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Correcting for contact area changes in nanoindentation using surface acoustic waves

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ABSTRACT

Nanoindentation is extensively used to quantify nano-scale mechanical behaviour. A widely-used assumption is that a well-defined, material-independent relationship exists between the indentation depth and indenter contact area. Here we demonstrate that this assumption is violated by ion-implanted tungsten, where pileup around the indenter tip leads to substantial changes in contact area. Using high accuracy surface acoustic wave measurements of elastic modulus, we are able to correct for this effect. Importantly we demonstrate that a priori knowledge of elastic properties can be readily used to compensate for pileup effects in nanoindentation without the need for any further measurements.

1. Main body

Most nanoindentation analysis methods assume that surface pileup or sink-in plays a negligible role in determining contact area for the calculation of hardness and modulus [1–3]. However, whilst applicable to a small number of materials, this is far from universally true. Methods for determining the *actual* contact area include post-test measurements using transmission electron microscopy (TEM) replicas [4], scanning electron microscopy (SEM) [5], or atomic force microscopy (AFM) [6]. However post-test examination has major restrictions. It is time consuming and must be carried out on individual indents that may not be representative. More importantly the examination is inherently carried out on the unloaded indentation impression, unlikely to represent the true contact area due to elastic recovery on unloading. This effect is particularly significant for small indents in elastically stiff and mechanically hard samples [7]. Nanoindentation performed inside the SEM can provide observations of changes in pile up morphology [8], however, with a single static viewpoint, it does not allow determination of contact area.

Here we propose an alternative approach that utilises simultaneous modulus and hardness measurements enabled by the Continuous Stiffness Measurement (CSM) method used on many nanoindentation systems [3]. If the modulus can be measured independently, changes in contact area due to pileup can be corrected for, allowing a substantially more accurate determination of indentation hardness.

Ion implantation is extensively used to mimic irradiation damage in nuclear reactor materials [9–12]. The ability to accurately probe mechanical properties of these modified layers is highly desirable. Bulk mechanical tests on heavy ion-implanted layers are not possible as the implanted layers are only a few microns thick due to limited ion penetration. Electron microscopy and atom probe tomography have been widely used to study the microstructural changes within these thin layers [13]. Their mechanical properties have been predominantly studied using nanoindentation to measure hardness changes. However the reliability of these measurements is questionable since even at low damage levels substantial variations in pileup morphology and hence contact area can occur. This has been observed for example in W-5 wt%Ta implanted with 2 MeV W^+ ions to damage level of 0.04 displacements per atom (dpa) [14], where pile up is significantly suppressed after implantation. Interestingly no changes in pileup morphology were observed in HT9 steel implanted with both helium-ions and protons [10]. Yet in Fe^+ implanted Fe-12 wt%Cr significant changes in pileup both as a function of indentation depth and indenter type have been reported after implantation [5]. However, the majority of nanoindentation studies on ion-implanted surfaces simply do not consider potential change in pile morphology and the effect they may have on the determined hardness and modulus values [15–17].

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Our previous nanoindentation measurements on tungsten alloys showed a large apparent increase of both elastic modulus and hardness after ion implantation [14,18]. This is surprising since recent density functional theory calculations and surface acoustic wave measurements both indicate that the elastic modulus of tungsten is reduced by helium-ion implantation [19]. Eqs. (1) and (2) show that changes in contact area will affect the measurement of both hardness and elastic modulus, with pile-up resulting in an overestimate of both quantities and sink-in an underestimate. This suggests that the reported large increase in modulus *and* hardness [18] is likely to be due to an *underestimation* of the true contact area during indentation. By measuring the elastic modulus of the implanted layer using an independent technique, such as surface acoustic wave (SAW) measurements [19–21], a correction factor for the contact area can be calculated allowing a correct hardness value, accounting for the pile up, to be determined. Here we employ this new approach to determine the true hardness of a 2.5 μm thick He^+ implanted surface layer in a W-1 wt% Re alloy sample.

The W-1 wt% Re alloy was produced by arc-melting of elemental powders: 99.9% W, (Sigma Aldrich, USA) and 99.99% Re (AEE, USA), as described in [18]. The resulting ingot was sectioned into 1 mm thick slices and polished using a final colloidal silica polishing step to produce a high quality surface finish, the composition was verified using electron probe micro-analysis. Helium ion-Implantations were performed at 573 K at the National Ion Beam Centre, University of Surrey, UK. Multiple ion energies (from 0.05 MeV to1.8 MeV), were used to produce an approximately uniform helium concentration of 3110 ± 270 appm and corresponding damage of 0.24 ± 0.02 dpa within a 2.5 μm thick surface layer, for full implantation details see Beck et al. [18]. The calculated implantation profiles, estimated using the Stopping Range of Ion in Matter (SRIM) code [22] with a displacement energy of 68 eV, are shown in Fig. 1.

