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Fracture toughness investigations of tungsten alloys and SPD tungsten alloys

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Abstract

Three different tungsten alloys (pure W, a lanthanum-oxide dispersion strengthened W-alloy – WL10 and a potassium doped tungsten alloy – WVM) in different fabrication conditions (sintered and rolled rods) were tested to characterize their fracture behaviour at different temperatures (from −196 to 800 °C). Additionally severe plastic deformed tungsten alloys were produced. It is shown that an ultra fine grained microstructure with a diameter of about 300 nm is able to decrease the brittleness at room temperature significantly. Such W-alloys are very attractive candidate materials for fusion applications.

1. Introduction

Tungsten and several of its alloys are widely used in high temperature applications. These materials offer high melting points, high erosion resistance, good thermal shock properties and good thermal conductivity, which are necessary in certain parts of future fusion reactors like ITER.

A major drawback of tungsten alloys is their high DBTT (ductile-to-brittle-transition temperature) which is influenced by parameters like cold work, grain size and impurities [1]. The divertor region in a fusion reactor is subjected to high thermal loads and temperature gradients resulting in thermal stress. During operation, these stresses could lead to cracks in the material. Thus, high ductility in the room temperature regime and above would be very useful for such structural applications.

Although used for nearly a hundred years in light bulbs, fracture toughness data of tungsten alloys are rare and studies have mainly focused on single crystals [1]. Polycrystalline tungsten alloys were therefore tested at various temperatures from −196 °C (liquid nitrogen) to 750 °C to examine their fracture behaviour. The influence of cold work was also taken into account, as is the orientation of the fracture surface.

High pressure torsion (HPT) is a method of severe plastic deformation (SPD) to produce bulk nanostructured materials with grain sizes sometimes below 100 nm [2]. This results in unique mechanical properties like increased hardness/yield strength and considerable or even increased ductility [3].

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2. Experimental

2.1. Tungsten alloys

The alloys used for both fracture mechanical tests and SPD are commercial materials (PLANSEE SE, Reutte, Austria). Table 1 shows fabrication conditions and the various tests performed.

W denotes pure tungsten (99.98%). WL10 is a 1% lanthanum-oxide-doped tungsten alloy, while WVM is a 0.005% potassium-doped tungsten alloy. All materials are produced in a powder-metallurgical route. The potassium and particle containing alloys are more resistant to creep than pure tungsten. The grain sizes of these alloys are in the range of several μm, depending on the degree of hot work.

2.2. Determination of fracture toughness

For fracture toughness tests, disk shaped compact tension (DCT) and single edge notched bend (SENB) specimens were prepared according to ASTM E399 from the materials in Table 1. The precrack was prepared by cuts with a diamond wire saw and a razor blade. Sharp precracks were introduced by cyclic compression with 15000–30000 cycles at stress intensity factors from 20 to 45 MPa$\sqrt{m}$ and $R = 20$ depending on the material and fabrication (WL10 and WVM require higher stress intensities). The achieved crack length was controlled with an optical microscope. DCT-specimens had C-R and SENB had L-R orientation. Tests at temperatures from 200 to 800 °C were performed in a vacuum device to avoid corrosion of the fracture surfaces. An inductive heater was used to heat the specimens and thermocouples were attached directly to the specimens to control the temperature. A potential drop method was used to measure crack extension (Keithley 2182 Nanovoltmeter and constant current source). During the experiments force F, temperature T and potential drop P were recorded.

With the exception of tests at higher temperatures (above 400 °C), all tests were valid according to the ASTM E399 standard. At higher temperatures, the plastic zone to specimen dimension-ratios are too high, the determined values are therefore too low and represent only a lower limit of fracture toughness at those test temperatures.

The smallest SENB-specimens were tested in a miniature testing machine.

