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Incipient plasticity in tungsten during nanoindentation: Dependence on surface roughness, probe radius and crystal orientation

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ABSTRACT

The influence of crystallographic orientation, contact size and surface roughness effects on incipient plasticity in tungsten were investigated by nanoindentation with indenters with a range of end radius (150, 350, 720 and 2800 nm) in single crystal samples with the (100) and (111) orientations. Results for the single crystals were compared to those for a reference polycrystalline tungsten sample tested under the same conditions. Surface roughness measurements showed that the R_a surface roughness was around 2, 4, and 6 nm for the (100), (111) and polycrystalline samples respectively. A strong size effect was observed, with the stress for incipient plasticity increasing as the indenter radius decreased. The maximum shear stress approached the theoretical shear strength when W(100) was indented using the tip with the smallest radius. The higher roughness and greater dislocation density on the W(111) and polycrystalline samples contributed to yield occurring at lower stresses.

1. Introduction

Tungsten (*W*) is a technologically important BCC metal with potential applications in the next generation of nuclear reactors, being a favoured choice for plasma-facing components in fusion reactors [1–3]. ISO:14577 specifies that *W* being very close to elastically isotropic (Zener anisotropy ratio is 1.01) while having a high elastic modulus makes it an important reference material for indirectly calibrating nanomechanical test instruments due to its high sensitivity to the instrument frame stiffness. It has the highest melting point of all the metals and its high temperature nanomechanical behaviour is beginning to be explored [4]. However, as yet relatively little attention has been given to the influence of crystallographic orientation, loading rate and surface roughness, and how these might influence size effects in incipient plasticity and hardness at the nano-/ and micro/-scale [5].

During nanoindentation, both BCC and FCC metals can show displacement bursts that are known as “pop-ins” [6–8]. Typically in BCC metals such as W, Cr, Mo and Ta, a single yield event is observed while for close packed metals multiple pop-in (“staircase”) behaviour is more common [9]. It is generally accepted that with a sharp indenter, incipient plasticity at the pop-in event occurs due to homologous dislocation nucleation and the shear stress required can approach the theoretical strength [10]. The presence of thick thermally grown oxide layers can modify the stress distributions under the indenter so that a pop-in may be associated with oxide fracture [6]. However, as the native oxide on tungsten is much thinner, of the order of ~0.7 nm thick at room temperature [11], oxide fracture is not thought to contribute to the observed behaviour [12]. Shim et al. [13] noted that the increase in strength as the size of the contact decreases can be considered to be a different type of indentation size effect to that commonly seen in hardness, since the latter depends on the yielding and work-hardening behaviour of the material and the former on the stress to initiate dislocation plasticity. In a fusion reactor, tungsten is subjected to intense bombardment from alpha particles and hydrogen ions which can cause indentation size effects [14]. Being able to deconvolute the origins of the different indentation size effects (ISEs) on the observed behaviour is essential since they will all contribute to the behaviour at a similar scale (e.g. within ~100 nm of the surface).

Yao et al. [1] reported a dependence on crystallographic orientation on electrochemically polished, vacuum annealed (12 h at 950 °C) and D-implanted single crystal tungsten with the critical load for pop-in with a R = 675 nm indenter being much larger on (100) and (110) surfaces than on the (111) orientation. Contrarily, they found no orientation dependence for hardness. Stelmashenko et al. [15] reported Vickers hardness measurements showing higher hardness and higher pile-up around the indentations for W(100). Pethica's group noted that after mechanical polishing a number of dislocation systems are active at low load in W(100) and a clear single pop-in was not observed [16]. They

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Abbreviations: BCC, Body centred cubic; FCC, Face centred cubic; ISE, Indentation size effect; NPL, National Physical Laboratory; RMS, Root mean square

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Nomenclature

a	Contact radius
Al	Aluminium
E or E_s	Elastic modulus of the material
E_r	Reduced elastic modulus
h^*	Characteristic length
h_c	Contact depth
$h_{\max}$	Depth under maximum load at pop-in
h_r	Residual indentation depth
H_0	Macroscopic hardness
L	Applied load
G	Shear modulus
Mo	Molybdenum
P_m	Mean contact pressure
p_0	Maximum contact pressure
R	End radius of the indenter
R_a	Arithmetic average surface roughness
Ta	Tantalum
W	Tungsten
$\tau_{\max}$	Maximum shear stress

also reported that the hardness of mechanically polished W samples determined at depths higher than the pop-in event was higher than that of electropolished samples.

Most studies on incipient plasticity of pure metals have used indenters of one or at most two radii, making the effect of tip radius difficult to establish accurately. There have been two recent reports using a wide range of tip radius. Shim et al. [13] studied the influence of indenter radius (R = 0.58 to 209 μm) on pop-in occurring in the FCC metal Ni(100) and reported that the critical loads and maximum shear stresses under the indenter increased as the radius decreased. Wu et al. [17] investigated the onset of plasticity in the BCC metal chromium using indenters with tip radius ranging from 60 to 759 nm and also found that the stress required for incipient plasticity increased with a reduction in tip radius.

