Development of tungsten fibre-reinforced tungsten composites towards their use in DEMO – Potassium doped tungsten wire

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Abstract. For the next step fusion reactor the use of tungsten is inevitable to suppress erosion and allow operation at elevated temperature and high heat loads. Tungsten fibre-reinforced composites overcome the intrinsic brittleness of tungsten and its susceptibility to operation embrittlement and thus allow its use as a structural as well as an armour material. That this concept works in principle has been shown in recent years. In this contribution we present a development approach towards their use in a future fusion reactor. A multilayer approach is needed addressing all composite constituents and manufacturing steps. A huge potential lies in the optimization of the tungsten wire used as fibre. We discuss this aspect and present studies on potassium doped tungsten wire in detail. This wire, utilized in the illumination industry, could be a replacement of the so far used pure tungsten wire due to its superior high temperature properties. In tensile tests the wire showed high strength and ductility up to an annealing temperature of 2200 K. The results show that the use of doped tungsten wire could increase the allowed fabrication temperature and the overall working temperature of the composite itself.

1. Introduction

Materials with advanced capabilities are essential for the successful design of the next step fusion reactor, i.a. DEMO, and are crucial for a fusion power plant. The use of tungsten is inevitable to suppress erosion and to allow operation at elevated temperature and high heat loads [1]. However tungsten suffers from an intrinsic brittleness below a certain temperature, the so called ductile-to-brittle transition temperature [2,3]. Depending on mechanical, chemical and (micro-)structural conditions this temperature is between 500 and 900 K. In addition W is susceptible to be further embrittled by overheating or neutron irradiation [4].

Tungsten fibre-reinforced tungsten composites (W_f/W) utilize extrinsic toughening mechanisms like crack bridging by intact fibres or frictional pull-out of broken fibres. Similar to ceramic fibre-reinforced ceramics [5] the overall toughness is increased and the brittleness problem of W is mitigated. Hence an application as a plasma facing material under thermal transients and neutron bombardment now seems feasible.

That extrinsic toughening works in W_f/W has been shown at the Max-Planck-Institute for Plasma Physics, Garching (IPP) in the past years [6,7]. As a key factor for the feasibility of this toughening mechanism the interface between fibre and matrix was investigated in a first step [8,9]. The feasibility of the toughening effect itself was shown on model systems consisting of a single fibre embedded in the matrix material. With this method the major contribution of the plastic fibre deformation to the toughening was shown [10]. In addition it was proven that the toughening mechanisms are still active after a full change of the microstructure by recrystallization [11]. Figure 1 shows a summary of the active toughening effects in W_f/W. In a further development step a fabrication method based on the chemical deposition of W was developed and first bulk samples were produced [12]. Mechanical tests on these samples revealed an intense toughening. Based on these results the material was chosen as risk mitigation PFC/HHF (plasma facing component/high heat flux) material in the EU Fusion roadmap [13]. In summary the idea of extrinsic toughening in W works in principle and the application as highly loaded divertor element is identified. Thus level 2 of

the so called technology readiness level (TRL) is reached (proof-of-principle + application formulated) (explanation of TRL concept in [14]).

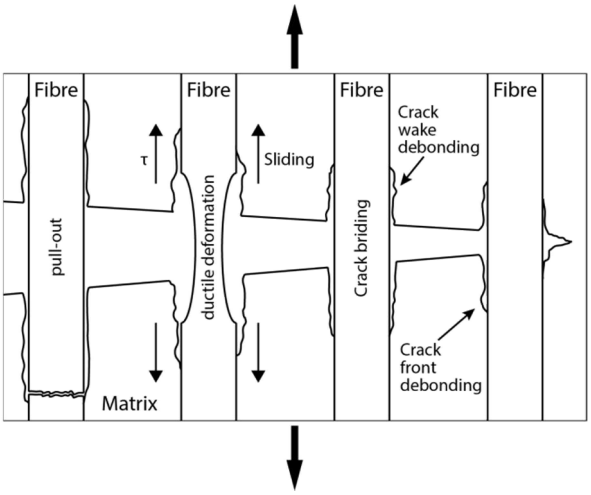

Figure 1: Toughening mechanism in tungsten fibre-reinforced composites.

