Relationship between Recrystallization Kinetics and Rate of Creep Cavitation in Bubble Strengthened Tungsten

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ABSTRACT

The kinetics of secondary recrystallization and high temperature creep fracture behaviour of bubble-strengthened (KSiAl-doped) tungsten wires have been investigated using emission electron microscopy and scanning electron microscopy. Two different doped wires were chosen that exhibited different creep properties, and their recrystallization and fracture behaviour were compared. In addition, creep and fracture behaviour of an undoped wire was also investigated. Under the applied creep conditions ($T = 2900$ K; $\sigma = 5$ MPa), fracture of the samples was caused by grain boundary cavitation, due to the coarsening and growth of the bubbles in doped wires, and due to the growth of deformation-induced microcracks in the undoped material. The results suggested that, in the case of bubble-strengthened tungsten, the high temperature fracture behaviour of the wire can be predicted from its recrystallization kinetics.

1. INTRODUCTION

The quality of a tungsten incandescent lamp depends primarily on the stability of the filament configuration during operation. To achieve the required good shape retention of the coil, creep of the wire must be suppressed. The solution of this metallurgical problem is equivalent to applying a suitable strengthening method by which the motion of dislocations can be impeded. Theory and experience indicate that under the operating conditions of lamp filaments (high temperatures and low stresses), the most effective way to retard the motion of dislocations and, hence, to reduce creep strain is to strengthen the wire by bubbles. Lamp-making factories accomplish this requirement by a KSiAl-doping procedure that introduces into tungsten wires a fine dispersion of potassium-filled bubbles /1/. This special kind of dispersion strengthening mechanism is especially suitable for high temperature operations, because – in contrast to elastically hard particles that repel the dislocations and, thus, dislocation climb over the particle may contribute to creep strain – the strong attractive bubble/dislocation interaction energy inhibits not only the glide but also the climb of the dislocations /2/.

In addition to the direct strengthening by bubble/dislocation interaction, the potassium bubbles, that are aligned in rows parallel to the wire axis, also exert an indirect strengthening effect by promoting the evolution of highly elongated recrystallized grains with interlocking boundaries that make grain boundary sliding difficult. While the bubble rows confer high creep resistance to the wire, besides this beneficial effect, the bubbles also play an important role in void formation and cavitational failure by coalescence and/or growth of the bubbles /3-6/.

Since in the case of bubble-strengthened (KSiAl-doped) tungsten wires both the recrystallization and the high temperature creep cavitation processes are influenced by the presence of the potassium bubbles /7,8/, it is reasonable to assume that the recrystallization behaviour of the wire contains information about the stability of the bubble structure against void (bubble) growth and premature failure. Therefore, one might expect that the "quality" of the bubble structure could be predicted from the recrystallization behaviour of the wire. In this work an attempt was made to search for a correlation between the recrystallization behaviour and cavitational fracture properties of doped tungsten wires.

2. EXPERIMENTAL PROCEDURE AND RESULTS

The comparative creep tests and recrystallization experiments were carried out on two different KSiAl-doped wires of 0.39 mm diameter, designated as wire A and wire B. The two materials with similar potassium contents (~70 µg/g) were chosen to represent different creep fracture properties. In addition, creep measurements were also made on an undoped tungsten wire. In the case of this technically pure material, no potassium was deliberately added; nevertheless the wire contained a relatively small amount of potassium (~5 µg/g). Prior to insertion in the creep apparatus, the 30 cm long samples were recrystallized by resistively heating at 2800 K for 10 minutes under vacuum. The samples were then cooled, a uniaxial stress of 5 MPa was applied by hanging an appropriate weight, and heated again to allow creep to occur at 2900 K. The elongation of the samples was measured through a window by means of a cathetometer.

