



# Development of a triple beam irradiation facility

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## Abstract

A triple beam ion irradiation facility has been developed to study the synergistic effects of displacement damage, helium and hydrogen atoms on microstructural changes of materials under irradiation environments simulating a fusion reactor. The system consists of a vacuum chamber and three beamlines, which are connected with each electrostatic accelerator. Samples can be irradiated in the wide temperature range from liquid nitrogen to 1273 K in the chamber by replacing two kinds of sample stages alternatively. An austenitic stainless steel was simultaneously irradiated with triple beam of nickel, helium and hydrogen ions at 573–673 K using this facility and TEM observations were carried out from a cross sectional view normal to the incident surface. It was shown that the number density of dislocation loops decreased in the region where hydrogen and helium were deposited in comparison with ones in the region where only displacement damage was induced to a similar damage level. © 1998 Elsevier Science B.V. All rights reserved.

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## 1. Introduction

Materials for a fusion reactor, which include structural materials, solid tritium breeder materials and ceramics materials as insulators, will be exposed to severe irradiation environment of heavy atomic displacement damage and large amounts of helium and hydrogen atoms including tritium, produced by transmutation reactions like  $(n, \alpha)$  and  $(n, p)$ . In order to develop materials for a fusion reactor, it is important to study the synergistic effects of both atomic displacement damage and transmutation reaction gas products as helium and hydrogen on properties and degradation of durability of fusion reactor materials. Therefore, to simulate the irradiation environment of a fusion reactor, a triple ion beam irradiation facility [1], which can simultaneously irradiate three species of ions on a target, has been developed and completed. We have developed a grade-up triple ion beam irradiation facility.

In this paper, the system of the facility is presented. Also, some results of microstructural changes in an austenitic stainless steel irradiated with triple ion beam are described.

## 2. Facility

The whole irradiation system is shown in Fig. 1. The facility consists of two main components: one is incident beam ports to induce the ion beams from the accelerators and another is a vacuum chamber for irradiation.

### 2.1. Incident beam ports

The incident ports have three beamlines as shown in Fig. 1. One is used to lead heavy ions such as nickel and oxygen to provide atomic displacement damage to materials. The other two beamlines are necessary to implant light ions as helium and hydrogen into materials. The beamline for heavy ions connects with a 3 MV tandem accelerator, those for hydrogen and helium ions with a 0.4 MV ion implanter and a 3 MV single-ended accelerator, respectively. These three kinds of beams are focused on a spot of the target sample. The beamline from the ion source is essentially normal to the target sample, whereas the planar angle between each remaining beamline and the center one is 15°. These beamlines are arranged at almost the same level from the floor.

In order to obtain a more uniform and wider irradiation area, scanners producing a triangle wave form in horizontal ( $X$ ) and vertical ( $Y$ ) direction are installed to all beamlines and its polarized wave is a triangle. The

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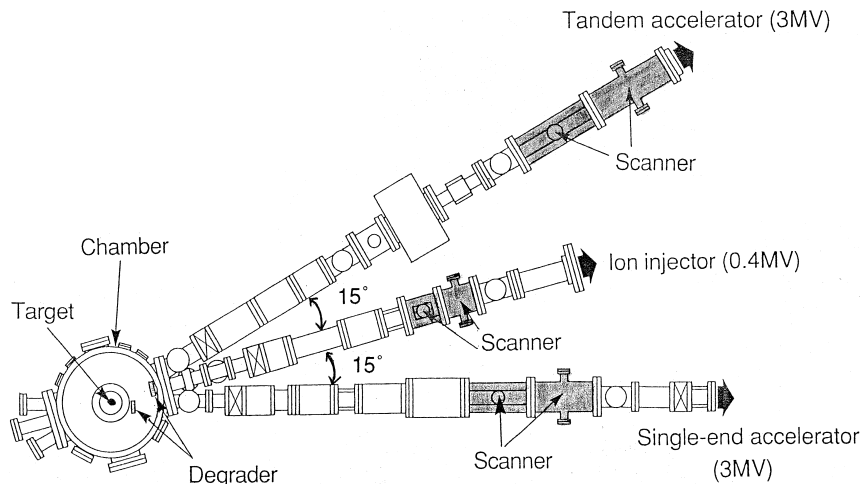


Fig. 1. A two-dimensional plane view of the chamber and the beamlines.

location of the scanner in each beamline is shown in Fig. 1. The frequency of each scanner is changeable in three modes: the modes in the  $X$  and  $Y$  direction are 10 k, 1 kHz and 100 Hz, and 1 k, 100 and 10 Hz, respectively. Furthermore, the scanner also possesses an offset-function in order to shift the beam up to  $\pm 10$  mm in the  $X$  or  $Y$  direction.

