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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 conception works in principle has been shown in the 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. We present microstructural as well as mechanical testing investigations. The results show that the use of doped tungsten wire allows increasing the synthesis temperature and can increase 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 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 seems now 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 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) material by the EU Fusion roadmap [13]. In summary the idea of extrinsic toughening in W is principally working and the application as highly loaded divertor element is identified thus the technology readiness level (TRL) 2 is reached (proof-of-principle + application formulated) (explanation of TRL concept in [14]).

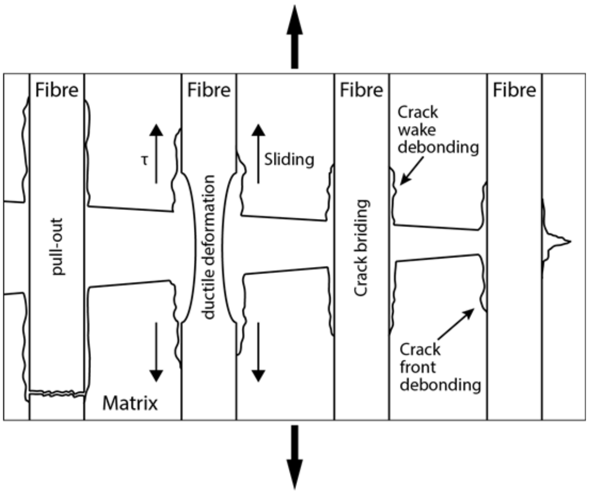


Figure 1: Toughening mechanism in tungsten fibre-reinforced composites.

Candidate materials for DEMO however need to reach TRL 6-8 before considered for design [15]. The first step in this development is to proof 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 or erosion and the behavior under neutron irradiation. TRL 6 will be reached by a prototype demonstration in a relevant environment i.e in wall tiles of existing fusion reactors e.g. on a manipulator or as long time 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 could for example be performed in the ion beam facility GLADIS in Garching, Germany [16,17] 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 i.e. toughness and thus the conceptional proof in one step. The concentration on one main test provides clear constraints for geometry and test methodology. This leads to a distinct structure of the whole project and 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).

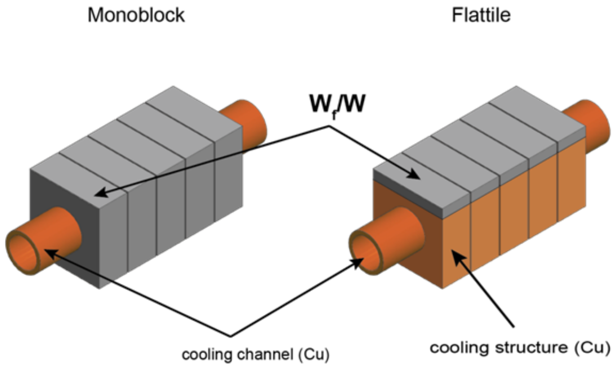


Figure 1: Monoblock and Flat tile mock-up for high heat flux tests

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 interface, this is a multilayer approach.

In this paper we exemplarily discuss the role of the tungsten wire used as fibre and especially the influence of annealing on the 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 at first a short review is given on matrix production techniques.

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 (< 600° C) and a force-less fabrication, and thus the preservation of the interface and fibre integrity as well as fibre topology. In this process the 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, gas flow and 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) specifically designed for the chemical deposition of tungsten for the matrix production in W_f/W was designed and installed at FZJ.

Although CVD processes have the advantage of low production temperature and the absence of mechanical impact PM routes would allow several interesting benefits: it can be faster than CVD, it is easier to implement alloying, e.g. self passivating tungsten [reference] and most important as it is the standard process for tungsten production the knowledge for production and processing is very sophisticated. Nevertheless there are also some drawbacks: it requires a high temperature and high mechanical impact.

PM investigations on single fibre composites have been started in order to understand the interaction between fibre, interface and matrix. Hot isostatic pressing was applied to produce a dense W matrix with and without embedded fibres. First samples at various HIPing temperature up to 1900˚C 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 are 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 a first investigation of this effect.

