MORPHOLOGICAL EFFECT OF GRAINS ON HIGH TEMPERATURE CREEP FRACTURE IN P/M TUNGSTEN FINE WIRES

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ABSTRACT

Some parameters characteristic of the configuration of secondary recrystallized grains grown in commercial tungsten fine wire are defined after making clear their contours three-dimensionally. The change in shape of the grains increases with an increase in heating rate up to an annealing temperature. The parameters reliably reflect such a change in three-dimensional shape of various grains. Creep tests are made using wires with various grain shapes which are arranged by changing the heating rate. It is then investigated through the grain shape parameters how the grain configuration influences the creep fracture mechanism at high temperatures. It is shown that one of the parameters can characterize the change of the fracture mechanism from rupture to intergranular fracture in the region of power law creep by the degree of interlocking of grains.

KEYWORDS

Secondary recrystallized grain, interlocking, grain boundary sliding, grain boundary cavitation, power law creep, grain shape parameter, rupture, intergranular fracture, filament.

INTRODUCTION

The elongated interlocking grain structure prevents a lamp filament from sagging by suppressing strains during creep at high temperatures (Warlimont *et al*, 1975; Tanoue *et al*, 1993a). It seems that two factors play an opposing role in improving the creep resistance. Namely, the elongated grain structure reduces creep strains resulting from the grain boundary itself such as grain boundary sliding and cavitation. On the other hand, the interlocked grain structure suppresses the rotative grain boundary sliding due to torsional stresses imposed on coiled wires under the influence of gravity (Raj and King, 1978; Pugh and Lasch, 1990). This is dealt with as a problem of sliding on a non-planar grain boundary (Raj and

Ashby, 1971). However, the increase in the degree of interlocking causes the area of grain boundary and thereby the cavitational fracture at grain boundaries to increase. It is therefore very interesting to know how the fracture mechanism changes depending on the degree of interlocking. The purpose of the present work is to characterize the change of the creep fracture mechanism due to the different degree of interlocking by some grain shape parameters.

EXPERIMENTAL PROCEDURE

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$\pm$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 2073-2473 K after heating at the rates of 0.2-880 K/s. The grain boundaries were revealed by etching in a solution of $30gK_3Fe(CN)_6$+2gNaOH+$100cm^3H_2O$ 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 0.5 or 1.0 mm in length using a three-dimensional image analyzer. 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 A to the cross sectional area A_0 of a perfect circle with a value of P, 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 done 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. 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. 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. Details of the procedure mentioned above are described elsewhere (Tanoue *et al.*, 1990, 1993a)

RESULTS AND DISCUSSION

The length H and the width W of about 200 grains were measured two-dimensionally to get the grain aspect ratio H/W. **Figure 1** shows the heating

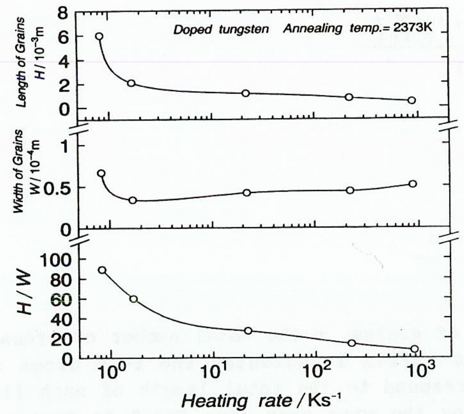

Fig.1 Variation of the length H, the width W and the aspect ratio H/W of secondary recrystallized grains with heating rate for doped tungsten wire.

