High-Temperature Creep Resistance Resulting from Grain Morphologies in P/M Tungsten Fine Wires

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The three-dimensional shape of secondary recrystallized grains grown in doped tungsten wires is constructed by an image analyzer. Some grain shape parameters characteristic of grain morphologies are then defined to clarify the morphological effect of grains on the minimum creep rate during diffusional creep without grain-boundary cavitation. While the creep rate decreases as the grain aspect ratio (GAR) increases in the range of $GAR < 30$, it increases as GAR increases in the range of $GAR > 30$. The Raj and Ashby's model gives a satisfactory explanation to the former although it does not give the one to the latter. It is proved that their model must be modified using the new grain shape parameters to explain the latter behavior because of the different grain configurations.

Keywords: bubble, secondary recrystallized grain, grain aspect ratio,
grain boundary sliding, creep rate, powder metallurgy, filament

I. Introduction

A number of bubbles existing on grain boundaries play an important role in controlling the morphology of secondary recrystallized grains⁽¹⁾. They produce an elongated and interlocked grain structure which reduces creep strains resulting from the grain boundary itself such as grain boundary sliding and cavitation⁽²⁾⁽³⁾. It seems that there are two main factors influencing the creep strains. One is the total area of grain boundaries included in a specimen. The other is the effect of nonplanar grain boundaries on grain boundary sliding⁽⁴⁾. We confirmed that this effect can be appreciated only when the area of grain boundary is not largely changed⁽⁵⁾. The purpose of the present paper is to define some grain shape parameters characteristic of grain morphologies and give an explanation using them to the morphological effect of grains on the creep rate mainly due to grain boundary

sliding.

II. Experimental

The material used was doped tungsten wire 0.13 mm in diameter of commercial grade. 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. In order to complete secondary recrystallization and get various grain configurations, the specimens were annealed for 300 s at 2373 K after heating at the rates of 0.2–880 K/s. The grain boundaries were revealed by etching in a solution of $30\text{gK}_3\text{Fe}(\text{CN})_6 + 2\text{gNaOH} + 100\text{cm}^3\text{H}_2\text{O}$ and the two-dimensional shape of a grain was measured every 20 μm along the wire axis. The grain contour was then constructed over about 500 μm in length using a three-dimensional image analyzer (NIKON COSMOZONE-S). The

grain boundary area per unit volume of a specimen S/V , the cross sectional area A and the shape factor $F(=4\pi A/P^2)$ on each cross section of a grain were measured, where P is the circumferential length of a cross section. As F is calculated as the ratio A/A_0 of the cross sectional area A_0 of a perfect circle with a value of P to A , the increase in F (<1) up to unity means that the grain shape becomes more cylindrical. Some of recrystallized specimens were cut to be 15 mm in length and mounted in a resin to measure the two-dimensional shape of grains along the wire axis.

Creep tests were carried out after the specimens with a gage length of 50 mm were attached to an Instron type tensile testing machine connected to a vacuum system and their secondary recrystallization was completed. A condition of creep was selected to be 2673 K and 28 MPa which give a diffusional creep with the stress exponent of 1 without the formation of voids at grain boundaries⁽²⁾⁽³⁾. The loads imposed on the specimens were kept constant by an automatic load controller. The constant power supply was specially designed to control the current so that the temperature of the specimens remained constant during tensile deformation. Details of the procedure are described elsewhere⁽⁵⁾⁽⁶⁾.

