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Fracture toughness of polycrystalline tungsten alloys

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ABSTRACT

Tungsten and tungsten alloys show the typical change in fracture behavior from brittle at low temperatures to ductile at high temperatures. In order to improve the understanding of the effect of microstructure the fracture toughness of pure tungsten, potassium doped tungsten, tungsten with 1 wt.% La_2O_3 and tungsten rhenium alloys were investigated by means of 3-point bending, double cantilever beam and compact tension specimens. All these materials show the expected increase in fracture toughness with increasing temperature. The experiments demonstrate that grain size, texture, chemical composition, grain boundary segregation and dislocation density seem to have a large effect on fracture toughness below the DBTT. These influences can be seen in the fracture behavior and morphology, where two kinds of fracture occur: on the one hand transgranular and on the other hand intergranular fracture. Therefore, techniques like electron backscatter diffraction (EBSD), Auger electron spectroscopy (AES) and X-ray line profile analysis were used to improve the understanding of the parameters influencing fracture toughness.

1. Introduction

Most studies related to the ductility of tungsten and tungsten alloys were performed in the sixties and seventies. A good example is Raffo et al. [1]. As fracture mechanics was not well established at that time studies on fracture toughness were scarce. Riedle and Gumbsch [2–5] extensively studied the fracture toughness of tungsten single crystals in the nineties. The effects of crystallographic orientation, crack propagation direction, loading rate and temperature were investigated. Compared to the single crystal, the fracture toughness of polycrystalline tungsten is not yet well examined.

We started an extensive investigation of the fracture toughness of pure tungsten (W), potassium doped tungsten (AKS-W), tungsten with 1 wt.% La_2O_3 (WL10) and tungsten–26 wt%-rhenium (WRe26). The results of a few selected microstructures are presented in this paper. A very large effect of the microstructure, especially below the ductile to brittle transition temperature, was observed. These investigations indicate that the change from transgranular to intergranular cleavage fracture plays an important role. Especially crystallographic analyses are presented to improve understanding of the interaction of these fracture processes.

This paper was also presented at the 12th International Conference on Fracture—July 12–17, 2009.

2. Experimental

The fracture toughness of W, AKS-W, WL10 and WRe26 were investigated by means of 3-point bending (3 PB — Fig. 1-a), double cantilever beam (DCB — Fig. 1-b) and compact tension specimens (CT — Fig. 1-c). All specimens were manufactured out of rods at different stages of the processing route. Fig. 1 shows specimens prepared to investigate the materials in rolling direction and radial direction. The experiments were performed in the range of −196 °C to 1000 °C.

In order to examine the local variation of the fracture resistance, DCB-specimens (Fig. 2-a) with a length of 30 mm, a height of 3.5 mm and width of 7.5 mm and CT-specimens with a length of 7.5 mm, a height of 3 mm and a width of 6 mm were manufactured out of W, AKS-W, and WRe-rods. The notches were prepared with a diamond-saw, refined with a razor blade and fatigue-precracked under cyclic compression [6]. The areas in front of the crack-tips were scanned using electron backscatter diffraction (EBSD) after a heat treatment of 2000 °C for an hour in hydrogen atmosphere (Fig. 2-b). Some specimens were then loaded under tension within the range of stable crack growth (Fig. 2-c). After that, the previously scanned areas were scanned again using EBSD (Fig. 2-d) to quantify changes of the grain orientation in the obtained orientation imaging maps (OIM).

The tests performed at room temperature were done with a microtensile-testing machine from *Kammrath & Weiss*, while the tests at elevated temperatures were done by use of a *ZWICK* universal-testing machine. The fractographic and crystallographic analyses were

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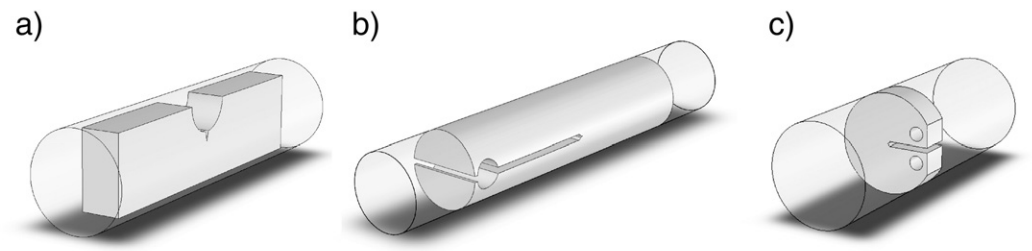

Fig. 1. Different specimen types manufactured out of tungsten rods. 3PB-specimen with crack direction in radial direction (a). DCB-specimen with crack direction in rolling direction (b). CT-specimen with crack direction in radial direction (c).

