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High temperature indentation of helium-implanted tungsten

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ABSTRACT

Nanoindentation has been performed on tungsten, unimplanted and helium-implanted to ~600 appm, at temperatures up to 750 °C. The hardening effect of the damage was 0.90 GPa at 50 °C, but is negligible above 450 °C. The hardness value at a given temperature did not change on re-testing after heating to 750 °C. This suggests that the helium is trapped in small vacancy complexes that are stable to at least 750 °C, but which can be bypassed due to increased dislocation mobility (cross slip or climb) above 450 °C.

1. Introduction

As the main plasma-facing material in a fusion reactor [1], tungsten will be exposed to a flux of alpha particles (essentially helium ions) from the plasma as well as 14 MeV neutrons. While the rate of helium production due to transmutation is negligible, this incident alpha particle flux will result in a high concentration of diffused-in helium that may combine with displacement damage from incident neutrons. The resulting defects are likely to produce significant mechanical property changes. There have been several studies on tungsten and tungsten alloys using neutrons or self-ion irradiations, in conjunction with either micro-indentation [2–4], nanoindentation [5,6] or micro-bending [7] experiments to probe the effect that displacement damage has on mechanical properties, such as hardness or yield stress. However all these studies have produced data at room temperature only; they also considered only the effects of displacement damage on mechanical behaviour and not any additional effects that helium might have.

Research into the effects of helium on tungsten has mainly focussed on determining the lattice position helium occupies at very low concentrations [8] and characterisation of 'nanofuzz' structures formed under conditions of high flux [9]. With regard to mechanical properties, work by Armstrong et al. [10] demonstrated the considerable hardening effect of helium in tungsten at room temperature even at relatively low concentrations (300 appm). Beck et al. [11] showed a similar hardening effect of implanted helium in tungsten–rhenium and tungsten–tantalum alloys. Beck also used TEM to show that the implanted helium does not form visible bubbles even at concentrations of approximately 3000 appm, suggesting that the implanted helium is in solution, or sits in stable vacancy-helium clusters below the resolution limit of the TEM.

However, in both Armstrong's and Beck's works the helium implantation took place at 300 °C, and mechanical properties were measured at room temperature. The expected steady state operating temperatures of tungsten are up to 700 °C at the first wall and up to 500 °C in the divertor [12]. During operation, these components will also be subject to fast neutron irradiation, producing displacement damage at a typical rate of 15 dpa/FPY [13]. As vacancy mobility becomes significant above 530 °C in tungsten [14], this will likely have an effect on the damage structures formed by the helium ions as well as the mechanical properties above this temperature.

This work aims to study pure tungsten (investigated previously [15,16]) implanted with helium ions at a reactor-relevant temperature of 800 °C. It also extends nanoindentation methods typically used to study effects of radiation damage [5] to tests performed at elevated temperatures, up to the 750 °C expected during standard service conditions. This is important to determine the mechanical properties of components during reactor operations, and also to study the hardening mechanisms that may influence component brittleness during maintenance periods.

2. Experimental methods

2.1. Ion implantation

Commercially pure tungsten (MCO, Cambridge, UK) was irradiated at 800 °C using 2 MeV He^+ ions at JANNuS (CEA Saclay, France) with a

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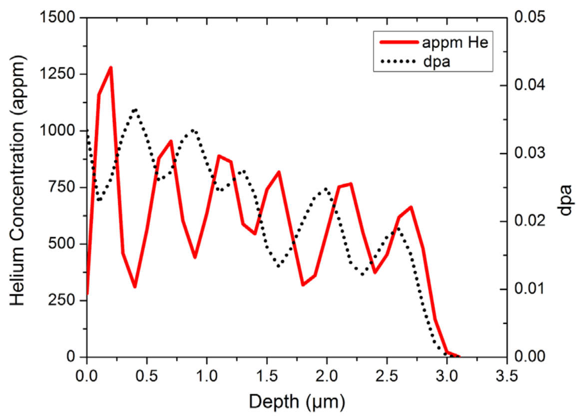

Fig. 1. Helium distribution and dpa profile in the ion-implanted later as predicted using SRIM-2008 (Stopping Range of Ions in Matter). The 'quick' Kinchin–Pease calculation of damage was used with a displacement energy of 68 eV. This gives an average concentration of 600 appm in the 3 μm implanted region. The multiple peaks were generated by implantation through a series of degrader foils from 0 to 6 μm.

series of aluminium degrader foils in place to produce the predicted depth distribution shown in Fig. 1. A dose of 1.21×10^{16} ions cm^{-2} was implanted at a flux of 7.51×10^{11} ions $cm^{-2}s^{-1}$. Five degrader foils (6 μm, 5 μm, 4 μm, 3 μm and 1.6 μm) were used in addition to the un-degraded 2 MeV beam. The profile has a peak concentration of 1250 appm at 0.15 μm depth, with an average concentration of 600 appm over the total depth of 3 μm. The temperature was monitored using a thermocouple mounted just behind the samples in the sample holder, and an infrared camera was used to check that a uniform temperature profile across the sample was achieved. Beam currents were measured periodically using a set of four Faraday cups.

