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Micro-tensile testing of reduced-activation ferritic steel F82H irradiated with Fe and He ions

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ABSTRACT

Micro-tensile tests were conducted on reduced-activation ferritic steel F82H specimens after irradiation with Fe and He ions. The displacement damage levels were 50 and 200 dpa, and helium concentrations were 0, 2000 and 5000 appm. The specimens had dimensions of 8 × 1 × 1 µm for the gage section and they were tension tested at room temperature in a vacuum. The yield strengths of the unirradiated specimens were close to the literature value reported for a large, unirradiated specimen. The increases in yield strength and ultimate tensile strength due to Fe ion irradiation were clearly observed. The loss of work hardening was confirmed for the 200 dpa specimens (0 or 2000 appmHe). Higher yield strength and ultimate tensile strength were confirmed for the 50 dpa/5000 appmHe specimen compared to the no helium specimen (50 dpa/0 appmHe). However, no apparent helium effects were confirmed for the 200 dpa/2000 appmHe specimen by the micro-tensile test at room temperature.

1. Introduction

Reduced-activation ferritic steel has been considered as one of the candidate materials for the blanket component of fusion reactors [1]. The fusion blanket structure will be subjected to severe heat loads and high fluxes of 14 MeV fusion neutrons. The radiation induced mechanical property changes of structural materials are a major issue in the blanket design for fusion reactors; especially, helium embrittlement of the structural materials is a major concern. However, suitable irradiation facilities to investigate the radiation effects of fusion neutrons do not exist presently. In the absence of intense fusion neutron irradiation facilities, an integrated understanding of the radiation effects of fusion neutrons by both experimental studies with fusion-relevant irradiation conditions using fission neutrons and ions and simulation or modeling studies are important.

To investigate helium embrittlement of the blanket structural materials, dual ion beam irradiation with Fe and He ions is a useful technique because it can achieve accurate injections of various helium concentrations in a broad temperature range with formation of radiation defects. However, the irradiated region is limited to a few microns below the surface due to the short penetration ranges of the ions. The nanoindentation technique is widely used to measure local hardness of specific regions of interest including ion-irradiated regions [2]. According to Tabor's relationship [3], yield stress, or strictly speaking flow stress, can be derived from hardness. Various research studies to obtain additional information on mechanical properties such as yield stress, Young's modulus and work-hardening coefficient, from load-displacement curve in nanoindentation experiments have been conducted [2,4]. However, it is difficult to estimate radiation-induced changes in post yielding behavior such as ductility deterioration from the hardness measurement. Ultra-small testing technologies (USTTs), such as micro-tensile testing and micro-compression testing [5,6], are useful to estimate the post yielding behavior of the ion-irradiated region because the specimen size is typically in the µm range for the usual ion irradiation range. Although there is a need to consider effects of specimen size [7,8] and focused ion beam (FIB) damage [9], most micro-sized specimens are fabricated using a FIB system, and radiation-induced mechanical property changes can be estimated by using USTTs [5,10].

In this study, the micro-tensile testing method that had been developed for characterization of grain boundary fracture [11,12] was applied to ion-irradiated reduced-activation ferritic steel F82H specimens to estimate helium effects on tensile properties.

2. Experimental procedure

Reduced-activation ferritic steel F82H IEA heat (Fe-0.1C-8Cr-2W-0.2V-0.04Ta) was used. Five plates, typically 6 × 3 × 0.6 mm in size, were cut from the larger steel plate and their surfaces were polished

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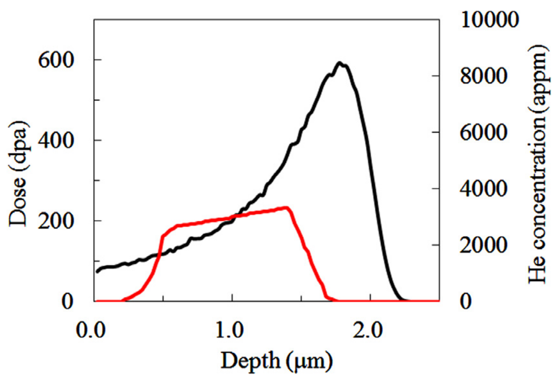

Fig. 1. Depth distributions of dose and helium concentration for the D2 specimen calculated by the SRIM code [13].

Table 1
Conditions of ion irradiation.

ID	Ion	Temperature (°C)	Dose (dpa)[a]	He conc. (appm)[a]
S1	10.5 MeV Fe^{3+}	360	50	–
S2	10.5 MeV Fe^{3+}	360	200	–
D1	10.5 MeV Fe^{3+}/1.05 MeV He^{+}	360	50	5000
D2	10.5 MeV Fe^{3+}/1.05 MeV He^{+}	360	200	2000

[a] Values at the depth of 1 μm.

mechanically and electrolytically to remove surface damage. Ion irradiations were carried out for the plates in the facility, Takasaki Ion Accelerators for Advanced Radiation (TIARA) in the National Institutes for Quantum and Radiological Science and Technology (QST). The plates were heated through the water-cooled specimen holder with the electron beam heater, and the temperature was maintained at 360 °C by monitoring with a radiation thermometer. Two of the plates were irradiated with 10.5 MeV Fe^{3+} ions and other two plates were irradiated with 1.05 MeV He^{+} ions simultaneously with the Fe ions. For the He ion irradiations, thin aluminum foils were used to degrade the He ions to the desired energies. To achieve uniform irradiation, a spot beam typically within 2 mm in diameter for Fe ions was raster-scanned. Fig. 1 shows the depth distributions of dose and helium concentration calculated by the SRIM code [13] using the Kinchin-Pease option with the displacement threshold energy of 40 eV. The plates are designated as S1, S2, D1 and D2 according to the dose and helium concentration. In the present study, the dose and helium concentration were defined as the values at 1 μm depth. The irradiation conditions are summarized in Table 1. The last plate was used for micro-tensile tests in the unirradiated condition.

