



# Loading rate dependence of the fracture toughness of polycrystalline tungsten

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## ABSTRACT

Crucial issues for the application of tungsten as a structural material for future fusion reactors are its inherent brittleness at low temperatures and its relatively high brittle to ductile transition temperature. The transition temperature is not an invariant material property but depends strongly on the loading rate and the fabrication route. To gain insight into the controlling factors of the brittle to ductile transition in polycrystalline tungsten, a comprehensive fracture mechanical study was setup for a common commercial material. To account for the anisotropic microstructure, three different crack orientations with respect to the rolling were tested from  $-150\text{ }^{\circ}\text{C}$  to  $950\text{ }^{\circ}\text{C}$ . Additionally, the loading rate was varied over two orders of magnitude. The results confirm that the transition temperature follows an Arrhenius relationship, giving an activation energy for the transition process. This activation energy can be employed to predict fracture toughnesses in a wide range of temperatures and loading rates.

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## 1. Introduction

Tungsten is considered as a plasma-facing material for highly loaded components of future fusion reactors due to its outstanding physical properties. Crucial issues for the application of tungsten as a structural material are its inherent brittleness at lower temperatures and the relatively high brittle to ductile transition temperature (BDTT). Particularly in brittle metals, the fracture behavior and the resulting fracture toughness strongly depend on the microstructure. Thus, it is necessary to gain a detailed understanding of the micro-mechanisms controlling the fracture process and their interaction with the microstructure to identify ways for improvement.

Tungsten, like other body-centered-cubic metals, fails generally brittle at low temperatures. With increasing temperature, a pronounced transition from brittle to ductile fracture behavior can be observed due to increasing crack tip plasticity. The BDTT is macroscopically marked by a steep increase in fracture toughness. The fracture toughness and its dependency on temperature, crack orientation and loading rate has been studied extensively for single crystalline tungsten by Gumbsch et al. [1,2]. They reported that the loading rate variation of the BDTT follows an Arrhenius law, giving an activation energy  $E_{\text{BDT}}$  of about  $0.2\text{ eV}$  for the transition process. Based on the comprehensive single crystal experiments together with discrete dislocation dynamic simulations [3], it has been established that the crack tip plasticity and the BDT in tungsten can be described as thermally activated processes controlled by

the dislocation mobility. Due to the low activation energy, Gumbsch et al. concluded that the mobility of non-screw (edge) dislocations is the controlling mechanism of the BDT in tungsten, in contrast to bulk plasticity in tungsten, which is believed to be controlled by the mobility of the screw dislocations ( $E_{\text{BDT}} > 1\text{ eV}$ ) [4]. Furthermore, the numerical simulations yield a scaling relation between loading rate and temperature at points of constant fracture toughness, which describes the single crystal experiments very well [3]. The authors state that the proposed scaling relation can be used to predict fracture toughnesses in a wide range of temperatures and loading rates, based on only a small number of experiments [3].

Commercially produced tungsten is almost exclusively processed through the powder metallurgical route and mostly possesses an anisotropic microstructure due to subsequent process steps like swaging or rolling. Works concerning the fracture behavior and the BDTT of such polycrystalline tungsten are relatively rare, and moreover, the anisotropic microstructure is seldom taken into account. One main objective of the present work is to examine to what extent the findings obtained for single crystal tungsten can be applied to a polycrystalline material.

## 2. Experimental

### 2.1. Material

In our studies on polycrystalline tungsten, rolled unalloyed 99.98% pure tungsten rods with a diameter of roughly  $14\text{ mm}$  are the starting material for our fracture mechanics specimens. This commercial material was produced by Plansee Metall GmbH,

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Reutte/Austria, in a powder metallurgical route. After sintering the rods were rolled which lead to elongated grains with an aspect ratio of roughly 1:3 in the rolling direction and a  $\langle 1\ 1\ 0 \rangle$ -fiber texture. To take the anisotropic microstructure into account, the specimens were extracted in three different kinds of crack orientations (see Fig. 1). Further details about the starting material and the sample preparation are given in [5].

## 2.2. Fracture mechanical testing

The fracture mechanical tests were conducted on notched specimens in 3-point-bending over the temperature range  $-150\text{ }^{\circ}\text{C}$  to  $950\text{ }^{\circ}\text{C}$ . The tests below room temperature were performed in a cooling nitrogen atmosphere while tests above  $350\text{ }^{\circ}\text{C}$  were conducted under high vacuum to avoid any oxidation of the specimens. The bend specimens ( $3\text{ mm} \times 6\text{ mm} \times 27\text{ mm}$ ) with 3 mm deep notches were extracted from the center of rods by electric discharge machining. Sharper notches were subsequently introduced by polishing with a razor blade. The final notches possess a well-defined tip radius of roughly  $20\text{ }\mu\text{m}$  and an overall notch depth of about 3.2 mm. During the experiments, force, displacement and temperature were recorded. The fracture toughness  $K_{IC}$  has been calculated following the ASTM E399 standard using the notch depth as crack length. To study the loading rate dependence of fracture toughness and the BDT, the bending experiments were performed in displacement control using three different loading rates, namely  $0.1\text{ }\mu\text{m/s}$ ,  $1\text{ }\mu\text{m/s}$  and  $10\text{ }\mu\text{m/s}$ . The applied loading rates correspond to stress intensity rates  $\dot{K}$  in the range of approximately  $0.05\text{--}5\text{ MPa m}^{1/2}\text{ s}^{-1}$ .

