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Characterization of ion beam irradiated 304 stainless steel utilizing nanoindentation and Laue microdiffraction

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ABSTRACT

Characterizing irradiation damage in materials utilized in light water reactors is critical for both material development and application reliability. Here we use both nanoindentation and Laue microdiffraction to characterize both the mechanical response and microstructure evolution due to irradiation. Two different irradiation conditions were considered in 304 stainless steel: 1 dpa and 10 dpa. In addition, an annealed condition of the 10 dpa specimen for 1 h at 500 °C was evaluated. Nanoindentation revealed an increase in hardness due to irradiation and also revealed that hardness saturated in the 10 dpa case. Broadening using Laue microdiffraction peaks indicates a significant plastic deformation in the irradiated area that is in good agreement with both the SRIM calculations and the nanoindentation results.

1. Introduction

Radiation damage in materials is a significant concern in advanced and conventional nuclear reactors, spallation sources, isotope production facilities and fusion technology applications. Embrittlement and environment interaction are key issues during lifetime extension or performance predictions for reactors [1–4]. Of particular interest is the effect of irradiation in stainless steels such as 304 and 316L due to their widespread use as structural materials in light water reactors [5]. However, when subjected to irradiation, austenitic steels harden due to the formation of defect clusters that act as obstacles to dislocation motion under an applied stress [6]. Depending on the material, irradiation temperature, and stacking fault energy of the system, these defect clusters can manifest themselves as stacking fault tetrahedra (SFT) and dislocation loops [7,8]. Furthermore, irradiation induced hardening results in a decrease in ductility, which has been well characterized in metals [9]. Therefore it is important to thoroughly characterize and understand both the defects that are introduced through irradiation and the corresponding change in mechanical properties.

Since reactor components become radioactive in service, especially stainless steels due to the high nickel content, ion beam irradiations are considered as a surrogate for reactor irradiations [10]. In addition ion beam damage has a significant higher damage rate than neutron irradiation and therefore high doses can be achieved in a matter of days rather than years. However, medium energy (2 MeV) light ions (*H*) have a limited penetration depth. Higher ion beam energies are possible but reduce the displacement per atom (dpa) rate while resulting in greater sample activation. One of the benefits of using a low energy proton beam is that it can be used to investigate radiation damage without highly activating the specimen. However one of the limitations to using a lower energy source is the limited penetration depth, therefore necessitating the use of nanoscale post irradiation characterization methods such as nanoindentation [11,12] and synchrotron radiation based X-ray Laue microdiffraction (μXRD) [13]. In this work, these methods are applied to investigate the change in mechanical properties as a result of ion beam irradiation and mapping of the microstructure induced by the ion beam irradiation.

In this study 304 stainless steel (304SS) was studied in two different dose conditions: 1 dpa, 10 dpa. Additionally, the 10 dpa sample was also annealed to determine whether the radiation damage can be annealed out at 500 °C. This paper reports the nanoindentation experiments of both configurations as well as

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the corresponding μXRD of the irradiated region. The purpose of this study is to characterize the hardening induced by ion irradiation and correlate that with the defect density estimated by μXRD.

2. Experimental

The irradiation experiment was conducted using 2 MeV protons in a Tandetron accelerator at the Michigan Ion Beam Laboratory (MIBL). Two specimens of 304SS were irradiated to 1 and 10 dpa, respectively, at 360 °C with a dose rate of $\sim 8 \times 10^{-6}$ dpa/s based on the SRIM calculation [14] (Full cascade option used with a displacement energy of 40 eV for Fe, Cr and Ni). The equivalent doses would be 0.5 and 5 dpa, respectively, if the K–P option is used in SRIM. The sample temperature was monitored using a two-dimensional (2D) thermal imager and the variation was kept within ± 10 °C during the course of irradiation. A detailed description of the proton irradiation procedure was published elsewhere [15]. The annealed specimen was subjected to 500 °C for 1 h in a vacuum furnace. Fig. 1 summarizes the dose profile calculated using SRIM where the x axis represents the penetration depth of the ion beam in cross section.

2.1. Sample preparation for nanoindentation

Both the 1 dpa and 10 dpa samples were polished in cross section with respect to the irradiated surface. During polishing each sample was mounted directly next to a thin piece of steel to prevent deformation and rounding near the edge of the specimen. The samples were planarized using SiC grinding paper with water as a lubricant and were then polished with 0.3 μm, 0.1 μm alumina polishing solutions, and 0.05 μm colloidal silica polishing solution.