Nanoindentation was performed on an MTS NanoXp with a Berkovich diamond indenter tip. Calibration indents in fused silica, using the continuous stiffness measurement (CSM) technique [3] with a 45 Hz and 2 nm oscillation, were used to fit a polynomial expressing contact area, A(d), as a function of indenter displacement, d, into the surface. Hardness, H (d), and indentation modulus, E*(d), were then computed using Eqs. (1) and (2) respectively:

$$H(d) = \frac{P(d)}{A(d)}, \tag{1}$$

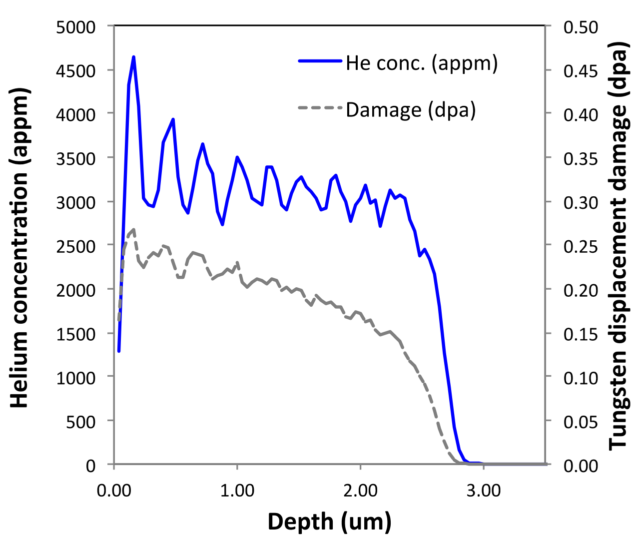

Fig. 1. Helium ion concentration and displacement damage caused by the ion implantation as calculated using the SRIM code.

$$E^*(d) = \frac{dP}{dd}(d)\frac{1}{2}\frac{\sqrt{\pi}}{\sqrt{A(d)}}, \tag{2}$$

where P(d) is the applied load as a function of displacement.

Indents were made to displacements of (a) 2000 nm and (b) 250 nm. Deep indents (a) allowed identification of the indent depths at which behaviour is dominated by the implanted layer. Shallow indents (b) served to study pileup morphology at these depths. Fig. 2a shows modulus vs indenter displacement and (Fig. 2b solid markers) hardness vs indenter displacement data. Hardness shows the expected behaviour with a small size effect seen in the unimplanted sample, and significant hardening after implantation, similar in magnitude to that seen in pure tungsten implanted with helium to similar doses [23]. In the unimplanted sample modulus varies little with indentation depth and the recovered value of 400 GPa is consistent with the literature value of Young's modulus for pure tungsten [24–26]. The implanted sample shows a significant increase in modulus to a maximum value of 475 GPa at indentation depths between 100 nm and 350 nm. At the maximum indentation depth of 2000 nm there is still an increases in modulus of 5% to a value of 420 GPa. This dramatic increase in elastic modulus is inconsistent with previous atomistic calculations and experimental measurements [19,27].

AFM micrographs of the 250 nm deep indents were recorded using a Digital Instruments Dimension 3100 microscope in contact mode (nominal tip radius 10 nm) and SEM micrographs were collected using a Zeiss Auriga FEG FIB/SEM (Fig. 3). In the unimplanted sample a small amount of pile-up is observed around the indent (Fig. 3a). The implanted sample

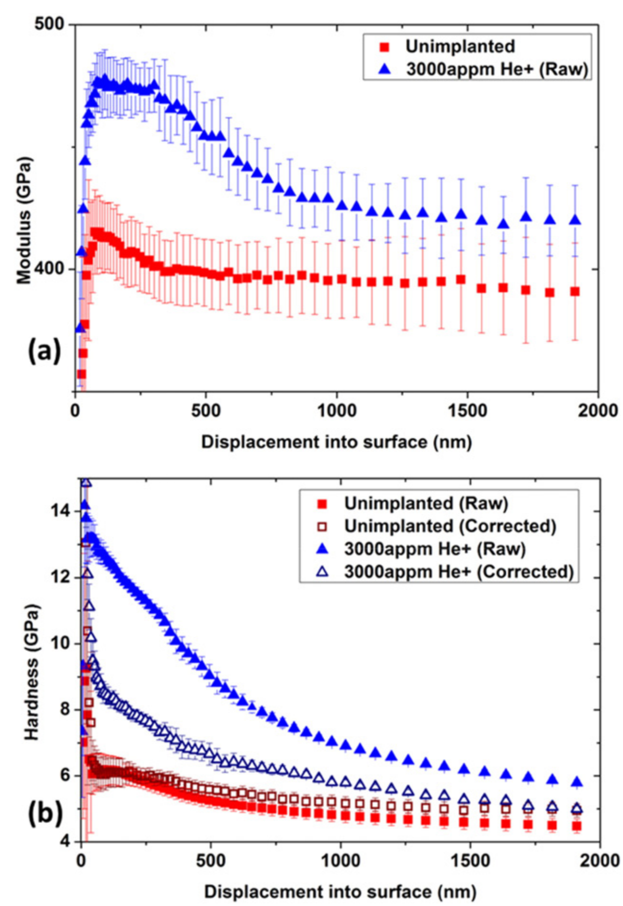

Fig. 2. (a) Indentation modulus as a function of depth for unimplanted (red) and He^+ implanted (blue) W-1 wt% Re. (b) Raw indentation hardness for unimplanted and implanted W-1 wt% Re (closed symbols). True indentation hardness values corrected using SAW measured modulus values (open symbols).