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The brittle-ductile transition of tungsten single crystals at the micro-scale

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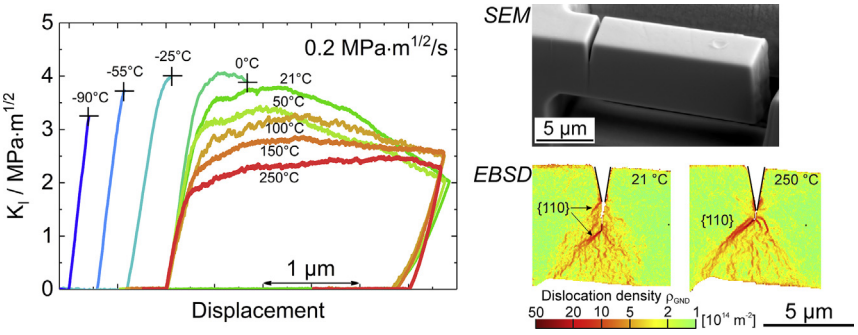
HIGHLIGHTS

- Determination of fracture toughness at the micro-scale as a function of loading rate, temperature and specimen size
- Measurement of J-Δa curves from in-situ micro-cantilever tests performed in the temperature range between -90 °C and 500 °C
- Discussion of activation energy for the brittle-ductile transition with respect to specimen size effects
- Analysis of crack tip plasticity by high-resolution electron backscatter diffraction and transmission electron microscopy

GRAPHICAL ABSTRACT



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ABSTRACT

The loading rate effect on the brittle-ductile transition temperatures of tungsten single crystals at the micro-scale was investigated by microcantilevers with a (100)[001] crack system. Specimens with a length to width to height of 15 µm/4 µm/6 µm were fabricated by focused ion beam milling. At low temperatures (−90 to −25 °C) the samples failed by brittle cleavage fracture, irrespective of the applied loading rate at a fracture toughness of 3.2 MPa·m$^{1/2}$. With increasing temperatures up to 500 °C and depending on the applied loading rate, the fracture toughness increased and significant crack tip plasticity and dislocation-controlled microcleavage were observed by means of high resolution electron backscatter diffraction measurements performed after testing. With respect to macroscopic specimens, a shift of the brittle-ductile transition to lower temperatures and a significantly lower activation energy of the brittle-ductile transition of 0.36 eV were found. We explain this by the increase in flow stresses due to sample size effects.

1. Introduction

Much progress has been made in recent years to understand the brittle-ductile transition (BDT) behaviour of materials. Initially beginning with semiconductors such as Si [1–5], and Ge [6], later investigations also focussed on intermetallics [7–10] and body-centred cubic (bcc) metals like α-Fe [11], Mo [12] and W [13–15]. The underlying deformation mechanisms controlling the transition from cleavage fracture below a critical temperature to effective crack tip shielding and plastic yielding above this temperature are often difficult to capture and sometimes controversially discussed. This is because not all materials show a sharp and distinct transition at a certain temperature like Si. A sharp transition facilitates the determination of the dislocation mechanism as the driving factor of the transition behaviour. Bcc metals show a pronounced loading rate influence on the fracture behaviour, as

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demonstrated in macro-scale experiments by Riedle et al. [13] and Gumbsch et al. [14,16] for single crystal W and supported by numerical simulations by Hartmaier and Gumbsch [17]. Unlike Si, a gradual increase of fracture toughness with temperature is found for W. For the examined (110)[1$\bar{1}$0] crack system, an activation energy of 0.2 eV led to the conclusion that the BDT was triggered by the mobility of edge dislocations. Giannattasio et al. [15] performed a similar study on W single- and polycrystals and calculated an activation energy of 1.0 eV. They concluded that the controlling factor for BDT in W with a (100)[001] crack system is the mobility of screw dislocations, whose motion is rate-controlled by the thermally activated formation of kink-pairs. In additional discrete dislocation dynamics (DDD) simulations by Tarleton and Roberts [18] looking more closely at the plastic zone and crack tip shielding effects, sufficient mobility rather than nucleation of screw dislocations was found to be the predominant mechanism for the BDT.

Small-scale effects resulting from small sample sizes play an important role in BDT behaviour. Korte and Clegg [19] performed micro-pillar compression experiments on ceramic phases that underwent plastic deformation at relatively low temperatures. A size-dependent shift of the BDT to lower temperatures was also found for the semiconductors GaAs [20] and Si [21,22]. The microcantilever testing technique introduced by Di Maio and Roberts [23] offers the great advantage of a local determination of fracture properties and fracture toughness of single phases [24–28], at interfaces [29,30] and grain boundaries [31–33]. The technique was further developed for testing of materials showing non-negligible plastic deformation by Wurster et al. [34]. In further studies on NiAl [35–37], W [38,39] and NiW films [40] fracture toughness determination of materials in the semi-brittle regime was elaborated. In this manner, environmental effects on the local mechanical behaviour could also be studied in detail [41–44].