Candidate materials for DEMO however need to reach TRL 6-8 before being considered for design [15]. The first step in this development is to prove that the concept of a W_f/W divertor element works (TRL3, proof-of-concept) and to validate it under relevant testing conditions (TRL4, validation). TRL 5 is typically associated with the validation in a relevant environment. In the case of W_f/W this is associated with the plasma interaction, e.g. regarding hydrogen retention or erosion, and the behavior under neutron irradiation. TRL 6 will be reached by a prototype demonstration in a relevant environment. This can be in wall tiles of existing fusion reactors, e.g. on a manipulator or as a long term wall tile.

As a first step it has been chosen to fabricate W_f/W components and test them under cyclic high heat flux conditions. These components will be designed close to the ITER reference-design. In figure 2 two possible versions for such a mock-up are shown. The loading will be performed in the ion beam facility GLADIS in Garching, Germany [16,17] and/or the electron beam facility JUDITH 1 [18]. Cyclic extreme loading allows the evaluation of the maximum strength, the fatigue strength and the damage tolerance/toughness and thus provides the conceptual proof in one step. The concentration on one main test provides clear constraints

for geometry and test methodology. This leads to a distinct structure and allows a target-oriented approach. For the successful realization and evaluation of such experiments the complete characterization of the material is essential. In summary this allows not only to show that the concept works but also qualifies W_f/W for future applications, e.g. in a DEMO divertor (TRL 4).

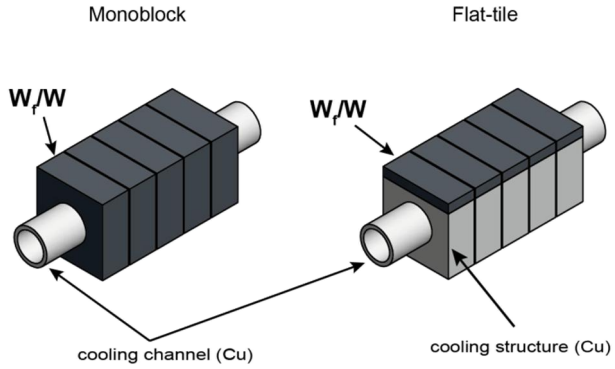

Figure 2: Monoblock and Flat tile mock-up for high heat flux tests

For the component production and the following testing all constituents of the composite, i.a. fibre, interface and matrix, and all manufacturing steps, i.a. interface coating, fibre positioning and matrix production, will be addressed. Utilizing the so far used techniques, a pure tungsten fibre, an oxidic interface and a chemically deposited matrix as a starting point, new techniques will be investigated and/or established techniques will be optimized. As all these aspects are strongly linked, e.g. the manufacturing temperature of the W matrix with the high temperature stability of the W fibre or the interface, this is a multilayer approach.

As an example we discuss in this paper the role of the tungsten wire used as fibre and especially the influence of annealing on its mechanical properties. A screening test on potassium doped tungsten wire is presented in detail. As this is strongly related to the allowed matrix manufacturing temperatures, a short review is given on matrix production techniques at first.

2. Fabrication of the tungsten matrix in W_f/W composites

The very high melting point and high temperature strength both for fibre and matrix do not allow for classical composite production routes (examples in [5]). It is furthermore important that the properties of fibre and interface are not degraded during the process. Chemical deposition techniques allow low processing temperatures (< 900 K) and a force-less fabrication, and thus the preservation of the interface and fibre integrity as well as fibre topology. In this process tungsten hexafluoride is reduced by hydrogen in a heterogeneous surface reaction and thus solid tungsten is formed.

So far surface deposition processes (chemical vapour deposition – CVD) have been used for the production of model systems containing a single fibre [8] and infiltration techniques (chemical vapour infiltration – CVI) have been investigated to produce larger samples. The infiltration process can be influenced by varying the fibre arrangement, the gas flow and the temperature. In a dual step infiltration process Riesch et al. were able to produce first bulk W_f/W for mechanical testing [12]. A key issue is the optimization of this tungsten matrix production process allowing the production of larger, reproducible samples. A chemical deposition device (WILMA) which has been specifically designed for the chemical deposition of tungsten for the matrix production in W_f/W was installed at the Forschungszentrum Jülich (FZJ).

Although CVD processes have the advantage of low production temperature and the absence of mechanical impact, powder metallurgical (PM) routes would allow several interesting benefits. The most important benefit is that the production and processing techniques are highly developed as PM is the standard process for tungsten bulk production. This would give potential for an easier optimisation and adoption of the matrix properties. For example it would be easier to implement alloying, e.g. self passivating tungsten. Nevertheless the

required high temperature and pressure might be a severe drawback regarding degradation of fibre or interface properties.