III. Results

The length H and the width W of about 200 grains were measured two-dimensionally to get the grain aspect ratio H/W which gives us some information about the shape of a grain. Figure 1 shows the heating rate dependence of these parameters. It is seen that H/W decreases with increasing heating rate, being caused mainly by the change in H . Figure 2 presents some typical stereoscopic views of secondary recrystallized grains of which the aspect ratio is an order of magnitude larger at (a) than at (b). It is clear that the irregularities of the grains increase with an increase in heating rate. Such a change in morphology and asperity is reflected by the changes in (a) the cross sectional area A and (b) the shape factor F along

the wire axes as shown in Fig.3 (a) and (b), respectively. In order to characterize such a change in A and F , three grain shape parameters f_1 to f_3 were defined as follows:

$$f_1 = \frac{1}{N} \sum_{j=1}^N \frac{\sum_{i=2}^n |A_i - A_{i-1}|}{n-1} / \sum_{i=1}^n A_i \quad (1)$$

$$f_2 = (\bar{F}) = \frac{1}{N} \sum_{j=1}^N \frac{\sum_{i=1}^n F_i}{n} \quad (2)$$

$$f_3 = \frac{1}{N} \sum_{j=1}^N \frac{\sum_{i=2}^n |F_i - F_{i-1}|}{n-1} \quad (3)$$

where $N(=2-6)$ is the number of grains, n the total number of cross section along the wire axis and the suffix i indicates the i -th cross section. Accordingly, f_1 and f_3 correspond to the total length of each line drawn through the data points along the wire axes as shown in Fig.3 (a) and (b), respectively. On the other hand, f_2 expresses the mean value along the wire axis. It is confirmed that f_2 remains almost unchanged while f_1 and f_3 increase drastically at the rapid rate of heating⁽⁵⁾⁽⁷⁾. Therefore, the product of f_1 and f_3 was used as a

Fig.1 Variation of the length H , the width W and the aspect ratio H/W of secondary recrystallized grains with heating rate for doped tungsten wire.

Fig.2 Stereoscopic views of secondary recrystallized grains for doped wire annealed for 300 s at 2373 K after heating at the rates of (a) 0.2 K/s and (b) 880 K/s.

Fig.3 Variations of (a) the cross sectional area A and (b) the shape factor F with the cross section number along the wire axes for doped wires annealed for 300 s at 2373 K after heating at the rates of 0.2–880 K/s.

shape parameter. The minimum creep rate $\dot{\epsilon}_m$, S/V and $f_1 \cdot f_3$ are plotted against the heating rate in Fig.4. The different heating rate dependences of $\dot{\epsilon}_m$

on both sides of 20 K/s ($H/W=30$ in Fig.1) seem to result from the different effects of S/V and $f_1 \cdot f_3$ on $\dot{\epsilon}_m$.

IV. Discussion

The analysis of Raj and Ashby⁽⁴⁾ demonstrates that the grain shape can have a profound effect on the rate of diffusional creep—that is, of sliding with diffusional accommodation—of a polycrystal. A polycrystal with elongated grains is schematically illustrated in Fig.5 in which the grains have height H and width W . We define the aspect ratio R of a grain as $R=H/W$ and the grain size d as $d=(HW)^{1/2}$. If the crystal is subject to equal, principal tensile and compressive stresses σ , these are equivalent to a shear $\tau_s=\sigma$ at 45° to the principal stresses. The sliding can be considered to occur on a path of wave-length $2W$ and of height $H/2$.

Fig.4 Heating rate dependence of $\dot{\epsilon}_m$, S/V and $f_1 \cdot f_3$.

The stress system results in a displacement of the chemical potential of vacancies by $+\sigma\Omega$ at the horizontal boundaries and of $-\sigma\Omega$ at the vertical ones, where Ω is the atomic volume⁽⁸⁾. The average gradient of potential between the two sorts of boundary is

$$\nabla\mu = 2\sigma\Omega/(H/4) \quad (4)$$

The flux of atoms (or counter flux of vacancies) through the grain is then

$$J_v = -\frac{D_v}{\Omega kT} \nabla\mu \quad (5)$$

and along the boundary is

$$J_b = -\frac{D_b}{\Omega kT} \nabla\mu \quad (6)$$

where D_v and D_b are the volume and boundary self-diffusion coefficients, k the Boltzmann constant and T the temperature. The net flow of matter within and round one grain to one of the horizontal boundaries is given using eqs.(5) and (6) by

