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Improved pseudo-ductile behavior of powder metallurgical tungsten short fiber-reinforced tungsten (W_f/W)

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A B S T R A C T

For the first wall of a fusion reactor unique challenges on materials in extreme environments require advanced features in areas ranging from mechanical strength to thermal properties. The main challenges include wall lifetime, erosion, fuel management and overall safety. For the lifetime of the wall material, considerations of thermal fatigue due to transient heat loading are crucial as severe mechanical and thermal loads during operation are expected.

Tungsten (W) is the main candidate material for the first wall of a fusion reactor as it is resilient against erosion, has the highest melting point of any metal and shows rather benign transmutation behavior under neutron irradiation. However, Tungsten has an issue related to intrinsic brittleness as well as operational embrittlement. To overcome this, a W-fiber enhanced W-composite material (W_f/W) incorporating extrinsic toughening mechanisms has been developed. Recently progress has been made in the powder metallurgical routes towards fully dense multi short-fiber W_f/W. For reasonable performance with respect to mechanical properties and hydrogen retention a fully dense pseudo-ductile W_f/W with is crucial. The properties of the used fibres are crucial. For the composite mechanisms to work a level of strength of the used fibres is required. In this contribution the change in ductility of the fibres is studied.

In this contribution it is shown that excluding or minimising the impact of carbon impurities during the sintering process can significantly improve the mechanical properties of the fibres. New test results on the behaviour of PM W_f/W with and without a diffusion barrier during the sintering show a clear benefit as the fibres can retain ductility. Not the grain growth during sintering but the carbon present during sintering is clearly identified as determining the mechanical properties of the fibres.

1. Introduction

Tungsten (W) is currently the main candidate material for the first wall and in particular for highly loaded components of the divertor of a future fusion reactor as it is resilient against erosion, has the highest melting point, shows rather benign behavior under neutron irradiation, and low tritium retention. Extensive work has been done to qualify current materials with respect to these issues for ITER [1–3]. For the next step devices, e.g. DEMO, or a future fusion power plant the limits on power exhaust, availability, lifetime and not least on fuel management are quite more stringent. Extensive studies and materials programs [4–6] have already been performed hence it is assumed that the boundary conditions [7] to be fulfilled for the materials are in many cases above the technical feasibility limits as they are set today [1,2]. Efforts to establish new advanced plasma-facing material-options are moving forward [2,8] focussing on crack resilient materials with low activation, minimal tritium uptake, long lifetime and low erosion. Many advanced materials base their improved properties on the use of a composite approach. One concept is based on the incorporation of fibres, energy dissipating mechanisms, like ductile deformation of fibres, fibre pull-out, and crack bridging and deflection are facilitated [9–11]. An issue not tackled in this contribution is the formation of radioactive and highly volatile W-oxide (WO_3) compounds during accidental air ingress. To suppress the release of W-oxides W-based self-passivating alloys can be incorporated into the composite approach [12–14]. In this contribution the focus lies on the improvement of the powder-

