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The effect of heat treatments on pure and potassium doped drawn tungsten wires: Part II – Fracture properties

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Advanced tungsten fibre-reinforced composites (W_f/W), showing pseudo ductile behaviour even at room temperature, are a promising option for future fusion reactors as the intrinsic brittleness of tungsten can be mitigated effectively. The drawn tungsten wires used as reinforcements are the key components of the composites, thus their mechanical properties and thermal stability define the allowed operation/fabrication temperature of the composite material itself. In this work, the room temperature fracture behaviour of the pure and potassium doped tungsten wires was investigated, focusing on the evolution of the fracture micromechanisms in respect to annealing. Single-edge-notched specimens were used, with the crack growth direction perpendicular to the drawing axis of the wire. The occurrence of either a fractographic brittle or a ductile response in the as-received state of both materials is a strong indication that the ductile-to-brittle transition temperature is about room temperature. Pure, annealed tungsten wires experience a tremendous deterioration of the fracture toughness with a very prominent transition of the failure mode. The observed embrittlement by annealing can be related to the loss of the fibrous, elongated microstructure. In contrast to this, the results of the annealed, doped wires demonstrate that the microstructural stability and preservation of the initial, beneficial grain structure is directly reflected in the crack resistance of the material. Predominately ductile behaviour, with characteristic knife-edge necking, is seen even after annealing at 1600 °C.

1. Introduction

The progress of the next generation of fusion reactors is strongly associated with the development of advanced high heat flux and plasma-facing materials, capable of enduring the challenging environment imposed by extreme operating conditions [1,2]. An extensive research regarding the first wall and divertor materials was conducted in recent years [3] leading to the choice of tungsten based materials as the most prominent candidates for these reactor parts [4,5]. Besides many advantageous properties of this refractory metal, the challenges associated with the use of tungsten (W) in high-temperature applications are its typically brittle nature and relatively high ductile-to-brittle transition temperature (DBTT) [6]. Further operational embrittlement is expected as a result of tremendous amount of neutron irradiation [7] and/or by annealing through recrystallization and grain growth [8,9]. Thus, one of the main goals in the development of the novel tungsten based material options is overcoming the problem of brittleness.

The broad range of conducted studies on the ductility and toughness enhancement of tungsten at lower temperatures led to the three main strategies being alloying (e.g. with Re [10,11] or Ir [12]), nanostructuring [13,14] and synthesizing composite materials. The third approach is a very promising option, in particular when thinking of tungsten fibre-reinforced tungsten composites (W_f/W) [15], where brittleness is mitigated by utilizing extrinsic toughening mechanisms [16]. W_f/W composites consist of commercially available drawn tungsten wires which are embedded in a tungsten matrix, either by a chemical deposition process [17] or by powder metallurgy [18]. Thus, key components which determine the structural integrity of these advanced plasma facing composites are the tungsten wires used as fibres, which sets the requirement of their exceptional properties and brings an interest in studying them.

Drawn tungsten wire was of great importance in the past century as it was originally developed for the illumination industry and used as the main filament element for light bulbs [19]. Initial studies were mostly focused on the production process and creep resistance at very high temperatures, which led to the discovery of the exceptional influence of doping of tungsten by a small amount of potassium (K) [20]. In such a way, the embrittlement of pure tungsten as a result of recrystallization,

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grain growth and thermal fatigue, which directly influenced lifetime performance of the lamps, was successfully mitigated. The advantageous effect of doping was correlated to the formation of K bubbles at the grain boundaries, which suppress the secondary recrystallization to higher temperatures and keep the initial, beneficial microstructure for a longer timespan [21]. The research in the following years was mostly dedicated to the optimization of the manufacturing process and the high temperature stability of the microstructure.

When used as reinforcement element in the composite materials, the performance of the tungsten wire and in particular its crack resistance properties at moderate temperatures are of fundamental importance. Investigations dealing with the fracture behaviour of W wire conducted over the past decades, focused on a wide range of different topics such as the typical failure modes occurring during the drawing process [22], complex mechanisms of lamp filaments deterioration [23], analyses of the wire splitting as a result of bending, stretching or coiling [24,25], just to name a few. Furthermore, several studies were addressing the influence of the microstructure on the occurrence of different fracture modes [26,27], as well as discussing the effect of grain structure of recrystallized wires [28].

However, despite the existing significant body of research on tungsten wires, there are still some open questions and gaps in the scientific knowledge, where a deeper microscopic understanding of the important properties is needed. This is particularly true for the crack resistance of the wire, where more comprehensive information regarding the fracture mechanisms, as well as correlation to the underlying microstructure is of the utmost importance. This holds true for both the as-received, as well as the annealed state of the wire.

Determination of the relationship between the microstructural features and the resulting fracture mechanical properties is one of the main goals of the performed work, as the variations in the grain structure are strongly reflected in the materials fracture behaviour. This contribution is the second part of the study conducted on pure and potassium doped wires. In the first part, the question of recrystallization phenomena and microstructural stability was addressed [29], through detailed analyses of the evolution of various aspects of the microstructure with annealing temperature. The main scope of this part of the study is the room temperature (RT) fracture toughness assessment of tungsten wires and the investigation of the evolution of the fracture mechanisms in respect to different heat treatments.

