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Crack bridging in as-fabricated and embrittled tungsten single fibre-reinforced tungsten composites shown by a novel in-situ high energy synchrotron tomography bending test

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ABSTRACT

Due to a unique property combination, tungsten is a promising candidate for highly loaded areas in advanced fusion reactors. However, tungsten suffers from its inherent brittleness at low temperature and its susceptibility to operational embrittlement. In tungsten fibre-reinforced tungsten composites (W_f/W) the toughness is enhanced by extrinsic mechanisms of energy dissipation allowing toughening in the absence of any plasticity. In the here presented work active extrinsic mechanisms of toughening were shown on a model system for as-fabricated and embrittled samples. The mechanisms were evaluated by means of mechanical bending tests in combination with high energy synchrotron tomography. For that a novel 4-point bending test for the in-situ use with high energy synchrotron tomography was developed. Despite the high X-ray attenuation in tungsten a sufficiently high resolution was achieved and clear images of crack extension and crack-fibre interaction were obtained. Several active toughening mechanisms were observed and quantified for the as-fabricated state and, in the case of a stable fibre-matrix interface, also in the embrittled state. The toughening contribution of the individual mechanism was estimated using the mechanical test results and compared with analytically derived values. Using the determined values a high toughening was estimated for as-fabricated and for embrittled bulk W_f/W. The results give hope that the composite material will retain toughness even if experiencing operational embrittlement when used in a future fusion reactor.

1. Introduction

Tungsten (W) is the main candidate for highly loaded areas in advanced fusion reactors due to its excellent erosion resistance and low H retention as well as high temperature strength and creep resistance combined with a high thermal conductivity and melting point [1,2]. However, as a typical bcc metal, tungsten exhibits a so called ductile-to-brittle transition (DBT) [3]. Below a certain temperature, the DBT temperature (DBTT), these materials show brittle behaviour. In the case of tungsten the transition takes place well above room temperature and therefore strongly restricts its use. The transition temperature is very much dependent on the composition, the fabrication process, and the pre-treatment and is typically between 500 K and 600 K [4] and up to 1200 K for material annealed at high temperature [5].

Microstructure and toughness are strongly related in tungsten. For example highly deformed tungsten, e.g. tungsten wire, shows ductility even at room temperature [6,7]. This phenomenon makes tungsten prone to embrittlement by a change of microstructure for example during thermal overload in operation [8]. In the case of fusion operational embrittlement induced by neutron irradiation is an even larger concern [9,10]. Reviews about possible solutions and unsolved problems are given by Rieth et al. [11] and Wurster et al. [12].

In brittle materials like tungsten (below the DBTT) or ceramics stress cannot be redistributed. Areas with large local strain experience high stress levels and are therefore prone to brittle fracture [13]. The only way to improve the toughness in brittle materials is by *extrinsic mechanisms* [14,15]. Mechanisms like crack bridging, crack deflection or fibre pull-out dissipate energy and relax local stress peaks. Such purely mechanical mechanisms which work without the need of any plasticity are widely used in ceramic fibre-reinforced ceramics [16,17].

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Fibre pull-out is in general seen as the most effective mechanism [18,19]. Starting with Aveston et al. [20] extensive theoretical studies have been performed to describe these mechanisms of toughening. Detailed overviews are given by Evans [18] and Steinbrech [21].

Using such energy dissipation mechanisms to solve the brittleness problem of tungsten was suggested by several authors [22,23]. Extrinsic mechanism of toughening are introduced by reinforcing tungsten with tungsten fibres made of drawn tungsten wire. These fibres are coated by engineered interfaces in order to optimise their behaviour within the composite [24]. More information about these tungsten fibre-reinforced tungsten composites (W_f/W) can be found elsewhere [25,26]. The plastic deformation of the tungsten fibre was shown to be an additional effective mechanism of energy dissipation in W_f/W [27]. As the toughening is achieved by mechanical mechanisms it works in the as-fabricated state having a ductile fibre as well as in a state were the fibre has been embrittled. This was shown for bulk materials in [28,29] and gives hope to mitigate the problem of embrittlement during operation. Tungsten wire has been used in the past as reinforcements in copper composites [30], superalloys [31] and in a plasma sprayed tungsten matrix by Hill and Banta [32]. The main objective of these applications was however to increase the strength and creep resistance but not primarily the toughness.

In this article we investigate the active toughening mechanisms in the as-fabricated and in an embrittled state of W_f/W in detail. We do this on single-fibre composite model systems consisting of a single fibre surrounded by a tungsten matrix. As chemically deposited tungsten used as matrix material is already very brittle [33] the fibres were embrittled to study the embrittled state. The ductility and high strength in the tungsten wire used as fibres is due to its special microstructure. A heat treatment above the recrystallization temperature changes this microstructure by recrystallization and grain growth and embrittles the fibres as it was shown for the here used fibres in [7]. As similarly high temperature treatment of bulk tungsten typically leads to embrittlement [8], it was expected that the brittle behaviour of the matrix materiel should be preserved. Thus, a composite consisting of fully brittle constituents is created. This state is called embrittled W_f/W in the following.

