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Synergistic effect of helium and hydrogen for defect evolution under multi-ion irradiation of Fe–Cr ferritic alloys

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Abstract

The purpose of this study is to evaluate the synergistic effect of helium and hydrogen on Fe–Cr ferritic model alloys, to provide basic understanding concerning development of fusion reactor components. Single, dual and triple ion-beams consisting of Fe^{3+}, He^{+} and H^{+} were used for irradiation, at temperatures 470–600 °C and dose to 50 dpa at 1 μm. The dual beam irradiation with He enhanced cavity nucleation extensively to swelling of about 0.4%, whereas the dual beam irradiation with H did not significantly affect the microstructure. In the case of triple ion irradiation, the synergistic effect of He and H was confirmed clearly; relative large void formation and enhanced swelling to almost 5%. The synergistic effect suggests that the role of H is important for void growth and dislocation bias.

1. Introduction

Ferritic steels are candidate materials for the first wall of a fusion reactor, because of excellent resistance to radiation damage, but one of the critical issues is the effect of gaseous transmutants produced by high-energy neutrons, and a second is the radiation embrittlement due to increasing in DBTT [1,2]. In these steels it has been confirmed that He can enhance the nucleation of cavity formation, but the role of H has been ignored till now [3]. In vanadium alloys, simultaneous irradiation of Ni, He and H ions enhance cavity formation and swelling [4]. However synergistic effect of He and H in ferritic alloy has not been demonstrated, because of magnetism in the microscope and the complex microstructure. Further more, the oxide dispersion strength (ODS) method for ferritic steels is being developed to improve swelling resistance, as well as high temperature strength [5,6], but the synergistic effects of He and H have not been studied.

The purpose of this study is to evaluate the synergistic effects of He and H on cavity formation in model alloys of Fe–Cr, and the final goal was to understand the cause of the irradiation-induced degradation.

2. Experimental procedure

Coupon-shaped specimens ($6\times3\times0.2$–0.3 mm^3 of Fe–9 wt% Cr and Fe–12 wt% Cr alloys were solution-treated in the ferrite phase region at 800 °C for 30–45 min. Fe–12 wt% Cr-ODS steel provided by JNC also was used for this experiment. One of the specimen surfaces was chosen for irradiation. Ion irradiations were performed under single, dual and triple ion beams in the Takasaki Ion Accelerator for Advanced Radiation Application (TIARA) facility at JAERI at temperature 470–600 °C to dose of 50 dpa at 1 μm. A single ion beam of Fe^{3+} was for producing radiation damage, and dual beams consisted of Fe^{3+} and He^{+} or H^{+}. Triple ion-beams were used to simulate a fusion environment. The accelerating voltages for Fe^{3+}, He^{+} and H^{+} were 10.5,

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1.05 MeV and 380 keV, respectively. The irradiations of He and H were controlled using an Al foil energy degrader to implant over the depth range from about 0.9 to 1.5 μm [7]. Injection rates for He and H were 10 and 40 appm/dpa, respectively. The damage rate was 1.6×10^{-3} dpa/s. After irradiation, thin foils for transmission electron microscopy (TEM) were prepared with a focused ion beam (FIB) micro-sampling system. The detail description can be found in [8]. Conventional TEM observation was carried out for the evaluation of cavity and dislocation structures.

3. Result and discussion

3.1. Depth distribution of damage structure

Fig. 1 shows the cross section view of the damage structures from FIB fabricated specimens, which were irradiated with the triple ion beam to 50 dpa at 510 °C. Voids were located from 0.8 to 2.0 μm in depth, and the damage distribution is consistent with the TRIM calculation. The profiles of He and H were also included in the figure, showing the profile smoothing from the energy degrader. Consequently, the He/dpa and H/dpa ratios were almost constant through 0.9–1.5 μm, where the damage level increased from 40 to 100 dpa, with an estimated average damage level of 50 dpa. Note that the cavity size and number density changed as a function of damage level, as shown in the TRIM data. All of the microstructural data were collected from corresponding depths in the FIB fabricated specimens.

3.2. Synergistic effect on cavity formation

Fig. 2 shows cavity number density, means size and swelling in the Fe–9Cr and Fe–12Cr alloys irradiated to 50 dpa at 510 °C for several conditions. Under single ion (Fe^{3+}) irradiation, a few cavities were observed in both ferritic alloys. The swelling of Fe–9Cr and Fe–12Cr was about 0.5%, which confirmed good resistance to irradiation damage. For dual ion ($Fe^{3+} + He^{+}$) irradiation to the 12Cr alloy, the presence of He produced many small cavities 20 nm in diameter at a number density of 6.4×10^{20} m^{-3}, with swelling of about 0.4%. This result is consistent with the general effect of He to enhance cavity nucleation. For dual ion ($Fe^{3+} + H^{+}$) irradiation, the presence of H did not affect the microstructures; no cavities and no obvious dislocation structure were formed. This result suggests that H has little affect on cavity nucleation. However, during triple ion ($Fe^{3+} + He^{+} + H^{+}$) irradiation for this alloy, the synergistic effect of He and H was shown clearly; relatively large cavities were formed, and the swelling was enhanced to almost 4%, which is the highest level detected in ferritic steels at this irradiation level.

