) must be replaced by σ_u and σ_c , respectively. To satisfy equilibrium with $d_f=d$

$$(2L-d)\sigma_u + d\sigma_c = 2L\sigma. \quad (17)$$

The combination of eqs. (15) to (17) gives

$$\frac{\sigma_c}{\sigma_0} = \frac{2(L/d)(\sigma/\sigma_0)}{\left(\frac{2L}{d}-1\right)X^{1/n}+1}, \quad (18)$$

where

$$X = 1 + \frac{2r}{d} \beta \left[\frac{1}{(1-f)^n} - 1 \right]. \quad (19)$$

Substituting eqs. (10) and (18) into eq. (16) leads to

$$\dot{\epsilon} = BP(T) \left(\frac{\sigma}{\sigma_0}\right)^n \exp(-Q_c/kT), \quad (20)$$

where

$$B = \frac{AbD_0}{k}, \quad (21)$$

$$P(T) = \left[\frac{2L/d}{\left(\frac{2L}{d}-1\right)X^{1/n}+1} \right]^n \frac{XG}{T}, \quad (22)$$

B is a constant independent of temperature, $P(T)$ a parameter containing all other factors dependent on temperature. The observation of voids as in Figs. 7 and 8 makes it possible to estimate $P(T)$. In order to obtain Q_c , the value of $\dot{\epsilon}_m/P(T)$ is plotted against the reciprocal of temperature on the logarithmic scale in Fig. 12. The resulting value of Q_c was about 200 kJ/mol independent of stress which is near the activation energy for surface diffusion (640 kJ/mol) as the mechanism of dislocations climb even if the effect of grain boundary cavitation on creep rates is removed. Similarly, Q_c is much less than the activation energy for breakaway of dislocations from bubbles which was estimated to be about 10^5 kJ/mol for the bubble diameter of 40–120 nm⁽¹⁾, $G=112373(2473 \text{ K}) - 123445 \text{ MPa}(2073 \text{ K})^{(12)}$ and the dislocation core cut-off radius of 5b from Fig. 3 in Ref. (8).

It is evident from Fig. 5 that the grain boundary sliding plays an important role in creep of non-doped wire in contrast with doped wire. Although the activation energy for grain boundary sliding is thought to be identical to that for lattice self-diffusion, a number of experiments

Fig. 12 Arrhenius plot free from grain boundary cavitation for doped wire.

have demonstrated that grain boundary sliding can proceed with a lower activation energy⁽²⁴⁾. Langdon⁽²⁹⁾ has proposed a model for grain boundary sliding which assumes that the rate-controlling process in grain boundary sliding is the climb of dislocations in the region of the grain boundary. Moon and Stickler⁽²⁴⁾ concluded on the basis of his model that the dislocations are able to climb either in the vicinity of, or in, the grain boundary with an activation energy close to that for surface diffusion in tungsten. If the effect of cavitation on creep rates of the present wires is similar, it seems that the creep behavior of non-doped wire is similar to that of 80 μm diameter wire⁽²⁴⁾ which gives $n=2$ and $Q=275 \pm 75$ kJ/mol because of grain boundary sliding. On the other hand, the grain boundary sliding is not important for doped wire as obvious from Fig. 5(a) and the interlocked grain structure⁽³⁰⁾. Therefore, the present value of $Q_c \approx 200$ kJ/mol can not be explained by the previous conclusion based on grain boundary sliding. It may be concluded that the low value of Q_c results from the deformation of the constraining surroundings (the surrounding cage in Fig. 11) which proceeds near the surface of doped wire because such a geometrical arrangement of the grains is almost satisfied with the present relation of $2L/d_t = 1.0$ – 1.5 and the ratio (≈ 3) of d_t to the wire diameter.

V. Conclusion

(1) The modified Monkman-Grant relation holds for the overall time, the strain to fracture and the minimum creep rate during creep in powder metallurgy tungsten

wires.

(2) The activation energy for steady state creep is obtained to be about 130 kJ/mol which is much less than the energies for surface, dislocation, grain boundary and lattice diffusion. The value does not approach the activation energy for lattice diffusion as the mechanism of dislocations climb even if the effect of grain boundary cavitation on creep rates is taken into account.

(3) The small activation energy for steady state creep must result from a combination of the effect of grain boundary cavitation on creep rates and the deformation of the constraining surroundings which proceeds near the surface of doped wire.

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