Typical creep curves are given in Fig. 1 showing that, under the same test conditions, wire A exhibited a shorter lifetime (time-to-fracture) than wire B. After fracture, the samples were examined by scanning electron microscopy to reveal the presence and features of the voids formed during the creep deformation. We found that under our test conditions the fracture of the two doped wires and the undoped material was equally caused by grain boundary cavitation. The fracture surfaces of the two doped samples showed coarsened

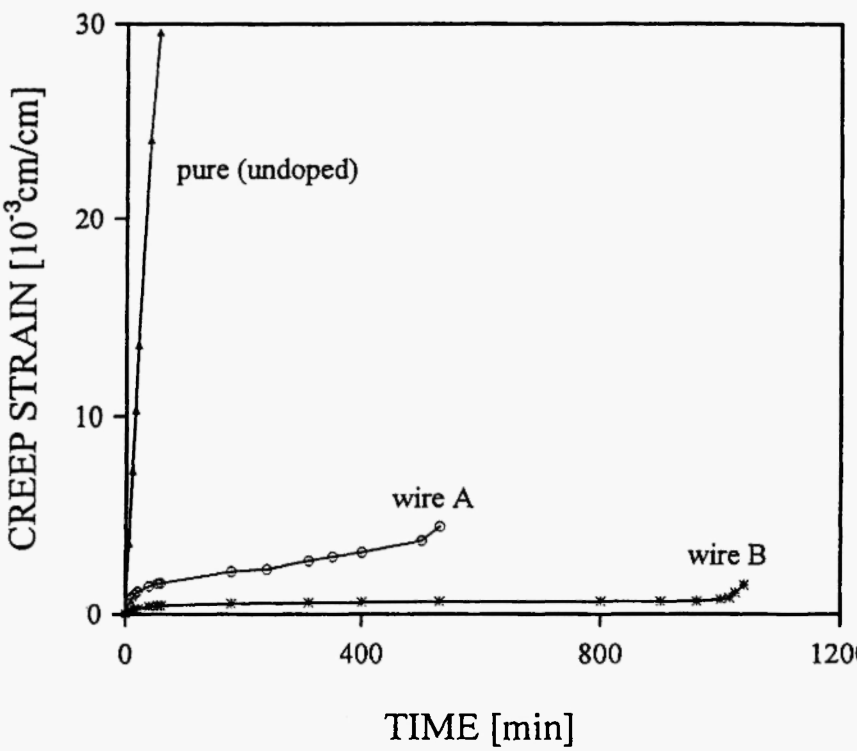

Fig. 1: Typical creep curves of an undoped and two bubble strengthened tungsten wires, at the same test conditions (T = 2900 K, σ = 5 MPa).

bubble structures on the grain boundaries, indicating that the creep voids in wire A and wire B were, in fact, coalesced bubbles (Fig. 2). Morphologically different cavitated grain boundaries were observed in the undoped wire (Fig. 3), where void formation could not be ascribed to bubble growth and, in contrast to the doped samples, no voids were revealed on those boundaries that were oriented parallel to the wire (stress) axis.

In order to get an insight into the details of the secondary recrystallization process, studies on grain growth were performed by emission electron microscopy /9/. The temperature of the sample was increased by self-resistance heating and the thermally emitted electrons were focused onto the fluorescent screen by an electrostatic lens. Direct observation of the grain growth process was allowed by orientation contrast associated with the differences in work function of the electrons for the different grains. This method was especially useful for kinetic studies, because thermal etching was not necessary to reveal the grain boundaries and, hence, the changing positions of the boundaries could be observed on the screen without delay. In this way the movement of the grain boundaries could be recorded by a video camera. To determine the recrystallization kinetics, the video tape recordings were evaluated during playback at a slower speed by measuring the time dependence of the volume fraction of the recrystallized material.

We found that there were certain characteristics of the recrystallization process that were common to wire A and wire B. In agreement with previous studies /10/, we observed that secondary recrystallization in these doped wires occurred only above a threshold temperature. Although this temperature (the lowest temperature at which secondary recrystallization started) was different for the two doped materials (~2000 K for wire A and ~2150 K for wire B), after completion of the recrystallization process no significant difference in the grain morphology could be detected between the two wires. Another well observable common characteristic of the recrystallization process of the doped wires was that, under isothermal annealing conditions, complete recrystallization could not be reached at (or slightly above) the threshold temperature, because after a shorter or longer period, the jerky motion of the grain