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 as the

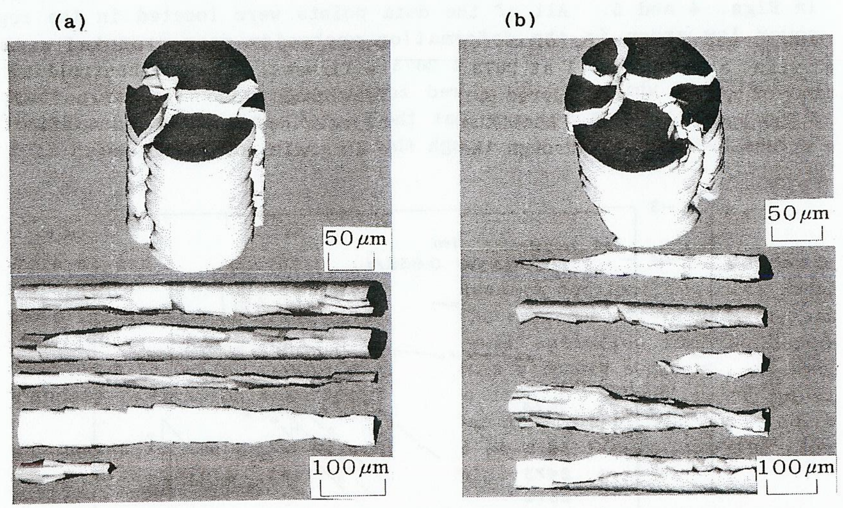

Fig.2 Stereoscopic views of secondary recrystallized grains for doped wire annealed for 300 s at 2373 K after heating at the rates of (a) 0.83 K/s and (b) 880 K/s.

heating rate increases. In order to characterize such a change in morphology and asperity, three grain shape parameters f_1 to f_3 were defined as follows:

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

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

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

where $N(=2\text{-}6)$ 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 (see Fig.3 in Tanoue, 1996). 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 (Tanoue *et al.*, 1995). Therefore, f_1 and f_3 were used as the grain shape parameters in the present study. The minimum creep rates $\dot{\varepsilon}$ obtained from creep tests are plotted against the stresses normalized by the shear modulus (Yih and Wang, 1979) in Figs. 4 and 5. All of the data points were located in the region of power law creep in the deformation mechanism maps except for the data points with a slope of 1 at 2673 K in the figures, the rate-controlling mechanism of which is considered to be diffusional (Tanoue *et al.*, 1990, 1995). The results suggest that the heating rate has a significant effect on the stress exponent n_s through the change in grain morphology.

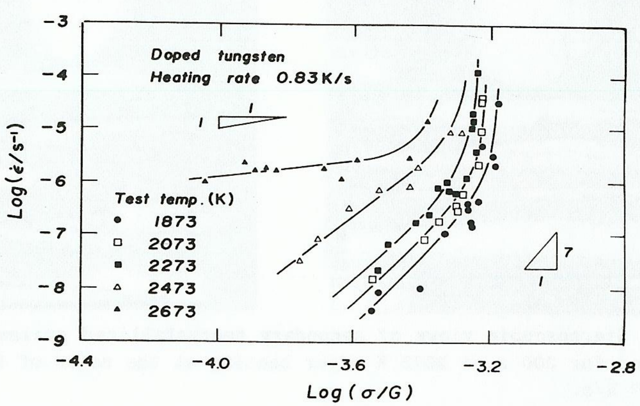

Fig.3 Logarithmic plot of the minimum creep rate against the modulus corrected stress for doped wire heated at 0.83 K/s and annealed for 300 s at 2373 K.

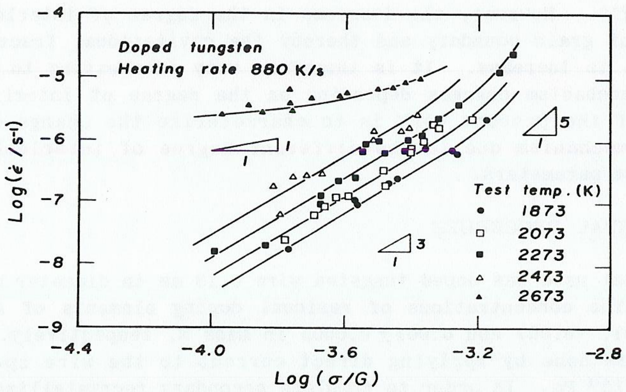

Fig.4 Logarithmic plots of the minimum creep rate against the modulus corrected stress for doped wire heated at 880 K/s and annealed for 300 s at 2373 K.

