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Synergistic effects of hydrogen and helium on microstructural evolution in vanadium alloys by triple ion beam irradiation

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Abstract

In fusion materials, irradiation of 14 MeV neutrons produces He and H atoms at a high generation rate. The objective of this study is to clarify synergistic effects of He and H on microstructural evolution in pure vanadium and two vanadium alloys including candidate ternary alloy V–5Cr–5Ti. The specimens are irradiated with 12 MeV Ni^{3+} ions at 873 K with simultaneous implantation of 1 MeV He^{+} and 350 keV H^{+} ions. Helium and hydrogen implantation ratios are independently controlled at two different He/dpa and H/dpa rates. Dual beam irradiation with heavy ions and He ions or H ions, and single beam irradiation experiments are also performed. Triple beam irradiation strongly enhances growth of cavities and swelling in pure vanadium compared with those under dual beam irradiation with He and single beam irradiation, whereas simultaneous implantation of H without He does not affect cavity growth and swelling. In V–5Cr–5Ti alloy, no cavities are detected without implantation of He. However, Ni, He and H triple beam irradiation is found to enhance swelling. These results are discussed in terms of dislocation and cavity evolution in the irradiated vanadium alloys.

1. Introduction

Vanadium alloys are potential blanket structural materials of a future DEMO reactor because of their excellent mechanical properties at elevated temperature and low induced radioactivity after neutron irradiation. Irradiation of 14 MeV neutrons produces He and H atoms at a high generation rate as well as displacement damage during irradiation. Microstructural evolution and resultant mechanical property changes in vanadium alloys are known to be strongly influenced by transmutation helium and hydrogen simultaneously produced during 14 MeV neutron irradiation. Therefore, the effect of high helium generation rate on microstructural evolution and mechanical property changes in vanadium has been investigated using dynamic helium charging experiments (DHCE) in fission reactors [1,2] and boron doping techniques [3,4].

Multiple beam irradiation technique has also been extensively applied to study synergistic effects of displacement damage and transmutation atoms under fusion irradiation conditions. However, very limited experiments are performed on vanadium alloys. Dual beam irradiation with heavy ions and H ions to pure vanadium was performed by Kano et al. [5]. They have found that hydrogen can affect cavity formation in vanadium, but that hydrogen effects on microstructural evolution vary in complex fashion with dose.

In this study, multiple ion irradiation experiments are carried out to understand the effects of helium and hydrogen on the mechanisms of microstructural evolution in pure vanadium and vanadium alloys by varying the combination of heavy ion, helium and hydrogen irradiation systematically.

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2. Experimental

We used the triple beam irradiation facility in the Takasaki Ion Accelerators for Advanced Radiation Application (TIARA), where three accelerators are simultaneously operated to irradiate small size specimens with heavy ions, helium ions and hydrogen ions [6].

99.8% pure vanadium and V–5Cr and V–5Cr–5Ti alloys are prepared by arc-melting and rolled to sheets of 0.2 mm thickness. These are cut into 3 mm disks and annealed at 1273 K for 1 h in high vacuum of 10^{-6} Pa wrapped within pure Zr foils followed by rapid cooling. Table 1 shows impurity levels in the pure vanadium.

These specimens are irradiated with 12 MeV Ni^{3+} ions to 30 dpa at 1700 nm from the surface with simultaneous implantation of helium and hydrogen by 1 MeV He^{+} and/or 350 keV H^{+} ions. Dual beam irradiation with 12 MeV Ni^{3+} and 1 MeV He^{+}, or 12 MeV Ni^{3+} and 350 KeV H^{+}, and single beam irradiation with 12 MeV Ni^{3+} are also performed. Irradiation temperature is dynamically controlled at 873 K during irradiation. Typical damage rate is 4×10^{-4} dpa/s.

Two different injection rates, 10 and 20 appm/dpa, are used for both helium and hydrogen irradiation. H and He implantation peak depths exist at about 1700 nm from the ion incident surface. All the displacement damage dose and H and He/dpa ratios are estimated at this depth. Total concentration of hydrogen at the damage peak depth is 300 and 600 appm at H/dpa ratios of 10 and 20, respectively, and these are well below the solution limit of hydrogen in vanadium [7].

About 1600 nm surface layer of the irradiated samples are sectioned away in a solution of 20% sulfuric acid and 80% methanol, and back-thinned to perforation to observe the microstructural evolution in the peak damage region by transmission electron microscope (TEM) operated at 200 kV.