PM investigations on single fibre composites have been started in order to understand the interaction between fibre, interface and matrix. Hot isostatic pressing (HIP) was applied to produce a dense W matrix with and without embedded fibres. First samples at various HIPing temperature up to 2200 K have been produced and mechanical testing including fibre push out has been performed. These tests show that the interface properties are critical for the path forward within the HIPing approach. A detailed description of these tests and their results is given by Jasper et al. [19].

In these tests pure tungsten wire was used as fibres. These fibres were fully recrystallized during the HIP process. As recrystallized fibres possess very poor mechanical properties compared to as-fabricated ones [20] this is a severe drawback for PM production route. As a loophole potassium doped fibres are known for their high temperature stability and could be a solution. In the following we present experiments on the mechanical properties after high temperature annealing to investigate this option.

3. Mechanical properties of W-wire used as fibre in W_f/W composites

A key benefit of W_f/W under cyclic high heat loads are the exceptional properties of the tungsten wires used as fibres: Pure as well as potassium doped tungsten wires show exceptional ductility and strength at room temperature in contrast to conventional bulk tungsten being brittle at room temperature. These are ideal properties facilitating the toughening in W_f/W as the high strength is important for the bridging effect and ductile deformation allows the dissipation of substantial amount of energy (compare mechanism in figure 1).

Pure tungsten wires have been investigated by Zhao et al. [20] by means of tensile tests. Wires in the as-fabricated state and after annealing for 3 h at 1273 K and 30 minutes at 1900

K were tested. The as-fabricated and the low heat treated fibres showed ductile behaviour and a strength of more than 2900 MPa and 1900 MPa respectively. In contrast to that the high temperature heat treated fibres failed in a brittle manner with a mean strength of approximately 900 MPa, and a high scatter. In the following we present similar experiments on potassium doped tungsten wire.

3.1 Sample preparation

Drawn tungsten wire doped with 60-75 ppm potassium was used for tensile tests. The wire was produced and provided by the OSRAM GmbH, Schwabmünchen. The measured diameter of the wire was 148.7±0.2 µm. The wire was cut into pieces and straightened by tensile loading until fracture (displacement rate of 100 µm/s). The straightened wires were cut into 80 mm long pieces to get rid of the damaged zone. The 80 mm wire pieces are called fibres in the following.

Fibres in the as-fabricated state and after annealing were tested. 5 different annealing temperatures are investigated (see table 1). The annealing was done in a tube furnace under hydrogen atmosphere. The holding time was 30 min in each case.

Temperature 1-3 are below the reported temperature of extensive grain growth in potassium doped wire (2100-2300 K [21]), temperature 4 is around this region and temperature 5 is well above it. In addition temperature 1 and 3 are similar to the annealing temperatures in pure tungsten wire studies [20] and therefore allow a direct comparison.

In figure 3 optical micrographs of longitudinal sections are shown for the different sample types. The extensive grain growth leading to very big grains is clearly visible for the sample annealed at 2573 K.