2.3. Severe plastic deformation

High pressure torsion is a method to produce ultra fine grained tungsten alloys. A pill-shaped specimen (the diameter is 6 mm, height is 0.8 mm) is inserted between two anvils (hardmetal with steel casing) which are pressed together with a high hydrostatic pressure of about 8 GPa. An inductive heating device is used to heat the specimen and

Table 1
Materials used for fracture toughness and SPD experiments

Material	Condition	Rod diameter (mm)	Nomenclature	Reduction (%)	Tests performed
W	As sintered	22.5	W_1	0	DCT ($W = 15$, $a = 5$ and $B = 6$ mm)
	Rolled (22.5 → 9 mm)	9.0	W_2	60	SENB ($W = 6$, $a = 3$ and $B = 3$ mm), DCT ($W = 6$, $a = 2$ and $B = 3$ mm) and HPT
	Rolled (9 → 3.8 mm)	3.8	W_3	83	SENB ($W = 3$, $a = 1.5$ and $B = 1.5$ mm)
WL10	As sintered	23.0	WL10_1	0	DCT ($W = 15$, $a = 5$ and $B = 6$ mm)
	Rolled (23 → 9.8 mm)	9.8	WL10_2	57	SENB ($W = 6$, $a = 3$ and $B = 3$ mm), DCT ($W = 6$, $a = 2$ and $B = 3$ mm)
	Rolled (9.8 → 4.4 mm)	4.4	WL10_3	80	SENB ($W = 3$, $a = 1.5$ and $B = 1.5$ mm)
	Rolled (48.5 → 9.3 mm)	9.3	WL10_4	81	HPT
WVM	As sintered	22.5	WVM_1	0	DCT ($W = 15$, $a = 5$ and $B = 6$ mm)
	Rolled (22.5 → 9.2 mm)	9.2	WVM_2	59	SENB ($W = 6$, $a = 3$ and $B = 3$ mm), DCT ($W = 6$, $a = 2$ and $B = 3$ mm)
	Rolled (9.2 → 3.8 mm)	3.8	WVM_3	83	SENB ($W = 3$, $a = 1.5$ and $B = 1.5$ mm)
	Forged (48 mm → 23 mm) Rolled (23 mm → 14 mm)	14.0	WVM_5	54	HPT

Notation see text.

anvils to 400 °C. Subsequently the lower anvil is rotated with a constant angular velocity. The degree of deformation (true Von Mises-strain ε_{VM}) can be evaluated with the simple relationship $\varepsilon_{VM} = \frac{2\pi}{\sqrt{3}}\frac{rn}{h}$, where r denotes the radius, n is the number of revolutions and h the height of the specimen. This equation shows that a linear relation between strain and radius exists, which makes the study of different degrees of deformation with in a single specimen possible. The produced microstructures show grain sizes in the range of 300 nm at $\varepsilon = 64$ and higher misorientation.

Fracture specimens of SENB-type were fabricated from the SPD-processed materials. Their approximate dimensions were $4.5 \times 1 \times 0.8$ mm. The necessary precrack was introduced with a focused ion-beam workstation (LEO 1540) instead of the usual precracking via applying a cyclic compressive load. These 'cracks' at the root of the notch measured approximately 100×15 μm. The specimens were tested at room temperature with a miniature testing machine. Because the yield strength σ_y is also increased by HPT from 1000 MPa up to 3000 MPa (estimated by Vickers microhardness H_V using the simple relation $\sigma_y = H_V/3$), the SENB fracture tests were valid at room temperature.

3. Results

3.1. Fracture toughness of tungsten alloys

The results of sintered tungsten alloys, denoted as W_1, WL10_1 and WVM_1, are shown in Fig. 1. As shown in Table 1 these samples were DCT-type with $W = 15$ mm, $a = 5$ mm and $B = 6$ mm.

The rolled material (denoted as W_2, WL10_2 and WVM_2) exhibits similar fracture toughness values up to 400 °C. The dimensions of DCT-specimens were $W = 6$ mm, $a = 2$ mm and $B = 3$ mm. Fig. 2 shows the results of the fracture toughness tests.

The same material as in Fig. 2 was also used for SENB-samples with a different orientation of the fracture surface. The dimensions were $W = 6$ mm, $a = 3$ mm and $B = 3$ mm. Fig. 3 shows the determined values.

