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Properties of drawn W wire used as high performance fibre in tungsten fibre-reinforced tungsten composite

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Abstract. High strength and creep resistance also at high temperature, combined with a high thermal conductivity and high melting point make tungsten (W) an ideal material for highly loaded areas in future fusion reactors. However, as a typical bcc metal tungsten features an intrinsic brittleness up to very high temperature and is prone to operational embrittlement. Tungsten fibre-reinforced tungsten composite (Wf/W) utilizes extrinsic toughening mechanisms similar to ceramic fibre-reinforced ceramics and therefore overcomes the brittleness problem. The properties of the composite are to a large extent determined by the properties of the drawn tungsten wire used as reinforcement fibres. W wire exhibits a superior strength and shows ductile behaviour with exceptional local plasticity. Beside the typical mechanisms observed for ceramic composites the ductile deformation of the fibres is therefore an additional very effective toughening mechanism. Tension tests were used to investigate this phenomenon in more detail. Results show that there is a region of enhanced localized plastic deformation. The specific energy consumption in this region was estimated and used to suggest optimisation options for Wf/W composites.

1. Introduction
The development of capable materials is essential for sophisticated future energy systems like a fusion reactor. Due to its high strength and creep resistance also at high temperature, combined with a high thermal conductivity and melting point tungsten is a very promising candidate for highly loaded areas in such a reactor [1]. However, as a typical bcc metal tungsten features an intrinsic brittleness up to a temperature of typically 500 K - 600 K [2] and up to above 1200 K for high temperature annealed material [3]. In addition, W is prone to operational embrittlement e.g. by grain coarsening [4] or/and neutron irradiation [5].
Tungsten fibre-reinforced tungsten composites (W_t/W) overcome the brittleness problem of tungsten by utilizing extrinsic toughening mechanisms [6]. Therefore, an application of W as a plasma facing material under thermal transients and neutron bombardment becomes feasible [7]. Hence W_t/W was chosen as risk mitigation plasma facing component and high heat flux material in the EU fusion roadmap [8, 9].

W_t/W composites consist of tungsten fibres made of commercial drawn tungsten wire embedded in a tungsten matrix produced either by a chemical process [10, 7] or by powder metallurgy [11]. The composite structure is determined and maintained (during fabrication and operation) by engineered interface systems between fibre and matrix [12]. The principle of extrinsic toughening in such a composite system has been shown in the past years at the Max-Planck-Institute for Plasma Physics, Garching (IPP) [13, 14]. Various active extrinsic toughening mechanisms, e.g. fibre bridging or fibre pull-out, have been shown in model systems consisting of a single W fibre embedded in a chemically deposited tungsten matrix in the as-fabricated case, as well as after embrittlement of the fibre by recrystallization and grain growth (called "embrittled" in the following) [15, 16]. In a further development step a fabrication method for bulk material was developed based on the chemical deposition process and first samples were produced. Mechanical tests on these samples revealed a very large toughness at room temperature and active toughening mechanisms in embrittled conditions [10, 17].

The idea of extrinsic toughening has been developed for ceramic materials and has been successfully used for many years [18]. Besides the typical mechanisms observed in ceramic composites the ductile deformation of the tungsten wire has been shown as a very effective additional mechanism in as-fabricated W_t/W [15]. A significant local plasticity combined with a large local strain of up to 1.36 for a length of 66 µm has been observed. The specific energy consumption in this region is therefore very high, being the reason for the high efficiency of the mechanism. As the specific deformation energy is highest in the necking region the formation, growth and extension of this zone are important for the optimisation of W_t/W composites.

In this contribution we give an overview of the tensile properties of tungsten wire and focus on the ductile deformation and fracture behaviour. Special focus is laid on the necking region. Results of standard tension tests and manual stepwise tests in combination with 3D observation in a confocal microscope are presented. The link between fibre properties and composite properties is discussed in detail.

2. Tensile properties of tungsten wire

Commercial drawn tungsten wire is used as fibres in W_t/W composites. The development of tungsten wire is strongly related with the illumination industry (more details in [19]). Starting with pure tungsten and a powder metallurgical process Coolidge realized the possibility to produce ductile tungsten wire by strong mechanical deformation at elevated temperature (more details e.g. in [20]). Later it was realized that W wire doped with several 10 ppm potassium (K) shows a very beneficial high temperature creep stability. During the drawing process K forms small bubble rows which are aligned on the grain boundaries and lead to a beneficial evolution of the grain structure at high temperature (more details e.g. in [21]).