Table 1: Annealing temperature

	T 0	*T 1*	*T 2*	*T 3*	*T 4*	*T 5*

Annealing temperature [K]	-	1273	1573	1873	2173	2573

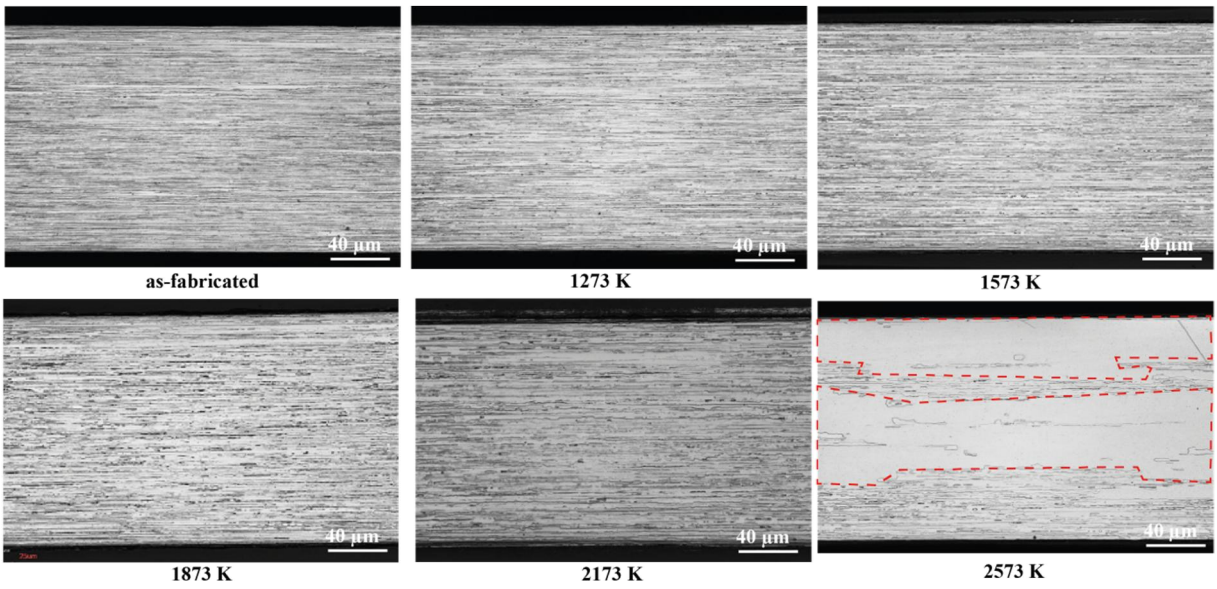

Figure 3 Longitudinal sections (optical) of potassium doped tungsten wire at different annealing stages. The dashed lines in the 2573 K case indicate large grains after secondary grain growth.

3.2 Experimental

The tensile tests were performed with a universal testing machine (TIRA Test 2820) at room temperature. The load is measured by a 200 N range load cell. The displacement was measured contactless by a laser speckle extensometer (LSE-4000 DE) using the interference pattern of two laser spots. This measurement requires a perfectly aligned sample. To align the fibre within the tension axis a preload (between 10-15 N ≙ 550-850 MPa) is applied and the lower sample holder is moved until a load minimum is reached. Still the displacement record was not always reliable.

The measuring length is defined by the distance between the two laser spots and was approximately 18 mm for all tests. Only if the fibre fractures within this zone the test is assigned as valid. To ensure that the fibre fractures within the measuring length both ends of the fibre are attached to paper by gluing (UHU Plus endfest 300). Thus the cross-section in these parts is enlarged and the probability of fracture is decreased. The uncovered part was approximately 20 mm for all tests. The tests were conducted in a displacement controlled mode with a displacement rate of 5 µm/s.

4. Results

Figure 4 (left) shows typical stress-strain curves of the tested wires. The region of elastic response was extrapolated to the origin (dotted line). Due to unreliable strain measurements the estimated Young's moduli show significant deviation between 280 and 440 GPa. To be able to compare the different annealing stages the strain axis was corrected to meet 400 GPa in the elastic region. However as the variations are quite significant we do not give any quantitative values besides the ultimate strength which is independent of the displacement measurement. Nevertheless trends are obvious for other mechanical properties like yield strength and fracture strain.

In all cases an elastic deformation is observed for low strain. Except for the 2573 K case this is followed by an extended phase of plastic deformation. If plastic deformation occurs the stress decreases moderately after reaching the ultimate strength. This goes on until a faster drop occurs near final fracture. Samples annealed at 1273K do not show this drop in 8 out of 24 cases and fail near to the maximum load. Typical curves for both cases are shown in Figure 4 (right). However yield strength and ultimate strength of these samples are comparable. The mean value for the ultimate strength of the different sample types is given in table 2. The errors are calculated by the standard deviation of the mean. For each temperature

6 valid measurements are considered. 16 samples have been taken into account for annealing temperature of 2573 K to give a better statistic in the brittle case.

In the as-fabricated case work hardening of approximately 2 % is observed. The work hardening becomes less with increased annealing temperature. For the samples annealed at 2173 K almost no work hardening is observed. The fracture strain drops with the first annealing step and stays almost constant for the next higher temperatures. For the annealing temperature of 2173 K it increases significantly and is even larger as in the as-fabricated case. However one out of 6 tested samples shows a lower fracture strain at this annealing stage. The yield strength as well as the ultimate strength decrease with rising annealing temperature.

