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Size-dependent fracture toughness of tungsten

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ABSTRACT

In order to understand the size-dependent fracture behaviour of tungsten at the microscale, micro-cantilever tests were performed on single crystalline and ultrafine grained tungsten. The elastic-plastic fracture behaviour and crack tip plasticity were found to be strongly influenced by the sample size. The smallest cantilevers with dimensions below one micron failed by pure cleavage fracture at a fracture toughness of 1.5 MPa $m^{1/2}$, which agrees well with the Griffith theory for brittle fracture. With increasing specimen size crack tip plasticity was observed, leading to a successive increase in fracture toughness and crack resistance behaviour. Using scanning transmission electron microscopy and electron backscatter diffraction, dislocation densities were analysed and measured. Pronounced plastic strain gradients were found at the crack tip for an intermediate specimen size, which also showed the highest crack resistance. When further increasing the specimen size, large plastic zones were still observed but the gradients in plastic strain vanished, leading to a reduction in the crack resistance behaviour. For those samples, a size independent fracture toughness of ca. 7 MPa $m^{1/2}$ was found, which is close to macroscopic literature data. Increasing the initial dislocation density in the single crystals through plastic pre-straining led to a reduced stable crack growth behaviour but did not affect the fracture toughness. Furthermore, the effect of grain boundaries on the fracture behaviour was studied by means of ultrafine grained tungsten. A fracture toughness lower than the one reported in literature was found, which is explained by the higher susceptibility to failure once a crack propagates at the micro-scale.

1. Introduction

The mechanical behaviour and fracture properties of W are well studied in literature and the material often serves as a model material due to its limited number of active slip systems and the elastically isotropic behaviour. Riedle et al. [1] studied the intrinsic fracture process for specific cleavage planes using macroscopic 3-point bend tests. In addition, the brittle-to-ductile transition was elaborated in detail for W single crystals [2]. However, little is known about the fracture behaviour of materials with limited plasticity at the micron length scale, where size effects are known to influence the plastic behaviour and are supposed to influence the fracture behaviour and fracture toughness. Testing methods for assessing the small scale fracture behaviour have been successfully developed and validated for brittle materials and interfaces [3–5]. Generally, micro-cantilevers with various shapes and FIB-notches are used in these studies, but double-cantilevers [6], pillars [7] and notched bulge membranes [8] can also be applied. Recent work on the intermetallic compound NiAl, which also shows limited plastic deformation depending on the investigated crack system, has shown that the fracture toughness is not size-affected even down to the micron length-scale [9–11]. This behaviour was discussed as being caused by the mainly brittle behaviour of NiAl and possible size effects in the yield strength leading to a reduction in the size of the plastic zone. It is known from experiments and simulations on un-notched micro-cantilevers with similar dimensions of a few microns that the yield and flow stresses increase with decreasing sample dimensions [12–14]. Consequently, the developing plastic zone at the crack tip of a notched cantilever of the same material is supposed to be more confined in micro- than in macro-scale specimens. In work by Preiβ et al. [15] the fracture toughness of freestanding gold thin films with thicknesses of a few hundred nanometres was investigated and measured to be ca. 2 MPa $m^{1/2}$. A significantly more brittle behaviour was therefore

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found in comparison to the fracture behaviour of bulk samples. The authors attributed this to the inherent effect of film thickness, which causes a highly localized plastic zone.

Fracture experiments on W at the micro-scale are also reported in literature. Wurster et al. [16] were the first to develop a J-Integral procedure adapted to the micro-scale, in order to analyse semi-brittle fracture processes and thus calculate the fracture toughness. Combined numerical and experimental investigations on W single crystals were performed by Bohnert et al. [17] and Schmitt et al. [18] by means of micro-cantilevers. The aim was to determine the fracture toughness and to describe the local fracture behaviour of their specifically oriented specimens. Armstrong and co-workers studied intensively the mechanical properties of ion-irradiated W [19]. They also investigated the fracture properties and found a clear correlation between the applied ion implantation conditions and the fracture toughness, which was in the range of 5–10 MPa $m^{1/2}$.

The present study focusses on the mechanisms linked with crack tip plasticity in small-scale W specimens. According to Irwin [20], the size of the plastic zone in a plane-strain stress state in front of a crack tip is given by:

$$r_{pl} = \frac{1}{3\pi}\left(\frac{K_I}{\sigma_y}\right)^2 \tag{1}$$

The macroscopic yield stress σ_y and the stress intensity factor K_I determine the formation of the plastic zone decisively. For large cantilevers, the plastic zone covers only a small portion of the sample volume (cantilever 1 in Fig. 1a). With decreasing sample size and a size independent yield stress, this plastic zone expands through a significant portion of the specimen (cantilever 2). Finally the plastic zone spreads over the whole specimen upon further miniaturization, so that dislocations are able to leave the crystal at all free surfaces.