As a consequence of the drawing process, tungsten wire features a unique microstructure consisting of elongated intertwined grains. The small grain size perpendicular to the wire axis together with the high dislocation density bestows high ductility (high density of mobile dislocations) and also considerable strength on the drawn wire (Hall-Petch strengthening). In figure 1 typical stress-strain curves of an as-fabricated doped tungsten wire tested at room temperature are shown (similar to the curves shown in [22]). After elastic loading a phase of significant work hardening occurs before the maximum stress is reached. The maximum stress is embedded in a region with only small variations in stress. A rapid stress decrease precedes the final fracture of the wire.
Figure 1. Typical tensile curves of three K doped W wire samples (diameter: 150 µm) (similar to [22]). The onset of apparent necking strain is shown in comparison to the ultimate strain and the fracture strain for each sample.

In figure 2 scanning electron microscopy pictures of a typical fracture surface of an as-fabricated pure tungsten wire after tensile testing is shown. The shown fibre has a diameter of 150 µm and was tested with a tensile speed of 5 µm s⁻¹ for a measuring length of 18 mm. The strong necking as an indication of the wire’s ductility is clearly visible in the images (figure 2, upper left corner). Besides some larger cracks with different sizes a typical knife-edge necking of individual grains leads to a fibrous structure (bottom row). There is no significant difference to fracture surfaces obtained for doped tungsten wire in previous studies (compare e.g. [7, 23]). Leber et al. [23] describes this behaviour as a consequence of the microstructure. During tensile loading the fine elongated grains produced in the drawing process debond at longitudinal grain boundaries due to the Poisson’s contraction. The resulting free-standing individual grains afterwards neck down in a knife-edge fracture mode leading to a typical fibrous surface.

Tensile test have been used to investigate the mechanical properties of both pure [24] and potassium doped tungsten wire [22]. As-fabricated and annealed samples with a diameter of 150 µm have been tested at room temperature for both grades. In the as-fabricated case and after annealing at 1273 K (3 h) pure W wire shows ductile behaviour and an ultimate strength of around 2900 MPa and 2000 MPa, respectively. A heat treatment of 1900 K (30 min) leads to embrittlement and a mean strength of approximately 900 MPa. Potassium doped W wire shows an ultimate strength of about 2700 MPa in the as-fabricated state and a ductile behaviour up to an annealing temperature of 2173 K (30 min). The tensile strength stays up to about 2000 MPa for this temperature. At an annealing temperature of 2500 K (30 min) rapid grain growth leads to embrittlement and a decrease of strength to 1300 MPa. For both grades the embrittlement is correlated with the loss of the fine elongated grain structure and not necessarily with the occurrence of (primary) recrystallisation. For pure tungsten wire this transition takes place at 1273 K, for potassium doped wire at 2173 K.

3. Experimental

Drawn W wire doped with 60-75 ppm potassium was tested in standard tension tests and in manual tests in combination with in-situ microscopy. In a first step the wire was cut into appropriate long pieces. For the standard test mechanically straightened wire was used (details
Figure 2. Fracture surface of a pure tungsten wire sample, fractured in a tensile test at room temperature (details of the testing procedure in [24]). In the side view (top row) the necking (left) and the fibrous fracture surface (right) are visible. The top views (bottom row) reveal the differently sized cracks and the typical knife-edge fracture of individual grains (right).

in [22]). To concentrate the fracture in the centre both ends of a wire sample were attached to paper by glueing (UHU Plus endfest 300) for the standard test or covered by the glue only for the manual test. The uncovered part was approximately 20 mm for the standard test and 2 mm for the manual test.

The standard tests were conducted similar to the procedure presented in [22]. The tests were performed with a universal testing machine (TIRA Test 2820) at room temperature. The load was measured by a 200 N range load cell. The displacement was measured contact-less by a laser speckle extensometer (LSE-4000 DE). The measuring length was in the range of 8 mm for 8 samples (short) and 18 mm for 6 samples (long). The tests were conducted in a displacement controlled mode with a displacement rate of 5 µm s⁻¹. More details can be found in [22]. Both pieces of each fractured sample were investigated in a confocal optical microscope (LEXT OLS 4000 3D Laser Measuring Microscope). In figure 3 the microscopic picture of one piece (left) together with a schematic drawing of the corresponding second piece (right) is shown for a typical fractured fibre. The necking diameter as well as the size of the zone of macroscopic necking were determined together with the necking elongation. Macroscopic necking was defined as the region where the diameter is smaller than 99% of the original diameter. This corresponds with the accuracy of the measurement in this investigation. The measurements were done for each side of a fractured sample separately and then summed up to determine the total value. The necking diameter was evaluated at the respective fracture surface. To determine the size of
the macroscopic necking zone the diameter for each pixel along the necking zone was determined as a first step. This zone starts at the 99% limit and ends at the tip of the fibre. The tip was defined as the point where the diameter shows a strong variation due to the roughness of the fracture surface. The necking length was then determined by counting the pixels between the 99% limit and the tip of the fibre. Assuming volume conservation the elongation during fracture was determined. For each pixel along the necking length a cylinder volume was calculated with the height of one pixel and the corresponding diameter (see figure 3). These cylinder slices were then summed up to calculate the total volume. By assuming a perfect cylinder for the undeformed sample the original size of the necking zone was obtained. The necking elongation was then calculated by subtracting the original size from the beforehand obtained size of the fractured sample.