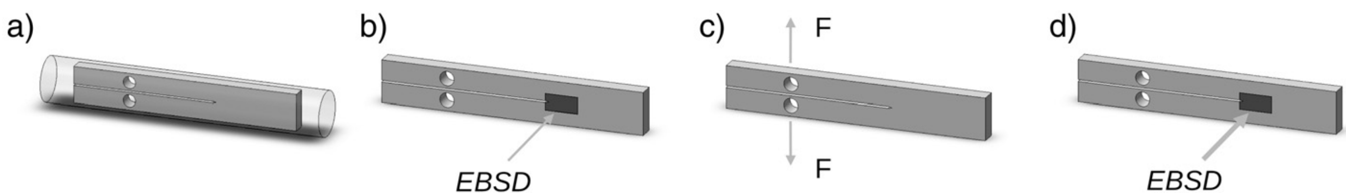

Fig. 2. Experimental setup to investigate the local variation of the fracture resistance: position of a DCB-specimen in a AKS-W rod (a). Scanning the area in front of the crack tip using EBSD (b). Apply a tensile load on the specimen (c). Scanning the previously scanned area again (d).

made by use of a Zeiss 1525 scanning electron microscope equipped with an EDAX EBSD system.

3. Results and discussion

3.1. Fracture toughness investigations

All tested specimens showed the expected increase in fracture toughness with increasing temperature, examples are shown in Fig. 3 with the associated values shown in Table 1.

At low temperatures, the fracture toughness was determined by use of linear elastic fracture mechanics whereas at temperatures above 600 °C the critical crack tip opening displacement *CTOD* [7] was used to determine the critical stress intensity factor. The fracture toughness determined by stereophotogrammetric techniques is then calculated by

$$K_{IC} = \sqrt{m \cdot \sigma_y \cdot E \cdot COD_C} \tag{1}$$

where σ_y represents the yield strength, E the Young's modulus and m a coefficient which depends on the work hardening factor n of the material. For low hardening m is about 1.5 [8,9].

σ_y for recrystallized and stress relieved W as a function of the temperature are shown in Fig. 4 as well as in Table 2 which show the expected decrease of the yield strength with increasing temperature [10].

Table 3 shows fracture toughness values of CT and 3 PB specimens tested at room temperature. The two letter code in the brackets describes the crack plane orientation of different specimens with respect to the geometry of the manufactured material. The first letter designates the *direction normal* to the crack plane, and the second letter the *expected direction of crack propagation* [11]. φ is the technically degree of deformation, referring to a rolling process, given by $\Delta A/A_0$ where ΔA is the cross-sectional reduction in area and A_0 is the original area of the cross-section. Due to the extreme differences of the determined K_Q values, the processing route seems to have a great influence on the results as well as the direction the specimens have been manufactured out of the rods. While sintered materials have equiaxed

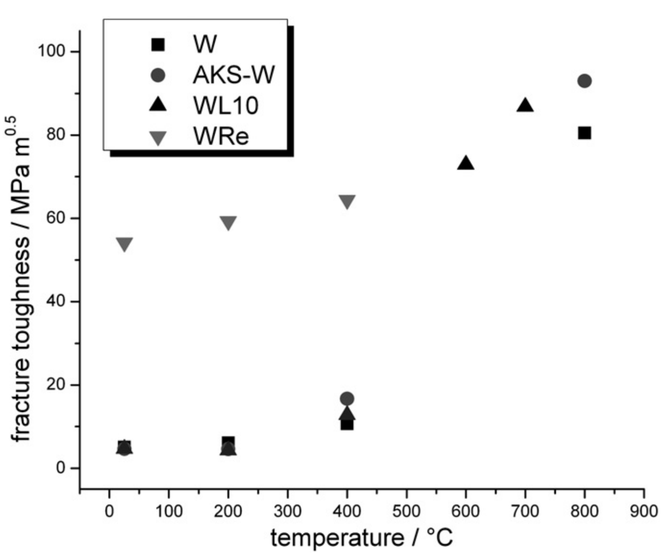

Fig. 3. Fracture toughness K_Q of W, AKS-W, WL10 (sintered) and WRe26 (rolled and stress relieved) as a function of temperature T. All values above 400 °C have been calculated from their COD_C values using Eq. (1).

