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Tungsten Composite Materials for Fusion First Wall applications

J.W. Coenen[1,*], J.Riesch[2], H.Gietl[2,3], B.Jasper[1], Y.Mao[1], T.Hoeschen[1], A.Terra[1], S.Sistla[4] and Ch Linsmeier[1], Ch. Broeckmann[4], R.Neu[2,3]
[1]*Forschungszentrum Jülich GmbH, Institut für Energie- und Klimaforschung - Plasmaphysik, Partner of the Trilateral Euregio Cluster (TEC), 52425 Jülich, Germany* [2] *Max-Planck-Institut für Plasmaphysik, Garching Germany* [3]*Technische Universität München, Boltzmannstrasse 15, 85748 Garching, Germany,* [4]*Lehrstuhl für Werkstoffanwendungen im Maschinenbau, RWTH Aachen, Aachen Germany*
* **j.w.coenen@fz-juelich.de**

Abstract
Material issues pose a significant challenge for future fusion reactors like DEMO, hence the development of advanced materials is essential for sophisticated energy systems like a future fusion reactor. When using materials in a fusion environment a highly integrated approach is required. Damage resilience, oxidation resistance during accidental air ingress as well as power exhaust are driving issues when deciding for new materials. Neutron induced effects e.g. transmutation adding to embrittlement are crucial to material performance. Here advanced materials e.g. composites allow the step towards a fusion reactor. W_f/W contributes to advanced material strength and crack resilience even with a brittle matrix. Tungsten fiber-reinforced tungsten composites (W_f/W) utilize extrinsic toughening mechanisms and therefore overcome the intrinsic brittleness of tungsten at low temperature and its sensitivity to operational embrittlement. W_f/W has been successfully produced and tested during the last years and the focus is now put on the technological realization for the use in plasma facing components of fusion devices. In this contribution, we present a way to utilize W_f/W composites for divertor applications by two main fabrication routes based on the chemical vapor deposition (CVD) of tungsten and powder metallurgy. A new device dedicated to the chemical deposition of W enhances significantly the available machine time for processing and optimization. Modeling shows that good deposition results are achievable by the use of a convectional flow and a directed temperature profile in an infiltration process. Recent developments in the area of multi-fiber powder-metallurgical W_f/W mark a possible path towards a component based on standard tungsten production technologies. Field Assisted Sintering Technology (FAST) is used as production route to achieve 94 % dense materials. Initial mechanical tests and micro-structural analyses show potential for pseudo-ductile behavior of materials with a reasonable (30 %) fiber fraction. In the as-fabricated condition samples showed step-wise cracking while the material is still able to bear rising load, the typical pseudo-ductile behavior of a composite. Yttria is used as the interface material in order to allow the energy dissipation mechanisms required.
The use of W_f/W could broaden the operation temperature window of tungsten significantly and mitigate problems of deep cracking occurring typically in cyclic high heat flux loading. Furthermore, W_f/W can potentially bridge the operational gap between the upper bound for strength of copper $\sim$ 620 K and the lower bound of DBTT for tungsten $\sim$ 850 K.

1 Introduction

Tungsten (W) is currently the main candidate material for the first wall of a 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 theses issues for ITER, especially for W as first wall and divertor material [1, 2]. For the next step devices, e.g. DEMO, or a future fusion reactor the limits on power exhaust, availability, lifetime and not least on fuel management are much more stringent. Extensive studies and materials programs [3, 4, 5, 6, 7, 8] have already been performed hence it is assumed that the boundary conditions [9] to be fulfilled for the materials are in many cases above the technical feasibility limits as they are set today. W_f/W as an advanced material has been successfully produced and tested during the last years and the focus is now put on the technological realization for the use in plasma facing components of fusion devices as well as the further improvement and development of production methods. In this contribution we present a way to utilize W_f/W composites for divertor applications by a fabrication route based on the both chemical vapor deposition or infiltration (CVD, CVI) [10, 11] of tungsten and powder metallurgy (PM) [12, 13].