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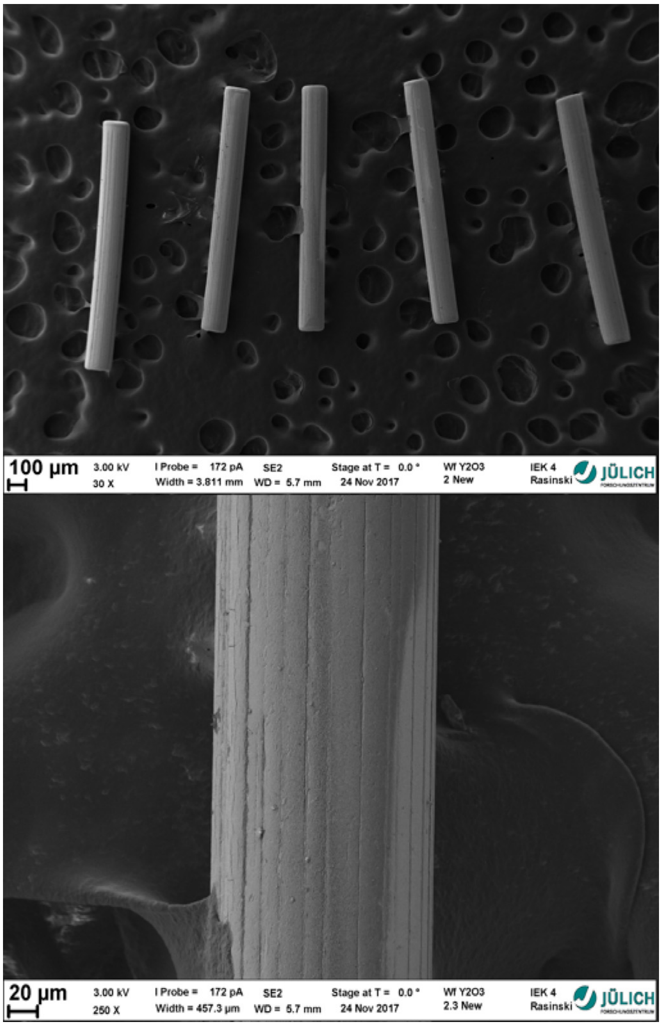

Fig. 1. Short W-fibres coated by 2.5 μ Yttria interface.

metallurgical (PM) production W_f/W as plasma-facing-material (PFM). The influence of the die material used is has been explored. It was shown that the ductile deformation [15] as well as the high strength [16] of the tungsten wire have a significant influence on the overall properties of W_f/W. Mueller et al. [17] show that one of the crucial aspects of the production process of any tungsten fibre composite is to control the amount of impurities e.g. carbon during the consolidation process. Carbon is of particular interested with respect to W and its mechanical properties [18].

2. Tungsten-fibre reinforced tungsten

To overcome the brittleness issues when using W, a W fibre enhanced W composite material (W_f/W), incorporating extrinsic toughening mechanisms can be used. The short fibres used in this PM version of W_f/W are shown in Fig. 1. Yttria is used as the interface material in order to allow the energy dissipation mechanisms to become active. Yttria is an ideal candidate as the interface material for the W_f/W composite due to its several advanced properties: good thermal and chemical stability, high mechanical strength and hardness [8,19]. Various methods of building and constructing W_f/W composites, either via Chemical Vapor Deposition (CVD) [20,21] or powder metallurgical processes [22,23] are available. Based on the work presented here and previous work [15,16,22,24,25], the basic proof of principle for CVD & PM W_f/W has been achieved. One of the crucial issues is to maintain as much of the properties of the constituents even after exposing the material to the production cycle and the fusion environment. This allows for better extrinsic toughening and pseudo-ductile behaviour. Here mainly the weak interface and the strength of the fibre is important. For PM = W_f/W the details of the underlying mechanisms are described in [26]. As the material should dissipate as much energy as possible it is hoped to at least start with the inclusion of ductile fibres. Even if the fibres loose their ductility the pull-out of fibres and the crack deflection can still to deliver some pseudo-ductility for a viable material option.

It can be expected that when using doped W-wires they will e.g. retain their ductility even at elevated temperatures (above 1500 K) [15] and all mechanisms necessary for pseudo-ductility will enabled [8,24,25]. Properties of the fibres might be degraded by various circumstances e.g. by impurities during fabrication, high-temperatures or neutron irradiation during operation [27,28]. In [17] it was found that all fibre samples categorised as brittle exhibit an increased C content compared to the fibres categorised as ductile.? All brittle samples have C content of 0: 0586 wt: % compared to the ductile samples with lower than 0.0013 wt %.

In the following we will describe that one aspect of the production needs to be controlled with particular care to minimise the degradation of the material properties of the fibres.

2.1. W_f/W – Material Production

For powder-metallurgical production of W_f/W as already described in [8,25] the homogenous introduction of powder between the fibres is required for good material properties, hence short fibres are used in contrast to e.g. woven preforms or parallel long fibres as used in the CVD process route. Based on results from [8] pressureless sintering of W_f/W was unsuccessful, additional external pressure during sintering of W_f/W is required to get a dense and crack-free sample. Field Assisted Sintering Technology (FAST) [29] provides such additional compaction during sintering. Details on the consolidation incl. HIP (Hot-isostatic-Pressing) as well as material properties can be found in [8,25].