$$I = J_v W + J_b \delta \quad (7)$$

where δ is the effective boundary thickness through which the boundary flux passes. This results in a normal displacement rate $\dot{U}_n$ at the horizontal boundaries of

$$\dot{U}_n = I\Omega/W \quad (8)$$

and a displacement rate at the vertical boundaries which is of opposite sign and smaller in magnitude by a factor W/H . Accordingly, the normal strain rate parallel to the principal stresses which is equivalent to a shear strain rate $\dot{\gamma}$ is obtained as⁽⁸⁾

$$\dot{\gamma} = \frac{2\dot{U}_n}{H} \approx \frac{16\tau_s\Omega}{kT} \frac{1}{d^2} \left(\frac{D_v}{R} + \frac{\delta}{d} \frac{D_b}{\sqrt{R}} \right) \quad (9)$$

If one estimates S/V to be roughly proportional to $1/d$, eq.(9) gives a good explanation to the heating rate dependence of the creep rate at the rapid rate of heating (or $GAR=H/W<30$ in Fig.1) in Fig.4. Namely, as is evident from Figs.1 and 4, $\dot{\epsilon}_m$ decreases with an increase in GAR and a decrease in S/V regardless of the grain asperities which are characterized by $f_1 \cdot f_3$. However, the behavior of $\dot{\epsilon}_m$ at the slow rate of heating (or $GAR>30$) can not be explained by eq.(9).

Fig.5 Schematic representation of a polycrystal with elongated grains and the flow of matter around them.

Fig.6 Effect of the grain morphologies on the creep rate due to grain boundary sliding.

It may be ascribed to the different shapes of the grains as shown in Fig.6. Equation (9) is applicable to the grains of $GAR < 30$ because they are approximately located so that the grain boundary sliding occurs at 45° to the tensile axis. On the other hand, the grains of $GAR > 30$ are too elongated to be explained by the model for eq.(9). Assuming that the shape of grain

boundary is sinusoidal, the diffusion-controlled sliding rate $\dot{U}$ is given by⁽⁴⁾

$$\dot{U} = \frac{8 \tau_a \Omega}{\pi k T} \left(\frac{\lambda}{h^2} \right) D_v \left(1 + \frac{\pi \delta}{\lambda} \frac{D_B}{D_v} \right) \quad (10)$$

where h is the total height of the boundary shape and λ is the basic

wavelength of the boundary as shown in Fig.6. When ε is small, the strain rate $\dot{\varepsilon}$ along the wire axis is given by

$$\dot{\varepsilon} \simeq \dot{\varepsilon} = \dot{U}/\ell_0 \quad (11)$$

where $\ell_0 (=50 \text{ mm})$ is the gage length of the specimen and $\dot{\varepsilon}$ the engineering strain rate. As the total height h and the "waviness" h/λ of a boundary can be regarded as f_1 and f_3 , respectively⁽⁷⁾, eq.(11) is rewritten to be

$$\dot{\varepsilon} \propto \frac{D_v}{f_1 f_3} \left(1 + \frac{\pi \delta}{\lambda} \frac{D_B}{D_v} \right) \quad (12)$$

It is clear from eq.(12) that the heating rate dependence of $\dot{\varepsilon}_m$ at the slow rate of heating (or $\text{GAR} > 30$) in Fig.4, viz. the increase in $\dot{\varepsilon}_m$ with an increase in GAR, results from the morphology of grains which is characterized by the change in $f_1 \cdot f_3$.

V. Conclusions

1. While the minimum creep rate decreases as the grain aspect ratio (GAR) increases in the range of $\text{GAR} < 30$, it increases as GAR increases in the range of $\text{GAR} > 30$.
2. The GAR dependence of the minimum creep rate can be explained by the change in the GAR and the area of grain boundary per unit volume of a specimen at $\text{GAR} < 30$. On the other hand, it results from the morphology of grains at $\text{GAR} > 30$.

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