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Strength and deformation mechanism of tungsten wires exposed to high temperature annealing: Impact of potassium doping

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ABSTRACT

Recent efforts dedicated to the assessment of mechanical properties of tungsten wires, as means for fiber-reinforced composites, have shown that potassium (K) doping in the as-drawn state does not modify the mechanical properties of the wire. High temperature annealing (T_a up to 2300 °C) leads to the severe embrittlement of the wire associated with the loss of fracture strength. In this work, we assess the transition behavior of pure and K-doped W wires exposed to the annealing in the temperature range of 1000–2300 °C to identify and recommend temperatures suitable for operation and fabrication of the fiber-reinforced composites. The results of mechanical tests performed in the temperature range of RT-500 °C are reported and substantiated by the electron microscopy analysis. Room temperature tests demonstrate that pure W wires become fully brittle after annealing above 1300 °C, whereas K-doped wires loses ductility above 2100 °C. With raising the test temperature to 300–500 °C, it is found that the strength of pure W wire reduces by a factor of two at T_a = 1000 °C (as compared to non-annealed wire), and goes down to 100 MPa at T_a = 1900 °C. The K-doping suppresses the reduction of the fracture strength at least up to T_a = 1900 °C, thus offering a temperature gap of ~600 °C for the use as reinforcement.

1. Introduction

Drawn tungsten wire originally developed for the illumination industry [1] has been used as reinforcing fiber in various composite concepts [2–4]. In this context, W wire recently got more attention to be used in advanced composites for fusion applications [5]. As a consequence of the drawing process, tungsten wire features a unique microstructure consisting of elongated intertwined grains [6,7]. The small grain size perpendicular to the wire axis together with the high dislocation density bestows high ductility (high density of mobile dislocations) [8] and also considerable strength (Hall-Petch strengthening) [9].

In its original application the focus was put on the creep resistance at very high temperature which has been significantly improved by doping pure tungsten wire with small amounts of potassium [10]. Further efforts in the research on W wire have been concentrated for many years in the development of the manufacturing process and high temperature stability of the microstructure. When applied as reinforcement element in composites the performance at lower temperature in general as well as basic mechanical properties like strength or fracture behaviour are becoming of essential importance. Besides the high strength, the ductile deformation is a crucial aspect to ensure durability of the composite under cyclic loads. Several studies were conducted to determine the mechanical properties and ductility at room temperature as well as at elevated temperature. Ability of plastic deformation in tungsten is not only important for the ductility, but also have consequences for the gas diffusion and eventually retention associated with the trapping on dislocations (see refs. [11–13]). Thus, investigation of the capacity of tungsten wires to experience the plastic deformation is important from a number of perspectives.

Recently, a set of W wires with and without potassium doping was studied in [14,15] by mechanical tests performed up to 600 °C. For the as-drawn wire, the potassium doping practically did not change the response to the mechanical load. The fracture occurred by the elongation and delamination of sub-grains (elongated in the direction of wire axis) showing the knife-edge necking fracture typically observed for pure W wire [16]. Both fracture stress and fracture strain monotonically decreased with increasing test temperature. The necking was essentially

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localized, and no diffuse necking deformation, usually causing extensive after yield elongation, was found for the wires in the as-fabricated condition irrespective of K doping.

The impact of high temperature annealing (T_a = 2300 °C) on the mechanical properties of the potassium-doped wire was studied at elevated temperature and followed by detailed microstructural analysis in [15]. The annealing induced severe embrittlement expressed in the strong reduction of the yield and fracture stress. The fracture mechanism was also different, namely: cleavage at 100 °C and ductile necking above 300 °C. The change of the deformation mechanism and thereby increased elongation was attributed to the onset of the 3D plastic deformation by movement of screw dislocations. The total elongation of the wire before the fracture was much higher as compared to the as-drawn wires. However, despite a positive result with respect to the total elongation, the actual fracture stress is found to be ~100 MPa which is very low value to enable reinforcement of the component.

A similar radical change in the deformation behavior is reported for pure tungsten wire treated at elevated temperature [17]. However, the temperature needed to drastically reduce the strength of W wire is significantly lower as compared to doped samples. To understand this difference, one needs to clarify possible microstructural changes associated with the heat treatment and deformation of the material at high temperature.

In pure tungsten wire recrystallization associated with a change in microstructure, i.e. a loss of the so-called Hosford structure as well as a complete removal of the dislocation structure, is reported to occur at 800 °C [17], as studied by scanning electron microscopy. However, after annealing up to this temperature, the elongated grain structure as well as the good mechanical properties are preserved. In contrast to that, annealing at 1627 °C leads to the formation of large equiaxed grains and to a severe degradation of the mechanical properties.

