Tungsten Fibre-Reinforced Composites for Advanced Plasma Facing Components

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Tungsten Fibre-Reinforced Composites for Advanced Plasma Facing Components

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Abstract

The European Fusion Roadmap foreseen water cooled PFCs in a first DEMO design in order to provide enough margin for the cooling capacity and to only moderately extrapolate the technology developed and tested for ITER. In order to make best use of the water cooling concept Cu and CuCrZr are foreseen as heat sink whereas as armour tungsten (W) based materials will be used. Combining both materials in a high heat flux component asks for an increase of their operational range towards higher temperature in case of Cu/CuCrZr and lower temperatures for W. A remedy for both issues brittleness of W and degrading strength of CuCrZr could be the use of W fibres in W and Cu based composites. Fibre preforms could be manufactured with industrially viable textile techniques. Flat textiles with a combination of 150/50 μm W wires have been chosen for layered deposition of W\textsubscript{f}/W-samples and tubular multi-layered braiding with W wire thickness of 50 μm were produced as a preform for W\textsubscript{f}/Cu tubes. Cu melt infiltration was performed together with an industrial partner in resulted in sample tubes without any blowholes. Property estimation by mean field homogenisation predicts strongly enhanced strength of the W\textsubscript{f}/CuCrZr composite compared to its pure CuCrZr counterpart. Tungsten fibre reinforced tungsten (W\textsubscript{f}/W) composites show very high toughness (about 10 times larger than pure W) and damage tolerance even at room temperature. Cyclic load tests reveal that the extrinsic toughening mechanisms counteracting the crack growth are active and stable. FEM simulations of the W\textsubscript{f}/W composite suggest the the influence of fibre debonding, which is an integral part of
the toughening mechanisms, and reduced thermal conductivity of the fibre due to the necessary interlayers do not strongly influence the thermal properties of future components.

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1 Introduction

In view of the severe working conditions for plasma facing components (PFCs) in future power producing fusion devices the development of advanced materials is mandatory. The materials not only have to withstand high steady state power loads but also thermal cycling and shocks. Moreover, the change of thermo-mechanical properties by damage, activation and transmutation through fusion neutrons has to be taken into account when designing PFCs and selecting the adequate armour and structural materials. Within the research along the European Fusion Roadmap, water cooled PFCs are foreseen in a first DEMO design in order to provide enough margin for the cooling capacity and to only moderately extrapolate the technology developed and tested for ITER [1]. In order to make best use of the water cooling concept copper (Cu) based alloys (as for example CuCrZr) are foreseen as heat sink whereas as armour tungsten (W) based materials will be used. Combining both materials in a high heat flux component bears the difficulty that their optimum operating temperatures do not overlap: W should be operated above 800°C in order to be in a ductile state to avoid brittle cracking under cyclic load, whereas CuCrZr should be operated below 300°C to provide enough mechanical strength. A remedy for both issues – brittleness of W and degrading strength of CuCrZr – could be the use of W fibres in W and Cu based composites. This contribution will present the investigations on both composite materials, tungsten fibre reinforced tungsten
(W/W) and copper (Wf/Cu). Section 2 will briefly report on the tungsten fibres and the latest developments of the W fibre preform production. Sections 3 and 4 will highlight the latest results for both composites Wf/Cu and Wf/W and finally section 5 will summarize the paper and point to investigations to be performed in the future, heading towards a plasma facing component to be built out of the two composites.

2 W wires and preforms

The W fibres used are drawn potassium doped tungsten wires as used in the lighting industry. They are characterized by very high strength (> 2500 MPa), ductility already at room temperature and recrystallization and embrittlement only above 1900 °C [2] in contrast to conventional bulk tungsten being brittle at room temperature and recrystallizing at 1300 °C. These are ideal properties for reinforcement of Cu and W. In Wf/Cu ductile W fibres strongly increase the strength of the composite at elevated temperatures combined with flexural flexibility enabling easy handling and high-curvature winding in the manufacture process (see below) [3]. In Wf/W the ductile deformation allows the dissipation of substantial amount of energy and the high strength is important for the bridging effect if a crack has formed in the matrix providing increased fracture toughness [4]. In Wf/W composites the fibres must be coated by a temperature stable material in order to avoid interdiffusion between W matrix and W fibres allowing for the intended extrinsic toughening. Typically, the fibres are coated in advance by magnetron sputtering with an interface layer with a thickness of 1 μm (see [5] for details). Whereas in earlier experiments an Er₂O₃ interface layer was used, future investigations will concentrate on Y₂O₃ or TiN as an interlayer because of their lower activation under neutron bombardment [6]. For the production of the Wf/Cu composite no further processing of the W fibres is necessary because W and Cu do not form compounds or alloys and Cu easily wets W [7] (see also Sec. 3).