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. Wire in the as-fabricated and after annealing for 30 minutes at 1273 K and 1900 K were tested. Whereas the as-fabricated and the low heat treated fibres showed ductile behaviour and a strength of more than 2900

MPa and 1900 MPa respectively the high temperature heat treated fibres failed in a brittle manner with a mean strength of approximately 900 MPa, but 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 the 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 (2100-2300K [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 [Zhao2015] 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

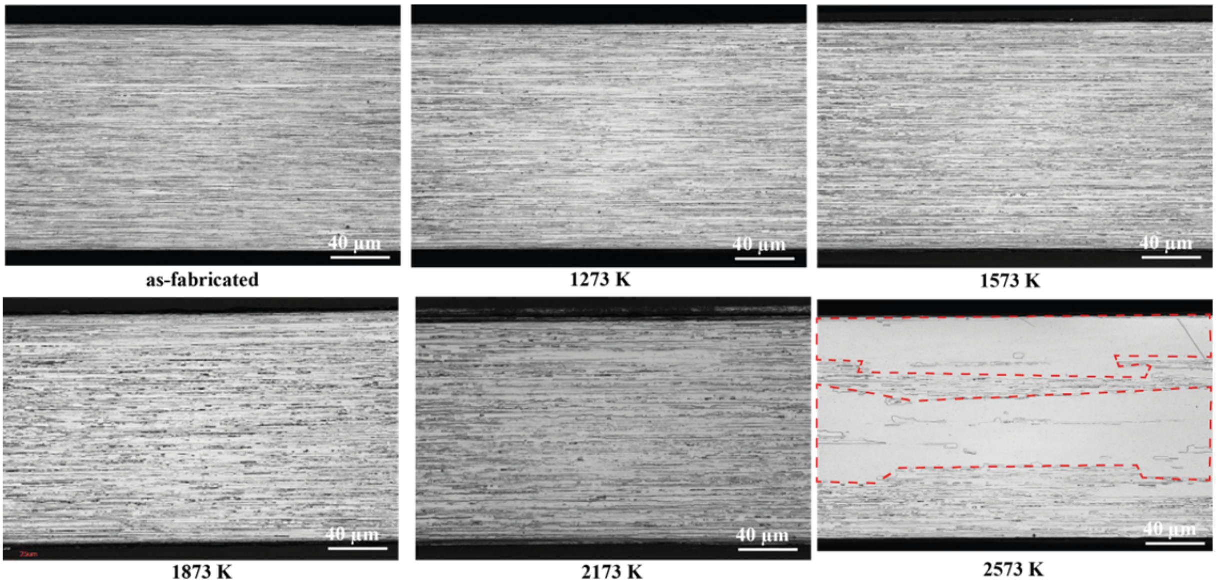


Figure 3 Longitudinal sections (Optical) of potassium doped tungsten wire at different annealing stages. The dashed lines in the 2573 K case indicate large grains after secondary grain growth.

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). Displacement measurements by laser speckle interferometry require 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 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 elastic line 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 beside 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 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 there have been samples at this annealing stage which show lower fracture strain. The yield strength as well as the ultimate strength decrease with rising annealing temperature.