2 Tungsten-fiber reinforced Tungsten (W_f/W)

Efforts to establish new advanced plasma-facing material-options are moving forward [2] focussing on crack resilient materials with low activation, minimal tritium uptake, long lifetime and low erosion. Brittle behavior is one of the limiting factors when operating any W based plasma facing components (PFCs) in a tokamak [14]. The operational gap ($\sim$ 620 K-850 K) between materials for cooling structures e.g. Cu, and the plasma-facing materials needs to be bridged [15, 2]. Cu needs to be strengthened and W ductilized. The use of

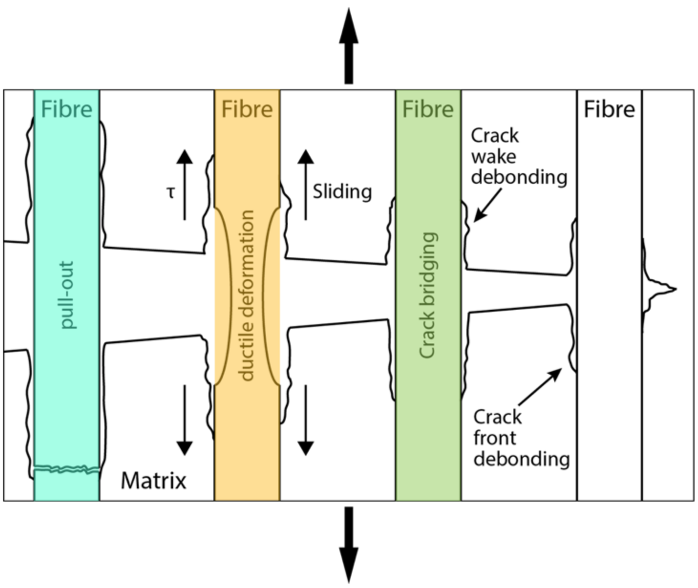


Figure 1: Energy dissipation mechanisms typically considered in W_f/W and other fiber-reinforced composites (based on [11])

tungsten fiber reinforced tungsten (W_f/W) could broaden the operation temperature window of tungsten significantly and mitigate problems of deep cracking occurring typically in

cyclic high heat flux loading. This is particularly crucial when considering material degradation from neutron-induced transmutation and embrittlement. Here W_f/W can mitigate these issues by utilizing extrinsic toughening mechanisms and therefore overcoming the intrinsic and neutron induced brittleness. An additional difficulty when using W in a fusion reactor is the formation of radioactive and highly volatile tungsten oxide (WO_3) compounds during accidental air ingress. In order to suppress the release of W-oxides tungsten-based self-passivating alloys can be incorporated into the composite approach [16, 17, 18, 19, 20].
Extrinsic toughening for W_f/W is achieved similar to other brittle composites by incorporating an interface between fiber and matrix allowing for additional energy dissipation without relying on intrinsic material properties such as ductility. With the incorporation of fiber, energy dissipating mechanisms, like ductile deformation of fiber, fiber pull out, and crack bridging and deflection are facilitated [21, 22, 23]. Figure 1 shows the typical mechanisms as discussed above.
A W_f/W composite is composed of commercially available drawn tungsten fibers, coated with an interface layer and integrated into a W matrix. For testing single fiber samples are used (Fig. 2).

