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Behavior of tungsten fiber-reinforced tungsten based on single fiber push-out study

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ABSTRACT

To overcome the intrinsic brittleness of tungsten (W), a tungsten fiber-reinforced tungsten-composite material (W_f/W) is under development. The composite addresses the brittleness of W by extrinsic toughening through the introduction of energy dissipation mechanisms. These mechanisms allow the reduction of stress peaks and thus improve the materials resistance against crack growth. They do not rely on the intrinsic material properties such as ductility. By utilizing powder metallurgy (PM) one could benefit from available industrialized approaches for composite production and alloying routes. In this contribution the PM method of hot isostatic pressing (HIP) is used to produce W_f/W samples containing W fibers coated with an Er_2O_3 interface. Analysis of the matrix material demonstrates a dense tungsten bulk, a deformed fiber and a deformed, but still intact interface layer. Metallographic analysis reveals indentations of powder particles in the interface, forming a complex 3D structure. Special emphasis is placed on push-out tests of single fiber HIP samples, where a load is applied via a small indenter on the fiber, to test the debonding and frictional properties of the Er_2O_3 interface region enabling the energy dissipation mechanisms. Together with the obtained experimental results, an axisymmetric finite element model is discussed and compared to existing work. In the HIP W_f/W composites the matrix adhesion is rather large and can dominate the push-out behavior. This is in contrast to the previously tested CVD produced samples.

1. Introduction

When considering a future fusion power plant multiple intertwined issues need to be evaluated. Some of the main challenges are linked to the materials exposed to the plasma and their lifetime considerations. An important challenge are effects caused by thermal fatigue by transient heat loading, as typically 10^9 (30 Hz) thermal transients (ELMs) during one full power year of operation are to be expected. Erosion of the first wall and the divertor will in addition require a significant armor thickness or short exchange intervals, while high-power transients need strong mitigation efficiency to prevent damage of the plasmafacing components [1]. Therefore materials with advanced properties in areas ranging from mechanical strength to thermal properties are required [2]. Tungsten (W) is currently 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 behavior under neutron irradiation [3], as well as low tritium retention. But extrinsic toughening mechanisms are required, since the high thermal and neutron loads during operation lead to an embrittlement of the tungsten materials. Composite approaches allow the combination of beneficial properties and would therefore be ideal to enhance material parameters and mitigate damage effects. Already today smart materials, fiber composites and alloys which adapt to the operational scenario are possible [2].

1.1. Tungsten fiber-reinforced tungsten W_f/W

One major disadvantage of W is its brittleness below the ductile-to-brittle transition temperature (DBTT), which ranges from

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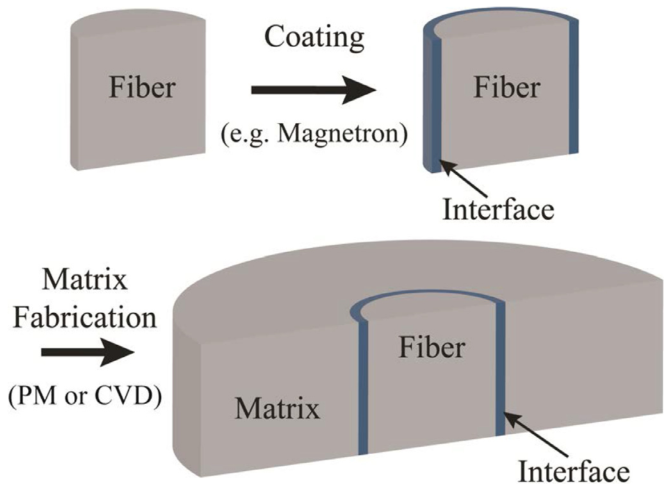

Fig. 1. Basic structure of a single-fiber W_f/W containing the interface, a fiber and the matrix.

400–700 K [4], depending on the preparation history of the material. To circumvent the issue of brittleness if using W, different composite approaches are investigated. Tungsten fiber-reinforced tungsten (W_f/W), as one example, utilizes fiber-reinforcement and comprises of a pure W matrix and an interface layer around a W fiber [5,6]. The basic idea is to introduce extrinsic toughening mechanisms which allow for crack energy dissipation. This is the only way to enhance the toughness in intrinsic brittle materials and commonly used to toughen ceramics [7]. A basic strategy to achieve pseudo-ductility is the incorporation of fibers and a weak interface into a matrix, which needs extensive development and validation [8]. In case of W, a W fiber-reinforced W composite material (W_f/W) can be used to overcome brittleness issues (Fig. 1). The incorporated fibers enable extrinsic mechanisms, thus stress peaks at crack tips can be reduced and further crack growth prevented.