It can be emphasized that the existence of H has an important role for cavity growth especially due to synergistic effects in the triple ion irradiation. Such high swelling could be explained by increase in defect collecting efficiency or in dislocation bias. The swelling was much higher in 9Cr than in 12Cr alloy. This result suggests that this synergistic effect is a general phenomenon in ferritic steels, affecting Cr concentrations.

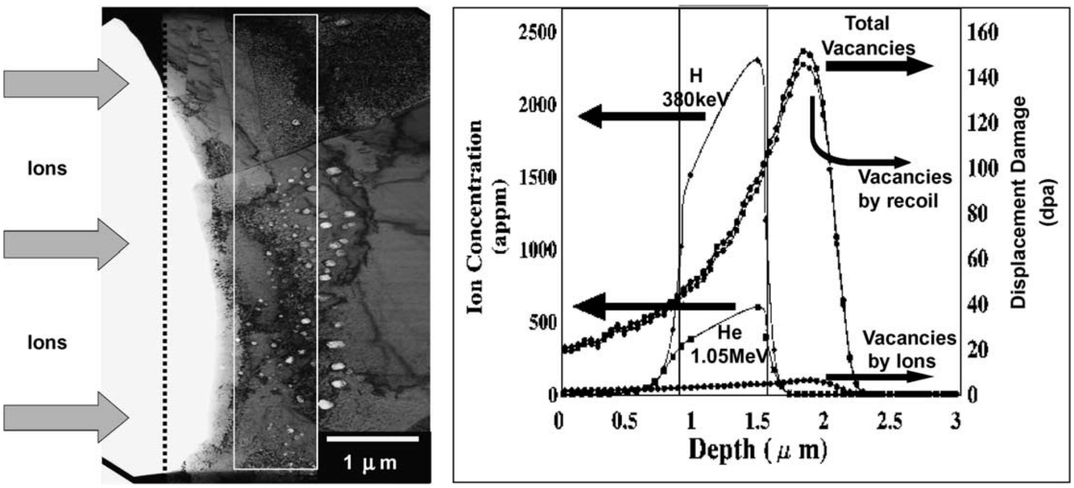

Fig. 1. Cross section view and TRIM calculations for an Fe–12Cr alloy irradiated to 50 dpa at 510 °C. A constant He/H/dpa ratio was achieved at hatched area.