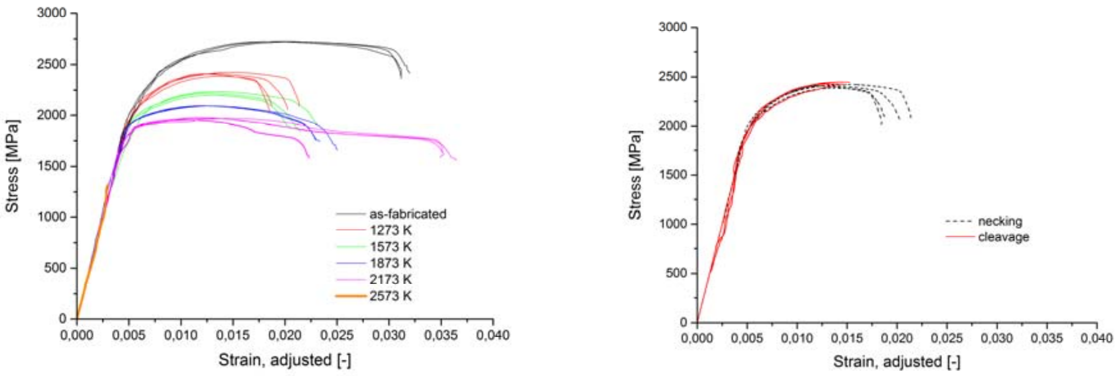
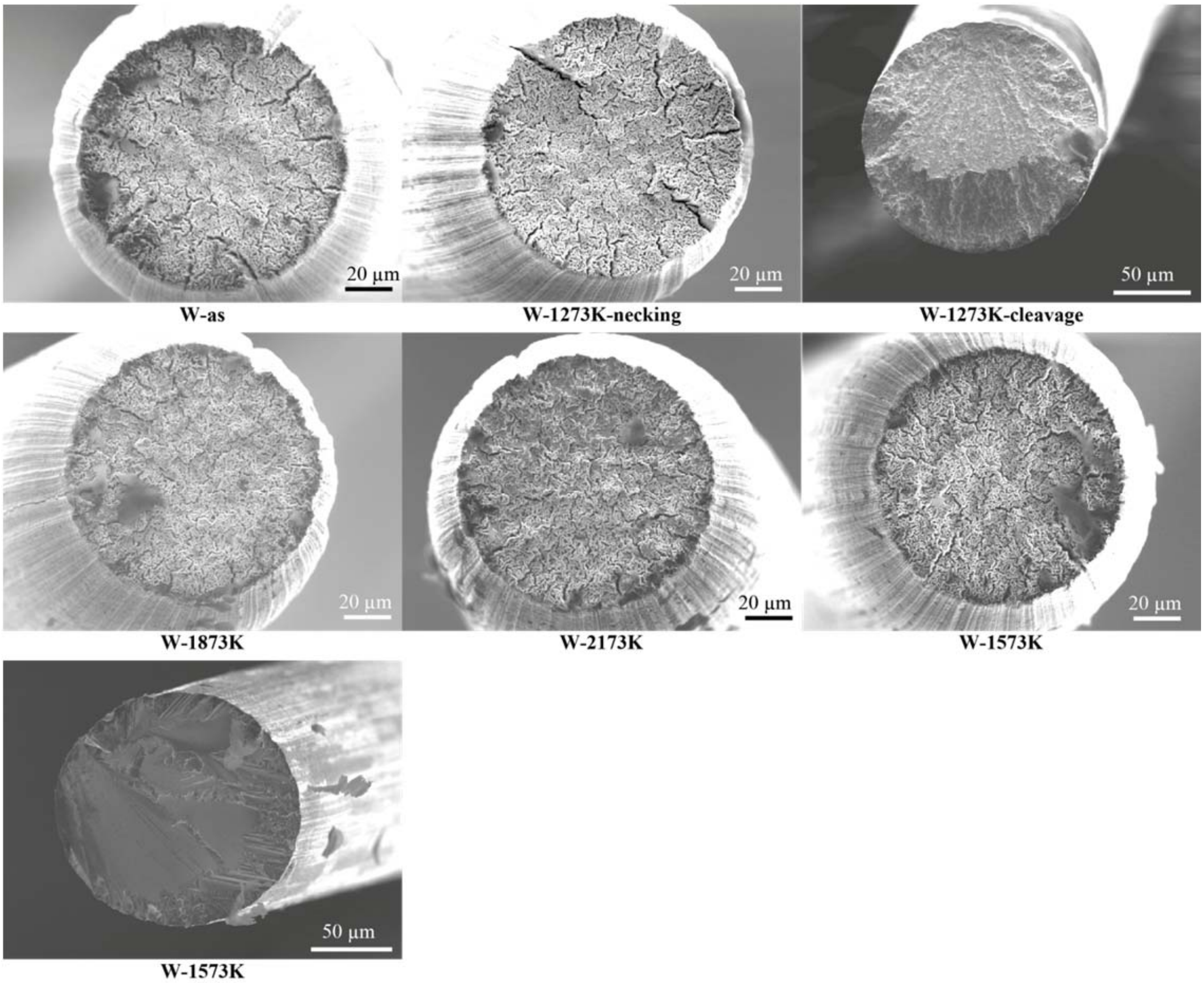