The recrystallization of K-doped tungsten is known to be suppressed by the potassium bubbles, which form rows of K particles at grain boundaries at the onset of the recrystallization thereby inhibiting grain interface movement in radial direction [18]. Thus, the elongated grain structure is retained upon grain coarsening until some grains reach the critical size (so called Hillerts criteria [19]) and rapid grain growth is promoted. Therefore, two distinct stages are identified: at first a relatively uniform coarsening sometimes referred to as primary recrystallization (starting in W at 800 °C) followed by a rapid growth of large elongated grains referred to as secondary recrystallization or extensive grain growth (starting in W at 1900 °C) [20].

In this work, we perform a set of parametric mechanical tests at room temperature (RT), 300 and 500 °C to investigate the fracture stress, uniform elongation, strain hardening and fracture mode of pure and K-doped W wires annealed in the temperature range of 1000–2300 °C. Thus, the actual transition of the mechanical properties as a function of the annealing temperature going from the dislocation recovery up to the recrystallization has been assessed to reveal limits for the critical reduction of the wire strength.

2. Experimental details

Drawn potassium doped (60–75 ppm) and pure tungsten wires, identical to the wires used in [14,15,21] were provided by the OSRAM GmbH, Schwabmünchen. The diameter of the wires was measured to be 148.7 ± 0.2 μm [21]. Measurements were performed by high resolution optical microscope. The K-doped and pure wires were cut into pieces of 100 mm and these were annealed at 1000 °C, 1300 °C, 1600 °C, 1900 °C, and 2100 °C. The highest and lowest annealing temperature was not used for pure and doped W wire respectively. For the doped W wire, the changes in the microstructure after annealing at 1000 °C were considered to be minor, based on the previously performed experiments. While, in the case of pure wire, experience has shown that the annealing at 2100 °C makes the wire completely brittle such that even clamping procedure may lead to the wire fracture. To perform the annealing, the wire was straightened prior to cutting. The straightened and cut wire pieces were then annealed in a tube furnace under hydrogen atmosphere at Osram GmbH. During this process the samples were placed on a shovel (carbon free) and kept at mentioned temperatures for 30 min.

The ends of the wire pieces (called fibers in the following) were clutched by two parallel mirror-polished stainless steel plates. The actual gauge section was 30 mm and the sample holder was equipped with a guide rail to ensure perfect alignment before and during the test. More details about testing procedure can be found in our earlier work [14]. To ensure constant temperature during the test, the gauge section of the sample and inner parts of the holders were placed inside the cylindrical furnace. The temperature of the sample was measured by a thermocouple, connected to a digital voltmeter. Accuracy of the temperature measurement was ~1 K. The samples were tested in air.

The deformation on the sample is applied via the pull rod driven by an electrical gear box, with a maximum load of 0.2 kN. A constant displacement loading with a displacement rate of 5 μm/s was applied until fracture. The load, measured by a strain gauge, was registered with a frequency of 0.3 Hz. The initial and final (i.e. after fracture) length of each sample was determined by the horizontal optical comparator with a precision of 1 μm[1]. The relative error on the measurement of the pull rod displacement is ± 0.1% and the absolute error on the stress measurement (considering the cross-section of the fiber to remain constant during the test) is 15 MPa.

To provide information on the morphology of the fracture surface and assist discussion on the deformation mode, the fractured samples were inspected by scanning electron microscopy (SEM, JEOL JSM-6610LV), using a secondary electron (SE) detector and operating conditions were: 20 kV accelerating voltage and 10–15 mm working distance. Each fractured sample was also investigated by the optical microscope Leica to characterize the necking geometry. However, in the following we put emphasis on the mechanical properties, while an in-depth microstructural analysis will be provided in the follow-up work.

3. Results and discussion

The test matrix is given in Table 1. At least five valid tests per condition were performed. Here, we provide examples of stress-strain curves and micrographs of few selected fibers, reflecting a typical result obtained for each tested condition. Table 1 thus contains the labels that refer to the ID of the tested fibers whose stress-strain curves and fracture surface images are presented as demonstration of the raw results.

Fig. 1 collects typical stress-strain curves obtained for the both types of fibers tested and annealed at different temperatures. The left column presents the plots for pure tungsten wire and the right one for the doped wire. The test temperature increases from top to down.

It was found that at room temperature, the pure W wires annealed above 1300 °C do not reach the yield point and break in the elastic limit, that is why Fig. 1(a) contains only data for T_a = 1000 and 1300 °C. As the T_t is increased to 300C°, the yield point and plastic deformation is registered for pure W wires annealed up to 1900 °C. It is however clearly seen from Fig. 1(b) and (c) that the strength of the pure wires annealed up 1900 °C drops below 200 MPa. Fig. 1(a–c) also reveals that at elevated temperature, the uniform elongation significantly increases reaching few percent for the wires annealed above 1000 °C. This is accompanied by a decrease in strain hardening, which is dominant in the case of the wire annealed at 1000 °C, and almost not present for wires annealed at higher temperature. This apparently causes the built up of the critical stress intensity sufficient to induce the crack propagation and fracture of the wire. This transition in the plastic behavior should be attributed to microstructural changes as

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[1] The fiber ends are placed in the special bed, where one end is abut and the length is measured by optical microscopy.