Effect of Grain Morphology on Brittle Fracture of P/M Tungsten Fine Wires

K. Tanoue

Department of Materials Science and Engineering, Faculty of Engineering, Kyushu Institute of Technology, Kitakyushu 804-8550, Japan

ABSTRACT: *A grain morphology index f_1 characteristic of the configuration of secondary recrystallized grains grown in tungsten wires is derived after making their contours clear in three dimensions. Tensile and hardness tests are made using the wires with different grain morphologies at room temperature. It is then investigated through f_1 how the grain morphology is connected to brittleness of doped wires. The fracture modes are classified to be ductile and brittle at each of the two regions, which are separated so that each of them is below and above a certain value of f_1 respectively. The morphological features of grains to prevent fatal brittleness of wires are demonstrated.*

INTRODUCTION

The doping elements such as Al, K and Si develop many arrays of small bubbles along the wire axis during fabrication. A number of bubbles exist on grain boundaries and play a role in controlling the morphology of secondary recrystallized grains [1, 2]. They produce an elongated and interlocked grain structure by impeding the transverse grain growth across the wire axis [3, 4]. 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 will suppress the rotative grain boundary sliding due to torsional stresses imposed on coiled wires under the influence of gravity [5]. Another beneficial effect of such morphology is to prevent the intergranular fracture with decreasing temperature after turning off the current. However, the highly elongated grain structure is apt to develop the so-called "bamboo structure" at both ends of the grains and reduce the ductility at room temperature [6]. Moreover, the increase in the degree of interlocking does not necessarily guarantee the prevention of the intergranular fracture because it is forced to accompany an increase in grain boundaries across a wire. The morphological effect of grains on fracture must therefore be clarified. The purpose of the present paper is to define a representative grain morphology index and thereby investigate the effect of grain morphology on brittle fracture of tungsten fine wires at room temperature.

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 <10, 42–94 and 10 in mass ppm, respectively. All of the heating was done by applying direct current to the wire specimens in a vacuum of 10^{-5} Pa. In order to complete secondary recrystallization and get various grain configurations, the specimens were annealed for 300 s at 2660 K after heating at the rates of 0.2–985 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 500 μm 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 obtained from the grain contours, where *P* is the circumferential length of a cross section. As *F* is calculated as the ratio A/A_o of the cross sectional area *A* to A_o 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 and the micro Vickers hardness.

The specimens with a gage length of 50 mm were attached to an Instron type tensile testing machine connected to a vacuum system and fully cooled in air from annealing temperatures to room temperature after their secondary recrystallization was completed. Tensile tests were then made at the strain rate of $3.3\times10^{-4}s^{-1}$ and room temperature. The micro Vickers hardness tests were carried out using a standard-type pyramidal diamond indenter at 19.9 N (200g). At least twenty impressions were measured at different grain interiors of each specimen to obtain a mean value.

RESULTS AND DISCUSSION

The length ℓ and the width *w* of about 200 grains were measured twodimensionally to get the grain aspect ratio $R(=\ell/w)$. Figure 1 shows the heating rate dependence of these parameters. It is seen that R decreases with increasing heating rate. Figure 2 presents some typical stereoscopic views of secondary recrystallized grains of which R ranges from 10 to 100. It is clear that the irregularities of the grains increase with increasing heating rate. Such a change in morphology and asperity is reflected by the changes in the cross sectional area A and the shape factor F along the wire axis. In order to characterize the change in A and F, three grain shape parameters f_1 to f_3 were defined as follows [3, 4, 7]: f_1 and f_3 corresponding to the total length of each line drawn through the data points of A and F along the wire axes, respectively and f_2 expressed as the mean value of F along the wire axis. The individual change of f_1 to f_3 is characterized by the schematic representation of Fig.5 of Ref.7. It is also confirmed that f_2 remains almost unchanged while f_1 and f_3 increase drastically at the rapid rate of heating [4,7], being connected to each other by the empirical equation of $f_1 = 0.24\, f_3^{\,0.3} \pm 0.014$ [8]. Accordingly, f_1 was used as a representative grain shape parameter in the present study.

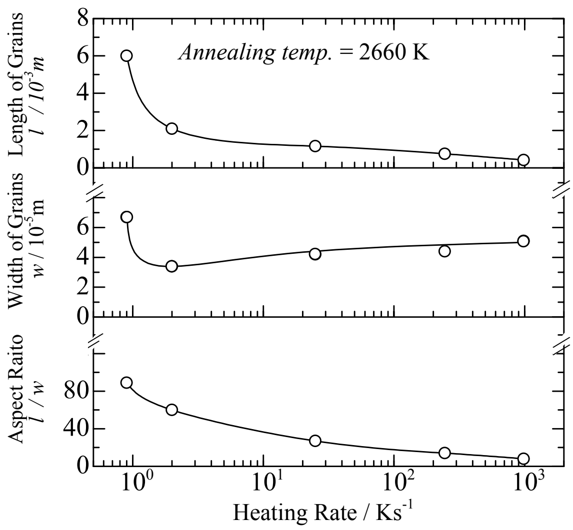

Figure 1: Variations of the length ℓ, the width w and the aspect ratio R $(=\ell/w)$ of secondary recrystallized grains with heating rate for doped tungsten wire with a potassium concentration of 75 mass ppm.

