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Small-scale characterisation of irradiated nuclear materials: Part II nanoindentation and micro-cantilever testing of ion irradiated nuclear materials

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ABSTRACT

This paper demonstrates the ability of advanced micro-mechanical testing methods, based on FIB machined micro-cantilevers, to measure the mechanical properties of ion implanted layers without the influence of underlying unimplanted material. The first section describes a study of iron–12 wt% chromium alloy implanted with iron ions. It is shown that by careful cantilever design and finite element modelling that changes in yield stress after implantation can be measured even with the influence of a strong size effect. The second section describes a study of tungsten implanted with both tungsten ions and tungsten and helium ions using spherical and sharp nanoindentation, and micro-cantilevers. The spherical indentation allows yield properties and work hardening behaviour of the implanted layers to be measured. However the brittle nature of the implanted tungsten is only revealed when using micro-cantilevers. This demonstrates that when applying micro-mechanical methods to ion implanted layers care is needed to understand the nature of size effects, careful modelling of experimental procedure is required and multiple experimental techniques are needed to allow the maximum amount of mechanical behaviour information to be collected.

1. Introduction

For the successful development and deployment of advanced nuclear reactors such as GEN IV fission types and fusion devices such as tokamaks or inertial confinement devices, there is a vital need to understand the effect of radiation damage from neutrons and charged particles on the mechanical properties of structural materials [1–4]. While some work can, and indeed must, be carried out on neutron irradiated samples, ideally irradiated at conditions as close to those seen in service as possible, this approach suffers from several difficulties. Firstly the neutron irradiated samples are by their nature active and must be handled with care and tested in hot cells. Secondly the cost and timescale over which such irradiation campaigns take place limit the number of variables which can be studied, and preclude rapid response to results. Finally there is no available source of 14 MeV neutrons, with sufficient flux to mimic fusion environments. Ion implantation can fill the gap here, allowing high damage levels to be produced quickly and comparably cheaply.

The use of ion implantation to study radiation damage and mimic neutron damage has a long history [5]. Transmission electron microscopy (TEM) has been widely used both with in-situ ion beams to study the formation of damage structures during irradiation and post implantation to study the final damage networks formed [6,7]. Alongside this, field ion microscopy and atom probe tomography have been used to investigate the damage formation and the chemical changes which can occur during irradiation [8,9]. These are further discussed in the paper of Edmondson et al. in this edition. [10]. The ability to measure the mechanical properties of ion implanted structures has lagged behind, due to the difficulties associated with measuring the mechanical properties of such thin layers (typically ∼0.2 to 2 μm deep) and then in relating them to macroscopic mechanical properties. Most of the work thus far has used micro and nano-indentation techniques to probe the mechanical properties of these layers. This comes with inherent difficulties. Firstly the hardness is not a fundamental mechanical property and different testing techniques can give different "hardness values" on nominally the same material [11]. Much work has been carried out using finite element and other empirical techniques to extract a yield or flow stress from indentation data, but due to the complex and ill-defined stress state no

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general method has been developed [12]. Secondly the ion implanted layers are nearly always on top of an unimplanted bulk material and deconvoluting the influence of this layer on the measured mechanical properties is difficult, particularly if the exact damage profile from implantation is unknown [13–16]. While methods used for analysing coating systems and their substrates can be used, the situation is more complex as the implanted layers vary in structure with depth and there is no longer a sharp boundary between the surface layer and substrate. Nonetheless indentation is an important tool in comparing implanted and unimplanted samples due to its comparative ease and speed of use and its ability to give quantitative data. A fuller discussion of indentation of irradiated layers can be found in the paper by Hardie et al. [17] in this special edition.

Development of micro-scale manufacturing methods such as focused ion beam machining, together with advances in indentation instrumentation, has allowed the manufacture and testing of micro-scale mechanical test structures with well-defined stress states, typically micro-pillars or micro-cantilevers. These have allowed measurement of basic mechanical properties such as elastic modulus [18,19], yield stress [20–24] and fracture properties [25–28], which are key parameters in engineering design. Either by manufacturing micro-scale test structures and then ion implanting them or by manufacturing the test pieces into pre-implanted layers it is possible to directly measure the mechanical properties of the damaged region without influence of the underlying material.