## 2.2. Vacuum chamber

The chamber is manufactured with 304 stainless steel. The dimensions are about 50 cm in diameter and 50 cm in height. The vacuum of the chamber was below  $2.6 \times 10^{-6}$  Pa without heating and below  $1.3 \times 10^{-5}$  Pa during heating of 1273 K.

The longitudinal sample stage to irradiate samples is installed in the center position of the upper hatch of the chamber. This is movable up and down in the  $Y$  direction to adjust the positions of sample holders and micro Faraday cups, which are described below. There are two kinds of sample stages to irradiate samples at lower or elevated temperature, which are removable alternatively: one is a cold stage available to irradiation in the temperature range from liquid nitrogen temperature to room temperature, another is a hot one for elevated temperature irradiation, which is available from 473 to 1273 K. Fig. 2 shows the heating sample stage. The sample stage possesses three micro Faraday cups and three sample holders. Each sample holder is capable of loading either seven samples of 3 mm in diameter for a transmission electron microscope (TEM) or one sample of 10 mm in diameter at one time by replacing the cap of a sample holder alternatively. Each micro Faraday cup essentially faces normal to each beamline to measure the beam current of each beamline and has seven round holes or one round hole alternatively. This system cannot directly measure the beam currents of each beamline

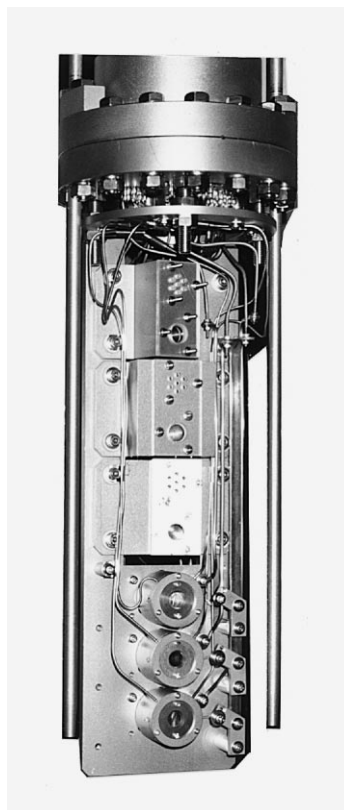


Fig. 2. The heating sample stage for elevated temperature irradiation: the upper three boxes with rectangular shape are micro Faraday cups. The lower three cylinders are sample holders. In the sample holders, the upper one is for TEM samples and the middle is for a disk sample of 10 mm in diameter.

during irradiation. However, the beam currents run sufficiently stable during irradiation and the correct dpa

as well as hydrogen and helium concentrations can be determined by often measuring the beam currents using the micro Faraday cups. The position and the diameter of each hole on it correspond to those of each sample of a sample holder. The sample stage can shift up and down in the vertical direction to irradiate samples and measure beam currents as described above. Therefore, the beam current cannot be measured during irradiation, unfortunately. The irradiation is available in the area  $15 \text{ mm} \times 15 \text{ mm}$  at maximum using the scanners.

A degrader is installed on each beamline of the ion implanter and of the single-ended machine in the chamber to inject light ions relatively uniformly in the depth direction of samples. It locates at the position of about 20 cm toward each accelerator from the target. The nickel foils are supplied to the degraders and its thickness is  $1 \mu\text{m}$  for each beamline of the ion implanter and the single-ended accelerator, respectively. A nickel foil has a rectangular shape and its dimension is  $68 \text{ mm} \times 50 \text{ mm}$ . The degrader can be anticlockwise rotated to the angle of  $45^\circ$  from the plane normal to the beamline.

Most of the action described above can be remote-controlled in the control room for electrostatic accelerators.

### 3. Microstructural changes

#### 3.1. Experimental

An austenitic stainless steel (JPCA) with the main chemical composition of Fe–16Ni–14Cr–0.25Ti–2.5Mo in wt% was used in this study. Samples were solution-annealed and then simultaneously irradiated with a tri-

ple ion beam of 12.0 MeV nickel, 1.0 MeV helium and 350 keV hydrogen ions in the temperature range of 573–673 K. The energies of the helium and hydrogen ions were selected appropriately in order to implant them at the depth behind the incident surface. The theoretical depth profiles of atomic displacement damage produced by nickel ions and the concentrations of both helium and hydrogen injected in 316 stainless steel were calculated using the TRIM code 89 [2] with threshold energy of 40 eV. In order to carry out TEM observations of the irradiated zone, cross sectional views normal to the incident surface were prepared from the irradiated samples [3].