The materials with 80% reduction were tested at room temperature only. The maximum stress intensity factors were 5.4 MPa$\sqrt{m}$ for W_3, 9.7 MPa$\sqrt{m}$ for WL10_3 and 13.5 MPa$\sqrt{m}$ for WVM_3. Extensive analysis of the fracture surfaces was done in the

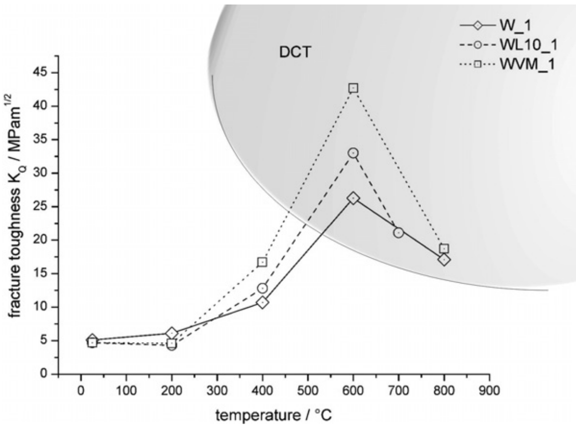

Fig. 1. Fracture toughness of tungsten alloys in the as sintered condition (DCT). The grey area marks the region where linear fracture mechanics is no longer valid.

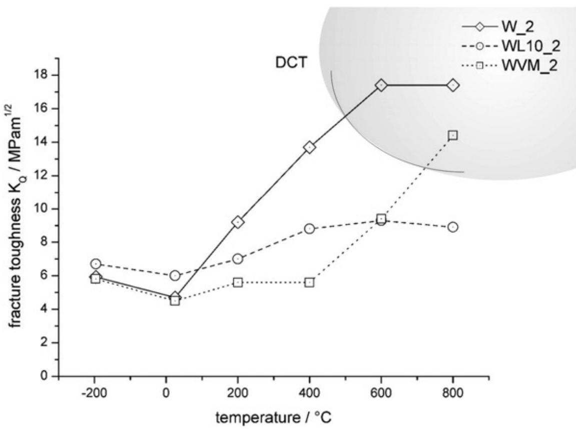

Fig. 2. Fracture toughness of tungsten alloys in the as-rolled condition (60% reduction, DCT). The grey area marks the region where linear fracture mechanics is no longer valid.

scanning electron microscope. The fractographs in Fig. 4(a) and (b) can only give a brief overview of the various morphologies that occur. A more detailed presentation of these results is in preparation (CTOD-measurements).

Fig. 4(a) shows a fractograph of sintered tungsten tested at RT. Although there is no big difference in the morphology, the K_Q-values differ significantly when going to higher temperatures (see W_1 curve in Fig. 1). The dominating kind of fracture is intercrystalline, with fracture at grain boundaries and sinter pores. Sintered WVM exhibits the same behaviour. Although the grain size is smaller (grain growth during sintering is retarded through potassium bubbles), the same fracture

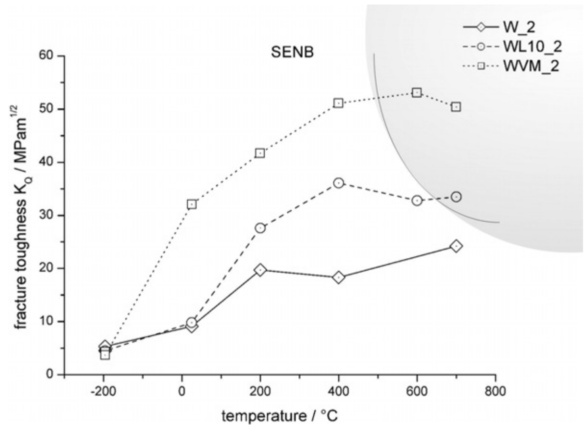

Fig. 3. Fracture toughness of tungsten alloys in the as-rolled condition (60% reduction, SENB). The grey area marks the region where linear fracture mechanics is no longer valid.

morphology occurs (intercrystalline). This results in elevated fracture toughness in almost every temperature region (see WVM_1 graph in Fig. 1).