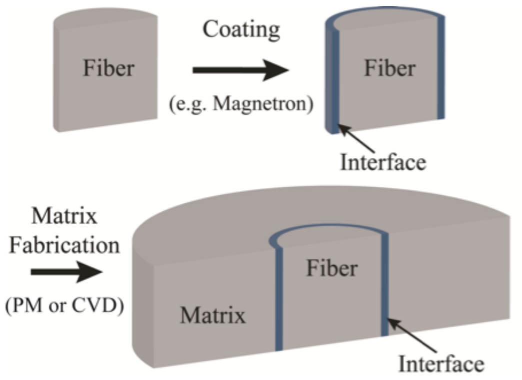
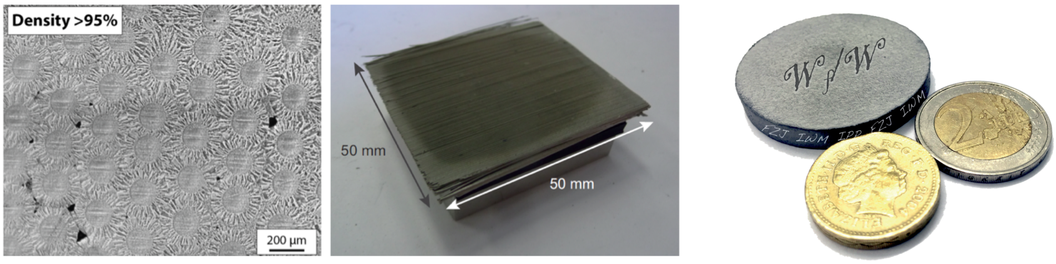


Figure 2: W_f/Wconsists of tungsten fibers, coated with an interface (e.g. Er2O3, Y2O3) and the W matrix.

For both fabrication routes, CVD/CVI [10, 11] or PM[12, 13] varying geometries for the introduction of fibers can be envisioned.

2.1 Production Routes

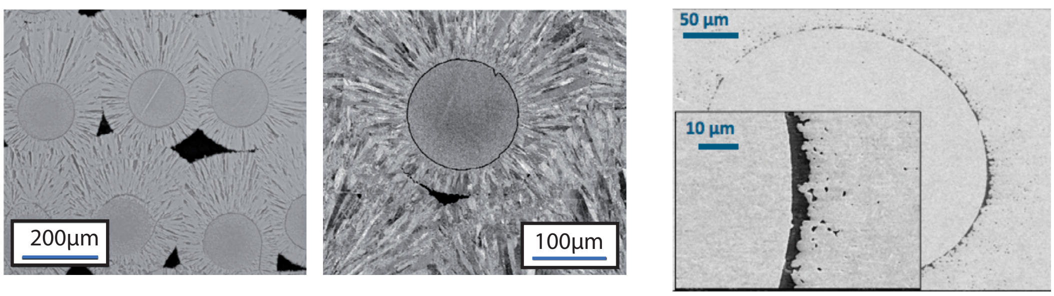
For the CVD route, the furthest developed method is a concept based on a layered deposition approach allowing the production of flat tiles in the required geometry. One fiber layer after the other is positioned and ingrown into the W-matrix until the final sample size is reached.
First large-scale W_f/W samples have been produced utilizing a layered deposition procedure (Fig. 3 (m)). Here the tile is produced by successive chemical vapor deposition (CVD) of W on equally distanced, unidirectionally orientated layers of tungsten wire (d=150 μm) (see also [24, 25, 11, 10]). A multi step process is utilized. To form the composite a fiber layer is placed on a heated surface where WF_6 and H_2 react and form a W deposit forming a closed layer. After reaching the required thickness the wires are cut and a second fiber layer is placed on top and also covered in CVD-W. This procedure is repeated until the desired sample thickness is reached. A vertical distance of the fiber layers slightly above 100μ m is achieved with a similar distances between the fibers in the layer. The sample size is hence determined by the number of layers and the original size of the fiber structure.