2. Materials and experimental methods

The investigated materials are commercially available drawn pure and potassium doped (K-W, 60 ppm) tungsten wires, with a diameter of 150 µm, which were provided by the OSRAM GmbH, Schwabmünchen. The typical production steps of tungsten-based materials involve a powder metallurgical process of sintering and swaging, while the final, small dimension of the wire is obtained by series of subsequent drawing steps.

In order to study the effect of heat treatments and annealing phenomena on the resulting fracture behaviour, the experiments were performed on both as-received and annealed samples. Based on the microstructural characterization conducted in the first part of the publication, two annealing temperatures were chosen for this study: 1300 °C and 1600 °C. The results indicate that the pure tungsten wire recrystallizes when annealed for 1 h at 1300 °C, while the highest temperature of 1600 °C induces substantial grain growth. In contrast, the elongated microstructure of the doped wire remains mostly preserved throughout the heat treatments. In such a way, selected temperatures enable investigating the relation between the microstructural evolution and the embrittlement by annealing and/or ductility conservation. The oxidation of the wires during heat treatments was prevented by using a tungsten based vacuum furnace (Leybold Heraeus PD 1000). The annealing was done at a pressure $< 10^{-5}$ mbar with the holding time of 1 h with subsequent furnace cooling. All the samples are assumed to have identical chemical composition, as they were cut from the same two spools of wires. Thus, the influence of the composition on the resulting properties can be excluded.

Single-edge notched tension (SENT) specimens were used for studying the damage tolerance of the investigated materials. Hence, the fracture experiments were conducted in one principal testing direction with the crack growth directions perpendicular to the drawing axis of the wires. The notches were introduced by a femtosecond (fs) laser (Origami 10 XP, Onefive GmbH), which is attached to the focused ion beam/scanning electron microscope (FIB/SEM, Auriga) workstation, enabling a precise positioning of the laser cuts. It has a pulse duration of about 500 fs and a focused spot size diameter of about 25 µm. Further details on the laser system can be found elsewhere [30]. The processing of the notches was performed under vacuum conditions with a fluence of 0.6 J/cm^2 and a pulse repetition rate of 100 kHz. The laser beam is guided across the sample surface by a galvanometer scanner. It was scanned repeatedly 14 times with a speed of 1 mm/s along a line, sufficient to cover the whole diameter of a wire. Hence, the time required for the processing of a single notch is below 4 s making laser cutting a very fast and therefore advantageous notch fabrication technique. Due to the ultrashort pulse duration, the heat influence on the material is negligible in the ideal case [31]. The typical sample length of the wire was about 20 mm with the notch depth being about one third of the wire diameter. Furthermore, in order to investigate the influence of a nearly atomically-sharp flaw in a material, some of the as-received samples also contained a pre-crack like notch introduced by a FIB workstation (FIB, Leo 1540, Zeiss). A crack-like structure, with the length of 50 µm, was milled in the centre of the laser notch using an ion current of 5 nA for 30 min. An example of a top view of a laser notch (perpendicular to the drawing axis) as well as the position of the FIB cut can be seen in Fig. 1a.

Fracture mechanical experiments were performed on a small scale tensile testing machine provided from Kammrath and Weiss, with a maximum load capacity of 200 N. All the tests were displacement

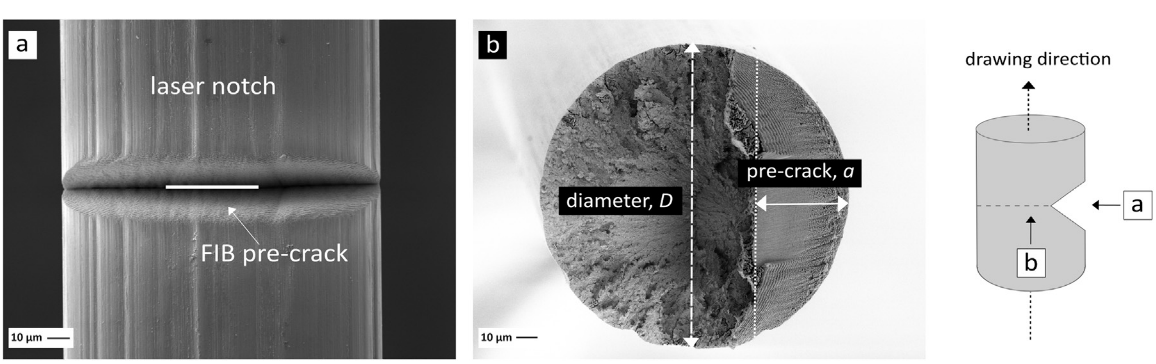

Fig. 1. Scanning electron micrographs of a) a top view of a laser notch with designated position of a FIB cut and b) an exemplary fracture surface showing how a minimal length of a pre-crack was measured. The schematic drawing on the right illustrates a characteristic SENT specimen with indicated viewing directions.