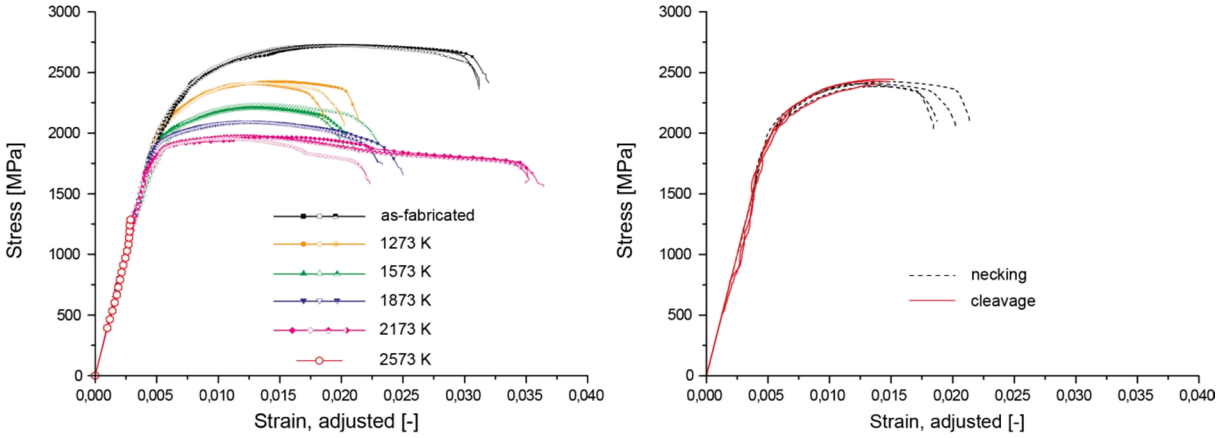

Figure 4: Typical stress-strain curves of tensile tests of potassium doped tungsten wire at different annealing stages (left). Three samples are shown for the as-fabricated case and an annealing temperature of 1273 K, 1573 K and 1873 K. A fourth sample having a much lower fracture strain is shown for annealing temperatures 2173 K. For the sake of clarity only one curve is shown for temperature 2573 K. For an annealing temperature of 1273 K fracture with necking as well as cleavage dominated occurred (right).

Table 2: Ultimate strength of potassium doped tungsten wire with different annealing stages determined in tensile tests.

	W-as	*W-1273K*	*W-1573K*	*W-1873K*	*W-2173K*	*W-2573K*
Ultimate Strength [MPa]	2721±1	2409±6	2220±5	2089±4	1968±4	1274±26

Typical fracture surfaces for each sample type are shown in figure 6. Almost all fibres show necking (reduction in cross-section) and a fibrous, knife edge necking dominated fracture mode. Samples annealed at 1273 K without necking (missing step stress drop at the end of tension test) and annealed at 2573 K show a cleavage dominated fracture.