In accordance with the final goal of building a complete, actively cooled plasma facing component and in order to provide the proof of concept, industrially viable textile techniques were developed with external partners. Since Wf/Cu is primarily foreseen as heat sink materials the preforms were chosen to consist of a tubular braiding in order to provide already a preform as close as possible to the shape of a cooling tube. The top part of Fig. 1 shows the photograph of a tubular W braiding using 50 μm thick W fibres with 23 layers and a braiding angle of 77° as deduced
form the fourier transform of the photograph (bottom part of Fig. 1). The braiding was performed at the Institut für Textil- und Verfahrenstechnik (ITV, Denkendorf, Germany) with the optimization criteria of high braiding angle to achieve a high strength in circumferential direction and a high fibre fraction in the final composite [8].

Fig. 1. Photograph of a braided W tube using 50 μm W fibres (top) and fourier transform of the photo for evaluation of the braiding angle (bottom)

For the W_f/W composites a flat preform was chosen in the style of a flat tile concept. Until recently the preform was produced manually by stacking frames with W fibres of 150 μm thickness with a horizontal and vertical distance of 100 μm (see for example [9]). Similarly as in the case of the preforms for W_f/Cu the application of industrially viable textile techniques for W_f/W preforms was evaluated. In this case the fabrics were produced with shuttle loom weaving machine at the Institut für Textiltechnik (ITA, RWTH Aachen, Aachen, Germany). The fabric was woven with a plain weave pattern using 60 parallel tungsten warp wires. The with a diameter of 150 μm and a distance of 100–150 μm. For the weft wires a diameter of 70 μm were chosen with distance in the range of 30-50 mm. With this weaving set up it was possible to produce a 15 mm wide pure tungsten fabric with a thickness of 230–250 μm after compressing/bending the warp fibres [9]. Differently to the production of W_f/Cu samples where the copper is infiltrated in the complete preform, the flat fabrics are stacked in the chemical infiltration device just before or during the deposition of the W-matrix (see below).
3 Wf/Cu Composite

Since W and Cu do not form compounds or alloys and Cu easily wets W, the Wf/Cu composites can be readily produced by Cu infiltration into the W preform. (Please note: Most of the experimental investigations presented here use pure Cu for infiltration, but the techniques should be easily transferable to CuCrZr). Wf/Cu composites have been already used in the sixties as an ideal model system for investigating fiber-reinforced metal matrix composites and reviews on the properties of Wf/Cu composites can be found in [3] and references therein. One feature of Wf/Cu composite materials is the fact that the mechanical and physical properties as for example yield stress and thermal conductivity can be calculated by mean field homogenisation over a wide range of fibre volume fractions due to the excellent interfacial adhesion. Figure 2 presents the result of such calculations using 2 × 23 braiding layers and a braiding angle of ±77° for different volume fractions of W fibres. It shows the stress strain behaviour in circumferential direction and the thermal conductivity of the tubes in radial direction (both at 300°). As it can be seen from the figure, the strength of the tube is strongly increased already at a moderate fibre fraction at the cost of a very moderate decrease in thermal conductivity. The calculations are performed at a maximum temperature of 300° because only up to this value the thermo-mechanical data for the W-fibres are available. It is however expected that the operational range of the Wf/CuCrZr composite could be extended to even higher temperatures.

![Fig. 2. Calculated stress strain behaviour at 300° in circumferential direction of a Wf/CuCrZr) tube with 46 braiding layers and a braiding angle of ±77° for different volume fraction of W fibres. The numbers in the diagram give the thermal conductivity of the tubes in radial direction.](image)

Figure 3 shows cross sections (left) and a micrograph of a first W fibre reinforced copper tube obtained in collaboration with the company Louis Renner GmbH (Dachau, Germany). For this first trial still W-fibres with a diameter of 150 μm were used,
which only allowed about 8 braiding layers and a lower braiding angle compared to the finally adopted one described above.

Fig. 3. Metallographic cross sections of a tungsten fibre reinforced copper (Wf/Cu) tube.

Already with this rather low number of fibre layers are rather homogeneous distribution of the reinforcing fibres could be reached. As can be seen from the micrograph, a perfect filling of the interstices with copper could be achieved leaving no disturbing cavities.