The micro-tensile specimens were fabricated as follows. First, electron backscatter diffraction (EBSD) maps were obtained using a scanning electron microscope (SEM; Carl Zeiss ULTRA-55) with the TSL DigiView EBSD detector to detect the location of boundaries (prior-austenite grain boundary, martensite packet and block boundary) and the crystallographic orientation at each plate surface. Next, a micro-piece, a small amount of material typically 15 × 10 × 5 μm in size, was removed from each plate surface by the FIB lift-out technique. The micro-piece was a single martensite block which contained some lath boundaries, and its orientation was selected to set the tensile direction in the subsequent micro-tensile tests to the ⟨001⟩ direction. The piece was sliced and then micro-tensile specimens were fabricated by FIB micro-processing.

The dimensions of a micro-tensile specimen are shown in Fig. 2. The position of the gage section corresponded to the depth between 0.5 and 1.5 μm from the plate surface. A part of the grip sections was made by

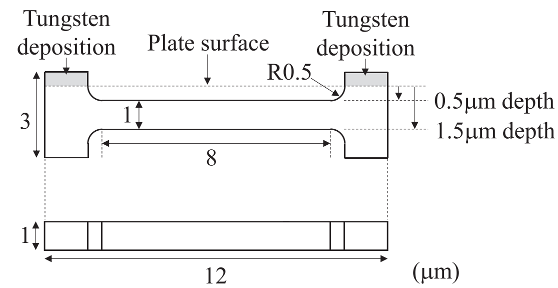

Fig. 2. Dimensions of micro-tensile specimen.

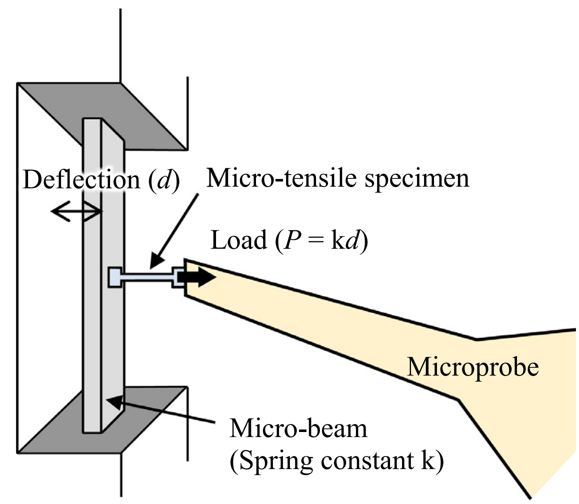

Fig. 3. Schematic image of micro-tensile test setup. The micro-tensile specimen was fixed to the micro-beam and microprobe by tungsten deposition.

using a tungsten deposition system equipped on the FIB system. Three specimens were fabricated from the unirradiated plate, and one specimen was fabricated from each of the ion-irradiated plates (S1, S2, D1 and D2).

Micro-tensile tests were carried out in the FIB-SEM system (Hitachi NB5000), in a vacuum at room temperature. A schematic image for the micro-tensile test setup is shown in Fig. 3. Details of the micro-tensile test procedure have been reported elsewhere [12]. The specimen was fixed to a microprobe and a double-supported micro-beam by tungsten deposition. The micro-beam was fabricated from a single crystal of silicon by FIB. Then, the microprobe was moved rightwards in Fig. 3 until the specimen failed. The applied load (P) was calculated from the spring constant (k) and deflection (x) of the micro-beam. The spring constant was measured by using the ultra-micro-hardness tester; an indentation was made into the center of the micro-beam with load up to 1000 μN. The load (P)-displacement (h) curve for the micro-beam is shown in Fig. 4. The relationship between dP/dh and displacement in the unloading part of the load-displacement curve is also shown in Fig. 4 together with the fitting curve. For Fig. 4(b), the displacements have been shifted laterally by subtracting the final displacement (h_f) marked by the arrow in Fig. 4(a) from the total displacement. The dP/dh was slightly increased as the displacement increased. This might mean that elastic deformation of the micro-beam appeared in the load-displacement curve. Therefore, the spring constant of the beam was determined as the following equation, $k = dP/dh\ (h \to 0)$. The maximum deviation from the fitting curve was 25 N/m. The spring constant was determined to be 582 ± 25 N/m. Both the deformation of specimen and the deflection of the micro-beam during the tensile test were estimated from scanning ion microscope (SIM) image records with a frame rate of 20 fps (frames per second) at 512 × 512 pixel resolution (14.45 × 14.45 μm). The resolution of SIM images corresponded to 28 nm, so that the resolution of the applied load was 16 μN. After the