#### 3.2. Results

The calculated depth profile of atomic displacement damage by nickel ions, and the concentrations of helium and hydrogen atoms in 316 stainless steel in this study are shown in Fig. 3. The depth distribution of the dislocation structures in JPCA irradiated with triple beam is given in Fig. 4. The irradiation condition is as follows: JPCA irradiated with triple beam to 56 dpa, 20570 appm H and 11270 appm He in the depth of  $1.6 \mu\text{m}$  from the incident surface. The displacement rate and the injection rates of H and He were estimated to be 3.1 dpa/s, 1.1 appm H/s and 0.6 appm He/s, respectively. High densities of dislocation loops and lines were observed from the depth of  $0.5 \mu\text{m}$  to the projected range of nickel ions. However, the region of lower dislocation density was observed between 1.2 and  $1.5 \mu\text{m}$  in depth behind the surface, while the dislocation density in the shallower or deeper region was higher as shown in Fig. 4. A comparison between the depth distribution of dislocation structures observed (Fig. 4) and that of displacement

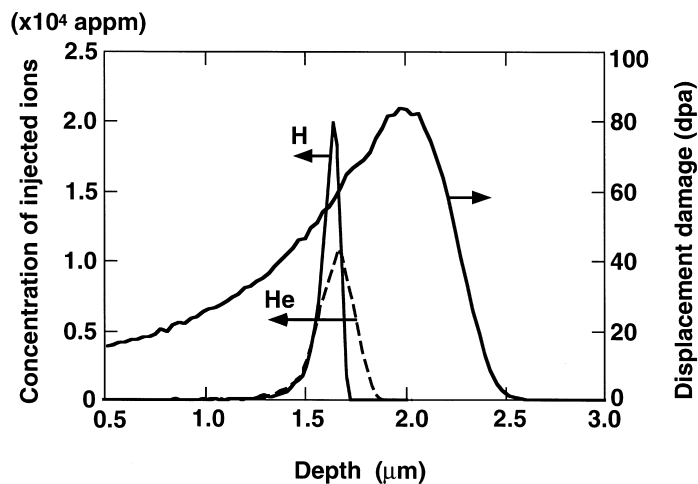


Fig. 3. Depth profiles of displacement damage by 12.0 MeV Ni ions and concentrations of 1.0 MeV He and 350 keV H in 316 stainless steel, calculated by using TRIM code.

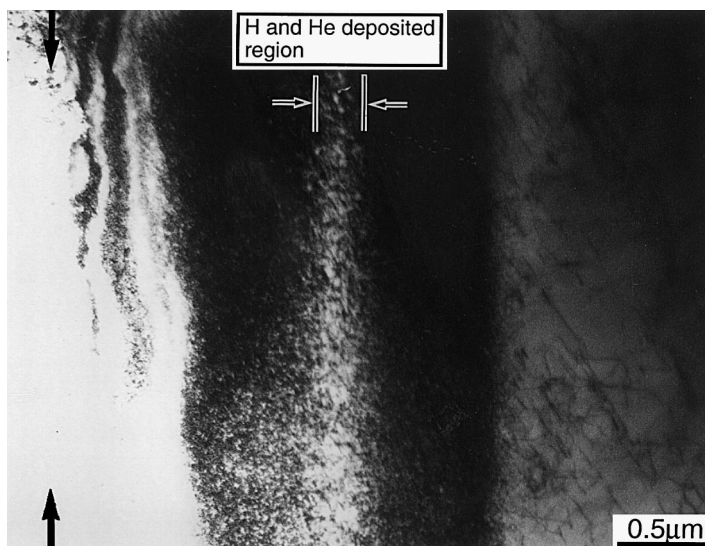


Fig. 4. The depth profiles of dislocation structures in JPCA irradiated with triple beam of 12 MeV  $\text{Ni}^{3+}$ , 1.0 MeV  $\text{He}^+$  and 350 keV  $\text{H}^+$  to 56 dpa, 20570 appm H and 11270 appm He at 573–673 K. The arrows in the left side indicate the incident surface.

