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In situ micro tensile testing of He^{+2} ion irradiated and implanted single crystal nickel film

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ABSTRACT

The effect of ion irradiation on the tensile properties of pure Ni single crystals was investigated using an *in situ* micro-mechanical testing device inside a scanning electron microscope. A 12.8 μm-thick Ni film with {001} plane normal was irradiated with 6 MeV He^{+2} ions to peak damage of 10 and 19 displacements per atom (dpa). Micro-tensile samples were fabricated from the specimens parallel to the plane of the film using a focused ion beam (FIB) instrument, and tested in tension along [100] direction, up to fracture. The peak strength increased from ~230 MPa for the unirradiated material to about 370 MPa and 500 MPa for the 10 dpa and 19 dpa samples respectively, while the ductility decreased with increasing dose. The surface near the peak damage regions fractured in a brittle manner, while the regions with smaller dose underwent significant plastic deformation. Slip bands extended to the peak-damage zone in the sample with a dose of 19 dpa, but did not propagate further. Transmission electron microscopy confirmed the stopping of the slip bands at the peak-damage region, just before the high He concentration region with voids or bubbles. By removing the peak damage region and the He bubble region with FIB, it was possible to attain propagation of slip bands through the entire remaining thickness of the sample. This material removal also made it possible to calculate the irradiation hardening in the region with peak hardness – thus enabling the separation of hardening effects in the high and low damage regions.

1. Introduction

It is well known that radiation damage can cause increase in strength and decrease in ductility, thus reducing the service life of structural parts in nuclear power reactors [1]. The effect of irradiation on the mechanical properties of structural materials has been the subject of intense study in the past few decades. Such studies have been integral to the worldwide effort to produce materials with greater resistance to higher irradiation doses for future designs of nuclear reactors [2,3]. Ion beam irradiation has been a method of choice to simulate the effects of neutron irradiation in a reactor for some time [4,5], since it enables the attainment of reasonable doses within hours, instead of years inside a reactor. One of the drawbacks of using this method is that the damaged region is very shallow (generally <20 μm), and mechanical testing of such thin layers is extremely difficult. This problem has been solved partially by using nanoindentation to probe the hardness changes of such thin irradiated materials [6–10]. However, the results of nanoindentation tests are difficult to interpret due to two reasons: (a) it is a test with a tri-axial stress state, hence deriving uniaxial tensile properties out of such tests is difficult, and (b) since the process creates a 3-dimensional plastic zone around it that grows with the depth of the indent in a manner which is material dependent, the hardness values measured by nanoindentation are an average of a somewhat uncertain volume of material around the indent [11,12]. Micro compression testing [13–15], which has a uniaxial stress state, has been conducted in the past on ion beam irradiated materials [6,16,17]. While yield stress and work hardening rate are accessible, compression testing comes with its own set of difficulties. Since the failure modes in compression are considerably different from the necking and subsequent fracture in tension, detecting a precise point of failure and measuring total compression is difficult, as the sample does not rupture, but is compressed with accompanying sliding and barrelling effects [16,18–20]. Tensile testing of thin regions of irradiated material can obviate

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these difficulties, and thus is a preferred method for studying the strengthening effects. However, until recently, there have been great technical challenges in the manufacturing of sensitive testing equipment and the fabrication of test samples. Only a limited amount of tensile tests have been performed in the past, such as those conducted on unirradiated material by Kiener et al. [21], Bhattacharyya et al. [22] and Wheeler et al. [23], and those conducted on electroplated and therefore FIB damage free tensile test specimens by Landau et al. [24]. These papers typically discuss either the sub micrometre scale in the irradiated condition, or the meso-scale dimension regime (i.e. a range of a few micrometre to tens of micrometres) in the un-irradiated condition. The meso-scale regime has not been evaluated in tension in the irradiated condition, especially on materials for engineering applications. The technical problems in applying tension at the mesoscale on irradiated materials have been overcome to a large extent in the past few years, and significant advances have been made in both the design and manufacture of *in situ* micro-mechanical testing equipment and the fabrication of micro-tensile samples [23]. In this paper, the authors have attempted to understand the mechanical response of a thin foil of single crystal Ni before and after irradiation by tensile testing of FIB fabricated samples in which the irradiated region is parallel to the gauge length. The methods adopted to perform these tests enable the simultaneous achievement of multiple experimental goals, viz. (a) Real-time visual observation and recording of the deformation behaviour in the high- and low-dose regions of the sample, (b) real-time recording of the stress–strain curves and their association with particular events as observed, such as load drops and strain jumps, (c) calculation of the radiation induced strengthening effects in different regions of the radiated/ion implanted material having different doses, and (d) post-test transmission electron microscopy examination for gaining insight into the slip propagation process. These methods show, for the first time, the distinctive effect of bubbles or voids in the obstruction to slip, resulting in remarkably different deformation behaviour with dosage. These methods together provide significant insights into the mechanical behaviour of ion irradiated material which will enable us to extend these results to the behaviour of bulk neutron irradiated materials in the future.