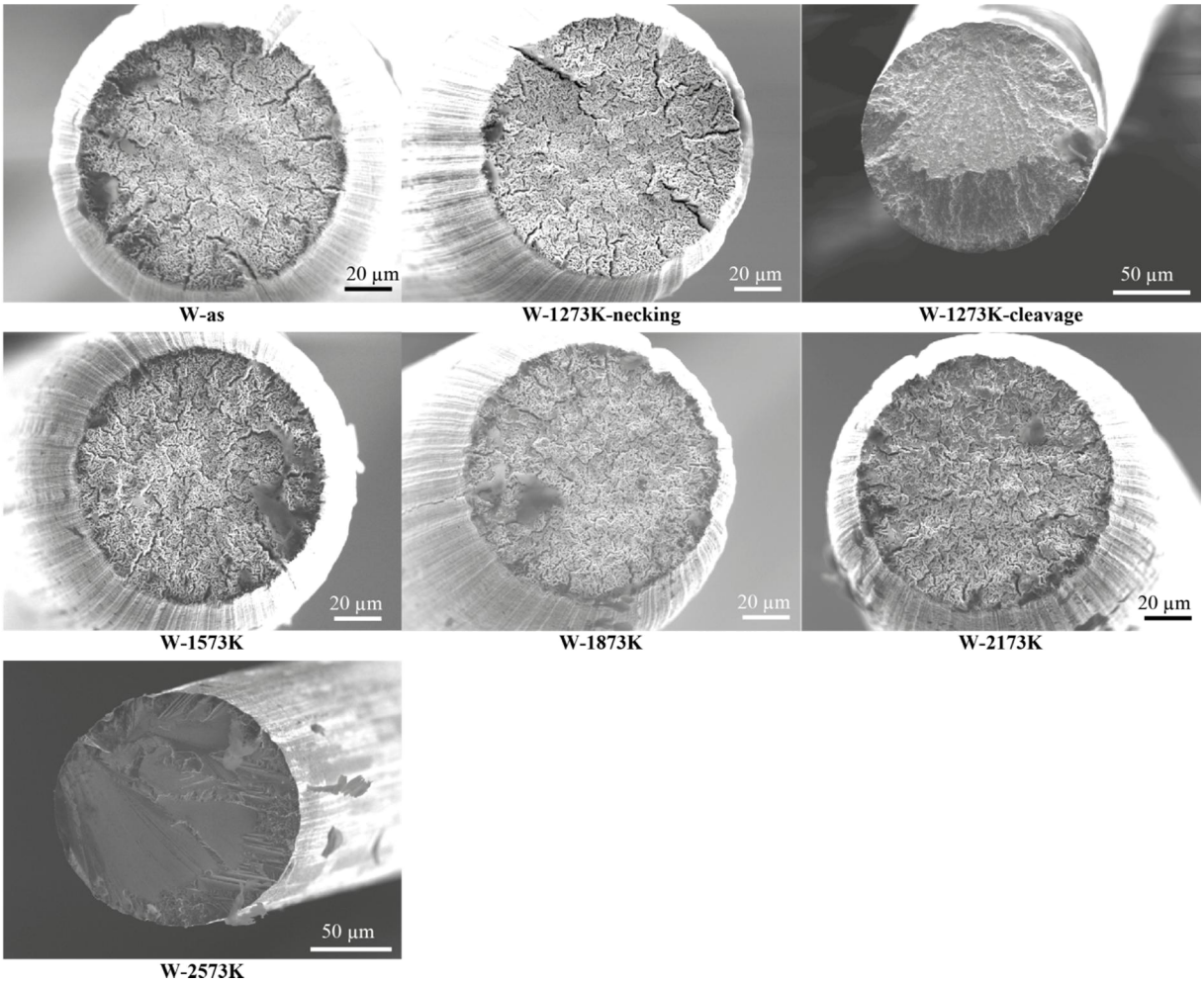

Figure 5: Typical fracture surfaces after tensile tests of potassium doped tungsten wire with different annealing stages.

5. Discussion

Most of the samples with an annealing temperature up to 2173 K show ductile behaviour with significant plastic deformation, a high fracture strain and pronounced necking. The fracture surfaces for these samples are typical for the ductile fracture of tungsten wire [22]. At first longitudinal grain boundaries debond leading to freestanding individual grains. Afterwards these grains neck down in a knife-edge fracture mode leading to a typical fibrous surface. For some samples annealed at 1273 K ductile behaviour without a dedicated necking regime is observed. These samples show a cleavage dominated fracture mode. All samples annealed at 2573 K show pure elastic i.e. brittle behaviour with a cleavage dominated fracture mode.

To understand this behaviour the annealing behaviour and accordingly the recrystallization behaviour are important. Recrystallisation is difficult to tackle in doped tungsten wire as the processes are different compared to pure bulk tungsten [23]. Two distinct stages are identified: at first a relatively uniform coarsening sometimes referred to as primary recrystallization (starting at 1100 K) followed by a rapid growth of large elongated grains referred as secondary recrystallization or extensive grain growth (starting at 2200 K) [24]. The first stage is assigned to polygonisation in which dislocations are organized into low angle boundaries [22] but also at this stage high angle boundary migration is active [25]. At the same time potassium bubble rows are formed at the grain boundaries inhibiting grain boundary migration in the radial direction. Thus the elongated grain structure is retained during grain coarsening until some grains reach the critical size (Hillerts criteria [26]) and show rapid grain growth. Also dragging of bubbles at this temperature can contribute to that effect [25].

As long as a relatively fine and elongated grain structure is preserved the amount of grain boundaries perpendicular to the tension load is small. Even if such boundaries debond they are bridged by adjacent long grains leading to a ductile behaviour. This seems to be the case until secondary grain growth occurs. The embrittlement is correlated with the occurrence of very large grains which facilitate cleavage.