The device used for the manual tests was small enough to be placed in the confocal microscope mentioned before allowing the detailed observation of the deformation process. The device consisted of two Teflon coated (to minimize friction) plates at which the wire was attached in the centre. At one side the plates were connected in a rotatable way and on the other side a screw with a fine thread (250µm pitch) allowed to move the plates apart. The attached fibre was thereby strained. The screw was moved by hand in 24 steps per turn. Due to the very small opening compared to the dimensions of the whole setup the fibre was mainly strained in tension. A microscopic observation was performed every 4-5 steps. If a change of the wire diameter was recognized, observations were performed every step or half-step. The microscopy pictures were used to determine the specific elongation for different regions of the wire. Surface features were used to divide the measuring length in distinct regions and a picture-to-picture comparison between the different steps was used to determine the specific elongation.

4. Results and Discussion

The size of the necking zone was determined to be around 250µm with an elongation of around 50µm. It has to be noted that there is a significant variation in the determined necking zone size (187µm - 369µm) and necking elongation (41µm - 69µm). The necking diameter was around 120µm which corresponds with to reduction in area of \( q = 0.36 \pm 0.02 \) and a zero gauge length elongation of \( \epsilon_0 = 0.6 \). The detailed values are shown in table 1. The rough fracture surface has an impact on the accuracy of the obtained values. However, as surface features on a typical fracture surface of a W wire (see figure 2) are small compared to the obtained values the influence is expected to be low. The reduction in area is very similar to tension test results for pure and doped W wire in previous studies [24, 23, 25]. This suggest that the observed mechanisms and behaviour seem to represent a typical behaviour of W wire. In comparison to the constraint wire in \( \text{Wf/W} \) the reduction in area is similar to results for moderate testing speeds [15], but larger than for samples tested at very high speeds [7]. The size of the necking zone is much larger than in the constraint case (250µm compared to 66µm in [15]), however, the elongation
Table 1. Overview of tension test results. The uncertainties are calculated as standard deviation of the mean.

<table>
<thead>
<tr>
<th>Size</th>
<th>Necking zone</th>
<th>Necking elongation</th>
<th>Necking diameter</th>
<th>reduction in area q</th>
<th>zero gauge length elongation $\epsilon_0$</th>
</tr>
</thead>
<tbody>
<tr>
<td>short</td>
<td>243 ± 12</td>
<td>50 ± 2</td>
<td>122 ± 3</td>
<td>0.34 ± 0.02</td>
<td>0.52 ± 0.06</td>
</tr>
<tr>
<td>long</td>
<td>259 ± 27</td>
<td>52 ± 5</td>
<td>118 ± 2</td>
<td>0.39 ± 0.02</td>
<td>0.63 ± 0.06</td>
</tr>
<tr>
<td>total</td>
<td>250 ± 13</td>
<td>51 ± 2</td>
<td>120 ± 3</td>
<td>0.36 ± 0.04</td>
<td>0.57 ± 0.09</td>
</tr>
</tbody>
</table>

Figure 4. Microscopy pictures of tungsten wire at different displacement steps during a manual tension test (left). Elongation over strain in a manual stepwise tension test (right). The total values as well as the values for distinct regions are given. Most of the elongation is concentrated in region $L_4$ which represents the region where apparent necking occurs.

is smaller (50 $\mu$m compared to 92 $\mu$m in [15]). The constraint seems to have a positive impact on plasticity, but results for constraint wire are few and the issue needs further investigation.

The results of the stepwise manual test are summarized in figure 4. In step 159 the diameter reduction exceeds 1 % corresponding with the onset of macroscopic necking. The reduction is also visible in the corresponding picture. To determine the local elongations the wire was divided in regions as shown in figure 4. Region $L_4$ was chosen in a way to include the region of macroscopic necking. The specific elongation of the different regions is shown in comparison to the total elongation over rising strain (see figure 4 right). Before necking was detectable the elongation was uniformly distributed along the regions (although the regions have a different length). When macroscopic necking occurs almost all the elongation is concentrated in region $L_4$ whereas the elongation in the other regions seems to stay constant. In step 159 already around 60 % of the total elongation is generated within $L_4$ which increases up to 70 % in step 162. Due to the limited measurement accuracy a clear mapping of the specific elongation before step 159 and beyond $L_4$ is difficult. However, the results indicate clearly that with the onset of macroscopic
necking most of the elongation -which is a measure of deformation- is concentrated in the region where this necking occurs. Up to this moment the elongation of the other measuring regions contribute significantly to the total elongation.