Potassium doped W-fibres with 150 μm diameter and 2.4 mm length (OSRAM), together with pure W-powders (OSRAM) (average particle size 5 μm) were used as raw materials. The FAST process gives rise to pressure and high temperatures temperature on the interface and can thus cause a thin interfaces to dissipate [30–32]. Here 2.5 μm thick yttria is applied for a viable interface similar to the work given in [25]. The fibres and powders were mixed homogeneously before sintering, in order to produce a W_f/W sample with a random fibre distribution and orientation. A density of ~ 94% was achieved after applying the sintering process at 2173 K (4 min) and 60 MPa (heating rate 200 K/min) [8,25]. In all cases a fibre-volume-fraction of 30% was used.

Samples have been prepared to establish if and how pseudo-ductility can be achieved in the case of a randomly distributed short fibre W_f/W and also which role the interface and impurity content may play. Here the main parameter that was changed in comparison to [8,25] is the addition of a tungsten foil to prevent as much as possible the interaction of the die material with the samples that are being sintered.

In Fig. 3 a sketch of the two different FAST procedures used is given. In one case the powder and die are separated by a graphite foil while in the other case a thin tungsten foil is used. Based on FAST samples with 20 and 40 mm diameter and a height of 5 mm were produced as shown in Fig. 2.

2.2. W_f/W – Material structure

Fig. 4, shows a representative cut though PM-W_f/W after consolidation as described above. The material is dense and a thin interface of yttria remains around the randomly distributed fibres. The

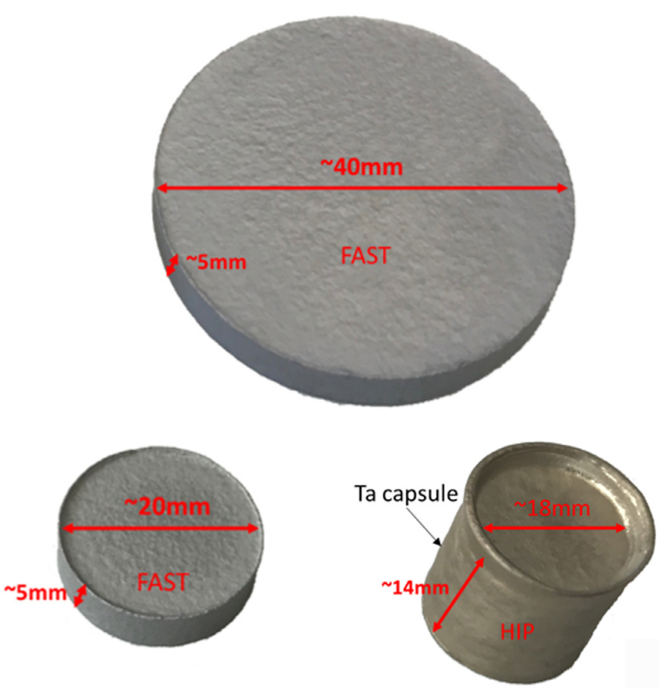

Fig. 2. Large 40 mm FAST W_f/W Sample displayed next to a small 20 mm FAST and HIP samples [25].

microstructure is identical for both consolidation procedures.

For both consolidation procedures shown the microstructure of the fibre was studied also. Fig. 5 shows for both cases similar grain size and structure after FAST. Originally the fibres have a very fine filamented grain structure as given in [33] with (0.3–1.5) µm in the directions perpendicular to the wire axis and roughly 10–90 μ along the axis. After consolidation of the W_f/W samples the average grain size along the original drawing direction of the fibre the is determined to be 2.322, and 2.502 μm respectively while perpendicular to the drawing direction the grains have an extent of around 6.5 μm . The grain size measurement is based on the Lineal Intercept Procedure (ASTM E 112) using SEM images. The average grain size is determined by the number of times a test line cuts across, or is tangent to, grain boundaries.