4 Wf/W Composites

The very high melting point and high temperature strength both for fibre and matrix of the Wf/W composite do not allow for conventional composite production routes (see examples in [10]). Furthermore it is 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 tungsten-hexafluoride is reduced by hydrogen in a heterogeneous surface reaction and thus solid tungsten is formed. First large samples of the Wf/W composites have been received by layered W chemical vapour deposition (CVD) [11] and W chemical infiltration (CVI) [2,12] performed at Archer Technicoat Ltd (High Wycombe, UK). The sample prepared for the bending tests described below is produced by CVI on a fibrous preform consisting of tungsten fibres with a diameter of 150 μm [12]) still using Er2O3 with a thickness of 1 μm as an interface layer. The sample was built by a two step infiltration process by which densities larger than 95% and a fibre volume fraction of about 0.3 could be achieved. Fig. 4 shows a schematic view of the sample geometry for a three point bending test (top) of Wf/W. A photograph of the sample during the bending test is shown at the
bottom, right and metallographic cross section of the sample after the bending test with marked failure characteristics of the W fibres is shown at the bottom, left.

As discussed above and described in more details elsewhere [4], key mechanisms for the toughening in W_f/W are debonding of the interface between fibre and matrix, crack bridging by intact fibres, ductile deformation of fibres, fibre pull-out, crack deflection and meandering. In order to check these toughening effects, cyclic bending tests have been performed using an in-situ surface observation by a scanning electron microscope (SEM). The testing conditions were similar to the ones described in detail in [12]. The test sample is cut according ASTM E399 [13] (see Fig. 4) to the dimensions of 2.3 x 2.6 x 25 mm$^3$ containing 10 fibre layers a 9 fibres. A careful surface preparation is required to allow the observation of the crack propagation and potential stopping. Therefore a combination of a mechanical and a mechanical-chemical procedure is used (details in [14]). The sample is prepared in such a way that the first layer of fibres is cut in half. This allows the observation of the interaction of the crack with the first fibre layer in the surface as shown in Fig. 4 (bottom right), where crack deflection and bridging is clearly visible. The sample is notched to concentrate the stress at the notch tip and to create a well-defined crack initiation point. As a sharper crack lowers the load needed for crack initiation [15], a three-step notching procedure with decreasing notch radii as described in [12] is used. The sample is loaded in a displacement-controlled manner while the
crack tip (respectively the notch tip) is observed in a fast scanning mode to capture any changes at the crack tip immediately. In parallel the force measurement unit was monitored for crack propagation indicated by small load drops. Figure 5 shows stress-strain curves of a cyclic 3-point bending test performed with the W_f/W sample described above. After an initial loading of 450 N, the displacement was reduced and 5 load cycles at 150 N, 250 N, 350 N and 450 N each, were performed. Finally the displacement was increased up to full fracture.

![Stress-strain curves](image)

**Fig. 5. Stress-strain curves of a cyclic 3-point bending test of the W_f/W sample shown in Fig. 4. After an initial loading of 450 N, the displacement was reduced and 5 load cycles at 150 N, 250 N, 350 N and 450 N were performed. Finally the displacement was increased up to full fracture.**

As can be seen from the small dips in the load curve, several cracks were already initiated before reaching the load of 450 N. Already at 150 N the first crack appeared – at this point a complete failure of the bulk material would have happened. On contrary, the cyclic loads up to 150 N and finally up to 450 N all show a similar slope and the displacement necessary for a certain load stabilizes during the cycles. This behaviour indicates that the mechanisms counteracting the crack growth are active and stable. Only when increasing the load further (above the initial value of 450 N) new cracks and multiple fibre crack events appear (as deduced from the acoustic perception) and finally lead to a the full fracture of the sample. Several of the characteristic toughening mechanism described above could be recognized during the SEM monitoring of the sample during the bending test and from the fracture surface after failure. In addition to the evolution of the cracks observed at the prepared side surface (see above) the fracture surface reveals the fully ductile fracture and the pull-out of several fibres (Fig. 4 bottom, left). The brittle failure of the fibres at the lower end of the samples could possibly explained by the sudden failure at the end of the loading curve.

Since the plastic deformation of the fibres is by far the largest contribution to the energy consumption during cracking [4], the fracture toughness can be estimated
from the number of ductile fibres involved (20, fibres volume fraction 0.15). A conservative estimate for the fracture toughness due to plastic deformation yields according to equations 2 and 10 of [4] $\Delta K_{pl} \approx 82 \text{ MPa m}^3$, which is about a factor of 10 larger than the value for the not reinforced W matrix material (see also [9]).