2. Methods

The tensile specimens tested in this study were manufactured in single crystal Ni foils (obtained from Princeton Scientific, PA, USA). The thickness of the foils was about 12.8 μm. The foils were of 99.999% purity or greater. The normal direction of the foils was <001> which is identical to the ion beam irradiation direction. The tensile axis was roughly parallel to the <100> direction, within ~±2–3°. The ion beam irradiation of the Ni foils was performed using the 2MV Tandetron STAR accelerator at the Australian Nuclear Science and Technology Organisation (ANSTO, Lucas Heights, NSW, Australia). One foil was preserved in the as-received condition, while two other foils were irradiated with 6 MeV He^{+2} ions to different doses, 2×10^{17} ions/cm^{-2} and 3.8×10^{17} ions/cm^{-2}. The irradiation temperature was approximately 25 °C ± 5–10 °C as monitored with a thermocouple mounted close to the foil on the aluminium sample clamp. SRIM calculations of the damage profiles [25] (Fig. 1) predict an average displacement damage of 0.2 dpa and 0.4 dpa through the depth of the two irradiated samples up to about 9.4 μm, with much higher damage peaks (up to 10 dpa and 19 dpa respectively) at the ion stopping point at ~11.85 μm depth. We define the "entry surface" for the ions as the "front surface", and the other, "exit surface" for the ions as the "back surface" for the sake of consistency. SRIM calculations show that very few, if any, ions actually exit at the back surface, since the sample thickness is greater than the maximum range of the 6 MeV He^{+2} ions used. Since the average thickness of the irradiated foils as measured with an SEM was ~12.8 μm, the peak damage stopping point region was predicted to be located within the sample volume. Thus a highly damaged region was formed about a micron inside the "back surface" of the ions. This situation is schematically depicted in Fig. 1, where the region with damage greater than approximately half the peak damage is shown shaded in grey. This region extends from a depth of about 11.6 μm to 12.1 μm. The SRIM calculations show that the peak He concentration occurs at a depth of ~12.0 μm, and is ~5% for the sample with fluence of 2×10^{17} ions/cm^2, and ~9.5% for that with 3.8×10^{17} ions/cm^2. This is plotted in Fig. 1 along with the damage profiles. It is clear that although the peaks of the damage profile are shallower than the He concentration profile by only about 150 nm, the damage profile starts to rise substantially before reaching the peak.

Tensile specimens of the geometry shown in Fig. 2(a) were fabricated in each sample using a Zeiss® Auriga 60™ focused ion beam (FIB). The gauge length was 25–30 μm and the cross sectional area was approximately 10 μm (width) × 13.0 μm ± 0.2 μm (thickness). A grip in the shape of a rectangular hole was milled at the free end of each specimen to provide a structure on which a tensile load could be applied via a hook type gripper (Supplementary Fig. 1). Surface imperfections and curtaining, as well as tapering of the specimen, was minimised by performing a low-current final polishing step at 30 kV, 240 pA. For some specimens, a shallow pattern of fiducial marks was lightly milled at 30 kV, 50 pA on the top surface to allow post-test high resolution digital image correlation on the movies.

The *in situ* tensile tests were conducted in a Zeiss® Ultra Plus™ SEM using a micro-test rig (MTR-3) developed by MicroTesting Solutions® (Hilliard, OH, USA) at the Institute of Materials Engineering in ANSTO. The samples were subjected to elongation by carefully raising a flat-faced silicon loading pin into the tensile grip, and applying a tensile load parallel to the specimen axis. Alignment between the loading direction and the sample axis was adjusted prior to testing to better than 0.5° in plane and 2° out of plane using a pair of motor-driven screws on either side of the sample stage. The displacement rate was 20 nm/s, corresponding to a strain rate of $\sim 8 \times 10^{-4}$ s^{-1}. High definition SEM images (2048 × 1535 pixels) were acquired at small strain intervals during the tests. The displacement or strain was held constant during the image acquisition by pausing the test. The pause at constant strain was accompanied by a small stress relaxation ranging from 0.1 to 1 MPa. For each image, the load recorded at the start of acquisition was used to calculate the engineering stress. A Matlab digital image correlation code was then used to calculate the engineering strain using the displacement of fiducial markers located on either side of the tensile specimen gauge length [13,22].

3. Results

Conducting the tensile tests *in situ* in an SEM allows real-time imaging for a detailed comparison of the deformation behaviour of the unirradiated versus irradiated samples. The SEM image in Fig. 2(a) shows a tensile test specimen before testing, while Fig. 2 (b) shows the final post-tension state of one of the two unirradiated specimens. A movie made from successive SEM images taken during the *in situ* tensile test of the unirradiated specimen is given in Supplementary Video 1. These specimens had an average ultimate tensile strength (UTS) of ~233 MPa and exhibited ductile behaviour with significant necking enabled by the formation of alternating slip steps on multiple slip systems (Fig. 2b and Supplementary Video 1). The corresponding engineering stress–strain