

# Synergistic effect of displacement damage and helium atoms on radiation hardening in F82H at TIARA facility

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## Abstract

Micro-indentation hardness was measured for the irradiated F82H steels by single (10.5 MeV Fe<sup>3+</sup>) beam or dual (10.5 MeV Fe<sup>3+</sup> and 1.05 MeV He<sup>+</sup> ions) beam at the TIARA facility in JAERI. The extra component of radiation hardening due to helium was slightly detected in the dual-beam (10 appmHe/dpa) irradiation at 633 K up to 33 dpa. As increased the ratio of He/dpa (100 appmHe/dpa), the extra component due to helium was increased. The microstructures in single/dual (10 appmHe/dpa) ion beam irradiated F82H steels consisted of interstitial loops and defect clusters at 50 dpa. However, at a higher ratio of He/dpa (100 appmHe/dpa), nano-voids were also observed in dual ion irradiated F82H.

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## 1. Introduction

Most crucial issues on R&D of reduced-activation ferritic/martensitic steels may be the effect of helium on the degradation of fracture toughness [1–4]. The synergistic effects of displacement damage and helium on mechanical properties and microstructure in F82H steel can be partially simulated by martensitic steels doped with <sup>10</sup>B or <sup>58</sup>Ni in a mixed spectrum fission reactor [5–8]. However, the control of the helium production rate during irradiation is difficult and the chemical effects of B or Ni doping on mechanical property are not necessarily small before and after irradiation. A multi-ion irradiation experiment is very important method to control of helium production ratio during irradiation [9–11]. Particularly, this method produces fusion relevant high-energy cascades at controllable helium production (He/dpa). Moreover, the effects of helium on radiation

hardening behavior can be evaluated by combining ion-irradiation experiments and an ultra micro-indentation technique [12,13]. It is considered that a comparison of radiation-induced hardening can approximately estimate a change in radiation-induced embrittlement because the positive correlation is generally observed between radiation hardening and radiation-embrittlement in a ferritic/martensitic steel. Therefore, finding the condition of generating an extra component of radiation hardening in matrix of F82H due to helium may be useful to evaluate the radiation-embrittlement of high-dose irradiated Ni-doped F82H steel in HFIR.

The purpose of this study is to examine the extra component of radiation hardening due to implanted helium atoms (up to ~3000 appmHe) in F82H under ratio of 0, 10, 100 appmHe/dpa.

## 2. Experimental procedure

The material used in this study was F82H IEA heat. The detailed description of the material has been published elsewhere [14]. The heat treatment was normalized

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at 1313 K for 0.5 h and tempered at temperatures of 1023 (Std) and 1073 (HT) K for 1 h. In this study, the purpose of the heat treatment was that the density of dislocations in F82H steel decreased with high temperature tempering (F82H HT) in order to compare with the radiation hardening after irradiation. The materials were cut to small coupon type specimens ( $6 \times 2 \times 0.8 \text{ mm}^3$ ). One of the  $6 \times 0.8 \text{ mm}$  sides was irradiated after polishing with SiC paper #4000 and  $0.3 \mu\text{m}$  alumina powder and finally to an electrolytic surface polishing.

The ion-beam irradiation experiment was carried out at the TIARA facility in JAERI. The specimens were irradiated at 543 and 633 K with  $10.5 \text{ MeV-Fe}^{3+}$  and  $1.05 \text{ MeV-He}^+$  ions. Helium implantation was performed using an aluminum foil energy degrader in order to control the helium distribution in the depth range of about  $0.5\text{--}1.3 \mu\text{m}$  from the specimen surface. The irradiation was performed to 3.3–33 dpa at the depth of  $0.6 \mu\text{m}$  from the irradiation surface. In previous study, the depth of the deformed region beneath the indent was about 4–5 times indentation depth from the results of TEM images [15]. The average deformed region contained 1.5–2 times indentation depth. In this study, the dpa was equal to the 1.5 times indentation depth. The damage rate was about  $1.0 \times 10^{-3} \text{ dpa/s}$ . The ratio of helium to dpa was 0, 10, and  $100 \text{ appmHe/dpa}$ .

The irradiated specimens were then indentation-tested at a load range of 10–25 mN using an UMIS-2000 (CSIRO, Australia) ultra micro-indentation testing system. The direction of indentation was chosen to be parallel to the ion beam axis or normal to the irradiated surface. The shape of the indenter tip was a Berkovich tip. The micro-indentation results were analyzed in the manner outlined by Oliver and Pharr [16]. Micro-indentation tests were performed at loads to penetrate about  $0.40 \mu\text{m}$  in this study.

The irradiated specimens were made into thin films, with a Hitachi FB-2000A focused ion beam (FIB) processing instrument with micro-sampling system at the Tokai Hot Laboratory. The details of the FIB micro-sampling procedure have been explained elsewhere [17]. The microstructural examination was carried out using a JEOL JEM-2000FX transmission electron microscope (TEM) operated at 200 kV.