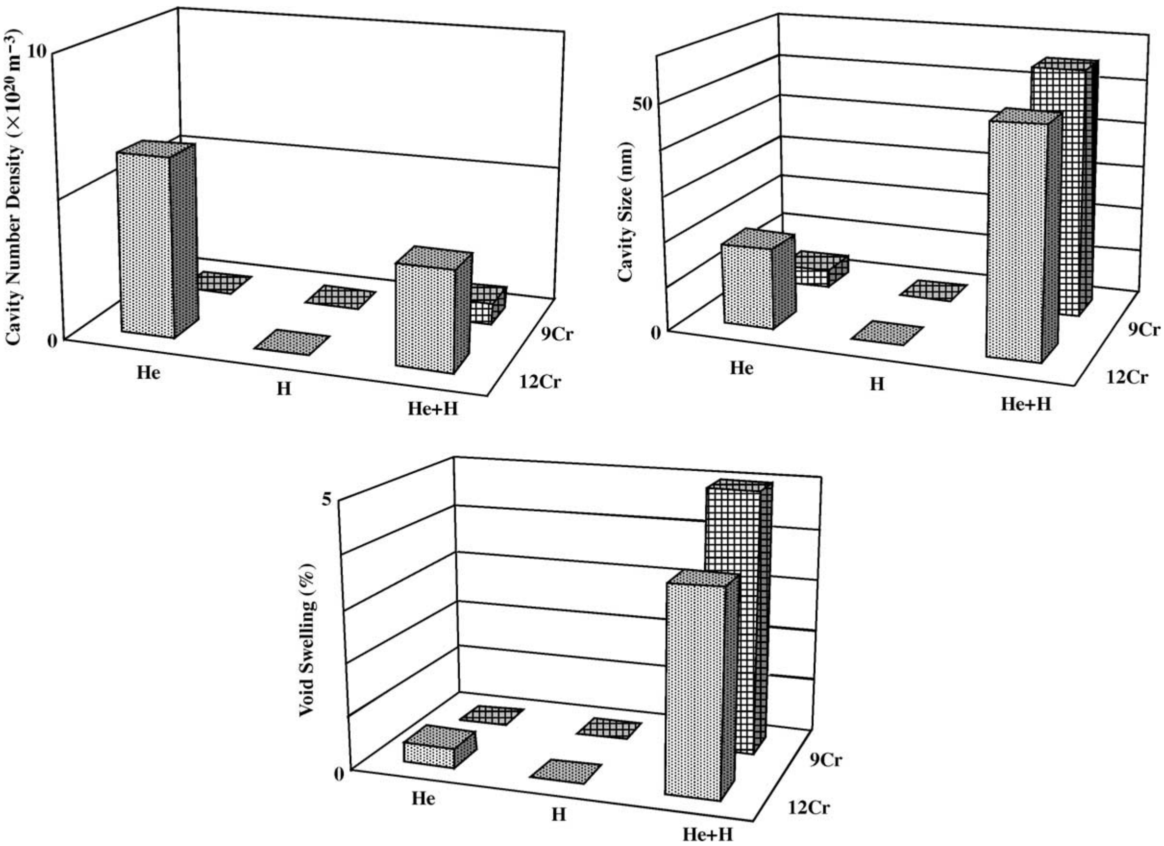

Fig. 2. Synergistic effects on cavity number density, size and swelling, developed in 9Cr and 12Cr alloys irradiated at several conditions.

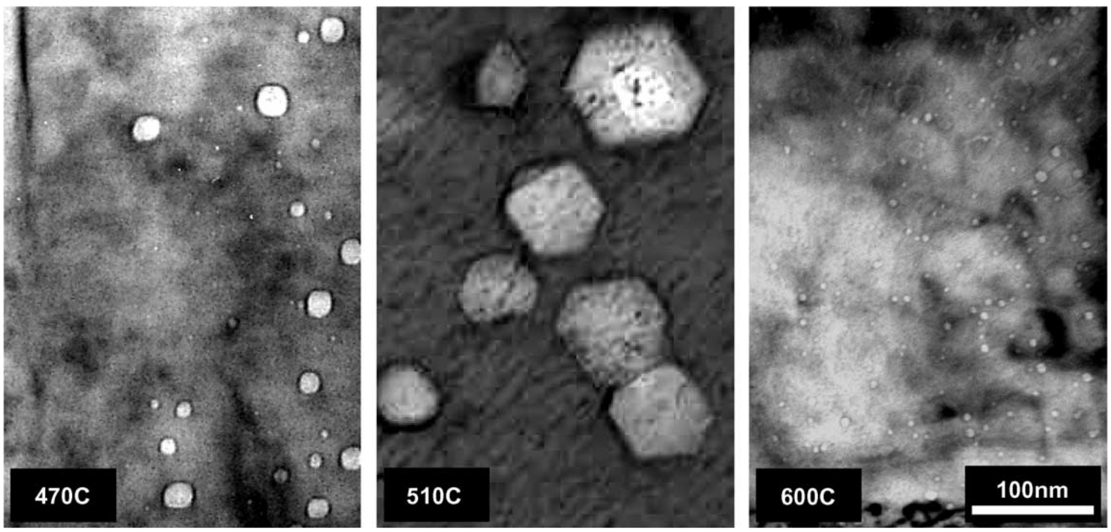

Fig. 3. Cavity structures in 12Cr alloys irradiated by triple ion beams at different temperatures. A bi-modal cavity distribution was confirmed at low and high temperatures.

3.3. Temperature dependence of cavity formation

Fig. 3 shows the cavity structures in an Fe–12Cr alloy irradiated with the triple ion beam at different temperatures. Relatively small cavities were formed at 470 °C, where a bi-modal size distribution was indicated. Large cavities with facetted shapes were developed at 510 °C, whereas images of small cavities were obscured by the large thickness needed to observe the large cavities. However, only tiny cavities, probably He bubbles, were found following irradiation at 600 °C, indicating that large cavities do not form at such high temperature. It should be noted that the size of the tiny cavities was almost constant, at about 5 nm, for all temperatures. This result is consistent with calculations, indicating that small cavities in the bi-modal distribution have a small temperature dependence. From these results, the peak swelling temperature is estimated to be 510 °C.