(a) (b)

Figure 3: W_f/W multi-fiber samples with 30% volume fraction - l: CVI W_f/W m: CVD W_f/W r: SPS W_f/W

The resulting fiber volume fraction is up to 30%. Samples 50 by 50 mm containing 10 fiber layers have been produced (Fig. 3 (m)).
For the PM route development is progressing towards the use of pressure assisted sintering methods like Field Assisted Sintering (FAST) and Hot Isostactic Pressing (HIP). PM production of W_f/W has started recently to achieved multi-fiber composites.
In Figure 3 (r) an as-produced PM-W_f/W sample is shown. Based on FAST a sample with 40mm diameter and a height of 5 mm was produced. Potassium doped W-fibers with 150 μm diameter and 1.5 mm length (OSRAM), together with pure tungsten powders (OSRAM) (average particle size 5 μm) were used as raw materials. The fibers and powders were mixed homogeneously before sintering, in order to produce a W_f/W sample with a random fiber distribution and orientation. A density of $\sim$ 94% was achieved after applying the sintering process at 2173 K (4 min) and 60MPa (heating rate 200 K/min). In addition to the large samples, samples with 20mm diameter for mechanical testing were produced based on the same parameters, but with varying composition. Two kinds of tungsten powders have been used: Pure tungsten powder (OSRAM) (average particle size 5 μm) and so called smart W-alloy powders (W-12Cr-0.5Y, provided by CEIT). The fiber size is also chosen differently in this case (240 μm x 2.4 mm). In all cases a fiber-volume-fraction of 30% was used. In case of the PM Route the samples have been prepared with the main aim to establish if and how pseudo-ductility can be investigated in the case of a randomly distributed short fiber W_f/W.
To provide a stable interface to the matrix the fibers are typically coated by magnetron sputtering with an interface layer (e.g. Y2O3, Er2O3) with a thickness of 1-3 μm (see [25, 12, 26, 27, 28]) prior to the matrix incorporation.



(a) (b)

Figure 4: Interfaces on W-fiber in W_f/W after consolidation - CVD-Layerwise (l) PM-FAST (r)

Figure 4 (l), shows W_f/W after consolidation via CVD and PM as described above. The impact of the FAST process in contrast to the CVD process can be clearly established. For the CVD layer deposition almost no forces act on the interface hence the 1μm Erbia interface is completely intact. After FAST the interface (start thiuckness 2.5 μm is now far thinner and shows the indentation marks of the surrounding powder. Further optimization may be required.
Apart from the maturity of the different production routes, distinct differences between CVD and PM tungsten can be pointed out. While for the CVD/CVI method a fiber preform is utilized, one can only achieve reasonable results with a short-fiber - powder mix on the PM route. There are different benefits for each route, where textile techniques can be used to optimize the tungsten wire positioning speed of preform production for the CVD route, alloying as seen above is a benefit of the powder metallurgical route. In both cases, K-doped fibers are used to optimize the impact of temperature induced recrystallization [11], minimizing the potential influence of fiber-embrittlement on component performance. For the CVD W_f/W tensile, bending and Charpy [29] tests already show enhanced performance of the material and preliminary bending test for the PM W_f/W show promising first results.

3 A new Divertor Component

In the brevity of this contribution mainly a brief summary on the production of W_f/W is reported, when trying to improve the performance of the divertor however not only the plasma facing material or the amour are important but also the cooling structure and potential joints in the component hence typically several material concepts need to be combined [2, 30]. A component [2, 30, 31] could comprise of tungsten fiber-reinforced tungsten (W_f/W) [29, 11, 13, 11, 12], smart W-alloy as the matrix material [32, 19, 18, 17] a copper based advanced- cooling tube (e.g. tungsten reinforced copper, W_f/Cu)[33] and integrated permeation barrier layers (e.g. Yttria) [34, 35].

