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Fracture toughness characterization of single-crystalline tungsten using notched micro-cantilever specimens

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The fracture toughness of tungsten is strongly influenced by microstructural features including crystal orientation and grain structure as well as specimen or component size. In order to gain insight into the mechanical response of individual grains, an experimental study using small scale fracture specimens was conducted and closely accompanied by finite element simulations. Free-standing notched single-crystalline tungsten micro-bending beams with an orientation of the $\{011\}\langle 0\bar{1}1\rangle$-crack system along the loading direction were loaded using a nanoindenter and modelled using crystal plasticity and cohesive zones. This combined numerical and experimental approach successfully demonstrates a procedure to determine the fracture toughness of such small non-standard specimens despite the occurrence of plastic deformation.

1. Introduction

Tungsten as the metal with the highest melting point has a number of outstanding mechanical and physical properties and is considered as a structural material in current and future high-temperature energy conversion systems. In those applications, material robustness, i.e. high strength in combination with sufficient toughness, is absolutely critical. Therefore, improving the fracture toughness of components made from tungsten represents the most important challenge since the brittle-to-ductile transition of tungsten is well above room temperature.

Previous studies have shown that the fracture toughness of single-crystalline tungsten depends on the crystal orientation and the active crack system (Riedle et al., 1996; Gumbsch, 2003). For polycrystalline samples, it was demonstrated that the toughness strongly depends on microstructural characteristics such as grain size and shape, and texture (Gludovatz et al., 2010; Rupp and Weygand, 2010) with crack propagation typically observed to occur along grain boundaries. Thus, extruded tungsten with elongated grains is highly anisotropic and exhibits significantly improved toughness for cracking perpendicular to the preferred grain orientation (Riedle et al., 1996). On the other hand, results from bending tests by Reiser et al. (2013) show ductile behavior of ultrafine-grained tungsten at room temperature. It was suggested (Reiser et al., 2013) that the ductility of the tungsten foils is possibly caused by (i) the high amount of mobile edge dislocations, (ii) the ultra-fine grain size, and (iii) the foil effect which leads to dislocation annihilation at the free surfaces. Interestingly, the ductility of the foil disappeared after a heat treatment, which led to recrystallization and grain growth pointing to the ultra-fine grain size as key for the observed behavior. Thus, it can be concluded that grain boundaries are not inherently brittle, and the

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macroscopically observed toughness is the result of the interplay between plasticity within grains and inter- or intra-crystalline crack propagation. In order to gain a deeper fundamental understanding of these mechanisms, the study of the fracture behavior of bi-crystalline sample is desirable. However, these should be conducted at an appropriate scale since size effects are known to strongly influence the deformation behavior in general (Kraft et al., 2010) and for the case of body-centered cubic (bcc) metals in particular (Kaufmann et al., 2011). Furthermore, for un-recrystallized swaged, extruded or rolled cylindrical rods made from tungsten, the grain size is typically of the order of several tens of microns in the elongated direction with an aspect ratio of the order of 4:1 (Margevicius et al., 1999), which requires fracture studies at this length scale and with a view to the application a micromechanical framework for describing the fracture behavior of tungsten.

In the literature, measurements of the fracture toughness on brittle materials have been reported using small-scale beam bending and focused ion beam-prepared notches since the last century. Notch geometries included double edge notches (Kraft et al., 1998) as well as top-down straight notches (Takashima et al., 2001). More recently, chevron notches were suggested as a means to reduce Ga contamination from the ion milling process and to promote a more stable crack growth (Mueller et al., 2015; Schmitt et al., 2013). Ion milling from the side represents another approach to minimize Ga implantation in the region of the crack (Kupka and Lilleodden, 2011). However, only few fracture-mechanical studies of metallic samples at the micro-scale have been reported. Wurster et al. (2012) performed fracture experiments using micrometer-sized notched cantilevers to study the semi-brittle fracture behavior of single-crystalline tungsten. They concluded that the behavior of micro-samples is more ductile with higher fracture toughness values compared to macro-sized single crystals. Similar conclusions were drawn by Ast et al. (2014) for NiAl. The fracture behavior of individual grain boundaries of an aluminum alloy was recently studied by combining crystal plasticity finite element simulation with cohesive zones and focused ion beam prepared microcantilevers (Kupka et al., 2014).

In this work, we combine experimental and numerical investigations in order to study the toughness of single-crystal small-scale tungsten samples. The development of the test procedure involves a new two-step fabrication procedure based on electrical discharge and focused ion beam machining. As an alternative to straight notches as used in Ast et al. (2014); Wurster et al. (2012), chevron notches were prepared, which offer, in particular at a small scale, the advantage of steady-state crack initiation and more controllable crack propagation under plane-strain conditions. In such small-scale fracture experiments the plastic deformation of the specimen during fracture cannot be neglected. To be able to distinguish between the contribution of plasticity and the contribution of cracking a finite element (FE) model was set up including crystal plasticity and cohesive zone elements. The comparison of the experimental results with the numerical ones allows for determining the toughness as well as orientation-specific effects of single-crystal tungsten for the crack system $\{011\}\langle 0\bar{1}1\rangle$.

2. Experimental approach

2.1. Method

Measuring the fracture toughness at the micrometer length scale requires a customized testing procedure, which differs from the standard fracture tests. Here, free-standing notched micro-cantilevers were tested using a nanoindenter (Nano-indenter G200 XP, Agilent, Inc.). To ensure comparability of the tungsten micro-cantilevers to macro-specimens, the sample geometry was based on the specifications of the standard ASTM sample and downscaled to maintain the proportions between the thickness W and the width B as well as the notch length a (Fig. 1). The notch was positioned close to the fixed end of the cantilever to minimize plasticity in the region between the fixed end and the notch. Two different notch geometries (Fig. 1) were investigated, i.e. the straight-through notch (STN) and the chevron notch (CN) (Schmitt et al., 2013), both experimentally and by simulation.

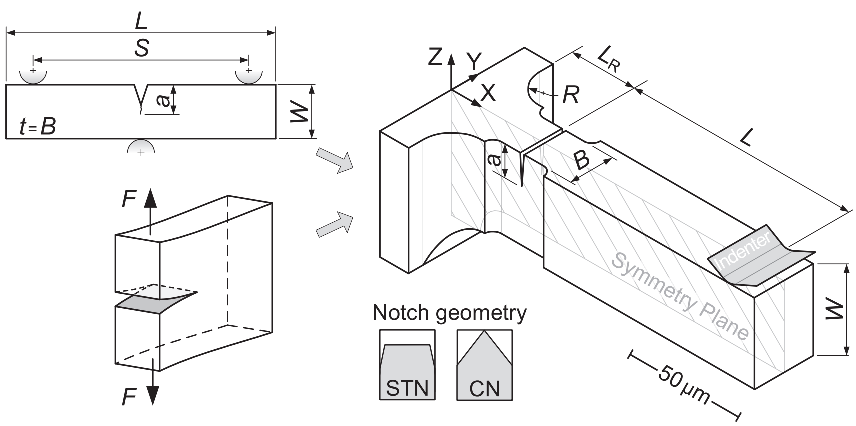

Fig. 1. Free-standing micro-bending geometry based on single edge notch bending mimicking crack opening mode I with straight (STN) and chevron notch (CN) geometries.