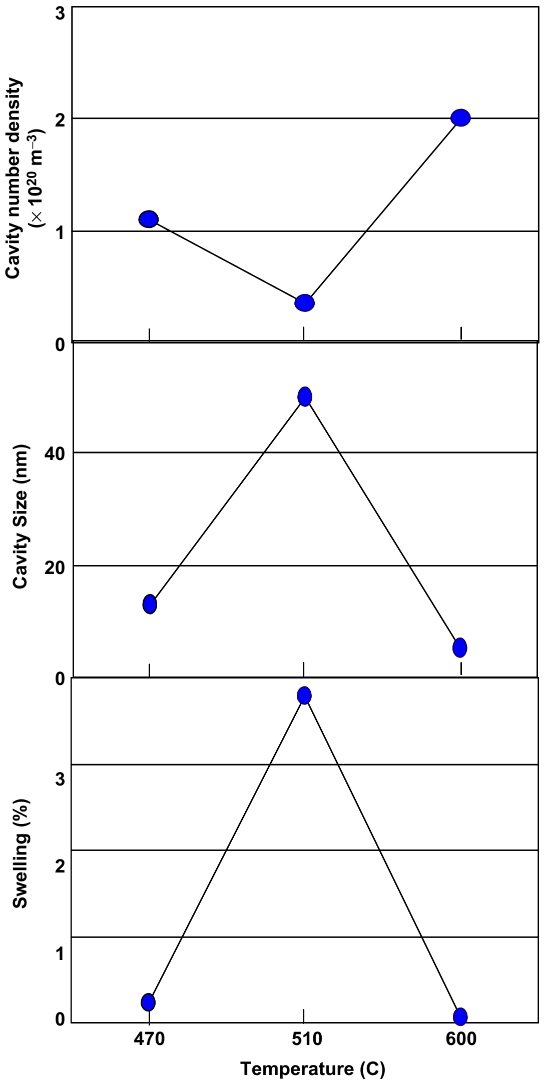

Fig. 4. Temperature dependence of cavity number density, size and swelling in 12Cr alloys irradiated by triple ions.

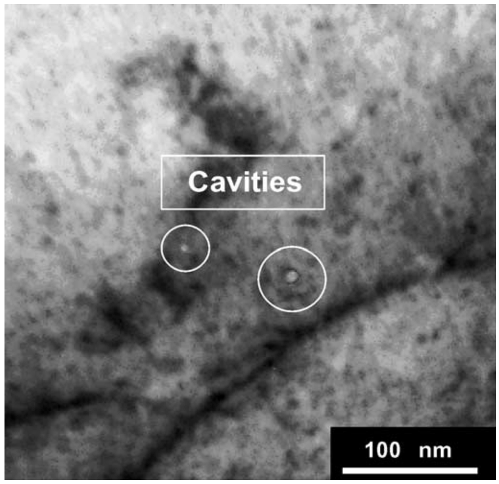

Fig. 5. Cavity structures formed in 12Cr ODS ferritic steel irradiated by triple ion beams to 50 dpa at 510 °C.

Fig. 4 shows the temperature dependence of cavity number density, mean size and swelling in Fe–12Cr alloy. The number density showed a minimum at 510 °C, but this may be an under-estimate due to the thickness of observed area, as noted above. Cavity mean size showed a peak at 510 °C, and only small cavities remained at higher temperature. As a consequent, the swelling peaked at 510 °C.

3.4. Cavity suppression in ODS steel

It is anticipated that ODS steels will have better swelling resistance than conventional ferritic steels, because of ODS particles at extremely high number density. Fig. 5 shows tiny cavities observed in an Fe–12Cr based ODS ferritic steel irradiated with the triple ion beam at 510 °C. The number density and mean size were 5.4×10^{17} m^{-3} and less than 10 nm, respectively. The swelling was quite limited (less than 0.01%). It should be noted that the dislocation density was quite low, with dislocation lines at low number density. ODS particles were mainly Y_2O_3 distributed homogeneously in the matrix, and showing as black dots consisted in this figure. The high resistance for cavity formation is probably due to the effect of the finely dispersed particles; point defect concentrations normally leading to cavity formation may be annihilated at nanoparticle surfaces.

4. Summary

Various ion irradiation experiments in Fe–9Cr, Fe–12Cr ferritic model alloys and ODS ferritic steel are carried out using TIARA facility in JAERI Takasaki.

(1) The highest swelling is observed in ferritic model alloys of Fe–Cr under triple ion irradiation. The synergistic effect of He and H irradiation in these alloys was confirmed by larger cavities and higher swelling under triple ion irradiation compared with dual ion irradiations.

(2) Under triple ion beam, the swelling peak was found to be at 510 °C in Fe–12Cr ferritic alloy. The maximum value of swelling was about 4% at 510 °C.

(3) In ODS ferritic alloy, the swelling was suppressed to less than 0.01% under triple ion irradiation at 510 °C to 50 dpa.

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