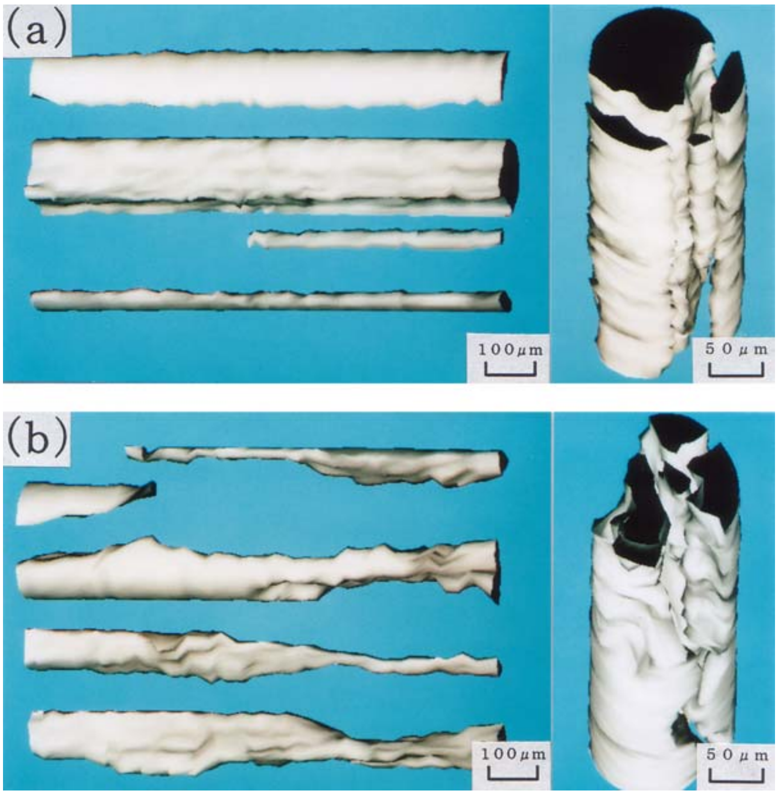

Figure 2: Stereoscopic views of secondary recrystallized grains for doped wire, which is annealed for 300 s at 2660 K after heating at the rates of (a) 0.5 and (b) 330 K/s, with a potassium concentration of 75 mass ppm.

The parameters f_1 and S/V were obtained from the grain contours of Fig.2 and given in Fig. 3 as a function of R. These parameters are connected to one another through the empirical equations, being almost independent of the annealing temperatures. Therefore, the quantitative estimation of the grain morphology is possible because all of the grain shape parameters are decided if one of them is known. One can again use f_1 as a representative grain morphology index to estimate the grain morphology quantitatively.

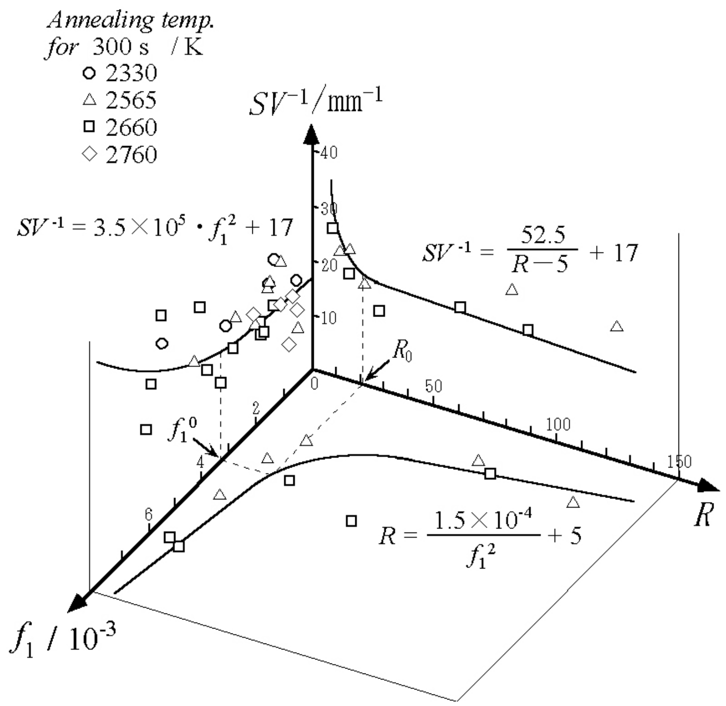

Figure 3: The relationship between the grain morphology index f_1, the grain aspect ratio R and the area of grain boundaries per unit volume of a specimen S/V. The critical value R_o indicates the minimum grain aspect ratio at which S/V begins to increase with a decrease in R.

