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Charpy impact tests of tungsten fiber–reinforced composite from −150 °C to 1000 °C[☆]

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ABSTRACT

Tungsten features a unique combination of properties that makes it a favorable plasma-facing material in fusion reactors. The main drawbacks of tungsten are its brittleness at room temperature and the potential embrittlement during operation. Tungsten fiber–reinforced composites have the potential to overcome these limitations. Charpy impact tests ranging from −150 °C to 1000 °C have been performed. To identify the ductile-to-brittle transition temperature and understand the behavior at high temperature a detailed microscopic investigation was used to identify the transition temperature of fiber and matrix, which was between −100 °C to −50 °C and 1000 °C, respectively.

1. Introduction

The materials that will be used in plasma-facing components in future fusion reactors will face severe challenges [1]. The materials need to have advanced mechanical and thermal capabilities and will be required to withstand operational degradation. Tungsten is currently foreseen as plasma-facing material in such components. Tungsten is very resilient against erosion, has the highest melting point of all metals, shows moderate activation, and has low hydrogen retention [1]. The main drawbacks when using pure tungsten is its intrinsic brittleness, which is below the ductile-to-brittle transition temperature (DBTT), and its embrittlement during operation (e.g., by overheating or neutron irradiation). These limitations are mitigated by using tungsten fiber–reinforced composites (W_f/W). In principle, toughening works in the as-fabricated state and after embrittlement due to high-temperature annealing at room temperature (RT), which is below the DBTT of bulk tungsten [2].

In this contribution, we focus on the Charpy impact behavior of chemical vapor–deposited W_f/W (CVD-W) at temperatures from −150 °C to 1000 °C. This is supplemented by microstructural investigations of the fracture surfaces to obtain the DBTT of the fiber and matrix.

2. Material and experimental procedure

Bulk W_f/W was produced with a layer-wise chemical vapor deposition process at approximately 650 °C with a K-doped tungsten fiber fabric as preform and yttrium oxide as interlayer material. A detailed description of the manufacturing process and the composite can be found elsewhere [3,4]. The density of every specimen was measured by processing the fracture surface images to distinguish between the pores (converted to black) and the dense material (converted to white). The

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density was mainly 99.8 % while the material tested at −50 °C and 600 °C had 97.9 % and 97.3 % density, respectively. The fiber volume fraction of 11 %. was calculated by dividing the sum of the fiber areas by the specimen area and was constant for all specimens.

KLST-type specimens (3 × 4 × 27 mm, notch depth of 1 mm) were chosen for comparison to previous experiments on tungsten [5–7]. All specimens were cut from one bulk sample with electrical discharge machining and tested without any further treatment. A Charpy impact pendulum device was used to test specimens below RT in air. The temperatures (−150 °C, −100 °C, and −50 °C) were controlled by a cooling unit, and the samples were automatically placed in the test position. As the samples were exposed to RT for a few seconds, neglectable heating is expected. For the tests at elevated temperatures (RT, 600 °C, 900 °C, and 1000 °C), a special drop-weight setup was used [5]. The elevated temperature tests were performed under vacuum (0.01 Pa). One specimen per temperature was tested, and one specimen was tested at 1000 °C after the temperature was held for 1 h to see if degradation of the material occurs. The support distance for all tests was 22 mm.

3. Results

In Fig. 1, the Charpy energy over temperature is shown. At a temperature of 1000 °C, a significant increase is visible.

In the following description, a distinction is made between fiber and matrix. The interlayer is not described as the volume ratio is negligible so that it does not make any noteworthy contribution to the impact energy. Note, the interlayer debonds in all tests despite the high loading speed and thus fulfills the debonding criteria described in a previous contribution [4].

In Fig. 2, the fracture surfaces of the Charpy impact tests performed at −150 °C, −100 °C, −50 °C, and RT are shown. At −150 °C, the fiber and matrix show a brittle fracture with trans- and intercrystalline failure. A pulled-out fiber can be identified at two points. The fracture surface at −100 °C shows ductile deformed fibers (see Fig. 2 d)), and brittle fibers are also visible. The matrix is brittle. At −50 °C (see Fig. 2 e)) all fibers show ductile failure. No sign of ductility can be seen in the matrix. At RT, no large change is visible.

The fracture surfaces of the Charpy impact tests at 600 °C, 900 °C, and 1000 °C are shown in Fig. 3. At 600 °C (see Fig. 3 b), the fiber shows further necking compared to the tests at RT. A brittle fracture behavior was identified for the matrix at this temperature. At 900 °C, the fiber diameter in the necking zone is further reduced (see Fig. 3 d). At this temperature, a beginning ductile deformation of the matrix with plastic deformation of individual grains was observed. However, most of the matrix shows brittle failure. For the tests at 1000 °C, the fibers show a ductile fracture with a further reduction in the diameter. The matrix shows ductile deformation in both cases. This is macroscopically characterized by a strong plastic deformation of the entire sample. Microscopically, the grains show a plastic deformation with a knife edge structure. No difference was identified between the samples tested at 1000 °C even if the temperature was held for 1 h.