It is clear that the sintering can have consequences for the grain structure as seen in the micrographs. The main message here is however that for both cases, with and without W-foil, a similar microstructure modification has taken place without a significant difference between the two procedures.

3. W_f/W – Pseudo-ductility

In previous studies, a series of tests have been performed without quantitative values and published in [8,25]. Fracture surfaces were analysed to establish if the desired mechanisms can be observed. In addition to that, small (27 × 2 × 3 mm) KLST type three point bending test samples were produced and a pre-notch introduced. Utilising an universal testing machine (TIRAtest 2820, TIRA GmbH) three point bending tests were performed. With an optical camera system, the accurate sample displacement was measured during the test, so that quantitative load displacement curves were taken. Typical curves are shown in Fig. 6. Here for the first time also quantitative numbers are given.

Even after crack initiation is observed an increased load can still be handled. This is a clear indication of pseudo-ductility in this simple model-system.

In Fig. 6 one major differences for the Wf/W produced with W-foil is the shape of the curve. The fall off in load is far smoother and more steps are visible indicating a different fracture behaviour of both samples.

In Figs. 7 and 8 thus the typical fracture surfaces are depicted to elaborate on the difference in production. Fig. 7 shows the clear brittle fracture of one of the constituent short fibres with a clear intra-granular cleavage fracture.

For the Fig. 8 a ductile behaviour of the fibre is observed allowing for more energy dissipation also indicative from the stress strain curve. The fracture shows fibre necking with knife edges and a sharp fracture surface. This behaviour is in line with the results by Mueller et al. [17]. It was found that carbon when present during the annealing process of

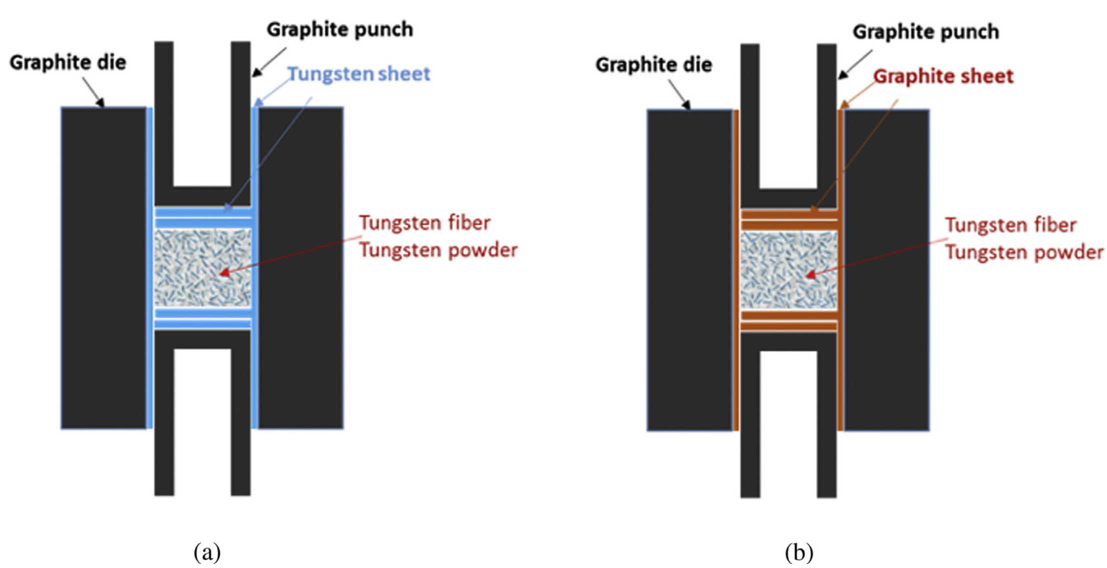

Fig. 3. Two different procedure for consolidation have been used to test the influence of the die material onto the final material properties (a) Tungsten foil as diffusion barrier, (b) graphite sheets for lubrication.