The change of grain morphology, which is formed by the change in heating rate, is shown schematically in Fig.4 as a function of f_1. It is seen that the interlocked grain structure begins to develop at $f_1/10^{-3} \geq 3.3$. Specimens were annealed so that they have such grain morphologies as in Fig.4 and deformed in tension at the strain rate of $3.3\times10^{-4}s^{-1}$ and room temperature. True stress-true strain curves and fractured surfaces are typically given in Figs. 5 and 6, respectively. It is clear that while the specimen with a value of $f_1/10^{-3} \approx 1.4$ is failed in a manner of ductile, the others are failed in a manner of brittle. In order to clarify the effect of grain morphology on brittleness of wires, the ultimate tensile strength σ_{UTS} and the total elongation ε_f were obtained from Fig.5 and plotted against f_1 in Fig. 7. The results of σ_{UTS} as well as the micro Vickers hardness had a tendency to increase with f_1 and the potassium concentration. One can see that σ_{UTS} and ε_f are very small for the specimen with a potassium concentration of 42 mass ppm. This implies that the fracture mode is brittle for the specimen with a potassium concentration below about 45 mass ppm because

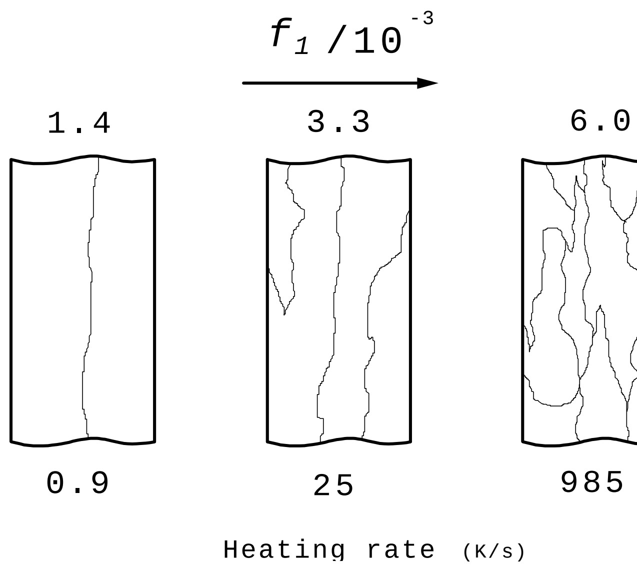

Figure 4: Schematic representation of the heating rate dependence of grain morphology expressed as a function of the grain morphology index f_1.

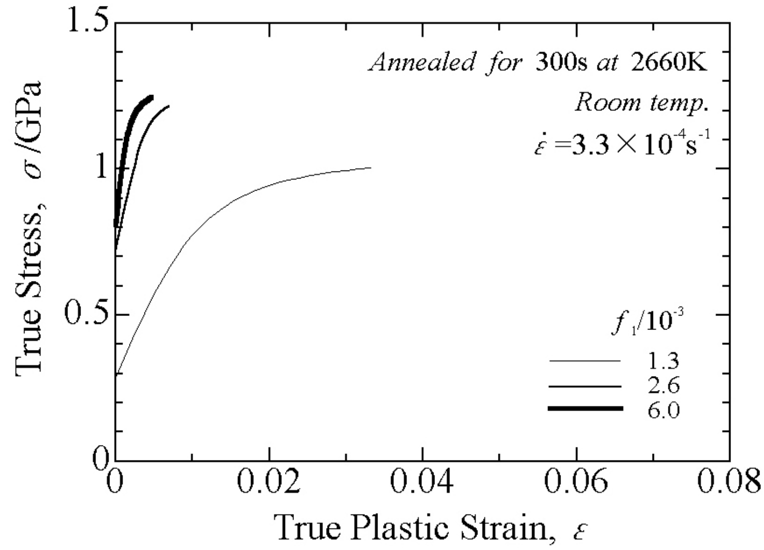

Figure 5: True stress-true strain curves for doped wire with a potassium concentration of 75 mass ppm.

the number of potassium bubbles is too small to play a role in controlling the shape of grains [9] and strengthening the matrix [10]. On the other hand, it is obvious that the fracture mode is relatively ductile at $f_1 < f_1^0$ ($=3.3\text{x}10^{-3}$) for some specimens with a potassium concentration above 70

