Microstructure characterization of reactor pressure vessel steel A508-3 irradiated by heavy ion

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Abstract. As one of the key structures used in nuclear power plants, the study of irradiation effects of pressure vessel steel (RPV) is of great scientific value to nuclear safety. The RPV steel was irradiated by Fe ions up to three different irradiation damage levels (0.08 dpa, 0.15 dpa, and 0.6 dpa). The transmission electron microscope was utilized to measure the irradiated microstructure and it was found that after the irradiation of 0.08 dpa, the density and size of dislocation loops in Fe ions irradiated samples was small and the dislocation loops were distributed near the surface. When irradiation dose was up to 0.15 dpa, many black dots were distributed in the whole irradiation region and some large size dislocation loops appeared. In the case of 0.6 dpa, a large number of dislocation loops were produced and the distribution of dislocation loops extended to the whole irradiation region owing to the production and growth of defects such as vacancies and black dots.

1. Introduction

The reactor pressure vessel (RPV) has been widely known as the only irreplaceable large structural component in the fission reactors, which is subjected to a large amount of neutron irradiation during the whole-life service of several decades [1-3]. Therefore, the safety and stability of reactor pressure vessel are crucial to the lifetime of the nuclear power plants. It is inevitable that the vacancies and dislocations induced by irradiation would cause the hardening and embrittlement of the RPV steels. In order to evaluate and improve the performance of RPV, it is necessary to conduct a lot of research on the microstructure and mechanical properties of PRV steel after irradiation [4-6]. At present, there are several major defects such as bulk damage, Cu-rich precipitations, and segregation of vacancies, impurity atoms or dislocations at grain boundaries.

Considering the low content of copper and other detrimental impurities in the RPV steel A508-3 used in China, the bulk damage including Frenkel pair defects, dislocation loops [7-10] has been reported as the dominant factor in the irradiation hardening and embrittlement. The irradiation defects hinder the movement of dislocations and the pile-up of dislocations would prevent the deformation of the steel,
resulting in the increase of brittleness. Therefore, the relationship between bulk damage and the reduction of the mechanical properties of RPV steel is still worth further study.

Currently, for simulating the neutron irradiation, the ion implantation has been widely used in study the microstructure evolution of materials under irradiation [4, 11, 12]. Moreover, the transmission electron microscope (TEM) has also been used to study the evolution of microstructure of ion implantation layer in materials.

In this study, the China A508-3 steels were irradiated with three energies (1.2, 2.4, 3.3 MeV) Fe+ ions at room temperature to reach three different damage levels (0.08, 0.15, 0.6 dpa). The microstructure of samples was characterized by transmission electron microscope (TEM) combined with the focused ion beam (FIB) technology. The selected irradiation damaged region characterization can be obtained from the cross section samples for investigating the distribution and characterization of irradiation defects. The aim of this study is to investigate the microstructure evolution of China A508-3 steel with various irradiation damage levels.

2. Materials and experiments
The chemical compositions of China A508-3 steel were shown in Table 1. The specimens were cut into 6 mm×6 mm×0.5 mm plates from a reactor pressure vessel shell. These specimens were mechanically ground by SiC grit paper of 600, 800, 1000, 1500, 2000 mesh and then polished with diamond suspension of 9 μm, 3 μm, 1 μm. Finally, the specimens were vibration polished with 0.05 μm polishing solution, followed by 15 minutes of ultrasonic in acetone, alcohol and deionized water, and blown dry.

The EBSD results of original samples were shown in Fig. 1. There is no preferred orientation in original samples with BCC structure. The average size of grain is about 15 μm.

The irradiation experiment was performed at a tandem accelerator in Wuhan University. Specimens were stick to the target chamber by conductive adhesive with good thermal conductivity. The uniform Fe+ ions beam coverage area in front of the target is controlled in the range of 6*6mm. Each specimen was irradiated by three energies (1.2, 2.4, 3.3 MeV) Fe+ ions with specific fluences at different energy to produce an irradiation damage plateau to some depths. The damage plateau was calculated by SRIM-2013 using Kinchin–Pease model, quick calculation. The threshold displacement energy $E_d$ of major elements Fe, Cr, Ni and Mo were selected into 40 eV. The corresponding simulation result was shown in Fig. 2.

The microstructure of RPV steel irradiated by Fe+ ions was characterized by TEM. The TEM specimens were prepared by using focused ion beam (FIB) technology on the FEI Helios NanoLab 600i with Lift-Out mode.

Table 1. The chemical compositions of China A508-3 steel

| Element | Fe | C  | Si  | Mn | S  | P  | Cr | Ni | Cu | Mo | V  |
|---------|----|----|-----|----|----|----|----|----|----|----|----|
| wt. %   | Bal. | 0.167 | 0.193 | 1.35 | 0.002 | 0.005 | 0.086 | 0.738 | 0.027 | 0.481 | 0.007 |

Fig. 1. EBSD results of the original RPV steels which showing almost random texture
3. Results and discussion

In Fig. 3, Fig. 4, and Fig. 5, there are TEM images of the A508-3 steel irradiated by Fe\textsuperscript{+} ions with 0.08 dpa, 0.15 dpa, 0.6 dpa. At irradiation damage valued 0.08 dpa in Fig. 3, there are few dislocation loops produced close to surface. It is noteworthy that there are no obvious defects around the precipitates with large size above 200 nm. At 0.15 dpa in Fig. 4, more dislocation loops appeared in shallow layer and many small size defects such as black dots are produced in deep layer. Finally, at 0.6 dpa in Fig. 5, with higher irradiation damage, the density of dislocation loops becomes larger and the distribution of defects such as black dots and dislocation loops gradually and evenly expands to the interior. The number of defects around the precipitates has a significant increase. However, the precipitates are still stable. In addition, different density network of dislocation loops can be formed due to irradiation. From 0.08 dpa to 0.6 dpa, the dislocation loops become easier to distinguish and the size of dislocation loops obviously increase. At the higher dose, the density of dislocation loops increased evidently and interaction between dislocation loops would be expected.

Fig. 3. Microstructure images of sample with plateau irradiation damage of 0.08 dpa
In this study, the microstructure features of RPV steel irradiated by Fe$^+$ ions showed dependence on the depth. When the irradiation damage of the plateau is 0.08 dpa, there was small density of dislocation loops distributed in the shallow layer and the dislocation loops were not clear. When the irradiation damage was up to 0.15 dpa, the black dots were mainly distributed in bulk and small density of dislocation loops with large size were produced. When the irradiation damage reached 0.6 dpa, the larger size and higher density dislocation loops appeared, due to the production and growth of defects such as vacancies and black dots [13, 14].

4. Conclusion
The microstructure in RPV steel irradiated by Fe$^+$ ions was investigated by Transmission Electron Microscope. With the increase of irradiation damage, the size and density of dislocation loops increased due to the production and growth of defects, such as vacancies and black dots. The distribution of defects such as black dots and dislocation loops also gradually expanded in the whole bulk owing to the accumulation of irradiation damage.

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