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Microstructure, mechanical behaviour and fracture of pure tungsten wire after different heat treatments

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ABSTRACT

Plastic deformation of tungsten wire is an effective source of toughening tungsten fibre-reinforced tungsten composites (W_f/W) and other tungsten fibre-reinforced composites. To provide a reference for optimization of those composites, unconstrained pure tungsten wire is studied after various heat treatments in terms of microstructure, mechanical behaviour and fracture mode. Recrystallization is already observed at a relatively low temperature of 1273 K due to the large driving force caused by a high dislocation density. Annealing for 30 min at 1900 K also leads to recrystallization, but causes a rather different microstructure. As-fabricated wire and wire recrystallized at 1273 K for 3 h show fine grains with a high aspect ratio and a substantial plastic deformability: a clearly defined tensile strength, high plastic work, similar necking shape, and the characteristic knife-edge-necking of individual grains on the fracture surface. While the wire recrystallized at 1900 K displays large, almost equiaxed grains with low aspect ratios as well as distinct brittle properties. Therefore, it is suggested that a high aspect ratio of the grains is important for the ductile behaviour of tungsten wire and that embrittlement is caused by the loss of the preferable elongated grain structure rather than by recrystallization. In addition, a detailed evaluation of the plastic deformation behaviour during tensile test gives guidance to the design and optimization of tungsten fibre-reinforced composites.

1. Introduction

Due to its very high melting point and superior high temperature strength, tungsten is an attractive material for high temperature components, including the plasma-facing components of fusion reactors [1]. However, due to its inherent brittleness and its susceptibility to embrittlement during operation, toughening of tungsten is necessary for this purpose [2]. Recently, the concept of reinforcing tungsten with tungsten wire has been proposed where toughening is achieved by extrinsic mechanisms of energy dissipation induced by the interaction between the wire, the matrix and the interface layer [3–8]. These mechanisms have been utilized in ceramic matrix composites (CMC) since the 1980s [9–11]. In the case of tungsten fibre-reinforced tungsten composites (W_f/W), the possible plastic deformation of tungsten wire (used as fibres) [12,13] provides an additional effective method of energy dissipation [6,14]. The toughening effect caused thereby has been estimated based on experimental tensile data showing that the mechanical behaviour of the wire has a strong influence. Therefore, a detailed investigation of unconstrained pure tungsten wire, with special attention to the energy consumed by plastic deformation of the wire (plastic work) and the necking geometry (which are both rarely reported in literature), will facilitate the optimization of W_f/W and other tungsten fibre-reinforced composites, like tungsten fibre-reinforced Cu composites [15,16], Zr-based metallic glass composites [17] and aluminium composites [18].

Bulk tungsten materials show brittleness below the ductile-to-brittle transition temperature of about 500–900 K (depending on microstructure, composition and testing conditions) and embrittlement by recrystallization [19]. Heavily worked, doped, as well as pure tungsten wires behave quite different and fracture in a ductile manner even at room temperature [20]. There have been many reports on the microstructural development of doped tungsten wires during annealing,

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which is particularly characterized by the preservation of grains with a high aspect ratio due to the dopants pinning the grain boundaries [12,13,21]. On the other hand, studies of pure tungsten wire addressing the question of microstructural evolution and embrittlement by annealing are rare.

In this study, pure tungsten wires with different thermal history were tensile tested and investigated regarding microstructural development, mechanical behaviour (plastic work, strength and necking geometry), fracture properties and the relationships between them. Knowledge concerning the necking geometry can provide an idea for an optimal debonding length, while determination of plastic work allows an estimation of the toughening effect, thereby assisting the design and optimization of W_f/W or other tungsten fibre-reinforced composites.

2. Experimental

One spool of pure tungsten[1] wire with a diameter of 150 μm was received from OSRAM GmbH, Schwabmünchen (company term: K-Draht Typ B). Beside *as-fabricated wire*, two other batches were produced. One batch was heated to a temperature of 1273 K and annealed for 3 h. In the following, this batch is referred to as *low-temperature heat-treated wire* (*L-heated wire*). Another batch was heated to the higher temperature of 1900 K and annealed for 30 min. This batch is referred to as *high-temperature heat-treated wire* (*H-heated wire*). Both heat treatments were done in a molybdenum (L-heated wire) and tungsten (H-heated wire) based vacuum furnace to avoid oxidation. The microstructures of the as-fabricated, the L-heated and the H-heated wires were investigated using a scanning electron microscope (SEM; Helios Nanolab 600, FEI) with an annular solid state backscatter electron (BSE) detector. Both cross-sections and longitudinal sections, were prepared by standard metallographic procedures and investigated by electron channelling contrast providing different brightness depending on the crystallographic orientation and local defects. A first assessment of the grain geometries in the cross-section was achieved by manually correlating with the scale of the sections using the software imageJ [22]. Only grains showing a distinct grain boundary are taken into account. Additionally, longitudinal sections were examined by electron backscatter diffraction (EBSD; HKL Nordlys II, Channel 5-Software). For each kind of wire, an orientation map of about 7000 μm^2 (as-fabricated), 5000 μm^2 (L-heated) and 6600 μm^2 (H-heated) was acquired with a step size of 0.25 μm. About 90% of the pixels in the patterns were successfully indexed by the software. The acquired orientation maps were post-processed by assigning the average orientation of the neighbouring pixels to an unindexed pixel, if seven or more neighbouring pixels showed the same orientation. The orientation maps obtained by EBSD were used to get a first estimation of the grain sizes in the longitudinal direction by using imageJ and refined by quantitative analysis of the orientation data. The texture of the wires was determined from pole figures calculated from the orientations acquired by EBSD.