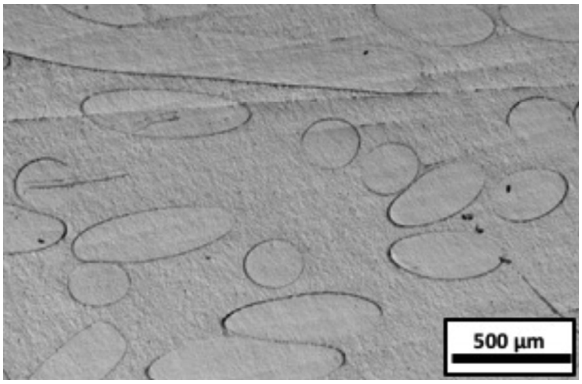

Fig. 4. Overview Microstructure after sintering, with-out tungsten foil.

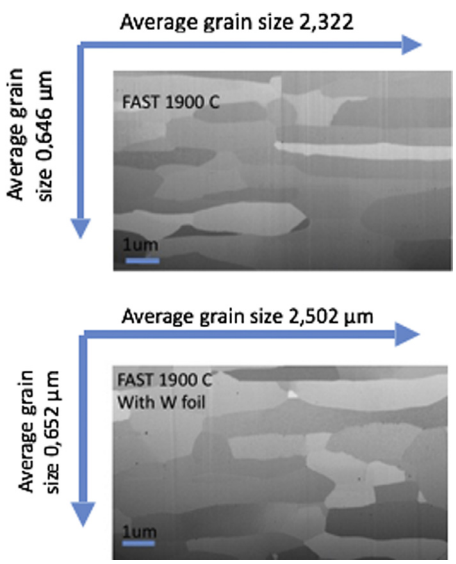

Fig. 5. Grainstucture and grain size of the fibres after sintering.

fibres can embrittle them already when diffusing in small quantities into the fibre. This is in line with findings from literature [18] where a link was established between the interstitial impurities such as carbon and the low temperature tensile properties of tungsten. In the given work above all presented test have been performed at room temperature to represent the worst case scenario for tungsten. Minimisation of

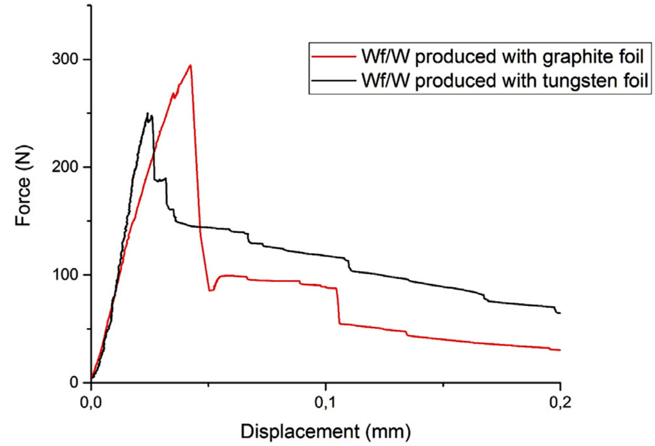

Fig. 6. (red) 3pt bending test results of W_f/W produce via FAST with W-Foil, (black) 3pt bending test results of W_f/W produce via FAST with C-Foil. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

the impact of the production process by excluding carbon is one step towards optimisation.

4. Conclusion and outlook

Based on the presented tests for PM-W_f/W with W-foil it can be said that the manufacturing path for W_f/W has been further improved. The presented approach utilising a W-foil mitigates the embrittlement of the constituent fibres during FAST processing.

Based on these results it can be seen that improved pseudo-ductile behaviour can be achieved for PM-W_f/W. It is planned to utilise this new route in developing prototype components for application in existing fusion devices. In order to also establish material performance under irradiation PM - W_f/W samples (cf. Fig. 2) are earmarked for neutron irradiation starting in 2018.