To assess the toughening mechanisms in both cases it is essential that the macroscopic mechanical behaviour can be related to microstructural effects. As these effects are active during the fracture process (e.g. fibre bridging) their characterisation requires in-situ techniques. Mechanical testing in combination with tomography is well suited to assess the interaction of a growing crack with the microstructure and thus allows studying these effects. Synchrotron tomography allows to do this with a spatial resolution in the range of a few microns [34]. This was intensively used to study SiC fibre-reinforced Ti matrix composites (SiC_f/Ti) [35–38]. Quasi static as well as cyclic tensile tests have been used to evaluate the interaction between the fibres and the matrix in general and under loading at room temperature or at elevated temperature. Due to the very strong X-ray attenuation of tungsten, high energy synchrotron radiation is necessary to perform tomography experiments in this material. The high energy beamline ID 15A at the European Synchrotron Radiation Facility (ESRF) allows fast tomography and beam energies up to 500 keV and was therefore used in this work.

The same beamline was used for studying the tensile behaviour of similar model systems of W_f/W [27]. Despite the high X-ray attenuation in tungsten, a resolution of 5 µm was achieved and clear images of crack extension and deformation were obtained. The crack bridging and ongoing plastic deformation were directly observed in tomographic observations and correlated to the load-displacement measurements. The amount of absorbed energy due to plastic deformation of the tungsten fibre was determined and compared with values obtained from single-fibre tensile tests. Based on this work we present in the following the results of an experimental campaign of bending tests monitored by in-situ high energy tomography based on these experiences. Bending tests are used as they allow to grow a crack in a more controlled way than tension tests. The as-fabricated and the heat treated and thus embrittled state are compared. Emphasis is given on the expected transition of plastic bridging to purely elastic bridging as a consequence of the loss of fibre ductility. A weak interface consisting of a porous tungsten layer and a very stable interface consisting of an Er_2O_3 layer are investigated to determine the influence of the interface system on the toughening.

2. Extrinsic toughening by reinforcements

The matrix in W_f/W produced by chemical vapour deposition behaves brittle and thus linear elastic fracture mechanics are applicable [26]. An energy approach using the energy release rate G or the stress state at the crack tip characterized by the stress intensity factor K can be used. For linear elastic materials the two parameters are related as follows [39]:

$$K = \sqrt{G \cdot E^*} \tag{1}$$

where $E^* = E$ for plain stress conditions and $E^* = E/(1-\nu^2)$ for plain strain conditions (E: Young's modulus; ν: Poisson's ratio). The resistance of a material against fracture and thus its toughness is described by a critical value G_c or K_c, respectively [39]. For materials featuring extrinsic toughening the following equation is used:

$$G_c = G_0 + \Delta G$$

where G_0 is the toughness of the matrix material and ΔG the gain in toughness due to the extrinsic mechanisms. In the case of fibre reinforced composites, this gain can be described by the traction t caused by the fibres on the crack faces withstanding the crack opening u [18]. The following equation describes the contribution of the fibres ΔG_f to the toughness:

$$\Delta G_f = V_f \cdot w_f^* = V_f \int_0^{u_{max}} t(u) \mathrm{d}u \tag{2}$$

with

V_f	: fibre volume fraction	[-]
w_f^*	: specific energy consumption	[J m^{-2}]
u_{max}	: maximum crack opening	[m]
$t(u)$	: traction of reinforcements on crack faces	[MPa]

Based on this relationship, Becher [40] gives an overview about toughening mechanisms in ceramic fibre-reinforced ceramics. He describes two main contributions, crack bridging by intact fibres $\Delta G_{f,\,br}$ and pull-out bridging $\Delta G_{f,\,po}$ (typically also referred to as pull-out only). He describes bridging as a consequence of a force equilibrium and expecting a frictional component, respectively. In the absence of a frictional component the contribution to the toughness is calculated as follows:

$$\Delta G_{f,br,eq} = \frac{V_{f,br} \cdot (\sigma_{f,fr})^2 u_{db}}{2E_f} = V_{f,br} \cdot w_{br,eq}^* \tag{3}$$

with

$V_{f,br}$	: fibre volume fraction bridging crack plane	[-]
$\sigma_{f,fr}$	: fibre fracture strength	[MPa]
u_{db}	: debonding length	[m]
E_f	: Young's modulus of the fibres	[MPa m^{-2}]
w_{br}^*	: specific work by bridging	[J m^{-2}]

with a frictional component as follows:

$$\Delta G_{f,br,fr} = \frac{V_{f,br} r}{3 E_f \tau_i} (\sigma_{f,fr})^3 = V_{f,br} \cdot w_{br,fr}^* \tag{4}$$

with

r	: fibre radius	[m]
τ_i	: frictional shear resistance	[MPa]

In contrast to Becher et al. we use a factor of 3 instead of 6 (see