Hot working of sintered alloys results in smaller grain sizes and closing of sinter pores. Generally, it can be stated that working results in increased fracture toughness, although Fig. 1 shows higher values (especially in the high temperature region) than Fig. 2. This can be linked to the specimen size, which is smaller in the case of the worked tungsten alloys. The tests of sintered alloys (bigger samples) therefore might give lower fracture toughnesses, due to smaller plastic zone/specimen size ratios. The fracture morphology looks different than that of sintered alloys (see Fig. 4(b)).

One reason is that the worked alloys consist of elongated grains parallel to the working direction, this results in an anisotropy of the fracture behaviour. Furthermore, some regions of the fracture surface show transcrystalline fracture. The specimens of SENB-type (not shown here) show a similar morphology, although the orientation of the fracture surface is different and the fracture toughness values are significantly larger (see Fig. 3).

Using the equation $y = \frac{A_1 - A_2}{1+\exp(T-T_0)} + A_2$ (A_1, A_2 and T_0 are fit parameters) as a fit function for the data in Figs. 1–3, one can easily determine a value for the ductile-to-brittle-transition temperature (DBTT), which is another characteristic of bcc metals, and corresponds to T_0. When doing so, an approximate value of 400 °C is obtained for the sintered materials. The as-rolled materials show values in the region between 80 and 200 °C.

3.2. Fracture toughness of SPD-W alloys

The evaluated fracture toughness values for HPT-strained samples are depicted in Fig. 5.

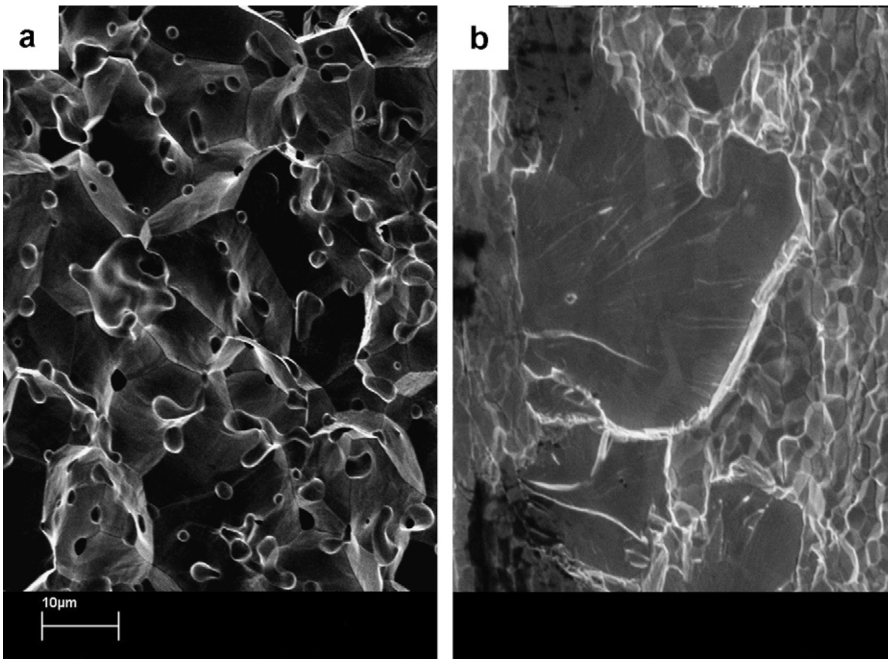

Fig. 4. Fractographs of CT-specimens tested at room temperature. (a) Sintered tungsten, (b) worked tungsten.

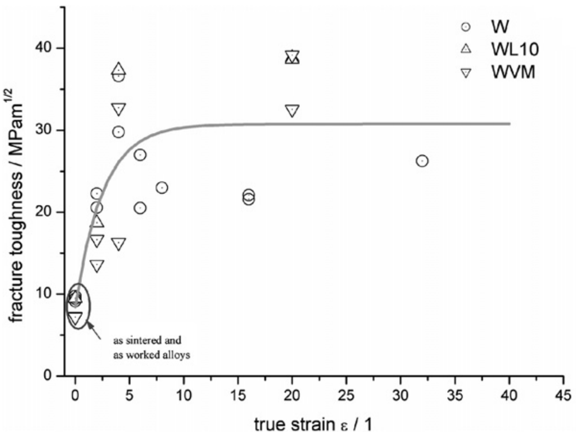

Fig. 5. The influence of high degrees of deformation on fracture toughness of tungsten alloys at room temperature.