In samples which where annealed at 1273 K, the reasons for the occurrence of cleavage and therefore the missing of necking are not obvious. For the occurrence of the knife edge fracture grain boundary debonding is necessary. The weakening of these boundaries by the formation of the potassium bubbles or by the interaction of longitudinal grain boundaries with these bubbles at increasing annealing temperature might be beneficial. In the as-fabricated stage the dislocations, which are present in a high amount, are able to move to the grain boundaries during loading and might have similar effects. The occurrence of cleavage at 1273 K annealing temperature is probably caused by a complicated interaction between annihilation of dislocations and formation of bubble rows and needs further investigation.

The as-fabricated wire shows a more pronounced strain hardening behaviour which can be attributed to the high density of dislocations due to the drawing process. With ongoing heat treatment the dislocation density decreases due to annihilation and grain coarsening. Therefore the blocking of dislocations is less pronounced and thus the work hardening rate is lower. For the same reason the yield strength decreases with rising annealing temperature. The strength stays around 2000 MPa until the very high annealing temperature where extensive grain growth and brittle behaviour occurs. All samples annealed at 1273 K show the same strength independent of the occurrence of necking. That means that there is no significant difference in the onset of ductile deformation. Leber et. al [22] also reported ductile deformation prior to cleavage fracture.

The strength in the as-fabricated state is slightly smaller than reported previously for pure tungsten wire in similar tests [20]. The strength is influenced very much by the deformation rate and the annealing stages during fabrication [27] and was also reported to be higher in doped W wire in some cases [28]. Different fabrication histories are therefore most probably the cause for the differences in strength. The effect of decreasing work hardening capability with rising temperature is similar in both cases. The gain in fracture strain observed for doped

wires is not reported for pure W wire. This might be attributed to having fewer annealing stages but could also be caused by the faster loss of the elongated grain structure. The fracture modes are similar, knife necking of individual grains in the ductile case and cleavage dominated fracture in the brittle case. In pure W wire the embrittlement was correlated with the loss of elongated grain structure and not necessarily with the occurrence of recrystallization. Samples annealed at 1273K showed clear evidence of recrystallization but also clear ductile behaviour. This was attributed to the preserved elongated grain structure. In samples annealed at 1900 K this elongated structure was lost and the samples showed brittle behaviour. The elongated grain structure was therefore identified to be the key factor deciding whether there is ductile or brittle behaviour. In contrast to that the size of the grains plays an additional role in potassium doped wire. Very large grains provoke brittle fracture even though they are elongated. This size effect was probably not observed in pure W wire as here the grains lose their elongated shape much earlier due to the absences of the grain boundary pinning by potassium.

In summary similar effects seem to be valid in both pure and potassium doped tungsten wire. Annealing and even recrystallization leads at first to a decrease in work hardening capability most probably due to the decrease in dislocation density but not necessarily to embrittlement. Both types show ductile behaviour as long as an elongated fine grain structure is preserved. The main difference is that due to the potassium doping grain boundaries are pinned and this structure is preserved to much higher temperatures compared up to pure tungsten wire.

6. Conclusion and outlook

Tungsten fibre-reinforced composites feature unique properties which could allow their use in highly loaded areas of a future fusion reactor. However to reach this goal further development steps are necessary. The first step will be reached by producing mock-ups and testing them under cyclic high heat load. For this a multilayer approach is necessary addressing all

composite constituents and relevant manufacturing techniques. As an example the role of the tungsten wire used as fibre is discussed as for instance the properties of the wire are not only important for the composite properties but are also constraining the fabrication process.

Potassium doped tungsten wire was investigated by means of tensile tests in the as-fabricated and annealed states in order to investigate its use as fibre reinforcement in W_f/W. The main findings are:

- Potassium doped W wire annealed up to 2173 K shows ductile behaviour.
- Tensile strength stays about 2000 MPa up to annealing temperatures of 2173 K.
- Secondary grain growth at 2573 K leads to embrittlement

Similar to pure tungsten wire embrittlement is correlated with the loss of the fine elongated grain structure. To achieve a better understanding of the correlation between microstructure, i.a. dislocation density, grain size and aspect ratio, and mechanical behaviour a detailed EBSD study could be performed on the samples.

As potassium doped wire does not lose its good mechanical properties and in particular its ductile behaviour up to very high temperature the fabrication temperature of W_f/W composites could be significantly increased. In addition the results are a strong indication that the application temperature of W_f/W might be increased if using doped wire. This has to be proven by investigating the mechanical properties of annealed W_f/W samples and by tensile tests of potassium doped W wire at an elevated temperature.

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agreement No 633053. The views and opinions expressed herein do not necessarily reflect those of the European Commission.

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