### 3. Results and discussion

#### 3.1. Temperature and dose dependence of radiation hardening without helium implantation

Fig. 1 shows the dependence of radiation hardening (Normalized hardening; Irra. Hardness/Unirra. Hardness) on irradiation temperature at 3.3 dpa in single ion irradiated F82H (Std) steel. The heavy radiation hard-

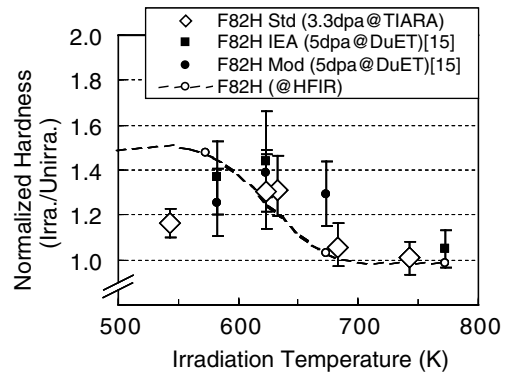


Fig. 1. Dependence of radiation hardening on irradiation temperature in F82H irradiated by  $10.5 \text{ MeV Fe}^{3+}$  ions.

ening occurred in irradiated F82H at below 633 K. This temperature dependence behavior agrees approximately with the reports of micro-hardness evaluation about ferritic/martensitic steels irradiated at the DuET Facility [15] and consistent results of radiation hardening used by the micro-indentation testing is consequently obtained.

The dose dependency of radiation hardening in F82H (HT) steel is shown in Fig. 2. At both irradiation temperature of 543 and 633 K, micro-hardness tended to increase with displacement damage up to about 30 dpa. Radiation hardening of F82H steel irradiated at 633 K is also larger than that of F82H steel irradiated at 543 K.

Fig. 3(a)–(e) show the microstructural development in F82H (HT) steel unirradiated and irradiated at 633 K between 3 and 50 dpa. The microstructures in irradiated specimens consisted of Interstitial-type loops (of diameter about  $20 \text{ nm}$ ), defect clusters ( $<5 \text{ nm}$  diameter) and network dislocations. Fig. 3(f) indicates the size distribution of defect clusters and I-loops in irradiated F82H (HT) up to 50 dpa. As a result, the size distribution is less affected by the irradiation dose, while the

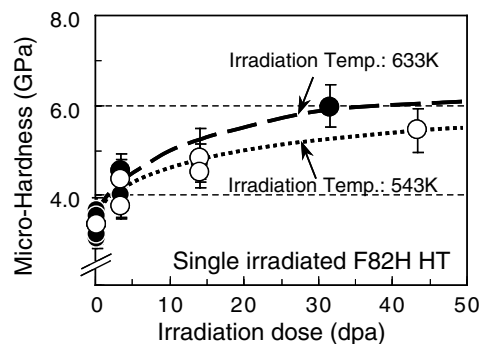


Fig. 2. Dose dependence on radiation hardening in F82H ( $\text{Fe}^{3+}$  single ion irradiation).

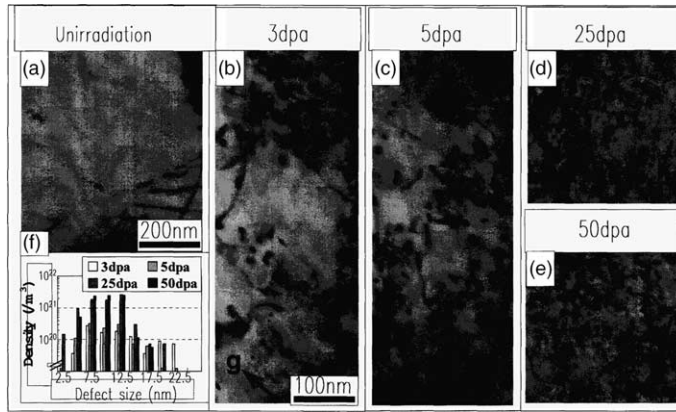


Fig. 3. Microstructure development in F82H HT irradiated at 633 K.

number density of I-loops tended to increase with the dose. It is suggested that the increase of I-loop (diameter range between 5 and 12 nm) density mainly contributes the increase of radiation hardening at 633 K.

3.2. Helium effect on radiation hardening and microstructure

The effect of He/dpa ratio on radiation hardening is shown in Fig. 4. Comparing single/dual (10 appmHe/dpa) ion-irradiated F82H at 633 K, the extra component of irradiation hardening due to helium was a hardly detectable. However, at 100 appmHe/dpa (~3300 appmHe; at 0.6 μm from surface), an extra component of

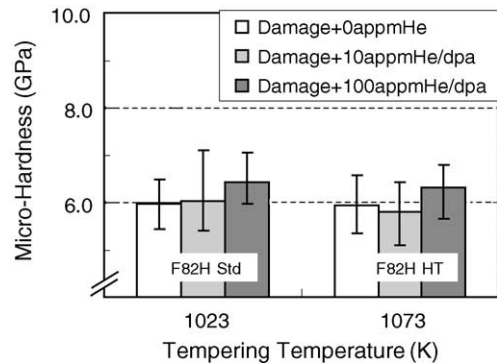


Fig. 4. Effect of helium/dpa ratio on radiation hardening of F82H at 633 K to 33 dpa.