Uniaxial tensile tests were conducted by using a universal testing machine (TIRA Test 2820) to acquire stress-strain curves. A sketch of the testing machine is shown in Fig. 1. The wires were cut into 80 mm long pieces. Both ends of such a tungsten wire specimen were covered by glue (UHU Plus endfest 300) to protect the ends from being damaged by the clamps and to provoke the sample to neck and crack in the central part. The free length was about 30 mm. Vertical alignment of the samples was achieved by adjusting the bottom clamp. The tensile tests were started with a pre-load of 5 N and conducted in a displacement-controlled mode at a cross head speed of 5 μm/s. The force was recorded by a 200 N load cell. The elongation of the sample was measured by a laser-speckle extensometer (LSE-4000 DE) as indicated in Fig. 1. Two spots on the sample with a distance of about 16 mm are illuminated by the laser system. The light is reflected on the optically rough wire surface and interferes with itself, resulting in a characteristic speckle pattern which is recorded by a CCD line camera (Sony XD-ES50). From the speckle movement, the elongation of the wire inside the measuring length is calculated. The gauge length thus corresponds to the laser spot distance. If the wire fractures between the two laser spots, both a uniform and a localized elongation (e.g. due to necking preceding fracture), are recorded by the laser-speckle extensometer. If the fracture occurs outside the measuring length, only the uniform deformation is detected.

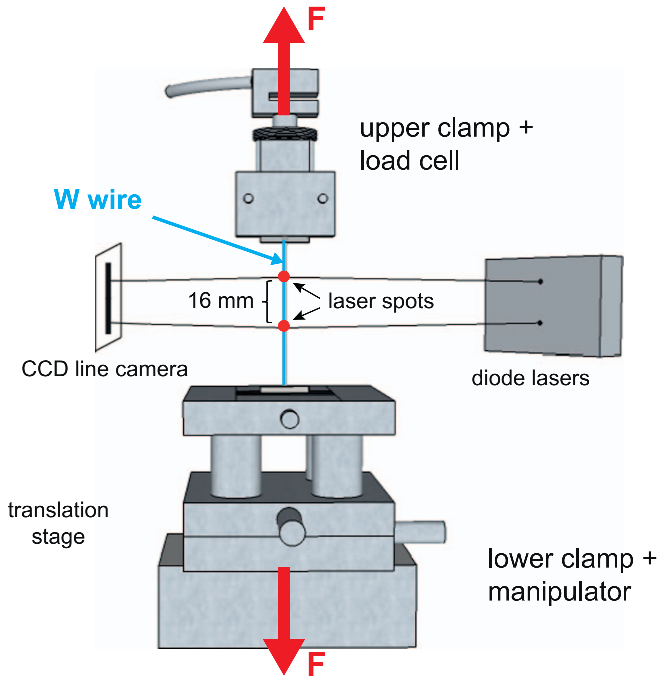

Fig. 1. A schematic diagram of the tensile testing machine where a laser-speckle extensometer is used to measure the elongation of a small sample.

Finally, the fracture surfaces were analysed by SEM and the geometry of the necking zone was characterized by a Confocal Laser Scanning Microscope (CLSM; OLYMPUS LEXT OLS 4000). By the combination of in-focus images at different heights, the confocal microscope allows images to be acquired with depth sensitivity. As the in-focus information from the laser provides a height value, the non-planar geometry of the necking region can be quantified. For this examination, the samples were put on a sample holder with the wire axis aligned parallel to the x-axis and roughly horizontal. The obtained 3D data were precisely levelled afterwards by a two-point method along the wire axis.

3. Results

3.1. Microstructure

Typical cross-sections and longitudinal sections of the as-fabricated wire are shown in Figs. 2 and 3, respectively. The contrast depends on the local grain orientation. This contrast is sensitive to subgrain boundaries with a misorientation below 1° [23]. In the cross-section (Fig. 2), bended grains forming a curled structure are observed. These structures are also known as “Van Gogh sky structures” for their similarity with his style used in painting sky [24]. The grains are not equiaxed and have sizes in the range of (0.1–0.4) × (0.5–1) μm^2 based on Fig. 2 (right) (note that all sizes reported in the observed sections are Feret diameters). The longitudinal section, shown in Fig. 3, displays extremely elongated grains. In the interior of the grains, significant changes in contrast can be detected, which are caused by elastic strains

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[1] 99.99 purity according to manufacturer specifications.