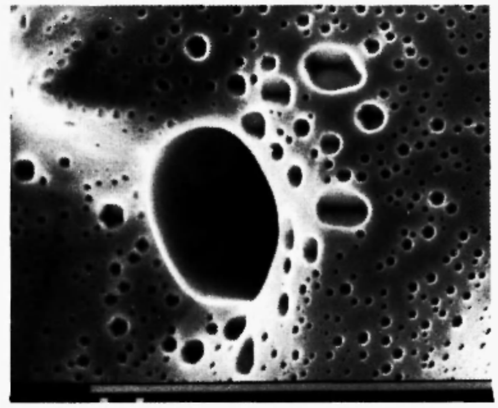

Fig. 2: Scanning electron micrograph showing grain boundary cavities formed from coalesced bubbles in doped tungsten. The micron marker represents 10 μm.

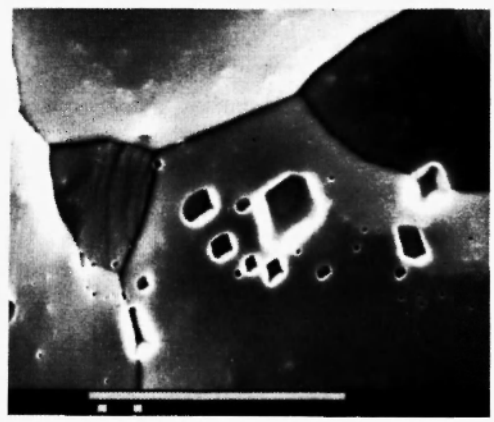

Fig. 3: Scanning electron micrograph showing grain boundary cavities growth from deformed-induced crack nuclei in undoped tungsten. The micron marker represents 10 μm.

boundaries came to a stop and further grain growth did not occur at the given temperature. Continuation of the arrested recrystallization required a temperature rise, the magnitude of which depended on the time during which the wire was further heated in its partially recrystallized condition. For the sake of simplicity, in this work we analyzed only the first (initial) stage of the recrystallization process.

Quantitative evaluation of the video tape recordings, made from the grain growth process as the samples were isothermally heated at their threshold temperatures, showed characteristic differences in the shape of the kinetic diagrams between wire A and wire B. Typical plots showing the time dependence of the secondary recrystallized volume fraction of the samples are given in Fig. 4. We observed that the recrystallization process in wire A usually stopped after a relatively prolonged stepwise boundary motion. In the case of wire B, however, the duration of the first stage of recrystallization was relatively short, because after a quick grain growth only a few occasional grain boundary jumps occurred at the given temperature. Accordingly, the kinetic plots for wire A consistently contained more breaking points and ledges than the plots for wire B. A similar description of the recrystallization kinetics of the undoped wire was impossible, because the process seemed to be a mixture of normal grain growth and secondary recrystallization; a certain part of the wire was repeatedly crossed by the migrating grain boundaries, while in other parts the small grained region was consumed by a growing grain in only one step. Fig. 5 shows some instances of the grain growth process for the two doped wires and for the undoped materials. The emission electron micrographs were obtained during video playback, taking photographs from the frozen still pictures that were visible on the TV screen.

3. DISCUSSION

Although the undoped wire was not totally free of bubbles, due to the presence of a small amount of potassium, their effect on high temperature strength seemed to be unimportant. Fig. 1 shows that the strain rate of the undoped wire was excessively high, because practically no dispersion strengthening mechanism worked against the movement of the lattice and grain boundary dislocations. Therefore, the grains in an undoped material can easily slide and, as the creep deformation proceeds, more and more void nuclei (growing microcracks) are formed in the grain boundaries as a result of the local stress concentrations. Since intercrystalline separation requires many growing voids that must be produced progressively during creep, this deformation-induced void nucleation mechanism