Pillars are the most widely used FIB machined micro- and nano-mechanical testing geometry used, due to their relative ease of manufacture and apparently simple stress state of uniaxial compression. Micropillars have been used in several studies of irradiated materials. Kiener et al. [29] produced pillars with diameters from 80 nm to 1500 nm in copper, irradiated to 0.8 dpa using protons, which were then compressed inside a TEM. They observed an increase of ≈100–250 MPa in yield stress for irradiated vs non-irradiated samples above 400 nm diameter; however, below 400 nm diameter, no difference between irradiated and unirradiated pillars was seen. This work allowed the deformation mechanisms to be observed directly but only allows small pillars (thin enough to be electron transparent) to be tested. Grieveson et al. [30] used 1 μm diameter micropillars to study the yield properties of pure iron in both the unimplanted state and self-ion-implanted to 6 dpa. While nanoindentation showed a clear increase in hardness in the implanted region, no significant change in the yield stress of the micro-pillars after irradiation was observed. Post-test examination showed that the unimplanted pillars failed by a giant shear type mechanism as commonly seen in other single crystal micro-pillars, while the failure of the implanted pillars was seen to be by multiple slip events on a stack of closely-spaced parallel slip planes. These differences in deformation behaviour were also apparent in the stress–strain curves, with the unimplanted material deforming by a small number of large strain jumps, and the implanted material by a larger number of smaller strain jumps. This was attributed to differences in behaviour of dislocations produced by the small number of dislocation sources in both sets of pillars. In the unimplanted pillars once the dislocation source is activated slip continues until it exits the pillar, producing a few discrete slip steps each with large associated strain, as has been commonly found in micro pillar deformation [24]. However the radiation damage in the implanted pillar acts as a dense distribution of obstacles to dislocation motion. Three mechanisms – cross-slip, stress concentrations due to pile up and stress-concentrations due to shear channels were suggested as reasons for the different flow behaviour in implanted micropillars.

The work by Grieveson et al. [30] also demonstrates one of the commonly seen weaknesses of micro-pillars in that an appreciable taper is seen on the pillars when produced by annular milling. This results in a more complex stress state making quantitative analysis of elastic modulus and yield stress difficult. Copper pillars have been manufactured by Gue et al. [31] using templating and electroplating. This allowed pillars of diameter 100–400 nm to be manufactured with no ion damage from FIB machining, and with a more uniform cross section than by FIB machining. After manufacture these pillars were then implanted with helium ions to a damage level of 0.7 dpa with a calculated helium concentration of 0.35 at.%. Although the 125 nm pillar showed little change in yield stress after irradiation, an increase in yield stress was observed in the 400 nm pillar; this contrasts with the work of Kiener et al. [29] who observed no increase in strength of <400 nm diameter copper pillars irradiated with protons. Gue et al. also saw a transition similar to that seen by Grieveson [30], where multiple short strain bursts occurred during compression after irradiation compared to fewer larger strain bursts in unirradiated material. Additionally they saw an increase in work hardening rate in irradiated material.

Halliday et al. [32] used nanoindentation and "waisted" micro-cantilever tests to measure the hardness and yield stress of a range of Fe–*X* wt%Cr alloys (where X ranges from 0% to 12%). Indentation tests showed an increase in hardness in all implanted alloys, but a significant effect from the underlying unimplanted material could be seen in hardness–displacement data. Analysis of tests on waisted micro-cantilevers, using simple elastic beam theory, showed that the yield stress in the implanted material increased only slightly on implantation to 0.35 dpa, but on implantation to 5.33 dpa both modulus and hardness significantly increased. The modulus of this heavily irradiated material, 374 GPa, is over a 100 GPa higher than that generally reported for pure iron. It seems likely that this is due to inaccuracies in either the measuring or manufacture of the waisted micro-cantilever and to the simplistic simple beam type analysis used; and as such is not thought to be physically realistic. This clearly demonstrates the need for accurate cantilever design, measurement and modelling if reliable stress–strain data is to be obtained.

Thus it is clear that while micromechanical testing techniques potentially offer considerable advantages over indentation methods for mechanical characterisation of ion implanted layers, this potential cannot be exploited without a better understanding of the deformation processes occurring during such tests, and without reliable techniques for analysis of micromechanical test data. Establishing that understanding and these techniques is an ongoing research effort; in this paper we will demonstrate the use of micro-mechanical testing techniques on two very different materials systems subjected to very different irradiation conditions, leading to the determination of mechanical data not possible using nanoindentation experiments alone.

2. Experimental work

In Part 1 an iron 12 wt% chromium alloy, the basis for a large number of nuclear ferritic alloys, is subjected to 2 MeV Fe+ ion implantation at 320 °C to a damage level of 6 dpa to act as an analogue of the damage produced by neutrons in an advanced fission or fusion reactor. In Part 2 tungsten samples are implanted with W^+ ions, and W^+ and He^+ ions simultaneously at 800 °C, giving damage levels of 1 dpa and 650 appm helium. This acts as an analogue for both the neutron and helium irradiation that a tungsten alloy will undergo in both structural and plasma facing rolls in future nuclear fusion reactors such as DEMO [33,34]. In both cases micro-cantilever bending and nanoindentation experiments were used to study the changes in the mechanical behaviour of these ion implanted layers. Use of these two rather different metals illustrates the utility of micromechanical test methods for study