The influence of temperatures as high as 600 °C on the micro-scale fracture and therefore the BDT behaviour was firstly treated by Jaya et al. [45] in Si using microcantilever experiments. Double-clamped beam bending tests were performed again in Si for temperatures up to 600 °C [46] and microcantilever experiments in Fe single crystals for room temperature and at −75 °C [47] to understand the increase and decrease in ductility in micron-sized specimens, respectively.

Whereas the above-mentioned studies showed the temperature influence on the micro-scale fracture behaviour, a deeper mechanistic understanding of the controlling deformation mechanism is still lacking. The loading rate-effect in combination with temperature influence on the fracture behaviour as shown by macroscopic tests needs further attention to understand thermal processes at the crack tip. It is the aim of the present study to provide a detailed conception of the BDT behaviour of the bcc metal W at the micro-scale in order to clarify the influence of scale-effects on the semi-brittle fracture behaviour. For this purpose we performed microcantilever fracture experiments applying different loading rates in the temperature range of −90 to 500 °C to describe the fracture behaviour and analyse crack tip plasticity using high resolution electron backscatter diffraction (HR-EBSD) and transmission electron microscopy (TEM). The activation energy, which was derived from the BDT in the micro-scale tests, is discussed in the context of size effects on the flow stress and the mobility of screw dislocations.

2. Experimental

2.1. Material and sample geometry

Tungsten single crystals with a (100)[001] crack system were investigated, where [001] is the crack propagation direction and (100) is the crack plane. The planes of type {100} are the primary cleavage planes in W [48]. Details on sample preparation and microcantilever preparation by focused ion beam (FIB) milling are given in an earlier publication [36]. The milling currents were subsequently reduced at the dual beam microscope (Lyra, Tescan) from 10 nA (coarse milling) to 0.3 nA (fine milling of notches). For all milling steps, an acceleration voltage of 30 kV was used.

A series of notched microcantilevers prepared at the sample edge is shown in Fig. 1(a). In Fig. 1(b) the geometric dimensions used for all specimens in this study, together with the crystallographic orientation of the single crystal, are presented. All samples have approximate length to width to height ratios of 15 μm/4 μm/6 μm, where B is the sample width, and W is the sample height. The applied loading span L varies between 11 and 14 μm and the initial crack length a between 2.0 and 2.5 μm. The specimens were prepared close to the edge in order to minimize material removal after testing for EBSD measurements.

2.2. Fracture testing at the micro-scale

The fracture experiments were performed in-situ inside a scanning electron microscope (Zeiss DSM962 SEM) using two different nanoindentation platforms (Alemnis AG, Switzerland). One of them was modified for application at sub-ambient and the other one at elevated temperatures. Thermal sensors and resistive heaters are attached to both sample and tip holders in order to allow local temperature sensing and tuning. The temperature of the sample surface and the indenter tip are matched after reaching a steady state in the frame. This is verified by analyzing repeated indents with drift measurement segments at different indenter temperatures and minimizing contact drift. This way, drift rates below 10 nm/min are achieved in the temperature range −90 to 600 °C. Tests were run in displacement-control, and cono-spherical diamond tips with a radius of ca. 1–2 μm were used.

2.2.1. Testing at sub-ambient temperatures

The high vacuum environment ($<10^{-5}$ mbar) inside the SEM is important to minimize condensation of water and organic molecules on the indenter and sample at low temperatures. During operation, liquid nitrogen is stored outside the vacuum chamber, evaporated on a fine copper grid and pumped in gaseous state through a cold finger attached to the back of the indenter frame with isolating shafts. The cold finger is linked to the sample and tip holders using copper braids. The frame and electronic components are thermally isolated from the cooled region by ceramic shafts. The noise floor of the low temperature indenter is approximately 1 nm RMS in displacement and 12 μN RMS in force at a 20 Hz sampling rate. Frame drift is minimized by holding the frame temperature constant using resistive heating and temperature feedback in a control loop. Contact drift is minimized by temperature matching of the indenter and the sample using independent resistive heaters and thermocouples. Further details on instrumentation and experimental set-up for testing at sub-ambient temperatures can be found in [49].

2.2.2. Testing at elevated temperatures

To provide a homogeneous and stable environment for testing at temperatures up to 600 °C and to prevent specimen and tip oxidation, the high vacuum inside the SEM chamber is critical. At elevated temperatures it is important to protect the sensitive electronic components of the Alemnis platform from overheating and to keep the heat affected zones small and localized (more details are found in [50]). To assure this, a water-cooled copper backing plate is installed, which is connected to a large external water reservoir and circulation pump. Constant frame temperatures are achieved in this way, which are important to minimize thermal frame drift. The load cell is directly connected to the backing plate by a low stiffness copper braid to draw out the conducted heat from the specimen heater. The heated tip is shielded with a ceramic block to avoid heat transfer into the displacement sensor.

2.3. Experimental procedure and evaluation

Constant displacement rates between 5 nm/s and 150 nm/s were applied, which translate into effective stress intensity factor rates (in the