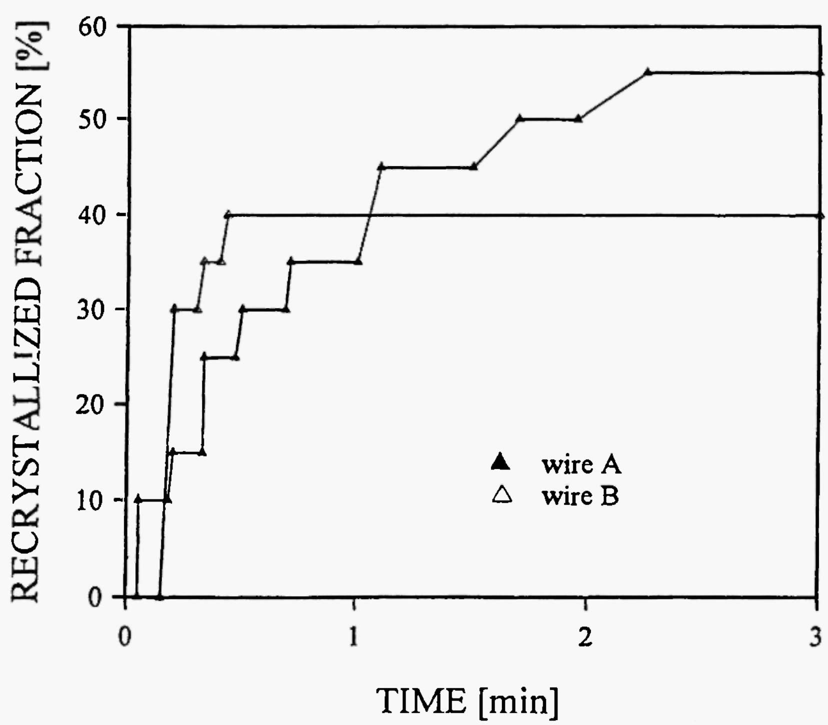

Fig. 4: Typical kinetic plots showing the time dependence of the secondary recrystallized volume fraction for wire A and wire B.

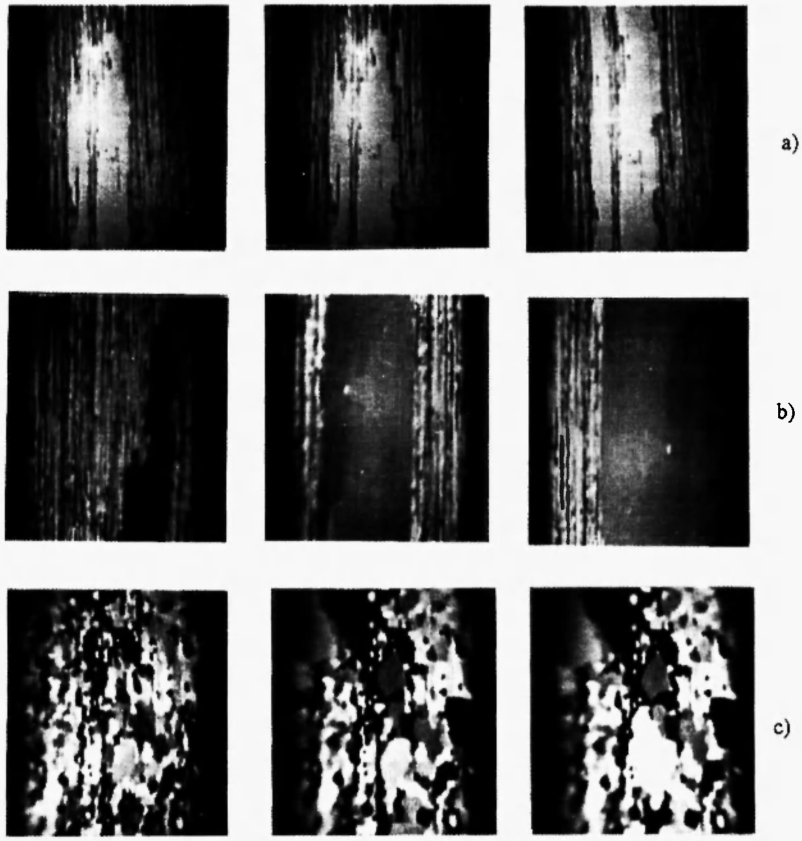

Fig. 5: Emission electron micrographs showing some instances of the recrystallization process occurring in wire A (a), in wire B (b) and in the undoped (c) sample. (Note that the second and third micrographs of series b represent increasing temperature.)

resulted in a relatively large strain at fracture, as shown in Fig. 1. In contrast to the undoped material, in doped wires the movement of dislocations is impeded by the closely spaced bubble rows /1/, which results in considerably reduced strain rates. At the same time, the interlocking grain boundaries are resistant to grain boundary sliding and, thus, deformation-induced void nucleation is suppressed. Since the coalescence of the bubbles /8/ and/or their growth /6/ by vacancy condensation does not require a significant amount of deformation, fracture in wire A and wire B occurred after considerably smaller strains compared to the undoped material.