damage by calculation (Fig. 3) shows a slight difference of the position in depth between the region of lower dislocation density observed in Fig. 4 and the region where hydrogen and helium atoms were calculated to be injected. The former is shallower than the latter. However, considering that there would be any interaction among damage structures and deposited helium and hydrogen atoms, the region of lower dislocation density observed in Fig. 4 could be regarded as the region where hydrogen and helium atoms accumulated. Fig. 5 shows the depth distribution of the number density of dislocation loops in the range of 0.8–1.8  $\mu\text{m}$  in depth in Fig. 4. Their number density varies in this depth range: while the loop density in the region of 0.8–1.0  $\mu\text{m}$  in depth and beyond the depth of 1.4  $\mu\text{m}$  was about  $2.0 \times 10^{22} \text{ m}^{-3}$ , it decreased to  $7 \times 10^{21}$ – $9 \times 10^{21} \text{ m}^{-3}$  in the region of 1.0–1.4  $\mu\text{m}$  in depth. Its depth profile is given in Fig. 6. This indicates that the number density of dislocation loop decreases in the region where hydrogen and helium were simultaneously implanted. Considering a strong interaction between dislocation and hydrogen in steels at the relatively lower temperature [4], this phenomenon seems to be due to strong interaction among defects produced by displacement, helium and hydrogen atoms.

Further, observation at high magnification for the region in which hydrogen and helium were simultaneously injected shows that very small cavities of high number density uniformly distribute in the matrix. Such small cavities of high number density were not found in any region other than that of about 1.0–1.4  $\mu\text{m}$  in depth. The average diameter and the number density of the cavities were 1.4 nm and  $1.5 \times 10^{24} \text{ m}^{-3}$ , respectively.

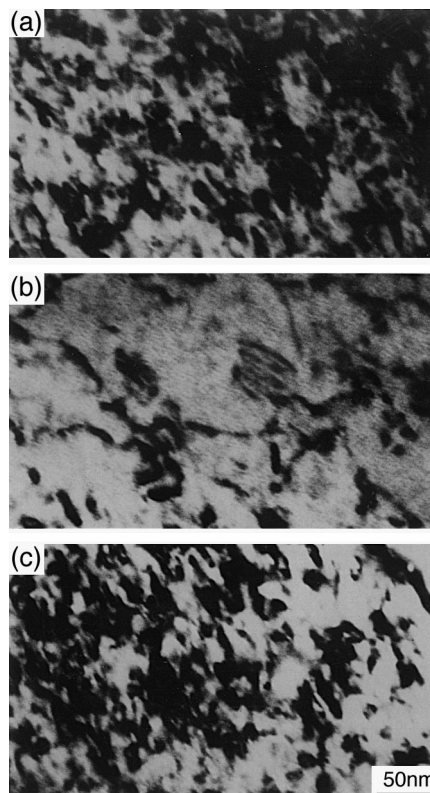


Fig. 5. Comparisons of the distributions of dislocation structures observed in the region of each depth (a) 0.8–1.0  $\mu\text{m}$ , (b) 1.1–1.3  $\mu\text{m}$ , (c) 1.4–1.6  $\mu\text{m}$ .

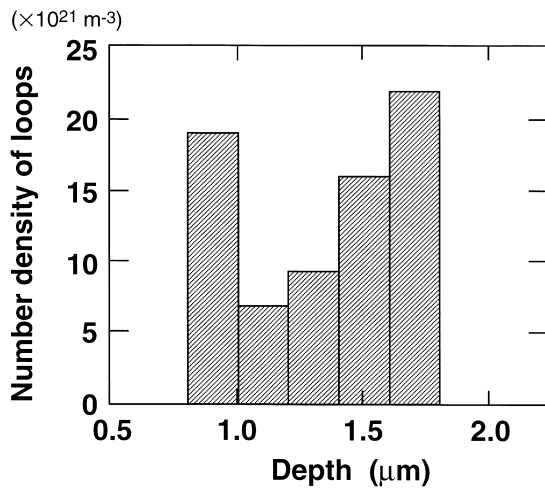


Fig. 6. Depth profile of the number density of dislocation loops in the range of 0.8–1.8 μm in depth.

#### 4. Summary

A triple beam ion irradiation facility has been developed to study the synergistic effects of displacement damage, helium and hydrogen atoms on microstructural changes of materials. An austenitic stainless steel was simultaneously irradiated with triple beam of nickel,

helium and hydrogen ions using this facility. A comparison of layers only Ni-irradiated with tripple beam irradiated regions has shown that the simultaneous irradiation of hydrogen, helium and nickel ions can reduce the number density of dislocation loops in the temperature range between 573 and 673 K. Very small cavities of high number density were observed in the region simultaneously irradiated with hydrogen, helium and nickel ions.

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