Table 1
Table of K_Q values of W, AKS-W, WL10 (sintered) and WRe26 (rolled and stress relieved) tested in the temperature range from room temperature to 800 °C.

Temperature [°C]	Fracture toughness [MPam$^{0.5}$]			
	W	AKS-W	WL10	WRe
RT	5.1	4.7	4.7	54.2
200	6.1	4.6	4.3	59.3
400	10.7	16.7	12.8	64.4
600	–	–	72.9[a]	–
700	–	–	86.3[a]	–
800	80.5[a]	93.0[a]	–	–

[a] These K_Q values have been estimated from the COD_C values using Eq. (1).

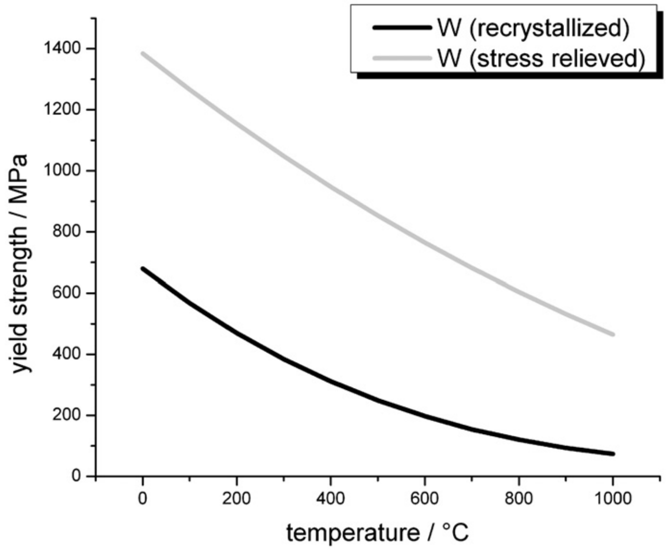

Fig. 4. Yield strength σ_y as $f(T)$ of recrystallized and stress relieved W [10].

grains and therefore the direction of the manufactured specimens doesn't play a role, especially rolled rods show very often large differences in K_Q for different crack propagation directions as a result of the elongated grains. An increasing degree of deformation leads to a stronger elongation of the grains. This makes it much easier for a crack to propagate along the axis of a rod than perpendicular to it. Additionally, it has to be mentioned that different specimens from the same rods and the same crack propagation direction sometimes show large differences. This is probably an effect of the location of the tested volume in the rod, as the texture changes from the center to the edge and also becomes more pronounced the thinner the rods are.

3.2. Examination of the local variation of fracture resistance

For a better understanding of the local variation of the fracture resistance, the DCB-specimens were tested with a microtensile-testing or a universal-testing machine as mentioned earlier. The crack path was recorded by EBSD. In the case of the potassium doped tungsten tested at *RT*, the inverse polefigures (IPF) and fracture surfaces show two types of fracture behavior (Fig. 5). On the one hand the crack propagates intergranular (ig) and on the other hand also transgranular (tg), though the fraction of transgranular crack propagation prevails. Orientation changes within single grains in the interior of an intergranular propagated crack (Fig. 5-a) as well as changes within grains in the case of a transgranular propagated crack (Fig. 5-b) were observed and are clearly viewable due to the gradual change in color in both pictures of Fig. 5. Such deformations are not frequently observable and are just very localized along the crack path of a *RT*-tested specimen.

Fig. 6 shows the correlation between the change in orientation and plastic deformation. A narrow band of geometrically necessary dislocations (GND) – generated by the crack tip – is arranged one after the next in a small angle tilt boundary. The volume elements below the boundary are rotated with respect to the elements above. This rotation can be associated with a tilt of crystals and can be used to determine GNDs [12].

Table 2
Yield strength σ_y of recrystallized and stress relieved tungsten as a function of the temperature in the range from room temperature to 1000 °C [10].

Temperature [°C]	Yield strength [MPa]	
	W (recrystallized)	W (stress relieved)
0	680	1385
200	470	1154
400	311	948
600	197	765
800	120	604
1000	74	465

Table 3
K_Q values of W and W-alloy specimens tested at room temperature.

