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Understanding the effects of ion irradiation using nanoindentation techniques

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ABSTRACT

The effects of ion irradiation in materials for research are usually limited to a shallow surface layer of the order of one micrometre in depth. Determining the mechanical properties of such irradiated materials requires techniques with high spatial resolution. Nanoindentation is a relatively simple method for investigating these shallow layers with the advantage that statistically rich data sets for elastic and plastic property values can be generated. However, interpretation of the results requires and understanding of the material response, including the extent of the plastic zone with respect to the irradiated layer, pile-up or sink-in of material around the indentation site that affect the calculated contact area and hence derived mechanical property values. An Fe^{+} self-irradiated Fe12%Cr alloy was investigated with three different indenter tip geometries, a cube corner, Berkovich and 10 μm radius indenter. Sharp indenters provide sufficiently small plastic zones to be contained within the irradiated layer but pop-in events and pile-up need to be taken into account for correct interpretation of the mechanical properties of the irradiated material.

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1. Introduction and background

Ion implantation can quickly produce high levels of displacement damage without inducing radioactivation of samples and is therefore a popular tool to investigate irradiation damage in solids. Several microstructural and chemical characterisation methods such as transmission electron microscopy (TEM), atom probe tomography (APT) and positron annihilation spectroscopy (PAS), require volumes of material which are inherently small relative to ion irradiated surface layers which are typically a few microns in thickness. Thus the limited irradiated volume produced by implantation presents little or no additional challenges when using these techniques. In contrast, there are few methods available for the characterisation of mechanical properties from such small volumes of material, and analyses regarding best practise and validity of using these techniques for irradiated materials are scarce. There have been several investigations which have used nanoindentation as a means to measure the effects of ion irradiation on mechanical properties [1–12]. The majority of investigations have been conducted using a Berkovich tip geometry and include various methods such as load–unload [1,2] and Continuous Stiffness Measurement (CSM) [8–11]. Within all investigations there is a lack of consistency or justification of indentation parameters used and various methods for data interpretation; this makes cross-comparison of such experiments difficult. Therefore there is a requirement to understand and optimise nanoindentation techniques specifically for the measurement of mechanical properties of shallow ion irradiated layers.

The work described in this paper investigates and compares several nanoindentation techniques available for the analysis of an 800 nm damage layer of ion-irradiated Fe12%Cr alloy implanted at the National Ion Beam Centre, Surrey UK. Data produced from a Continuous Stiffness Measurement (CSM) nanoindentation technique with cube corner and Berkovich pyramidal tips and a partial-unload technique with a spherical tip are directly compared and critically evaluated. Micro-mechanical techniques offer an alternative method for testing ion irradiated layers; in comparison to nanoindentation, these methods are more intensive on resources, time and expertise and are not described here. Testing using several micro-cantilever techniques on the same material have also been conducted and are reported in Ref. [13] in this journal edition.

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2. Experimental details

2.1. Material and sample preparation

The material and irradiation details are identical to that reported in [10], where full details have been given. Briefly, an Fe12%Cr alloy was manufactured by Cambridge Metals Crystals and Oxides (CMCO) with final cold rolling into sheet at a thickness of approximately 1 mm. A sample was cut into a rectangle sample of approximately 4 × 8 mm, and then annealed within an evacuated sealed quartz silica tube at 830 ± 10 °C for 72 h to develop a large equiaxed grain structure. A series of lapping stages using SiC abrasive papers from FEPA P120 to P4000 grades was used to produce a smooth surface with a thin layer of polishing damage. Finally a chemo-mechanical polish with a colloidal silica suspension (0.05 μm) was used to provide a surface with minimal polishing damage and of a quality suitable for electron backscattered diffraction (EBSD). Grain diameters were determined by EBSD to be in the range of 20–450 μm, with a mean grain size of 189 μm.

2.2. Ion implantation

The alloy was irradiated with Fe^+ ions at 320 °C to an average dose of 6.18 dpa (0–800 nm depth). Two implantation energies of 2 MeV and subsequently 0.5 MeV were used in an attempt to produce a uniform distribution of damage with depth. The ion beam and environmental conditions for the implantation are shown in Table 1. Calculations of damage with depth into the sample surface are presented in Fig. 1, these were produced using the SRIM 2013 code [14] and a threshold displacement energy of 40 eV for iron [15].