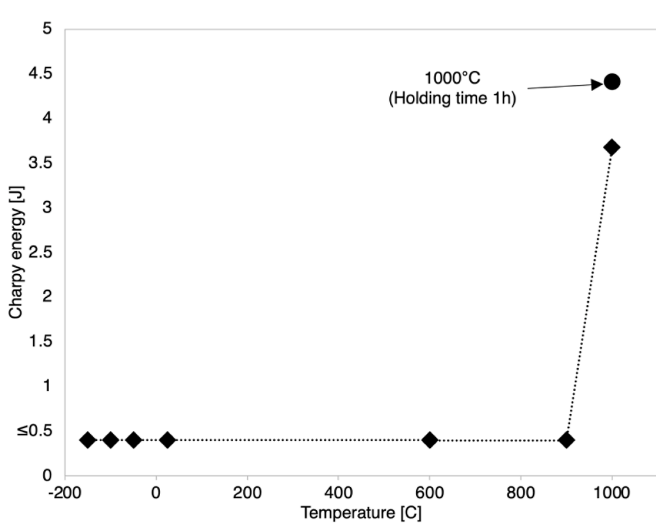

Fig. 1. Absorbed energy during Charpy impact test at different temperatures.

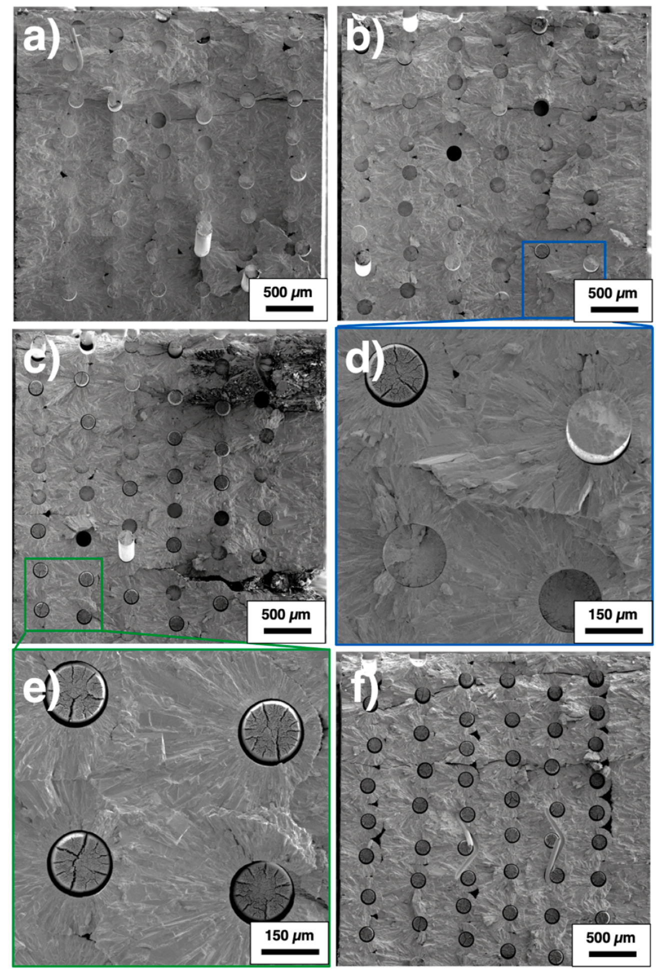

Fig. 2. Fracture surfaces of specimens tested at −150 °C (a), −100 °C (b), d)), −50 °C (c), e)) and RT (f). The notch is always on the bottom of the specimens.

The values and characteristics of the fracture surfaces are summarized in Table 1.

4. Discussion and conclusion

For the tested samples, the E_{KV} can be divided into the energy the matrix or fiber absorbed. In the event of a brittle fracture, almost no energy is absorbed. Thus, the E_{KV} between −50 °C and 900 °C is dominated by the fibers. The deformation energy of pure tungsten fibers with a diameter of 150 μm at RT is 3.4–5.8 mJ [8,9]. Thus, the E_{KV} at RT with a fiber volume fraction of 11 % (~54 fibers) can be calculated as 0.2–0.3 J. However, measured values below 0.5 J are outside the resolution range of the test equipment. To confirm the assumption that the fibers have the main energy consumption during the experiment, the experiment can either be carried out with a more sensitive apparatus or a significantly larger sample with more fibers. In addition, an increase in the fiber volume fraction from currently 11 % to higher values, leads to a higher E_{KV} at RT. If, for example, a fiber volume fraction of 40 % is used, the expected Charpy energy is calculated between 0.70 and 1.18 J. Thus, an increase in fiber volume fracture would lead to measurable Charpy