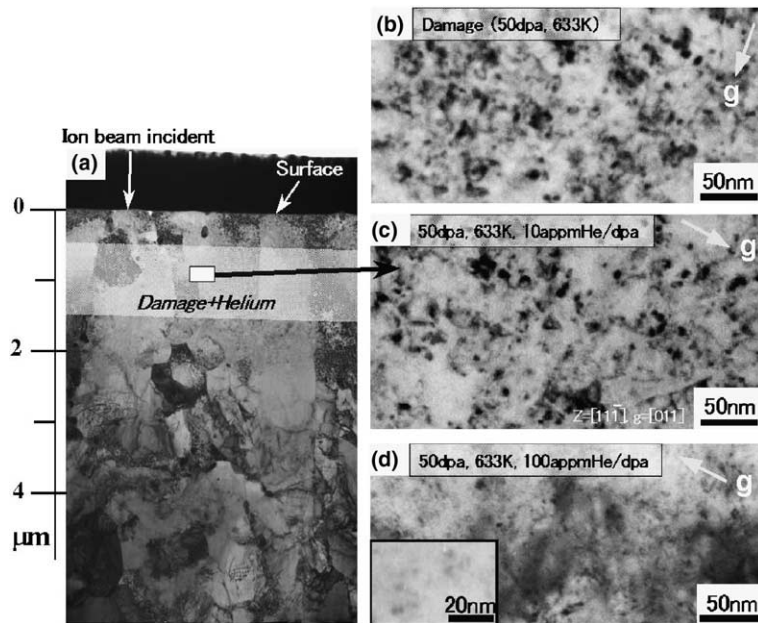


Fig. 5. Cross-sectional TEM bright field images in the single/dual ion-irradiated F82H HT (633 K, 50 dpa).

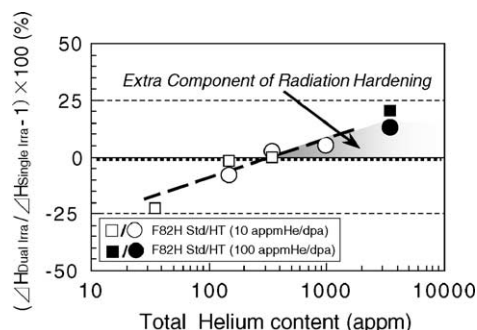


Fig. 6. Relationship between extra component radiation hardening due to helium and total implanted helium content in irradiated F82H at 633 K.

radiation hardening about 10% is obviously caused by implanted helium atoms in F82H Std and HT.

Fig. 5 provides the effect of He/dpa ratio on a microstructural evolution. Fig. 5(a) is low magnification example of the microstructure in the thin film produced by the focused ion beam (FIB) processing technique. Fig. 5(b) and (c) show a cross-sectional TEM bright field images for the single/dual ion-irradiated F82H HT (633 K, 50 dpa), respectively. The dual-beam irradiated region was observed in the range of 0.5–1.3  $\mu\text{m}$  in depth from the irradiated surface. The microstructures for the single/dual ion beam irradiated specimens consisted of similar fine interstitial loops and defect clusters at 50 dpa (the depth of 1000 nm) and no cavities were observed. The dual-beam (100 appmHe/dpa) irradiated microstructure is shown in Fig. 5(d), nano-voids and fine defects were observed. It is suggested that the formation of nano-voids causes an extra radiation hardening by higher He/dpa. Similar results of the extra-hardening due to helium was reported in Ref. [18,19].

An extra component of radiation hardening as a function of helium content was summarized in Fig. 6. For F82H, an extra component of radiation hardening occurred about 600 appm co-implanted helium. Therefore, the result of this study shows that an extra component of radiation hardening is slightly caused by implanted helium below 1000 appm in F82H.

#### 4. Summary

- (1) As a result of single irradiated F82H (Std), the heavy radiation hardening occurred in irradiated F82H at below 633 K, the micro-hardness tended to increase about 30 dpa.
- (2) The microstructure in F82H steel irradiated at 633 K between 3 and 50 dpa consisted of interstitial loops (of diameter about 20 nm), defect clusters (<5 nm

diameter) and network dislocations. The number density of I-loops tended to increase with the dose.

- (3) In case of single/dual (10 appmHe/dpa) ion-irradiated F82H at 633 K, the extra irradiation hardening was a hardly detectable. However, an extra radiation hardening is obviously caused by implanted helium atoms (100 appmHe/dpa; total:  $\sim 3300$  appmHe) in F82H.
- (4) The dual-beam (100 appmHe/dpa) irradiated microstructure showed that nano-voids and fine defects were observed. It is suggested that the formation of nano-voids causes an extra radiation hardening by helium co-implantation.

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