## 3. Results and discussion

Comparing the three different crack orientations, one main distinction can be drawn from their fracture behavior at low temperatures in the brittle regime. While the fracture of the L-R specimens is dominated by transgranular cleavage, the two transverse specimen types C-R and R-L failed primarily by intergranular fracture. Furthermore, the L-R specimens exhibited much higher fracture toughnesses and a much lower BDTT (about  $200\text{ }^{\circ}\text{C}$ ) than the transverse types. The fracture toughness increased gradually with temperature for all investigated crack orientations and loading rates till the BDT is marked by a steep increase in fracture toughness. In contrast to the two transverse specimens, the L-R specimens exhibited a distinct change of their fracture behavior with increasing temperature. Intergranular fracture became increasingly dominant in the semi-brittle regime. Above  $200\text{ }^{\circ}\text{C}$ , delamination ahead of the notch tip was observed, leading to crack growth along the rolling direction, normal to the notch. First results about the fracture morphology have already been published in [5]. A more detailed electron-microscopic analysis of the fracture process will be presented elsewhere.

In this paper we focus on the loading rate dependence of the fracture toughness. Figs. 2 and 3 show the fracture toughnesses

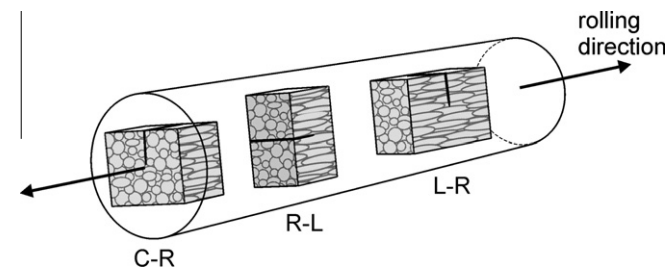


Fig. 1. Illustration of crack orientation and texture of the investigated specimens.

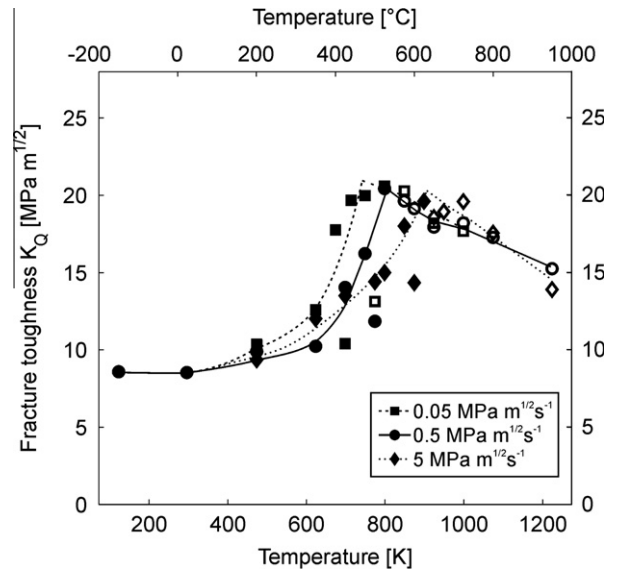


Fig. 2. Fracture toughness of the C-R orientation as a function of temperature  $T$  for different stress intensity rates  $\dot{K}$  ( $\text{MPa m}^{1/2}\text{ s}^{-1}$ ). Values out of the limits of the ASTM test standard are marked by the open symbols. Lines are guide to the eye.