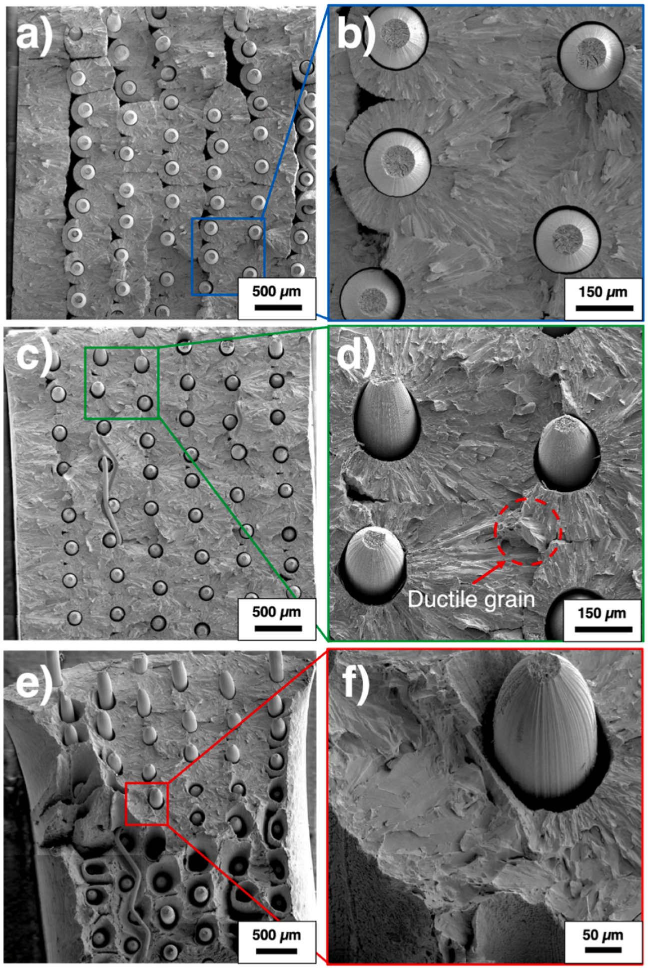

Fig. 3. Fracture surfaces of specimens tested at 600 °C (a), (b), 900 °C (c), (d) and 1000 °C (e), (f). The notch is always on the bottom of the specimens.

Table 1
Summary of the results.

Temperature [°C]	E_{KV} [J]	Fiber	Reduction in area of fiber [%]	Matrix
−150	≤ 0.5	Brittle	—	Brittle
−100	≤ 0.5	Brittle/first signs of ductility	0–27	Brittle
−50	≤ 0.5	Ductile	31 ± 3	Brittle
RT	≤ 0.5	Ductile	38 ± 3	Brittle
600	≤ 0.5	Ductile	79 ± 2	Brittle
900	≤ 0.5	Ductile	90 ± 1	Brittle/first signs of ductility
1000	3.7	Ductile	91 ± 1	Partially ductile
1000 (1 h)	4.4	Ductile	91 ± 1	Partially ductile

energy at RT.

The DBTT of the fiber is between −100 °C and −50 °C, which is also consistent with fracture toughness investigations on tungsten foil [10]. The plastic deformation of the matrix at 1000 °C correlates with the increase in impact energy (E_{KV}), and thus the DBTT of this CVD-W is between 900 and 1000 °C. Compared to previous Charpy impact experiments on pure tungsten, our highest values of ~4 J are relatively low compared to the previously reported upper shelf energy of 6–12 J [6,7]. This suggests that if the test temperature is further increased, the E_{KV} could increase further. To determine the maximum E_{KV} of the matrix, the impact tests must be carried out at even higher temperatures, which was not possible with the testing equipment used. In addition, a holding time of 1 h at 1000 °C does not change the E_{KV} and the higher value is related to scatter of data between different samples. The DBTT of the CVD-W is relatively high compared to other tungsten materials [6,7]. Murphy et al. [11] produced CVD-W from WF_6, and in four-point bending tests, no ductile deformation was reported at temperatures up to 967 °C. It was assumed that the 10–100 ppm fluorine contaminants that accumulate at the grain boundaries weakens them and leads to grain boundary failure [11]. Bryant et al. [12] produced CVD-W with the additional introduction of oxygen [13] and found a DBTT of 205–250 °C with a fluorine and oxygen content of 1–2 ppm and 5–20 ppm, respectively.

The results showed that the DBTT of the used tungsten fibers is very low compared to bulk tungsten (minimum 350 °C [7]), but it is relatively high for the CVD-W. The low DBTT of the fibers is caused by their special microstructure resulting from wire drawing, but mechanisms are not fully clear yet. The ductilization of the W fibers is currently under investigations and could be different compared to W-foils as the manufacturing routines are different (fibers: wire drawing vs. foils: rolling). The high DBTT of the CVD-W might be caused by fluorine impurities, which could possibly be suppressed by an optimization of the deposition process.

CRediT authorship contribution statement

Hanns Gietl: Conceptualization, Methodology, Investigation, Visualization, Formal analysis, Writing – original draft. **Johann Riesch:** Conceptualization, Supervision, Writing – review & editing. **Till Höschen:** Resources, Methodology. **Michael Rieth:** Resources, Writing – review & editing. **Jan W. Coenen:** Writing – review & editing. **Rudolf Neu:** Funding acquisition, Supervision, Writing – review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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