According to the presented results the plastic deformation in the examined potassium doped tungsten wire can be classified in 3 regions (the boundaries of the different regions are according to figure 1):

- Between the yield point and the ultimate strain: plastic deformation without macroscopic necking and rising load (Ris)
- Between ultimate strain and the onset of macroscopic necking: plastic deformation without macroscopic necking and decreasing load (Dec)
- Between onset of macroscopic necking and final fracture: plastic deformation with macroscopic necking and localized deformation (MaN)

In the following we present an interpretation based on the microstructure of drawn tungsten wire. As it was mentioned before (chapter 2), the grain structure of tungsten wire consists of long elongated grains with a high aspect ratio [23, 25]. Hence the majority of grain boundaries lies parallel to the wire axis and only a small fraction is oriented perpendicular to the fibre axis. The weakness of grain boundaries in tungsten materials has been reported elsewhere [2]. Considering the highly stressed perpendicular grain boundaries and assuming a high concentration of defects in a particular wire region, it is likely that this perpendicular grain boundaries fail in this region by delamination at stresses around the ultimate strength. Even if such boundaries debond they are bridged by the adjacent long grains leading only to a slight decrease in stress and a preservation of to a ductile behaviour (region: Dec). Due to the perpendicular direction the impact of this debonding on the diameter reduction is expected to be low and therefore below the measuring limit in this investigation. However, the delamination would lead to a weakening in the particular area and thus promote the occurrence of macroscopic necking in this area (region: MaN). After macroscopic necking has occurred, the deformation of the drawn tungsten wire could follow the procedure suggested by other researchers [23, 25] and fail in the typical knife-edge fracture of individual grains. Although this model coincides with the experimental results of this study, the deformation of tungsten wire needs further investigation.

The macroscopic necking is correlated with the stress-strain curves for the three samples shown in figure 1. At first the necking elongation is normalized with the measuring length. Following the elastic line the onset of the apparent necking is found at the stress-strain curve (see figure 1). It is remarkable that the onset of the macroscopic necking deviates significantly from the ultimate strain which is typically seen as the starting point of necking [26]. The macroscopic necking seems to be correlated with the crook at the end of the tensile curve.

The above presented results suggest that the plastic deformation is very large in the region of macroscopic necking. For a first estimation the specific plastic deformation energy is determined by integrating the stress-strain curve from the onset of the macroscopic necking until the fracture and by normalizing the energy with the necking length rather than the measuring length. For comparison, the specific energy until ultimate strain is also calculated by integrating the stress-strain curve (the elastic fraction is subtracted).

\[ \bar{w}_{t,pl,Ris} = (0.032 \pm 0.002) \, \text{J m}^{-3} \]
\[ \bar{w}_{t,pl,MaN} = (0.56 \pm 0.07) \, \text{J m}^{-3} \]

The error in this estimation especially for the necking region is not negligible. Nevertheless, it is significant that the specific energy consumption in this area is more than one order of magnitude higher in comparison to the part where the deformation is distributed uniformly along the whole fibre (uniform deformation). The region between uniform strain and the onset
of the macroscopic necking (region: Dec) probably shows a mixture of localized deformation and evenly distributed elongation. Further research is necessary.

If the analysed wire is used in Wf/W composites the plastic deformation plays a significant role for the toughness of the material. The amount of energy dissipation is determined by the actual length being deformed and the specific plastic deformation energy [15]. Due to the large local plasticity in the zone of macroscopic necking the size of this zone is a good guiding value for the optimum debonding length. Based on the obtained results a debonding length in the range of 250µm would be ideal. A crack opening of 50µm would be sufficient to allow a full plastic deformation and therefore a maximum energy consumption.

5. Summary and Outlook
The large toughness of Wf/W composites is strongly related to the large specific deformation energy and high strength of the tungsten wire used as reinforcing fibre. Tension tests were used to investigate the deformation of potassium doped tungsten wire in detail. The necking diameter as well as the size of macroscopic necking and the corresponding elongation were determined. Tests with in-situ observation revealed that the elongation preceding fracture predominantly occurs in a small region as soon as macroscopic necking occurs but not necessarily when ultimate strain is reached. It was shown that the specific deformation energy in this region is much higher than in the other parts of the wire. Based on the size of this area an optimum debonding length in Wf/W composites was suggested.

The investigations showed that the deformation behaviour is strongly related to the microstructure and its evolution. Further research is essential to relate the macroscopic response to the microscopic behaviour. An optical strain measurement setup allowing the determination of local strains will be used together with a detailed microscopic analysis in a first step. Furthermore, tension tests with in-situ tomographic observation are planned.

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