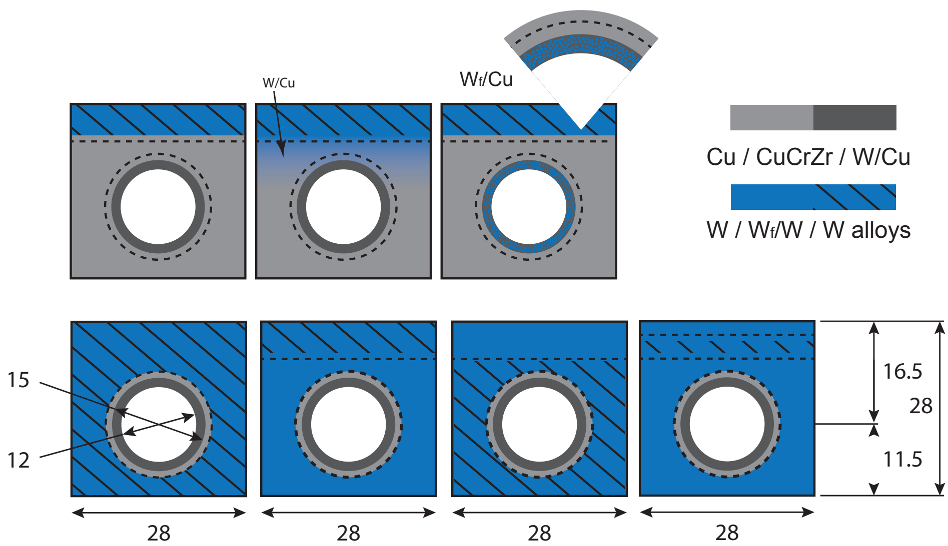


Figure 5: Component design, incorporating W_f/W and W/Cu solutions at various points in the structure, based on [36, 29]

Figure 5. shows only a small variety of potential options that could potentially be used based on the conventional ITER-like divertor design only. The top row assumes a copper

cooling structure and a flat tile of tungsten as armour material. The copper tube can be strengthened via introduction of fibers and the mechanical stresses on the copper structure attenuated by the introduction of a graded transition between Cu and W. [37, 31, 4, 33].
It is essential that the exhaust capability of an advanced component is similar to conventional designs and does in addition show resilience against e.g. embrittlement, failure due to thermal stresses and cyclic loading. We hence propose to utilize the W_f/W composite approach together W-alloying concepts to maximize the potential of W-based-PFCs on top of the advanced cooling options. The lifetime influenced by erosion, creep, thermal fatigue, and embrittlement, needs to be compatible to the requirements from steady state operation. This means that erosion determined by the top layer needs to be close to pure tungsten. Potentially various options introducing the composite need to be considered. Here thermal stress analyses will provide hints for potential applications [3].

4 Conclusion and Outlook

For W_f/W as a new material class several further development steps are planned to enhance availability and performance. Tungsten fiber-reinforced composites feature unique properties which could allow their use in the divertor areas of a future fusion reactor.
Based on initial tests for PM-W_f/W it can be stated that a potential development path for enhancement of tungsten has been opened in addition to the established W_f/W production via CVD. The multi-fiber approach allows now the quick prototyping and testing of new material combinations, fibers, interfaces and alloys.
Especially for the CVD route a dedicated device for the chemical deposition of W enhances significantly the available machine time for processing and optimization. For both options - CVD and PM -, interface optimization needs to be undertaken.
W_f/W on its own can however not solve the issues of heat-exhaust in the divertor of a future fusion reactor. Here also the improvement of the typically used copper cooling structure needs to be considered. Results on W/Cu new materials are reported elsewhere [37, 31, 4, 33]. In combination both can be used to develop a new divertor component. Here rigorous testing and qualification is required with respect to heat-exhaust, thermal fatigue, cyclic loading and plasma wall interaction.
The next steps include further dedicated mechanical tests as well as initial plasma exposures and high heat-flux performance tests. Neutron irradiation will be a crucial undertaking for the final qualification of the material class for fusion. Mock-ups based on the ITER typical design can be realized by the implementation of W_f/W-flat tiles or mono-block like approaches.
It is planned to have prototype components available within 5 years for application in existing fusion devices. In order to also establish material performance under irradiation PM - W_f/W samples (cf. fig. 3) are earmarked for irradiation in a nuclear reactor starting in 2017.
Applications in industrial fields with high temperature demands are considered in parallel to the fusion application.

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References

[1] Philipps, V. *Journal of Nuclear Materials*, **415** (2011), 1, 2–9.

[2] Coenen, J. et al. *Physica Scripta*, **2016** (2016), T167.