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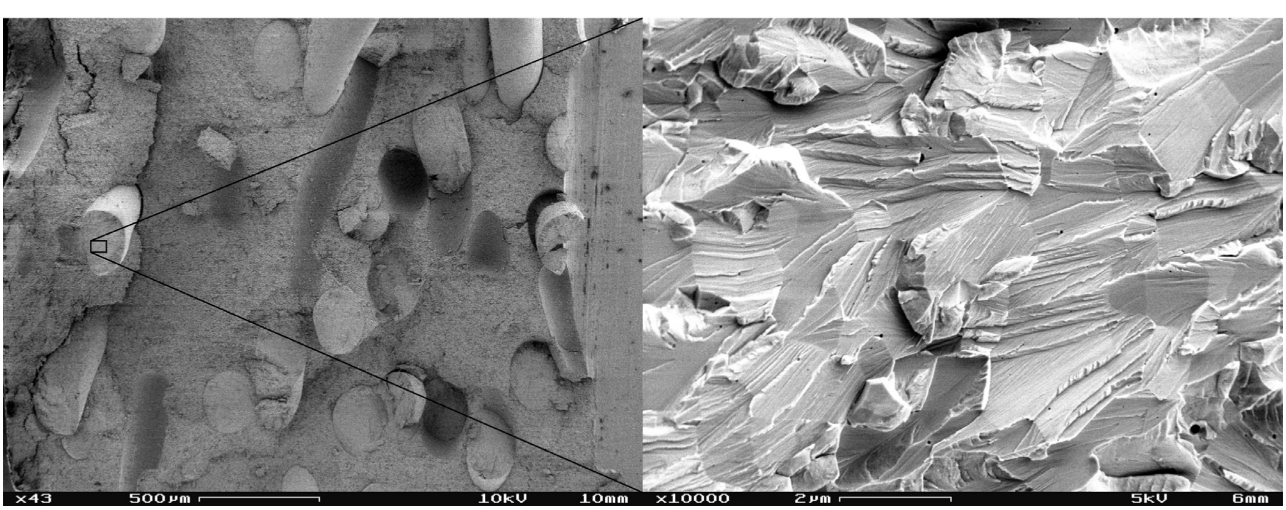

Fig. 7. Fracture Surfaces of a W_f/W samples consolidated with C-foil .

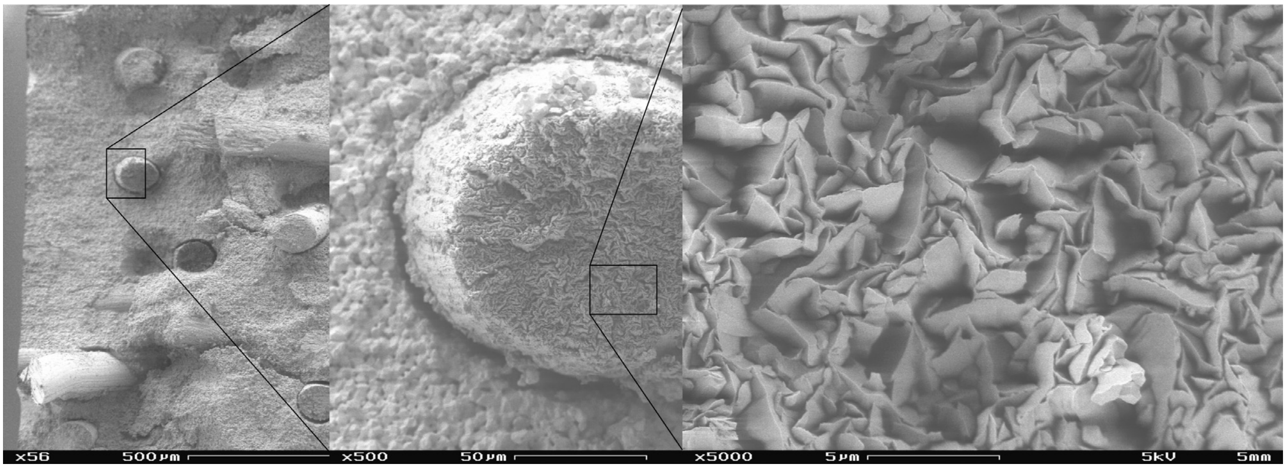

Fig. 8. Fracture Surfaces of a W_f/W samples consolidated with W-foil.

expressed herein do not necessarily reflect those of the European Commission.

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