Previous work on tungsten implanted with helium of the same energy range [10] used continuous stiffness measurement nanoindentation [17] to determine the depth to which nanoindentation results are unaffected by the unimplanted substrate. This analysis showed that values of hardness extracted <400 nm into the surface are dominated by the ion-implanted layer, while hardness values extracted from >1500 nm into the surface are dominated by the hardness of the bulk material. These critical depths are used here in the analysis of the load-unload data at high temperatures.

2.2. High temperature micro-indentation

High temperature micro-indentation has been widely used to study the hardness dependence on temperature on a variety of materials. In the case of tungsten there is a well-documented "knee" in the hardness drop at around 300 °C, where the rate of change in hardness significantly decreases [18,19]. To study the accuracy of the high temperature nanoindentation experiments, high temperature micro-hardness tests were performed over the same temperature range. A single-crystal sample of tungsten from the same source used for the nanoindentation study was used. Indentations were performed using a sapphire Vickers indenter and a 200 g load held for 15 s to allow for any indentation creep effects. Five indents were performed at each temperature from 23 °C to 700 °C

2.3. High temperature nano-indentation

High temperature nanoindentation was performed on an unirradiated sample and a helium-implanted sample using a MicroMaterials NanoTest nanoindenter. This instrument is housed in a vacuum chamber that attains a vacuum level of $<10^{-5}$ mbar. A cubic boron nitride indenter with standard Berkovich geometry was used. Tungsten samples were mounted on a furnace using FortaFix Autostic FC6 high temperature cement. A thermocouple was cemented to the surface of the sample to monitor the temperature of the material in contact with the indenter. A second thermocouple in the indenter was used to equalise sample and indenter temperatures during the indentation process. At least 36 indents were performed at each temperature. Each indent was performed using twenty load-unload segments, evenly distributed in depth from 50 to 2000 nm so as to determine hardness as a function of depth. This produces load–displacement curves as shown in Fig. 2. The form of the curves – apart

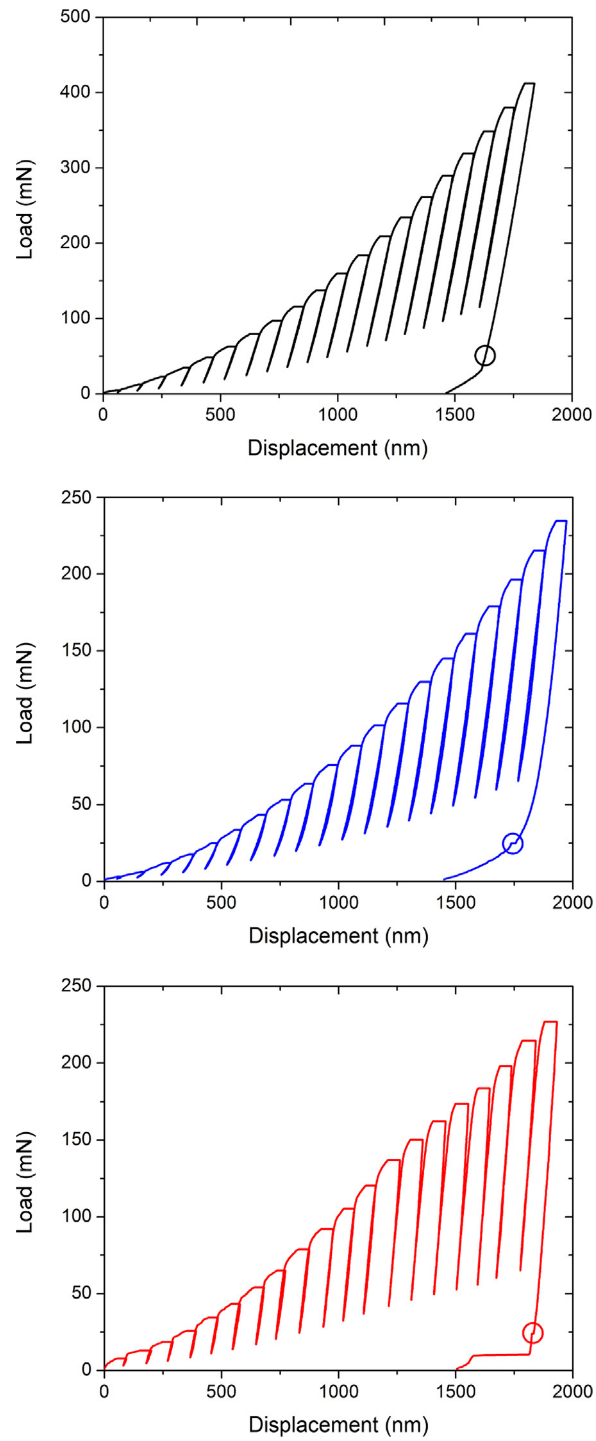

Fig. 2. Typical load–partial unload curves for indents performed at 50 °C (black), 350 °C (blue) and 750 °C (red). No differences in form – except for the peak load – are seen between the three curves. Post-indentation drift is measured at 80% unload; these regions are circled on the curves. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)