Similarly, the change in grain morphology makes a change in the fracture mode from rupture to intergranular fracture with increasing heating rate, as seen in **Fig.5**. It should be noticed that both doped wires are not much affected by grain boundary sliding compared with non-doped wire (Tanoue *et al.*, 1993b).

The modified Monkman-Grant equation (Dobeš and Milička, 1976) gives the relation between the overall time t_f and the strain ε_f to fracture and $\dot{\varepsilon}$ in creep tests as follows:

$$\varepsilon_f / t_f = K_o \dot{\varepsilon}^{m_o} \quad (4)$$

where m_o and K_o are constants which are close to unity and independent of temperature, respectively. If ε_f is assumed constant, eq. (4) reduces to the original Monkman-Grant relation. The experimental results of Tanoue *et al.* (1993b) showed that eq.(4) was well satisfied for the doped wire annealed at 880 K/s, giving m_o=1.0. This strongly suggests that the grain boundary cavitation takes part in the fracture mechanism of the wire of which the grains are fully interlocked as in Fig.2(b). However, eq.(4) was not useful to the doped wire annealed at 0.83 K/s because the plot gave m_o=0.9 with a less linear relationship. This implies that the grain boundary cavitation does not play an important role in the fracture mechanism of the wire of which the grains are very elongated as in Fig.2(a). The creep stress σ is plotted against the overall time t_f in Fig. 6. It is clear from eq.(4) that if m_o=1.0 and ε_f is constant, the slope in Fig. 6 gives $-1/n_s$ because the relation of $\dot{\varepsilon} \propto \sigma^{n_s}$ holds in the range of the power law creep. Namely, such a plot as Fig. 6 is another expression of the original Monkman-Grant relation combined with Figs. 3 and

(a) (b)

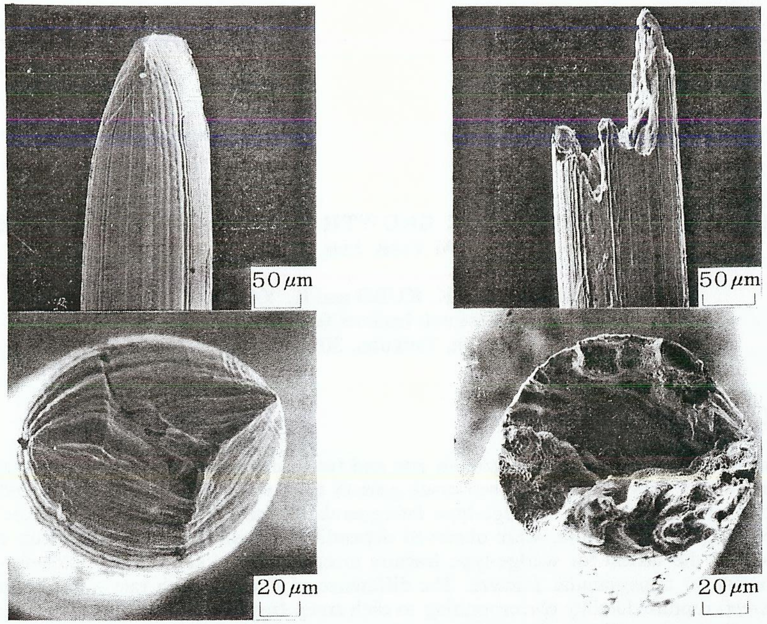

Fig.5 SEM photographs of fractured surface for doped wire crept under conditions of (a) 71 MPa, (b) 50 MPa and 2273 K. Each secondary recrystallization was completed by annealing for 300 s at 2373 K after heating at the rates of (a) 0.83 K/s and (b) 880 K/s, respectively.

4. Although the slope gives -1/3 for the data points at 880 K/s, it does not give -1/7 for them at 0.83 K/s. It follows that the original relation is also satisfied approximately unless the grains are very elongated. Figure 7 shows some cavities grown on a grain boundary facet in a wire annealed and crept after heating at the rapid rate of 880 K/s. A void cluster is formed along the boundary in the figure, the shape of each void being polyhedral (Horacsek, 1989). This is an evidence showing that the grain boundary cavitation takes part in the fracture mechanism of the wire in which the Monkman-Grant relation is satisfied. Accordingly, it is clear that the fracture mode changes depending on the degree of interlocking within the range of the power law creep.