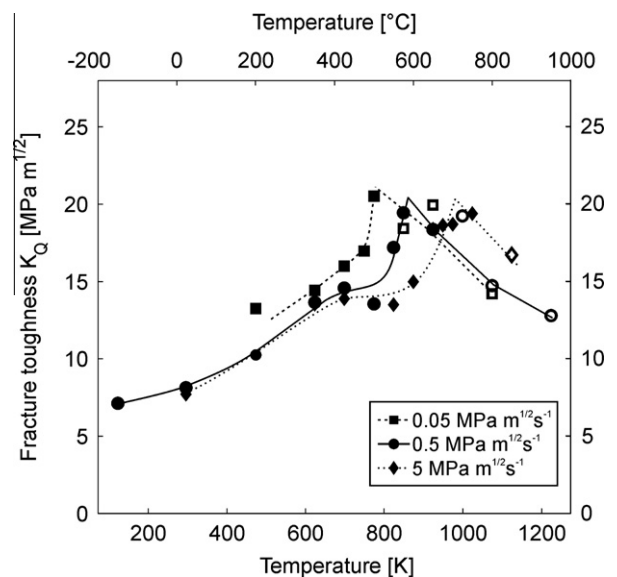


Fig. 3. Fracture toughness of the R-L orientation as a function of temperature  $T$  for different stress intensity rates  $\dot{K}$  ( $\text{MPa m}^{1/2}\text{ s}^{-1}$ ). Values out of the limits of the ASTM test standard are marked by the open symbols. Lines are guide to the eye.

of the two transverse specimen types C-R and R-L as a function of the temperature for three different stress intensity rates. The BDT of both specimen types is clearly shifted to higher temperatures with increasing loading rate and a strong dependence of the fracture toughness on the loading rate can be observed in the semi-brittle regime. At low temperatures, in contrast, the fracture toughness is almost independent of the loading rate. Compared to the single crystal experiments of Gumbsch et al. [1], where similar stress intensity rates were applied, the polycrystalline material responds much less sensitive to the variation of the loading rate and the BDT occurs generally at higher temperatures.

The transition from brittle to ductile response usually correlates with the maximum in fracture toughness. However,  $K_{IC}$  loses its validity as a failure criterion with increasing ductile behavior, and thus, the calculated fracture toughness values above the

transition regime can only give a lower bound (denoted by open symbols). As a result, the resistance of the material against crack propagation pretends to decrease at higher temperatures. The temperature at this turning point was taken as the BDTT, analog to the BDTT determination in the single crystal experiments of Gumbsch et al. [1]. Fig. 4 shows the experimental BDTT data obtained for the C-R and R-L specimens in an Arrhenius plot. The best fit to the data gives an activation energy  $E_{BDT} = 1.32$  eV for the R-L specimens and for the C-R specimens  $E_{BDT} = 1.44$  eV.

For the third orientation (L-R) no results are shown as the variation of the loading rate dependence showed no clear trend. This is probably due to the scatter in the experimental data. Furthermore, it is very likely that the rate dependence is obscured by the observed change of fracture mode, which occurred just in the semi-brittle regime, where the largest effect of loading rate is expected.

Compared to the activation energy of the BDT ( $E_{BDT} = 0.2$  eV) measured in the single crystal experiments of Gumbsch et al. [1], we found much higher activation energies for the investigated polycrystalline material. However, in recent experiments of Giannattasio and Roberts [6] conducted on tungsten single crystals and high purity polycrystals, an activation energy of about 1.05 eV was deduced. They attributed the low activation measured by Gumbsch et al. to the particular geometry of the investigated  $\{110\}\langle 1\bar{1}0\rangle$  crack system.

The discrete dislocation dynamics simulation of crack tip plasticity, carried out by Gumbsch et al. [3], showed that loading rate and temperature are strongly correlated at points of constant fracture toughness. The relation between these two quantities is given by:

$$T_2 = \left[ \frac{k_B}{E_{BDT}} \ln \frac{\dot{K}_1}{\dot{K}_2} + \frac{1}{T_1} \right]^{-1} \quad (1)$$

where  $k_B$  is the Boltzmann constant. This relation can be used to transform fracture toughness data obtained for the loading rate  $\dot{K}_1$  into data for the loading rate  $\dot{K}_2$  by rescaling the temperature axis. Provided that the scaling relation is valid, the results for different loading rates are projected on a single master curve. The scaling relation has been applied to the fracture toughness data of the transverse specimens (given in Figs. 2 and 3) by rescaling the temperature axes of the two lower stress intensity rates (0.05

and  $0.5 \text{ MPa m}^{1/2} \text{ s}^{-1}$ ) using the deduced activation energies of 1.32 eV and 1.44 eV. Figs. 5 and 6 show the obtained master curve of the fracture toughnesses for the stress intensity rate of  $5 \text{ MPa m}^{1/2} \text{ s}^{-1}$  for the R-L and the C-R crack orientation. As can be seen from both rescaled plots, the fracture toughness at a higher loading rate can be reasonably well predicted with data stemming from lower loading rates by employing the proposed scaling relation. However, in the case of the C-R specimens, there seem to be some significant outliers, but these already differ from the overall trend in the original unscaled data set (see Fig. 2). Particularly in the transition regime, such scatter of fracture toughness data can be expected.

