Orientation dependence of the fracture behavior of single-crystal tungsten

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Abstract

Polycrystalline tungsten at room temperature shows a brittle fracture behavior, which is also strongly influenced by the grain structure and texture as well as sample dimensions. To gain insight into the mechanical response of individual grains, an experimental program has been set up to test small scale samples under microbending starting with a notched tungsten single crystal oriented with the \{110\}<110> crack system along the loading direction. Related to this experimental program a finite element study has been performed to analyze the crack propagation in such single-crystal tungsten micro cantilevers. The aim of the present numerical work is to investigate the influence of the single-crystal orientation on the fracture process.

A finite element (FE) model of the notched microbeam was created taking plastic deformation at the crack tip into account. Plastic deformation is implemented using a crystal plasticity approach formulated by Asaro (1983) and written by Huang (1991). Furthermore, the fracture process with crack propagation is described by a cohesive zone model. The simulations of microbending allow for evaluating the details of the fracture process more accurately. The results reveal details of the developing plastic zone as well as the current crack propagation and the $J$-integral in dependence of the crystal orientation and notch geometry.

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Keywords: Single-Crystal Tungsten, Microbeam, Fracture Toughness, FE Model, Crystal Plasticity, Cohesive Zone Model

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1. Introduction

Tungsten has been used as a functional material in the lighting industry. Due to its distinct properties it shall be applied as a structural material in future energy applications. Its main advantages are the very high melting point, the high Young’s modulus, the high density and the good thermal conductivity. Improvement of the fracture toughness represents one of the challenges due to the brittle-to-ductile transition of tungsten above room temperature.

Rupp et al. (2010) as well as Gludovatz et al. (2010) found a strong influence of the microstructure on the fracture morphology and toughness as well as on the brittle-to-ductile transition temperature in polycrystalline tungsten. The same holds for tungsten single crystals; Riedle (1996) and Gumbsch (2003) found that the fracture toughness of tungsten single crystals varies from 6.2 to 20.2 MPa m$^{0.5}$ by changing the crystal orientation. Until now, investigations have mainly been carried out on the macro-scale. Fracture studies using micro-specimens are very rare with few exceptions such as Wurster et al. (2010 and 2012).

To deepen the insight into the fracture mechanisms in tungsten, an experimental programme has been setup where single-crystalline notched micro-cantilevers are bent with a nanoindenter, performed by Schmitt et al. (2013).

2. Experimental and Simulation Approach

2.1. Micro-cantilever bending

To determine the fracture toughness free-standing single crystal micro-cantilevers are used. At the scale the experiments are conducted, the sample geometry (e.g. the crack ratio $a/W$) is limited by the manufacturing process and, thus, the requirements of the ASTM standards (ASTM E399 and E1820) cannot be completely fulfilled. In relation to the cantilever dimensions the plastic zone is very large. The geometry, which was developed, is somewhat related to the specifications of the ASTM standard and allows the preparation of different notch shapes. As schematically shown in Figure 1, the width $W$ of the cantilever is 40 $\mu$m, the thickness $B_0$ 22 $\mu$m, the length $H$ approximately 150 $\mu$m. The crack length $a$ is typically 10 $\mu$m.

In the present paper, two different notch types are analyzed: a V notch and a Chevron notch. In the latter case, stable crack growth is expected after crack initiation.

To manufacture several free-standing micro-cantilevers in a row, tungsten single crystals of 3x3x5 mm$^3$ size were aligned in a specific crystal orientation. The basic sample geometries are carved by a micro electrical discharge machining ($\mu$-EDM) process. The notch is then prepared with a focused ion beam (FIB) perpendicular to the surface. Finally, the gage section is milled with the FIB to a smooth surface finish as shown in Figure 2. Due to the manufacturing process the final geometry differs from the original design dimensions and is inspected by scanning electron microscopy (SEM) prior to testing. More details about the preparation steps are given by Schmitt et al. (2013). In the present experimental study, the specimens are orientated in such a way that fracture occurs in the $\{110\}<11\bar{2}>$ crack system.
A commercially available nanoindenter (Agilent G200) is used to bend the cantilever at a defined distance from the notch. Numerical studies with different indenter tip geometries have shown that the wedge indenter is most suitable (Bohnert et al. (2013)). This is due to the fact that localized penetration is prevented with a wedge tip. In the displacement-controlled bending test, the displacement of the wedge indenter tip and the necessary force are recorded simultaneously. The broken cantilevers are further analyzed using a SEM.

2.2. FE-Modelling

The notched micro-cantilever is represented by a three-dimensional finite element (FE) model shown in Figure 3. Due to the symmetry of the crystal lattice and of the geometry only half of the specimen is modeled and meshed with 8-node brick elements with linear function (C3D8). The specimen is in contact with the wedge indenter which is modeled as a rigid body. The friction coefficient between specimen and wedge is assumed as 0.2.