Since the creep fracture of the pure and the two doped wires was equally caused by growth of grain boundary voids, the measured difference in lifetime between the three wires reflects the difference in the rate of cavitation occurring in these materials. Fig. 1 shows that growth of the deformation-induced nuclei (microcracks) in the undoped wire resulted in the highest rate of cavitation (shortest lifetime), while bubble growth in wire A and wire B occurred at considerably lower rates. The difference in the lifetime between the two doped materials indicates that the cavitation process in wire A occurred at a higher rate than in wire B. This means that the bubbles in wire A were more liable to coarsen and to grow than in wire B. As will be shown below, in the light of the difference in the shape of the kinetic plots (Fig. 4) one can recognize that the lower stability of the bubble structure of wire A also manifested itself during recrystallization.

The recrystallization in doped tungsten is determined by the driving force, F_d, that arises from the deformation stored energy consisting of dislocation and grain (fiber) boundary energies, and by the pinning force (Zener drag), F_p, exerted on the moving boundary by the bubbles. The pinning force depends on the dispersity of the bubble structure: the higher the dispersity, the stronger bubble barriers act against the moving boundary. Although, for lack of knowledge of the microstructural parameters, it is difficult to make exact calculations of the driving and pinning forces, semi-quantitative estimates have shown that in doped tungsten wires the average driving and pinning forces are of the same order of magnitude ($F_d \sim F_p \sim 10^7$ dyn cm^{-2}) /7,11/. Thus, the progress of recrystallization is governed by local fluctuations in the driving and pinning forces. The manifestation of these fluctuations is the characteristic jerky motion of the grain boundaries. Where the moving boundary comes to a stop, a local balance sets up between F_d and F_p. If the bubbles in the wire can easily coarsen and, as a result of the increased bubble diameter and decreased density of the bubbles, the pinning force, F_p, locally decreases, then the grain boundary can overcome its blocked position, which appears on the screen as a jerk and on the kinetic plot as a ledge.

From the consideration presented above, it is clear that the number of the breaking points and ledges of the kinetic plot is closely associated with the stability of the bubble structure; the greater the number of breaking points and ledges on the kinetic plot, the greater the tendency of the bubbles to coarsen and to grow at high temperatures. This interpretation is compatible with the results of Schade, who found that the onset of secondary recrystallization in doped tungsten wires correlates with the growth of the bubbles /12/.

Fig. 4 shows that the kinetic plot of wire A has more breaking points and ledges than the plot of wire B. This means that, during the recrystallization process, the grain boundaries in wire A were more frequently able to overcome their blocked positions than in wire B. consequently, the first stage of recrystallization in wire A lasted for a relatively long time. In contrast, the bubble rows in wire B proved to be very stable barriers because, despite the higher threshold temperature that would promote bubble coarsening, after a quick grain growth and subsequent few boundary jumps, no further grain growth occurred. The observed difference in the recrystallization kinetics between wire A and wire B suggests that the stability of the bubble structure in wire A is lower than in wire B. As shown in Fig. 1, this prediction is supported by the results of our high temperature creep experiments, indicating that cavitation in wire A occurred at a higher rate than in wire B.

4. CONCLUSIONS

From the results of recrystallization and creep experiments, it was concluded that the high-temperature fracture behaviour of bubble-strengthened tungsten wires can be predicted from the kinetics of secondary recrystallization. A correlation between the number of ledges in the kinetic diagram and the rate of cavitation was found: the greater the number of the ledges in the kinetic plot, the higher the rate of the creep cavitation associated with a low stability of the bubble structure.

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