The original material (in sintered and rolled condition) can also be found in this graph.

It is clear that the high deformation has a considerable impact on the fracture toughness. The shape of this graph is similar to the influence of HPT on microhardness/yield strength. Intercrystalline fracture seems to be typical for HPT-tungsten, although severely deformed WL10 sometimes fails in a transcrystalline manner.

4. Discussion

Although many other parameters influence the fracture of bcc-metals, the DBTT is usually associated with the thermal activation of dislocation kink pairs. Below this characteristic temperature the separation of a screw dislocation into three partial dislocations (which cannot easily recombine and are therefore more or less immobile) is responsible for the brittle behaviour. Increasing temperature leads to thermal activation of the kink mechanism and increased ductility due to shielding of the crack tip. This increase is generally observed in all specimens, independent of fracture orientation and alloy. However, the effect of microstructure on the K_{IC}-value in the low temperature regime and the DBTT cannot be explained solely by this mechanism. It was also found in [1] that fracture toughness increases when the material was deformed. Furthermore, the crack propagation direction with respect to the hot working direction was also found to be of importance for fracture. Specimens with L-R orientation of the fracture surface (like the SENB-specimens in our study, compare Fig. 3 with Fig. 2) also exhibited higher ductility in most cases. This could be an indication for lower grain boundary ductility in the rolling direction. Riedle [1] also found that the change of orientation (from L-R orientation to C-R) results in a lower amount of transcrystalline regions on the fracture surface. This result is not comparable with those of our study, because in our case both orientations (L-R and C-R) showed trans- and intercrystalline fracture, although most of them failed intercrystalline.

Varying the temperature also influences the morphology. Danylenko [4] distinguishes between five different types of fracture due to the influence of temperature (cleavage, brittle intergranular, cleavage with delamination, dimple and high temperature fracture). Furthermore there is a strong dependence of strain, leading to four distinct types in fracture toughness vs. strain graphs and nonmonotonic behaviour at low strains. The HPT-deformed materials, which had much higher degrees of deformation than the materials in [4], can be linked to region IV, where fracture toughness also shows a strong increase due to reduced cell size. The reason for this behaviour is also connected to the increased misorientation, because the stress which dislocations have to overcome at the cell boundaries increases with misorientation.

One explanation for the extremely high fracture toughness of SPD-tungsten could be the generation of additional edge dislocations. Schafler [5] has found via X-ray measurements, that in highly strained materials more edge dislocations are present. Because edge dislocations are mobile (even at RT) in bcc-metals, their increased number could also be an explanation for their extraordinary high fracture toughness of SPD-deformed tungsten alloys.

From this investigation it can be concluded that, at least in the interesting temperature regime from room temperature to 800 °C, some of these microstructures have critical fracture toughness that is better than in the sintered condition. Nevertheless it must be stated that due to the number of parameters (cell/grain size, impurities, etc.) a satisfying general explanation of the fracture behaviour cannot be given.

An important objective for future work will be to find a better explanation and understanding of the fundamental mechanism to develop a material design concept in order to optimize the fracture toughness.

5. Summary

Three different commercial tungsten alloys (pure W, WVM and WL10) have been tested to study their fracture behaviour at different temperatures (−196 to 800 °C) in different fabrication conditions. Furthermore, ultra fine-grained tungsten alloys have been produced by high pressure torsion and showed increased fracture toughness at room temperature.

The general fracture behaviour and the parameters influencing the character of fracture can be summarized as follows:

- Increasing the temperature generally leads to increased fracture toughness.
- Decreasing the cell/grain size also leads to increased fracture toughness.
- L-R orientation has a higher ductility compared to C-R orientation.

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