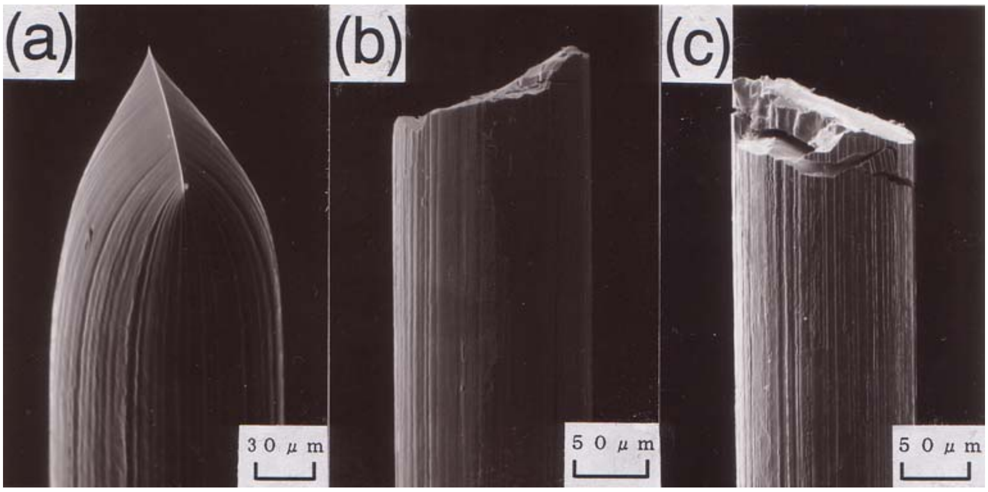

Figure 6: SEM photographs of fractured surface. The photographs (a) to (c) correspond to each specimen with a value of $f_l/10^{-3}$ =1.4, 3.3 and 6.0, respectively, in Figs.4 and 5.

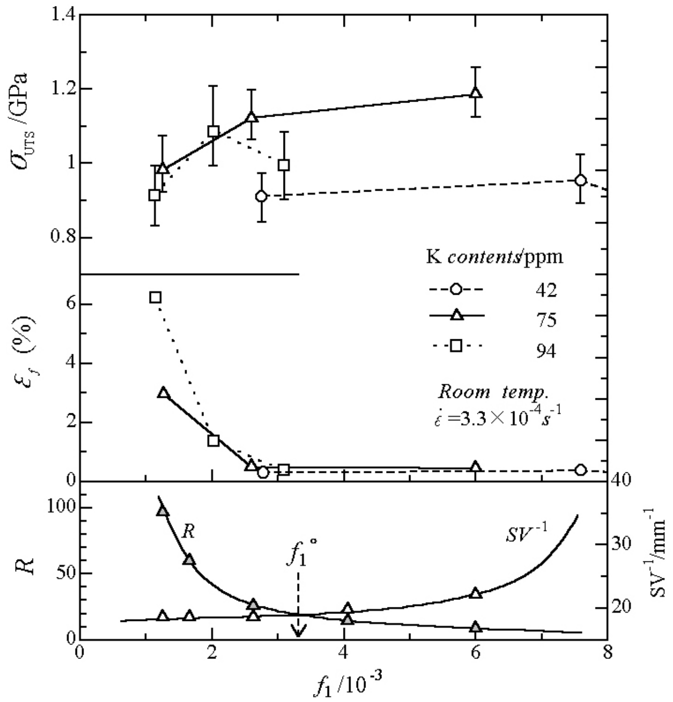

Figure 7: Variations of the ultimate tensile strength σ_{UTS} and the total elongation ε_f with the grain morphology index f_l for doped wires annealed for 300 s at 2660 K after heating at the rates of 0.2-985 K/s.

mass ppm, where f_l^0 is the critical value of f_l corresponding to R_0 (=20) in Fig.3. Namely, all of the specimens become brittle when f_l exceeds f_l^0 because the fully interlocked grain structure is accompanied with the drastic increase in *S/V* as seen in Fig.3. Therefore, the grain morphology must be arranged under the condition of $f_l < f_l^0$ (or equivalently $R > R_0$) to prevent the fatal brittle fracture after secondary recrystallization. However, when $f_l/10^{-3}$ comes close to one, it means the development of highly elongated grains or a single crystal, which is apt to develop "bamboo structure" and fracture at both ends of the grains. It is thus clear that the optimum value of $f_l/10^{-3}$ ranges from 1.5 to 2.0 which is almost equivalent to R=20-70.

CONCLUSIONS

1. The potassium concentration below about 45 mass ppm is too low to control the shape of grains and strengthen the matrix of tungsten fine wires.

2. The grain morphology must be arranged under the condition of $f_l < f_l^0$ (or equivalently $R > R_0$) to prevent the fatal brittle fracture after secondary recrystallization. The optimum grain morphology is given by $f_l/10^{-3}$=1.5–2.0 which is almost equivalent to R=20-70.

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