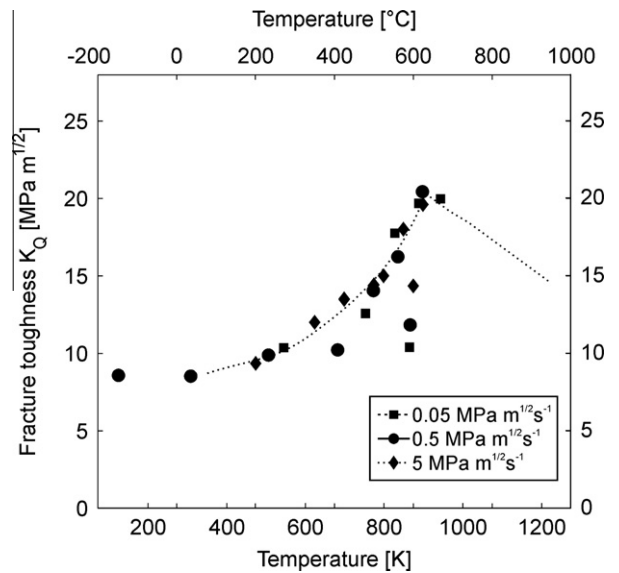


Fig. 5. Experimental data of the C-R orientation from Fig. 2 displayed in a fracture toughness – temperature plot with rescaled temperature axis. The reference stress intensity rate is  $5 \text{ MPa m}^{1/2} \text{ s}^{-1}$ .

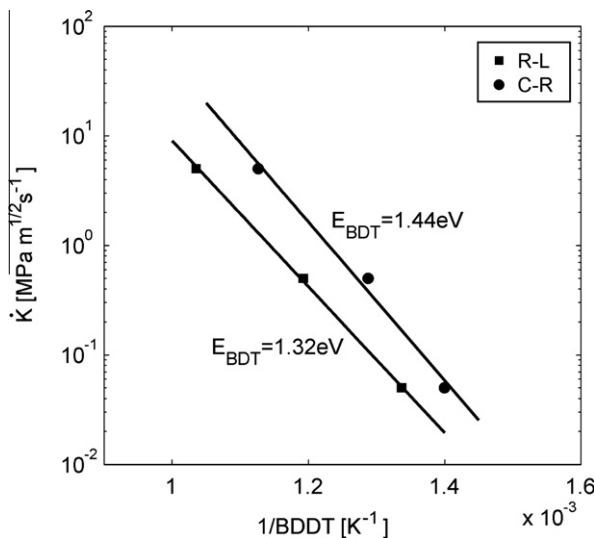


Fig. 4. Arrhenius plot of the stress intensity rate versus  $1/BDTT$  for the two transverse specimen types C-R and R-L.

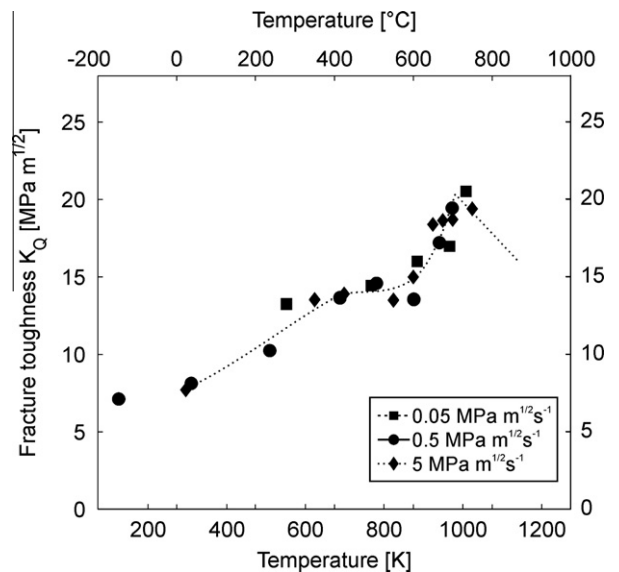


Fig. 6. Experimental data of the R-L orientation from Fig. 3 displayed in a fracture toughness – temperature plot with rescaled temperature axis. The reference stress intensity rate is  $5 \text{ MPa m}^{1/2} \text{ s}^{-1}$ .

#### 4. Summary

Commercial polycrystalline tungsten has been tested to study the dependence of fracture toughness and the BDT on the loading rate. Three different crack orientations were investigated to account for the anisotropic microstructure with respect to the rolling direction. In the case of the R-L and C-R specimens, the BDTT strongly depends on the loading rate, giving an activation energy for the BDT of  $E_{\text{BDT}} = 1.32$  eV and  $E_{\text{BDT}} = 1.44$  eV, respectively. In contrast to these two transverse orientations, no clear trend was identifiable for the L-R orientation. The experimental results reveal that the findings obtained for single crystal tungsten also hold for polycrystalline tungsten. In particular, the practical use of the apparent activation energy for the prediction of fracture toughnesses at different loading rates could be demonstrated.

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