In parallel to experimental investigations, finite element simulations were initiated to interpret and to extrapolate the thermomechanical behaviour of W-fibre reinforced components. The simulations were performed using the same boundary conditions as in [16] where the deep cracking of ITER mock-ups at 20 MW/m$^2$ was investigated. In [9] it was suggest that using a fibre fraction of only 13% effectively could suppress the growth of these cracks. For this model system further thermal modelling was performed in order to quantify the effect of fibre debonding of fibres and of a reduced thermal conductivity at the fibre interface (simulated by reduced thermal conductivity of the fibres).

Table 1 gives the temperature response of the surface and the fibres for different debonding length. Only for very large debonding lengths (2 mm debonding is already very long as observed in bending tests) of all fibres slightly increased maximum temperatures can be observed. Similarly, also only a reduction of the thermal conductivity in the range of 50% of the whole fibre volume would yield an appreciable temperature increase. However since only the thin interlayers will have a reduced thermal conductivity their effect on the maximum surface temperature should be negligible. In summary, the FEM simulations suggest that the fibres
should have only a very moderate influence on the thermal properties of PFCs using W/W as armour.

5 Summary and Outlook

Within the research along the European Fusion Roadmap, water cooled PFCs are foreseen in a first DEMO design in order to provide enough margin for the cooling capacity and to only moderately extrapolate the technology developed and tested for ITER [1]. In order to make best use of the water cooling concept copper based alloys (as for example CuCrZr) are foreseen as heat sink whereas as armour tungsten based materials will be used. Combining both materials in a high heat flux component bears the difficulty that their optimum operating temperatures do not overlap: W should be operated above 800°C in order to be in a ductile state to avoid brittle cracking under cyclic load, whereas CuCrZr should be operated below 300°C to provide enough mechanical strength. A remedy for both issues, brittleness of W and degrading strength of CuCrZr, could be the use of W fibres in W and Cu based composites. The development of these W-fibre reinforced W and Cu components comprises investigations on W-fibres, development of wire preforms, selection and deposition of viable thermally stable interlayers (in the case of Wf/W) and finally manufacturing of the compounds. Potassium doped W wires behave ductile in a large temperature range starting at least at room temperature and reaching up to 1900 °C. Preforms could be manufactured with industrially viable textile techniques. Flat textiles with a combination of 150/70 μm W wires have been chosen for layered deposition of Wf/W-samples and tubular multi-layered braiding with W wire thickness of 50 μm were produced as a preform for Wf/Cu tubes. The Cu melt infiltration was performed together with an industrial partner in resulted in sample tubes without any blowholes. Property estimation by mean field homogenisation predicts strongly enhanced strength of the Wf/CuCrZr composite compared to its pure CuCrZr counterpart. Tungsten fibre reinforced tungsten (Wf/W) composites produced by chemical vapour deposition or infiltration using WF₆ as processing gas allow the production of samples with a density up to 95 %. Mechanical tests show very high toughness (about 10 times larger than pure W) and damage tolerance even at room temperature. Cyclic load tests performed in the frame of this work reveal that the extrinsic toughening mechanisms counteracting the crack growth are active and stable. FEM simulations of the Wf/W composite suggest the the influence of fibre debonding, which is an integral part of the toughening mechanisms,
and reduced thermal conductivity of the fibre due to the necessary interlayers do not strongly influence the thermal properties of future components. Future activities will aim at optimization of the compounds and their production routes. In the case of Wf/Cu a centrifugal casting process could be used to readily produce the reinforced cooling tube. Further, the use of short W-fibres is envisaged for the Wf/Cu heat sink in a flat tile design. In order to optimize the CVI/CVD process for Wf/W a strong collaboration with the Forschungszentrum Jülich, which recently commissioned the CVD device ‘WILMA’ is ongoing [9]. In parallel to these activities, the influence of the fibres in the W armour on the hydrogen retention and the interaction with helium have to be assessed in order to satisfy the integrated approach necessary for the material’s application in a future fusion reactor [17]. The final goal of the activities is to provide actively cooled mock-ups of plasma facing components to test their integrated behaviour under high thermal load (for example in the high heat flux test facility GLADIS [18]) and under high power plasma exposure using the divertor manipulator of ASDEX Upgrade [19].

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