Other options include composite laminates made of commercially available raw materials [9,10]. The link between W_f/W and laminates is the similarity of fibers and foils. Both show a special microstructure of highly deformed and elongated grains, hence showing high strength and ductility even at room temperature [11,12,13]. Accordingly, even in the brittle regime, below the DBTT, these materials allow for a certain tolerance towards cracking and damage in general. Even if a crack has been initiated inside the composite material the extrinsic energy dissipation mechanisms allow further load to be applied towards the component. In comparison, conventional tungsten would fail immediately. After reaching the ultimate strength the mechanisms also lead to a controlled failure rather than a catastrophic one in the brittle case. Assuming embrittlement by high-temperature operation and neutrons however it can be expected that ductility will be lost.

W_f/W in contrast to W-laminates hence has the benefit of utilizing extrinsic mechanisms and still working in the embrittled case [14]. First W_f/W samples have been produced, showing extrinsic toughening mechanisms similar to those of ceramic materials [15,16]. They exhibit the necessary mechanisms to mitigate effects of operational embrittlement due to neutrons and high operational temperatures.

A component based on W_f/W can be produced with both chemical vapor deposition (CVD) [6] and a powder metallurgical path through hot isostatic pressing [17] (Fig. 3). Crucial in both cases is the interface between fiber and matrix, since both are made out of tungsten. The interface is typically a thin layer (Fig. 1) with targeted properties: weak enough to enable the toughening mechanism, as strong as possible to maximize the dissipated energy [5]. This is an idea based on enabling pseudo-ductile fracture in inherently brittle material, e.g. SiC ceramics [18].

2. Single fiber W_f/W characterisation

The main experimental characterization method of this work is the push-out test. It provides a quantitative insight into important parameters of the fiber-interface-matrix system, e.g. the interfacial shear strength (τ_d). The load-displacement curve, obtained during the test, displays several important features, e.g. the load that is needed to initially break or debond the interface or the maximum load which the sample is able to withstand before complete debonding. For W_f/W samples produced via chemical vapor infiltration this test was extensively used [19] and important parameters characterizing the interface were retrieved. Depending on the underlying theory one can for example determine τ_d by obtaining the maximum load for a series of samples from the same batch with different thicknesses (H) by fitting according to Eq. 1. A more basic approach can be found in [20].

$$F_{max} = \pi \cdot \frac{d_f * \tau_d}{\alpha_2} \cdot tanh(\alpha_2 \cdot H) \quad (1)$$

where F_{max} is the maximum load, d_f the diameter of the fiber, α_2 the elastic constant, a parameter in shear-lag theory and τ_d the interfacial shear strength.

2.1. Sample preparation and experimental

The samples presented in this work are hot isostatically pressed (HIPed) in an argon atmosphere at 200 MPa for 4 h, if not indicated otherwise. Carbon heating filaments are used to achieve the desired temperatures of 1500 °C. Powder provided by Plansee SE was chosen to produce the PM W_f/W samples. The mean particle diameter of the powders is 10.60 ± 7.78 µm. The fibers used for this work are made of undoped and pure tungsten with a diameter of 150 µm (made available by Osram GmbH) and possess a as produced tensile strength of typically 2900 MPa. The fibers are not stabilized against recrystallization by potassium doping, hence tend to lose their ductility at temperatures above 1000 °C. For more details on the sample production refer to [17] and for detail on the actual fiber to [13,16].

Before testing and analysis, all samples were prepared in size and shape appropriate for the specific method. In addition, all samples are polished with a Saphier 550 - Rubin 520 polishing machine (ATM GmbH) using silicon carbide paper and 3 µm and 1 µm diamond polishing suspensions for the final polishing steps. A $K_3[Fe(CN)_6]$ etching solution is used to reveal the grain structure and enhance the visibility of the fiber for precise positioning before the push-out test. All scanning electron microscopy (SEM) images presented here are either taken with a DSM 982 Gemini or a Crossbeam 540 Gemini II (Carl Zeiss AG), which is also a focused ion beam (FIB) device. Density measurements are carried out on a pure W section of the sample after Archimedes principle with a Cubis MSA225S scale and the density measurement kit YDK01 (Satorius AG) in 99.9% ethanol. All W_f/W single fiber samples are showing a density of 99% or more.

2.2. Push-out test

The push-out tests are carried out with the help of a setup containing a special sample holder (Fig. 2(a)) and a micro-indenter attached to an Instron 3342 universal testing machine (Instron GmbH) with a 500 N load cell. The fiber of the single fiber push-out samples, polished on both sides, is positioned over a small hole (diameter L = 0.2 mm) in the sample holder and then carefully

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