The relation between f_1, f_3 and S/V is examined and shown in Fig.8. The parameter f_3 increases drastically in contrast with the slight increases in f_1 and thereby S/V in the range of H/W>25. It is therefore obvious that f_3 is a grain shape parameter which represents the small asperity of grains with the condition that f_1 remains approximately unchanged (Fig.2(a)). On the other hand, f_3 as well as S/V increase as soon as f_1 begins to

increase in the range of H/W<25. The increase in f_1 implies a development of complicated shape of grains (Fig.2(b)) accompanied with an increasing area of grain boundary, suggesting that it can be a measure characteristic of the interlocking grain structure. Accordingly, the parameter f_1 could classify all of the fracture into two modes of rapture and intergranular fracture depending on the degree of interlocking of grains, as seen in the two regions separated by the broken line of H/W=25 in Fig.8.

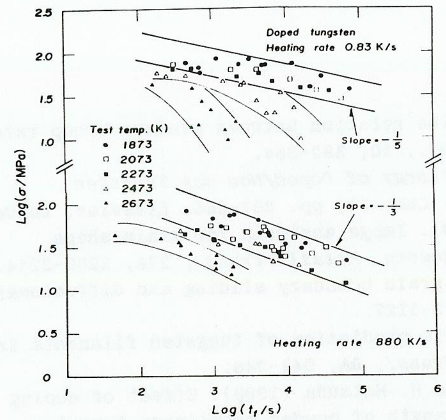

Fig.6 Logarithmic plot of the creep stress σ against the overall time t_f to fracture for the wires annealed for 300 s at 2373 K after heating at 0.83 and 880 K/s.

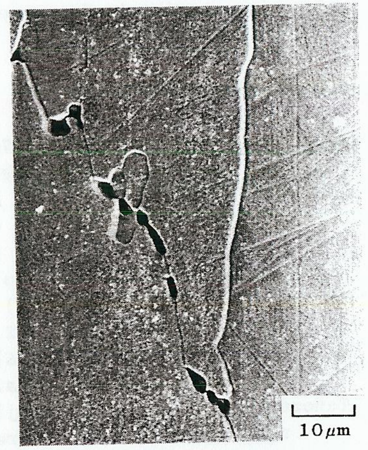

Fig.7 A cluster of voids on the grain boundary of doped wire crept for 1.5 ks under a stress of 51 MPa at 2273 K after heating at 880 K/s and annealing for 300 s at 2373 K.

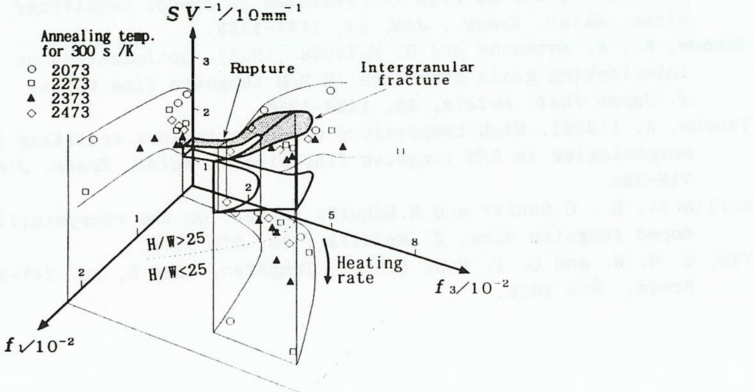

Fig.8 Effect of the grain morphology on the fracture mode expressed by some grain shape parameters.

CONCLUSIONS

1. While the Monkman-Grant relation holds for the wire of which the grains are fully interlocked, it does not hold for the wire of which the grains are very elongated.

2. The fracture mode changes from rupture to intergranular fracture as soon as the grain shape parameter f_1, which expresses the degree of interlocking of grains, begins to increase.

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