There has been recent interest in the influence of the surface state on the load required for pop-in [8,9,12,14]. Although it is generally accepted that pop-in events require highly polished surfaces, it is a common practice in the literature for either the surface roughness to not be quoted or for only an approximate measure of R_a to be provided. On Al(001), Shibutani et al. [8] observed that the critical load scaled inversely with surface roughness. A reduction in R_a from ~2.5 nm to under 0.5 nm resulted in the critical load increasing by a factor of 3. Bahr et al. [6] reported that, as opposed to electropolished surfaces, mechanically polished W single crystals did not show pop-ins. Biener et al. [9] reported that on Ta(001) there was no difference between electropolished or mechanically polished surfaces provided a high-quality surface finish was obtained. They found a tight distribution in the critical load for pop-in for a Ta(001) surface with the RMS roughness well below 1 nm. Introducing surface roughness on Ta by low-energy Ar^+ ion bombardment suppressed the linear elastic regime and the pop-in behaviour.

This work reports novel findings obtained from nanoindentation experiments performed on tungsten samples. The objective of this study was to investigate the contribution of size effects to incipient plasticity in tungsten using a wide range of indenter radius (0.15–2.8 μm). Alongside this, the influence of crystallographic orientation, loading rate and surface roughness were also studied on single crystals of tungsten with the (100) and (111) orientation and a reference polycrystalline tungsten sample. Nanoindentation data at a lower load were supplemented by measurements to 500 mN to determine the conventional indentation size effect in hardness.

2. Experimental

2.1. Materials

Two high purity polished tungsten single crystals and a high purity polycrystalline tungsten certified reference sample were tested. The sample with (100) orientation was provided by KRISS (Korea), originally for the VAMAS TWA22 Intercomparison on nanoindentation, being supplied by Goodfellow (USA) and polished by KRISS. The sample with (111) orientation was supplied by Goodfellow (UK) and was of thickness 2 mm and diameter 6 mm, and was polished on one side to better than 1 μm (W 002166). The quoted elastic modulus and Poisson's ratio of the samples were 411 GPa and 0.28 respectively. The polycrystalline certified reference tungsten sample ("JGA-105", Instrumented Indentation Reference Block, DataSure-IIT, NPL, Teddington, UK) was obtained from NPL, based in the UK. Its elastic modulus and Poisson's ratio were determined by NPL in accordance with BS EN 843-2:2006. The certified value of E obtained by NPL was 411.5 ± 1.9 GPa and the Poisson's ratio was 0.2806 ± 0.0017. The density of the polycrystalline sample was 1.9259 g cm^{-3}. The sample was coarse-grained with an average grain size in the region of 10 μm. The tungsten samples were tested as-received and no further attempt was made to modify surface roughness or near-surface defect density by further polishing or annealing steps. Surface roughness was measured over a line profile using the Surface Topography option in the Scanning Module of the NanoTest using (i) a spheroconical diamond probe with a nominal end radius of 5 μm (the actual end radius was separately determined as 4 μm) (ii) a well-worn Berkovich indenter with an end radius of 1 μm. Surface roughness of the single crystal samples was also measured at the 5 μm × 5 μm scale by AFM (NanoSurf Nanite B). Table 1 summarises the surface roughness data. The AFM images revealed the presence of very fine polishing marks on the surface of the (111) oriented W which were absent on the (100) oriented W.

2.2. Nanoindentation

Nanoindentation testing of the tungsten samples was performed with a commercial nanomechanical test instrument (NanoTest Platform 3, Micro Materials Ltd., Wrexham, UK) which had been calibrated in accordance with the ISO 14577-4. The polycrystalline W was used to determine the frame compliance of the instrument which was confirmed by measurements in other reference metallic samples. The end radii of the diamond indenters were calibrated by fully elastic nanoindentation measurements into fused silica and sapphire reference samples. Three of the indenters used were Berkovich indenters of different end radius and one was spheroconical diamond with a nominalend radius of 5 μm. The fused silica was a nanoindentation intercomparison reference sample (obtained from KRISS, Korea) with a nominal elastic modulus of 72.5 GPa and Poisson's ratio of 0.17. Its

Table 1
Surface Roughness of the measured samples.

	R_a surface roughness (nm)		
	AFM (5 μm × 5 μm area)	Line scan with R = 1.0 μm diamond (over 10 μm length)	Line scan with R = 4.0 μm diamond (over 10 μm length)
W(100)	1.4 ± 0.6	2.0 ± 0.3	2.3 ± 0.5
W(111)	4.0 ± 0.7	3.1 ± 0.4	5.5 ± 1.6
Polycrystalline W	Not measured	5.5 ± 1.4	6.9 ± 2.1