[3] Li, M. and You, J.-H. *Nuclear Materials and Energy*, (2016), –. In Press.

[4] You, J. et al. *Nuclear Materials and Energy*, (2016), –.

[5] Stork, D. et al. *Journal of Nuclear Materials*, **455** (2014), 1-3, 277–291.

[6] Rieth, M. et al. *Journal of Nuclear Materials*, **442** (2013), 1-3 SUPPL.1, 173–180.

[7] Reiser, J. and Rieth, M. *Fusion Engineering and Design*, **87** (2012), 5 - 6, 718–721. ISFNT-10.

[8] Stork et al, D. Assessment of the EU R&D programme on DEMO structural and high-heat flux materials, final report of the materials assessment group,. Technical Report EFDA(12)52/7.2, EFDA (December 2012).

[9] Bachmann, C. et al. *Fusion Engineering and Design*, **98-99** (2015), 14231426.

[10] Riesch, J. et al. *Physica Scripta*, **2014** (2014), T159, 014031.

[11] Riesch, J. et al. *Physica Scripta*, **2016** (2016), T167, 014006.

[12] Jasper et al, B. *Material Science Forum, Trans Tech Publications*, **825-826** (2015), 125–133.

[13] Jasper, B. et al. *Nuclear Materials and Energy*, (2016).

[14] Linke, J. et al. *Nuclear Fusion*, **51** (2011), 7, 073017.

[15] Zinkle, S. and Ghoniem, N. *Fusion Engineering and Design*, **5152** (2000), 0, 55–71.

[16] Calvo, A. et al. *Nuclear Materials and Energy*, (2016).

[17] Koch, F. and Bolt, H. volume T128, pages 100–105 (2007).

[18] Wegener et al, T. In PFMC-2015 (2015). Poster at this conferencxe.

[19] Wegener, T. . et al. *Nuclear Materials and Energy*, (2016). ICFRM Conference 2015.

[20] Wegener et al, T. (2016). Talk, Soft2016 (O2B.2).

[21] Launey, M. and Ritchie, R. b. *Advanced Materials*, **21** (2009), 20, 2103–2110.

[22] Czel, G. and Wisnom, M. *Composites Part A: Applied Science and Manufacturing*, **52** (2013), 0, 23–30.

[23] Shimoda, K. et al. *Composites Science and Technology*, **68** (2008), 1, 98–105.

[24] Riesch, J. et al. *IOP Conference Series: Materials Science and Engineering*, **139** (2016), 012043.

[25] Jasper, B. et al. *Nuclear Materials and Energy*, (2016).

[26] Du, J.; You, J.-H. and Hoeschen, T. *Journal of Materials Science*, **47** (2012), 11, 4706–4715.

[27] Du, J. et al. A feasibility study of tungsten-fiber-reinforced tungsten composites with engineered interfaces. Ph.D. thesis, PhD Thesis (2011).

[28] Du, J. et al. *Composites Science and Technology*, **70** (2010), 10, 1482–1489.

[29] Riesch, J. et al. *Nuclear Materials and Energy*, (2016), –.

[30] Neu, R. et al. *Fusion Engineering and Design*, (2016), –.

[31] You, J.-H. (2016). Poster , SOFT 2016 (P1.116).

[32] Litnovsky, A. et al. *PSI 2016*, (2016).

[33] von Mueller, A. (2016). Poster , SOFT 2016 (P1.104).

[34] Engels et al, J. (2016). Poster Soft 2016(P3.180).

[35] Causey, R.; Karnesky, R. and Marchi, C. S. In Konings, R. J., editor, Comprehensive Nuclear Materials, pages 511–549. Elsevier, Oxford (2012). ISBN 978-0-08-056033-5.

[36] W., C. J. et al. *Submitted to Nuclear Materials and Energie*, (2016). Presented at PSI2016.

[37] You, J.-H. et al. *Journal of Nuclear Materials*, **438** (2013), 13, 1–6.