Figure 4: Typical stress-strain curves of tensile tests of potassium doped tungsten wire at different annealing stages (left). Three samples are shown for the as-fabricated case and annealing temperatures of 1573 K, 1873 K and 2573 K. Four samples are shown for annealing temperatures of 1273 K and 2173 K. For the annealing stage 1273K fracture with necking as well as with cleavage occurred (right).

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

	W-as	*W-1273K-D*	*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 knife edge necking dominated fracture mode. However areas showing cleavage are observed for annealing temperature between 1573 and 2173 K. These areas seem to grow with rising annealing temperature Samples annealed at 1273 K without necking (missing step stress drop at the end of tension test) and annealed at 2573 K show full cleavage fracture.



W-as **W-1273K-necking** **W-1273K-cleavage**

W-1873K **W-2173K** **W-1573K**

W-1573K

Figure 6: Typical fracture surfaces after tensile tests of potassium doped tungsten wire with different annealing stages.

5. Discussion

Almost all samples up to 2173 K show ductile behaviour with significant plastic deformation, a high fracture strain and a pronounced necking zone. For some samples annealed at 1273 K ductile behaviour without a dedicated necking regime is observed. Only the samples annealed at 2573 K show pure elastic i.e. brittle behaviour. The embrittlement is correlated with the occurrence of secondary/extensive grain growth and therefore the loss of the elongated grain structure. A reason for that might be that as long as the elongated grain structure is preserved the fraction of grain boundaries being perpendicular to the tensile load is small. Even if such boundaries debond they are bridged by adjacent grains. The final fracture occurs when these bridging grains are necked down leading to the for tungsten wire typical fibrous, knife-edge dominated fracture surface [22].

In pure W wire the embrittlement was also correlated with the loss of elongated grain structure and not necessarily on the occurrence of recrystallization. Recrystallisation is hard to be tackled in doped tungsten wire as the processes are different [23]. Two distinct stages are distinguished: at first a relatively uniform coarsening sometimes referred as primary recrystallization followed by rapid growth of large elongated grains referred as secondary recrystallization or extensive grain growth depending on the author [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 the grain boundary migration in 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].

Reasons for the occurrence of cleavage and therefore the missing of necking in samples which where annealed at 1273 K are not obvious. As for the occurace of the knife edge fracture grain boundary debondig 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. Therefore cleavage might be more beneficial at this very low annealing temperature.

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 curing. Therefore the blocking of dislocations is less pronounced and thus the work hardening rate is lower. Similar effects are reported for pure W wire. For the same reason the yield strength decreases with rising annealing temperature.

The strength in the as-fabricated state is slightly smaller than reported previously for pure tungsten wire in similar tests [20]. 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 the 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 with an increasing fracture of cleavage with rising annealing temperature. In summary similar effects seems to be valid/active in both pure and potassium doped tungsten wire. Annealing 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 its use in highly loaded areas of a future fusion reactor. However to reach this goal some development steps are necessary. The first step will be reached by producing mock-ups and test them under cyclic high heat load. For this a multilayer approach is necessary addressing all composite constituents and relevant manufacturing techniques. Exemplarily the role of the tungsten wire used as fibre is discussed. The properties of the wire are important for the production techniques as well as for the composite properties.

Potassium doped tungsten wire was investigated by means of tensile tests in the as-fabricated and in 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 show ductile behaviour.
- Tensile strength keeps about 2000 MPa up to annealing temperatures of 2173K.
- 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. Potassium doped wire do not lose their good mechanical properties and in particular their ductile behaviour due to temperature induced effects during PM relevant temperatures. 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 elevated temperature.

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