The deformation behavior is described with the rate-dependent formulation of crystal plasticity by Asaro (1983) and implemented in the commercial finite element code ABAQUS as user material subroutine by Huang (1991). In this constitutive law plastic deformation is the result of slip on distinct slip systems. The driving force for crystallographic slip is the resolved shear stress on the slip plane in slip direction (Schmid stress). If this stress reaches a critical value, yielding starts (Schmid 1931) followed by hardening. This so called Schmid law describes the deformation of face-centered single crystals very well while in body-centered materials also deviations from this law have been observed (Argon et al. (1966)). However, in this work possible non-Schmid effects are neglected and the deformation of tungsten is assumed to be governed by slip on \{110\}<\{111\> slip systems.

The implemented material parameters are taken from literature (Yao (2012)). Based on experimental microindentation tests on tungsten single crystals, Yao (2012) identified the initial hardening modulus $h_0 = 1350$ MPa, the initial yield stress $\tau_0 = 210$ MPa and the stage I stress $\tau_s = 530$ MPa which are used in the present work.

To enable crack initiation and propagation, a cohesive zone model (CZM) was implemented in the finite element code as user defined finite elements (UEL) developed at GKSS Research Centre Geesthacht by Scheider (2006). They are marked in Figure 3 as blue zone ahead of the crack plane. A partly constant traction separation law (TSL) with two additional shape parameters $\delta_1$ and $\delta_2$ was chosen to mimic the fracture process at the crack tip. The main parameters of the separation law are the cohesive strength $\sigma_0$ and the critical separation $\delta_c$. They are related to the separation energy $\Gamma_0$ via

$$
\Gamma_0 = \sigma_0 \delta_c \left( \frac{1}{2} - \frac{1}{3} \frac{\delta_1}{\delta_c} + \frac{1}{2} \frac{\delta_2}{\delta_c} \right)
$$

(1)

with the shape parameters defined by $\delta_1/\delta_c = 0.01$ and $\delta_2/\delta_c = 0.75$ (Scheider 2006). A reasonable value for $\Gamma_0$ can be taken from fracture tests performed by Riedle (1996) on the macro-scale. In the present work, two crystal orientations (CO) were studied in more detail, namely CO1 with the crack system \{011\}<\{011\> and CO2 with the crack system \{100\}<\{011\>. Based on Riedle (1996), for CO1 and CO2, the fracture toughness $K_{IC}$ was 12.9 MPa m$^{0.5}$ and 6.2 MPa m$^{0.5}$, respectively. Since the separation energy should not vary with crystal orientation, the average toughness value of $K_{IC} = 9.5$ MPa m$^{0.5}$, which corresponds to a separation energy for the cohesive zone of $\Gamma_0 = 0.2$ N/mm, was used in this study. This value is kept the same in all simulations while the cohesive strength is varied from 750 MPa (about 1.8 times the macroscopic yield stress $\sigma_Y$) to 1500 MPa ($\approx 3.6 \sigma_Y$).

Fig. 3. Three-dimensional finite element model of the micro-cantilever illustrating the mesh and the boundary conditions.
3. Results

The presented model was applied to simulate displacement-controlled micro-bending tests of differently orientated tungsten cantilevers with two different notch geometries. In these numerical experiments the wedge indenter was placed 110 μm from notch and moved with a speed of 20 nm/s in the negative z-direction (Figure 1).

3.1. Crystal orientation and notch geometry – a parameter study

Two crystal orientations were studied, i.e. {011}<011> (CO1) and {100}<011> (CO2). In the first analysis the notch is a V-notch with a length \( a_0 = 10\mu m \) \((a/W = 0.25)\). Figure 4 shows the resulting force-displacement curves for these two crystal orientations and for a cohesive strength \( \sigma_0 \) varying from 750 MPa to 1500 MPa. In addition, the gray curves stem from simulations without CZM and crack propagation. In specimens with the crystal orientation CO2 a lower force \( RF \) is needed to obtain the same displacement \( u \) of the cantilever. This is due to the fact that in CO2 the slip systems are oriented more favorably leading to yielding at a lower stress level, see gray curves. Figure 4 reveals that an increasing cohesive strength \( \sigma_0 \) leads to a higher force \( RF \). If the cohesive stress \( \sigma_0 \) is reached in a CZM, the fracture process starts leading to a lower stiffness of the specimen which results in a decrease of the reaction force \( RF \). As expected, both crystal orientations show the same behavior in this respect.