2.2. Nanoindentation measurements

Nanoindentation measurements were performed at the Nuclear Materials Laboratory at the University of California, Berkeley on the Micro Materials NanoTest™. The nanoindenter was calibrated against fused silica before each indentation run to allow for cross comparison between samples and indenters [16]. Indents were 200 nm deep and a minimum of 4 μm apart from each other to ensure no interaction of the plastic zone around the indents. An array of 10×8 indents was set near the irradiated edge and an array of 8×8 indents was set on the opposite side where no beam had hit the surface. Comparing the irradiated with the unirradiated edge of the sample ensures that there were no edge effects, and observed differences can be attributed to ion beam irradiation. The indent field in the irradiated region were tilted towards the edge intentionally to increase the depth resolution and potentially resolve the stopping peak, which is shown in Fig. 2 [17]. The indents were all performed in depth-control mode with a loading and unloading rate of 2 mN/s and a dwell time of 5 s. The hardness was obtained by the Oliver–Pharr method [18]. In support of the nanoindentation measurements, an indentation size effect study was performed on the surface of a 10 dpa specimen in both the control and irradiated region. Indents were measured at various depths. A set of three indents was performed at each unique depth. The size effect study was performed on a Hysitron Triboindenter using a Berkovich tip.

Each indentation field was examined using scanning electron microscopy to verify the location of each indent with respect to the sample edge. The data reported (hardness vs. depth from the irradiated surface) reflects the SEM measurements.

2.3. μXRD study

Synchrotron radiation based Laue microdiffraction experiments were conducted at Beamline 12.3.2 of the Advanced Light Source (ALS) at Lawrence Berkeley National Laboratory [19]. A polychromatic X-ray beam (5–24 keV) was focused to $\sim 1 \times 1$ μm^2 by a pair of Kirkpatrick–Baez (KB) mirrors. A schematic of the Laue diffraction setup can be found in the study presented by Kunz et al. [20]. The samples were mounted on a high resolution x–y scan stage with its polished cross-section facing up, and then tilted 45° relative to the incident X-ray beam. For each sample, a fast fluorescence scan was conducted to position the sample edge so as to ensure that all Laue diffraction scans covered the region from the sample edge to about 100–150 μm towards the sample matrix. In this study the diffraction scanning step size was 2 μm and the exposure time at each position was 1 s. Diffraction patterns were recorded in reflection mode with a 2D Pilatus detector mounted at 90° to the incoming X-ray, approximately 140 mm from the probe spot. The detector has 1043×981 pixels and each pixel is about 170×170 μm^2 in size. Calibrations for sample-to-detector

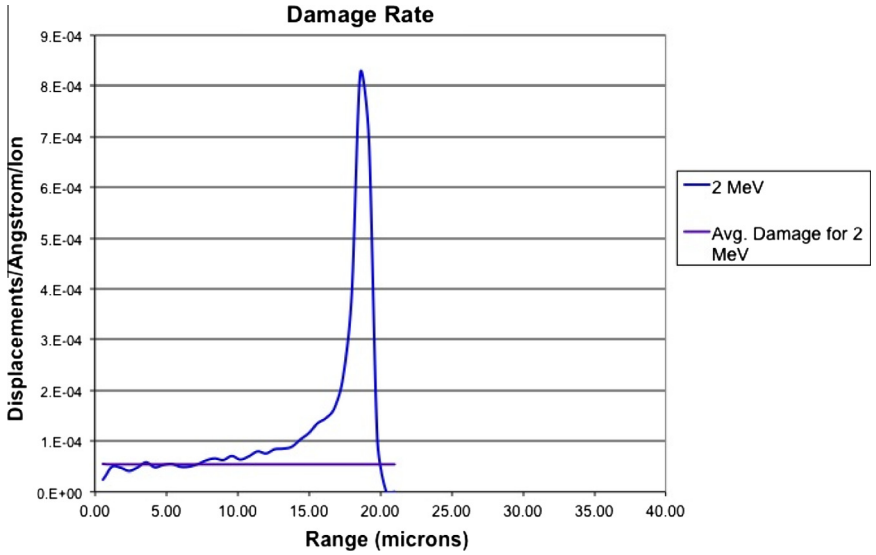

Fig. 1. Calculated SRIM dose profile showing the damage rate.