It is expected and has been often shown that the measured fracture toughness first increases as the specimen size decreases, since the plastic zone is confined by the sample dimensions and consequently has a stronger shielding effect on the crack tip. This is also generally observed when going from a plane strain to a plane stress state [21]. However, in micro-cantilever testing all dimensions are equally decreased and not only one (generally the thickness). The critical height W_{crit} according to the ASTM standard E 399 [22] is approximately 25 times larger than the plastic zone size. However, if the specimen size is so small that most dislocations emitted from the crack tip are directly slipping through the whole sample and leaving the crystal at the surface, then they can have no shielding effect on the crack tip and correspondingly the fracture toughness decreases again, finally reaching the very low level of the Griffith fracture toughness. Such a behaviour has also been found in thin films [15].

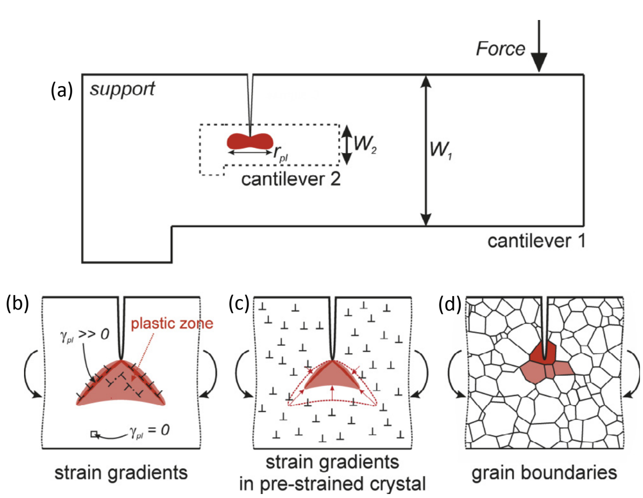

Fig. 1. Schematic presentations for the description of the plastic zone ahead of the crack tip in a loaded cantilever: (a) "classical" model according to Irwin [20] applied to two sample sizes and effects of (b) plastic strain gradients, (c) plastic strain gradients and a high initial dislocation density and (d) grain boundaries on shape and size of the plastic zone for specimens with dimensions below a critical height W_{crit}.

For small-scale samples, microstructural effects in combination with inhomogeneous loading conditions and strain gradients must also be considered. Plastic strain gradients acting at the crack tip and creating large shear strains γ_{pl} (Fig. 1b) can become important once the sample dimensions are significantly decreased [9,23,24]. These strain gradients can influence the development of the plastic zone and may significantly change the stress state in micron-sized samples [25]. Furthermore, an increased inherent dislocation density can additionally effect the expansion of the plastic zone by interacting with dislocations, which are emitted from the crack tip or from dislocation sources close to the crack tip as shown in Fig. 1c. Grain boundaries acting as obstacles for dislocation motion, as schematically depicted in Fig. 1d, also affect crack tip plasticity and are known to strongly influence the fracture behaviour in micron-sized specimens [26].

Thus, a detailed analytical and mechanical understanding of deformation mechanisms linked with fracture in micro-scale specimens is still lacking. This study focuses on the size dependent fracture behaviour of the transition metal W by means of notched micro-cantilevers. The influence of the specimen size, ranging from some tens of microns down to the submicron regime, is systematically studied. The recently presented continuous J-Integral approach was chosen to determine continuous R-curves [11]. This allowed for an accurate analysis of first crack growth events, which is important for the determination of the fracture toughness and the crack resistance behaviour. Scanning transmission electron microscopy (STEM) and electron backscatter diffraction (EBSD) measurements were performed after testing to analyse dislocation arrangements at the crack tip and to explain the observed fracture behaviour. Furthermore, the influence of a pre-existing dislocation density produced by plastic pre-straining on the fracture properties was studied. Ultrafine grained W with many grains along the crack front was investigated to gain insights into the interplay between grain boundaries and the onset of fracture. Finally, experiments with un-notched micro-cantilevers were performed to obtain a better estimate for the yield stress of W in micro-bending, which is needed for the assessment of the theoretical size of the plastic zone.

2. Experimental

2.1. Material and sample preparation

Tungsten was investigated in three different microstructural states as presented in Table 1. The fracture behaviour of single crystals (SX) was studied focussing on the ⟨100⟩{100} crack system, where ⟨100⟩ denotes the crack propagation direction and {100} the crack plane. Cantilevers with different sizes were tested to investigate a size-dependent fracture behaviour. A sample, denominated as "SX-pl.", was pre-strained to 11% plastic strain in a macroscopic compression test prior to micro-cantilever preparation resulting in a dislocation density of ca. 1.0×10^{14} m^{-2}. This was done in order to explore the effect of a pre-existing dislocation density on the fracture process. Furthermore, an ultrafine grained (UFG) sample of commercial purity and produced by high pressure torsion was also investigated. Details on the fabrication process leading to a homogeneous microstructure with a median grain size of ca. 790 nm