As in the experimental program it has not been possible to introduce a sharp crack via fatigue, another notch geometry has been investigated, namely a Chevron notch (Schmitt (2013)), and also implemented in the numerical studies. This type of notch leads to a more controlled crack growth as the resistance against crack propagation increases with increasing crack length. The simulations were repeated for a specimen with a Chevron notch of crack length at surface \( a_{SUR} = 10 \mu m \) with an angle of 50°. Figure 5 shows the resulting force displacement curves obtained for CO1 and for various cohesive strengths. As the average crack length is smaller, the Chevron notched micro-cantilevers have a higher force level compared with the V-notched cantilevers. In addition, all curves show a strong force reduction after crack initiation.

3.2. Crack growth analysis

One advantage of the present simulations is that the crack can also be observed “inside” the material. We picked the three simulations with a cohesive strength of 1500 MPa and analyzed the crack growth in these three specimens: A (V-notch, CO1), B (V-notch, CO2) and C (Chevron, CO1), see Figure 6.

The point of crack initiation is marked as (1) in the force-displacement curves (Figure 6a).
Furthermore, Figure 6b) shows the crack growth process by means of several crack paths (1-6) owing to increasing indenter displacement. In these three specimens the following characteristics can be observed:

A) As also observed experimentally, the crack initiates in the center of the sample. This is due to the fact that in the bulk of the cantilever plane strain conditions dominate leading to higher stress triaxiality compared to the surface where plane stress conditions occur. The significant advancing crack in the sample center is characteristic for this crystal orientation CO1.

B) Again, first cohesive zone elements fail in the sample center. Compared to A, however, the crack grows slowly with increasing indenter displacement and the difference in growth between center and surface is not as distinct.

C) Contrary to the expectation, the crack does not start in the symmetry plane. The initial crack front has not been modelled straight but owing to variation in the manufacturing process a bit wavy. At this weak point the crack initiation takes place. As soon as the crack reaches the sample center, a large number of elements fail instantaneously resulting in a strong reduction of the force. As Figure 6b) shows, this results in a straight crack (marked as (3)) which propagates similar to A with increasing indenter displacement.

3.3. Crack resistance by J-R curve

The J-integral can be determined from the force-displacement curve according to ASTM E1820. The crack growth Δa is determined for increments by averaging over the total area of “broken” cohesive elements divided by the current B. The corresponding J-Δa or J-R curves are shown in Figure 7 for V-notched cantilevers and two crystal orientations. Due to the cohesive energy value $\Gamma_0 = 0.2$ N/mm used in all simulations crack initiation occurs at a J-value of 0.2 N/mm in all investigated cases.
The $J$-$R$ curves are approximately identically for both crystal orientations in case of the low cohesive strength of 750 MPa. The cohesive strength is easily reached ahead of the crack tip; the cohesive elements begin to separate while no further slip activities are needed in the surrounding material. This is indicated by the rather flat $J$-$R$ curve.

In case of higher cohesive strength (1500 MPa), slip activities play a more important role and the curves of CO1 and CO2 deviate. Figure 6 shows the accumulated slip on all slip systems at the surface of CO1 and CO2 at an average crack growth of 0.5 $\mu$m. It reveals that more plasticity occurs in CO2 leading to a higher value for $J$. Thus it becomes clear that the crystal orientation has an influence on the fracture toughness of single crystal tungsten.

4. Conclusion

In this paper, we presented a three-dimensional finite element model of a notched single crystalline tungsten micro-cantilever closely resembling actual samples tested in a parallel experimental program. The cantilever is deflected by a nanoincidenter equipped with a wedge tip. In this model, crack initiation and crack growth are handled with cohesive zones (CZM) while deformation is described with crystal plasticity (CP). The model was applied to study the influence of cohesive strength, different crystal orientations and different notch geometries on the fracture behavior. First, the computed force-displacement curves show that there is a direct influence of the crystal orientation: the $\{110\}<11\overline{0}>$ crack system orientation leads to higher force levels compared to the $\{100\]<011> orientation. Second, the notch geometry also influences the $F$-$U$ curves especially after crack initiation. Here, the Chevron notch results in a stronger force reduction in comparison with the V-notch. Another result is the influence of the slip activities on the $J$-integral. It was shown that this increase due to plasticity strongly depends on the crystal orientation.

In future, the material parameters for the CP and the CZM will be estimated based on experimental observations from tensile tests on unnotched and notched small-scale specimens. After the parameter validation ($F$-$U$- and $J$-$R$-curve), the model shall ultimately be used to describe deformation and fracture of tungsten at different length scales.

Acknowledgements

The authors would like to thank the Karlsruhe University of Applied Sciences and Karlsruhe Institute of Technology (KIT) for providing the necessary equipment.

The research was financially supported by a fellowship of the “Kooperatives Promotionskolleg: “Gefügestrukturenalyse und Prozessbewertung” funded by the Postgraduate Research Grants Programme of Baden-Württemberg (Germany).

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