Fig. 2 shows a TEM cross-section of the radiation damage layer at the sample surface (as reported in Ref. [10]). The micrograph includes the protective Pt layer deposited in the FIB before foil thinning, a region of dense dislocation loops forming the damage layer and the underlying un-irradiated substrate. It is evident that the visible damage extends to a depth no more than 600 nm at the sample surface which is slightly less than that predicted by the SRIM calculations. The sample clamp used during ion-irradiation produced a sample with areas both exposed to and shielded from the beam. The region of the sample exposed to the beam was clearly visible in secondary electron images after implantation as it was darker in contrast compared to the un-implanted region of the sample. This enabled the FIB milling of marker lines which were visible under an optical microscope, at the boundary between implanted and un-implanted regions. This enabled mechanical testing adjacent to the boundary of the implanted and un-implanted regions within same grain and provided the advantage of eliminating differences in observed mechanical properties due to any anisotropy associated with crystallographic orientation, variation in the quality of polishing and sample mounting conditions.

Table 1
Ion beam conditions at the Surrey Ion Beam Centre (UK).

Implantation energy	0.5 MeV	2 MeV
Implantation dose (ions/cm^2)	1.5×10^{15}	3×10^{15}
Radiation damage (avg.)	6.18 dpa	
Radiation damage (max.)	8.36 dpa	

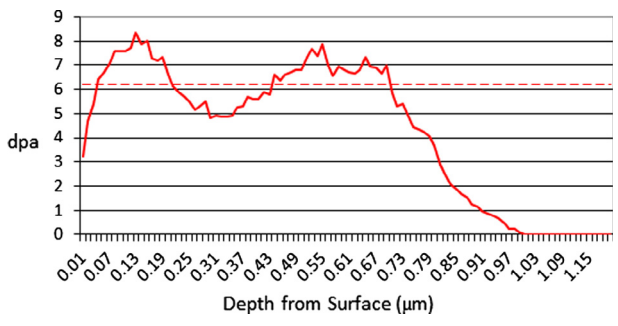

Fig. 1. Irradiation damage versus depth from the sample surface as calculated by SRIM with a displacement energy of 40 eV for iron [15].

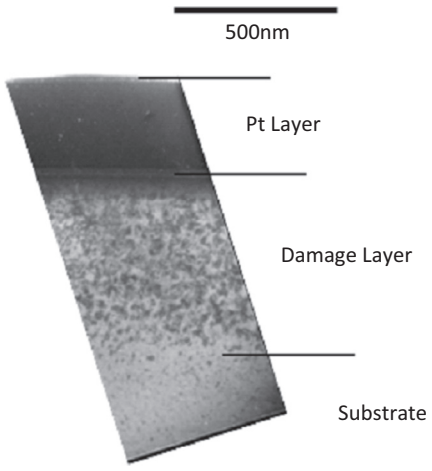

Fig. 2. Cross-sectional TEM image of the damage layer produced in Fe12%Cr by Fe+ implantation at 2 MeV and 0.5 MeV (reproduced from Ref. [10]).

2.3. Nanoindentation techniques

For nanoindentation testing the alloy sample was mounted on an aluminium stub using a thin layer of Crystalbond® resin. Three diamond indenter tip geometries were used for comparison of indentation response. These were a pyramidal cube corner tip with a centreline to face angle of 34.3°, a pyramidal Berkovich tip with centreline to face angle of 65.3° and a spherical tip with a nominal radius of 10 μm. The geometry of pyramidal indenter tips are described by an area function, $A = f(h_c)$, and were calibrated by indentation of a reference fused silica sample with a known elastic modulus of 72 GPa according to the methods described in Ref. [16]. The spherical indenter was calibrated using the multiple reference material method [17].

Continuous Stiffness Method nanoindentation (CSM) [18] was carried out with the pyramidal tips using an MTS NANO Indenter XP fitted with the NANO CSM system (MTS NANO Oak Ridge Tennessee, USA). A small sinusoidal oscillation in the load signal measures stiffness dynamically during the indentation sequence and the corresponding displacement signal is monitored. Contact stiffness can be calculated by measuring the phase difference or the amplitude of the displacement signal, accounting for the response of the entire nanoindentation system by using a dynamic model (as given in Ref. [16]).

The stiffness of the contact, dP/dh, has contributions from both the sample (E) and the indenter tip (E_i). This is described as the reduced modulus, (E_r), which is calculated from the stiffness, S, of the initial part of the unloading curve and the area of contact between indentation tip and sample surface, A, by:

$$S = \frac{dP}{dh} = \frac{2}{\sqrt{\pi}} E_r \sqrt{A} \tag{1}$$

where P is the indentation load on the surface and h is the indentation displacement into the surface. The sample modulus is therefore calculated by correcting for tip deformation by:

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