Material	Condition	Rod ϕ [mm]	φ [%]	Tests performed	K_Q [MPam$^{0.5}$]
W	As sintered	23	0.0	CT	5.1
	Rolled	9	84.7	CT (C-R)	4.7
	Forged	25	75.9	3PB (L-R)	8.0
	Rolled	9	84.7	3PB (L-R)	9.1
	Rolled	4	97.0	3PB (L-R)	5.4
	Rolled and drawn	1	99.8	3PB (L-R)	35.1
WL	As sintered	23	0.0	CT	4.7
	Rolled	9	84.7	CT (C-R)	6.0
	Forged	25	76.9	3PB (L-R)	16.6
	Rolled	9	84.7	3PB (L-R)	9.8
	Rolled	4	97	3PB (L-R)	9.7
AKS-W	As sintered	23	0.0	CT	6.4
	Rolled	9	84.7	CT (C-R)	4.5
	Rolled	9	84.7	3PB (L-R)	32.1
	Rolled	4	97	3PB (L-R)	13.5
	Rolled and drawn	1	99.8	3PB (L-R)	32.1
WRe (26%)	Forged	25		3PB (L-R)	54.2
	Recrystallized	18		CT (C-R)	22.8

Measurements of the misorientation in the case of the intergranular propagated crack show a maximum value of about 4° whereas the transgranular propagated crack leads to a maximum orientation change of about 5°.

The DCB-specimens tested at elevated temperatures are shown in Fig. 7-b/-c and compared with the specimen tested at *RT* (Fig. 7-a). It can be seen that the amount of plastically deformed areas within single grains does not significantly increase from a *RT*-tested specimen to a 300 °C-tested specimen or a 600 °C-tested specimen. Misorientation measurements within the grains also show that the values always are in the range between 4 and 6°. However, the frequency of deformed grains is increasing with increasing temperature. In the case of *RT* and 300 °C-tested specimens the deformation was just observed directly along the crack path, whereas the 600 °C-tested specimen showed plastically deformed areas also in a wider vicinity of the propagated crack.

The compact tension specimens manufactured out of a WRe26 alloy were fractured at room temperature and at elevated temperatures. Fig. 8 shows scans of all specimens after fracture. Compared to the AKS-W-specimens these IPF show many more plastically deformed areas. In the RT-tested specimens (Fig. 8-a) plastically deformed grains were observed just in the direct vicinity of the propagated crack. The 300 °C

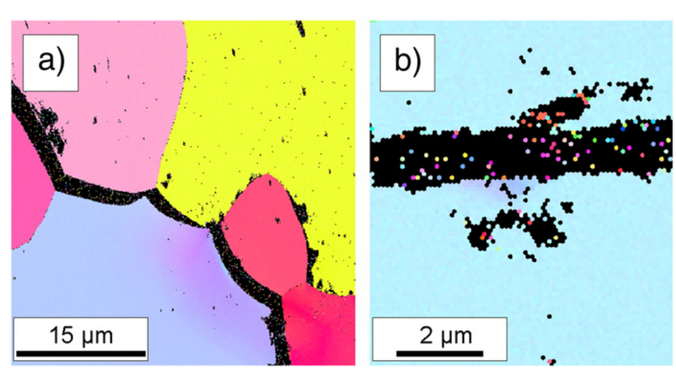

Fig. 5. Intergranular (a) and transgranular (b) propagated crack of a DCB-specimen manufactured out of an AKS-W-rod and tested at *RT*.

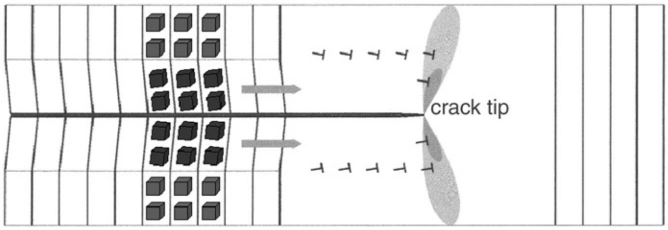

Fig. 6. The propagating crack generates a band of geometrically necessary dislocations (GND). These GNDs have an effect similar to a series of small angle boundaries. The rotation of the crystals can be used to determine the density of the GNDs and the size of the plastic zone. Additionally, they can be used to estimate the local plastic work spent during crack propagation.

and 600 °C-tested samples (Fig. 8-b and -c) also show plasticity in the wider vicinity of the crack flanks. Measured misorientations of about 10 degrees for the 300 °C and about 14° for the 600 °C-tested specimens are much higher than the values obtained from the AKS-W-specimens.

In summary, a comparison of the EBSD analyses shows that recrystallized tungsten and tungsten alloys mainly cleave intergranularly at room temperature. However, they have a small amount of transgranular cleavage as well. EBSD analyses, taken along the crack path of the propagated crack, show plastic deformation close to the crack flanks only at selected places. This occurs in both transgranular and intergranular fracture. Most plasticity can be found at crack bridges which occur along the whole crack path. This indicates an extreme variability in deformation during cleavage processes at low temperatures. With increasing temperature the amount of plastic deformation rises which is clearly visible in the EBSD data. Plastic deformation can be seen along the whole crack path, in both intergranular and transgranular fractured grains. Grains neighboring the propagated crack show plastic deformation as well as grains in the wider surrounding of the crack path. This indicates a much higher amount of deformation during propagation of the crack, which can also be seen in the higher fracture toughness values. Compared to all other tungsten materials tungsten rhenium alloys show significantly higher amounts of plastic deformation, even at room temperature.

In order to quantify the effect of locally varying fracture resistances, further investigations on different microstructures will be performed and described in a forthcoming paper.

4. Conclusion and summary

The fracture toughness of pure polycrystalline tungsten, potassium doped tungsten, tungsten with 1 wt.% La_2O_3 and tungsten rhenium alloys were investigated by means of 3-point bending, double cantilever beam and compact tension specimens. Tests were performed in the temperature range of −196 °C to 1000 °C and the obtained values show the expected increase of fracture toughness with increasing temperature. However, the values obtained in the low temperature regime show a particularly strong dependence on the microstructure and therefore the production route.

Investigations of fracture surfaces, as well as EBSD-scans of propagated cracks, always show intergranular as well as transgranular fracture of all tested materials, although intergranular fracture behavior is dominant in most cases. Detailed EBSD investigations along the crack path show plastically deformed areas within single grains for both types of fracture. Tests performed at room temperature show such plasticity only locally while experiments at elevated temperatures lead to a greater amount of deformed areas in the grains neighboring the crack and also grains in the wider vicinity of the crack.

Fracture toughness tests of tungsten rhenium alloys show significantly higher values than all other tungsten materials. EBSD-scans along the crack path of the propagated crack show very pronounced plastic deformation even at room temperature.

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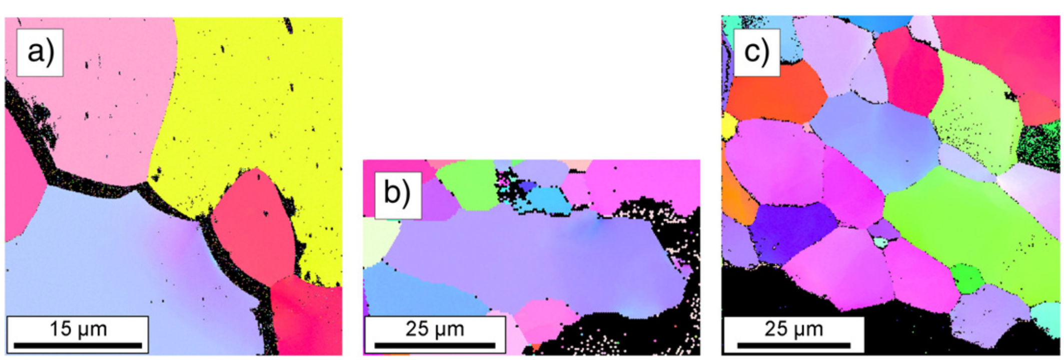

Fig. 7. Plastically deformed areas along the crack paths of potassium doped tungsten DCB-specimens tested at *RT* (a), 300 °C (b) and 600 °C (c).

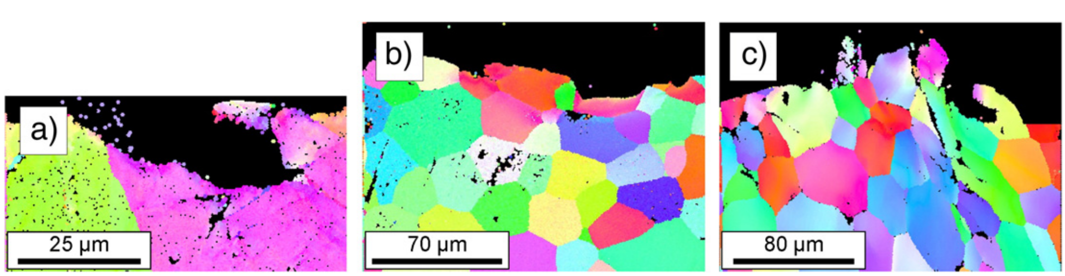

Fig. 8. Plastically deformed areas along the crack paths of tungsten rhenium CT-specimens tested at *RT* (a), 300 °C (b) and 600 °C (c).

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