3. Results and discussion

3.1. Microstructural evolution in pure vanadium

Cavities are observed in all the pure vanadium specimens irradiated with single, dual and triple ions to 30 dpa at 873 K. Under dual beam irradiation with helium and heavy ions, helium enhances swelling mainly by increasing cavity number density. However, dual beam irradiation with 12 MeV Ni^{3+} and 350 keV H^{+} ions does not seem to change cavity microstructure compared with the single beam irradiation case, suggesting that hydrogen does not play any significant role in cavity formation.

In the case of triple beam irradiation, however, swelling is found to be enhanced especially at 10 appm He/dpa and 10 appm H/dpa, a typical rate of fusion irradiation conditions. These results cannot be explained by simply adding the helium and hydrogen effects observed in the dual beam irradiation experiments, since hydrogen dose not contribute to swelling under dual beam irradiation.

Fig. 1 summarizes cavity volume fractions in pure vanadium irradiated to 30 dpa at 873 K. Average cavity diameter and cavity density in pure vanadium are also shown in Figs. 2 and 3, respectively. It is clearly observed that Ni and H dual beam irradiation does not affect cavity density and size strongly, whereas Ni and He co-implantation increases nucleation of cavities. In the case of dual beam irradiation with 20 appm He/dpa rate without hydrogen, high density of fine cavities are formed and average cavity size is slightly smaller than that at 10 appm He/dpa rate.

It should be noted that enhanced swelling under triple beam irradiation at 10 appm/dpa for both He and H mainly comes from the growth of cavities, since the cavity number density is lower for triple beam irradiation compared with that of dual beam irradiation with

Table 1
Impurity levels of 99.8% pure vanadium specimens used in this study (wt.ppm)

H	14	C	36	Al	200
O	120	Cr	300	Si	100
N	6	Fe	400	Cu	200

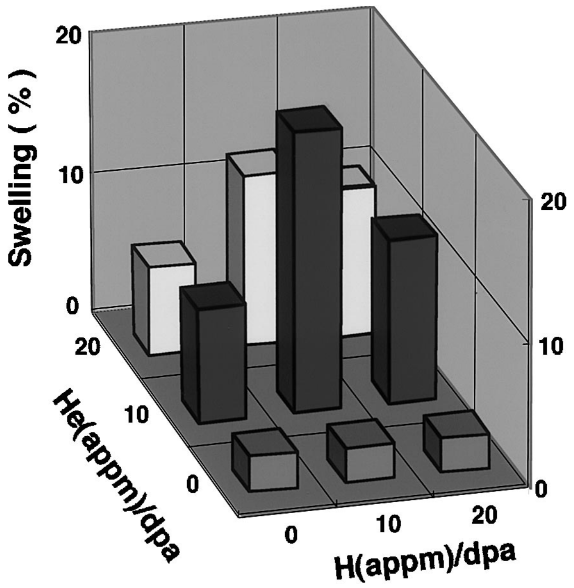

Fig. 1. Cavity volume fraction in pure vanadium irradiated with 12 MeV Ni^{3+} ions to 30 dpa at 873 K with and without simultaneous irradiation of He and H.

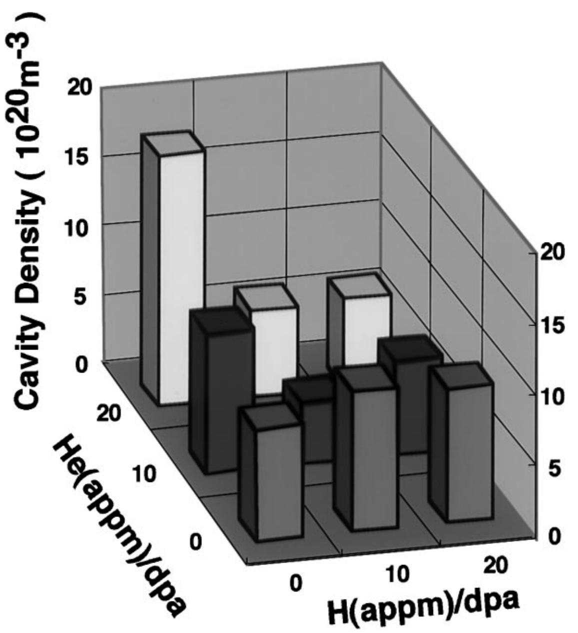

Fig. 2. Cavity density in pure vanadium irradiated with 12 MeV Ni^{3+} ions to 30 dpa at 873 K with and without simultaneous irradiation of He and H.

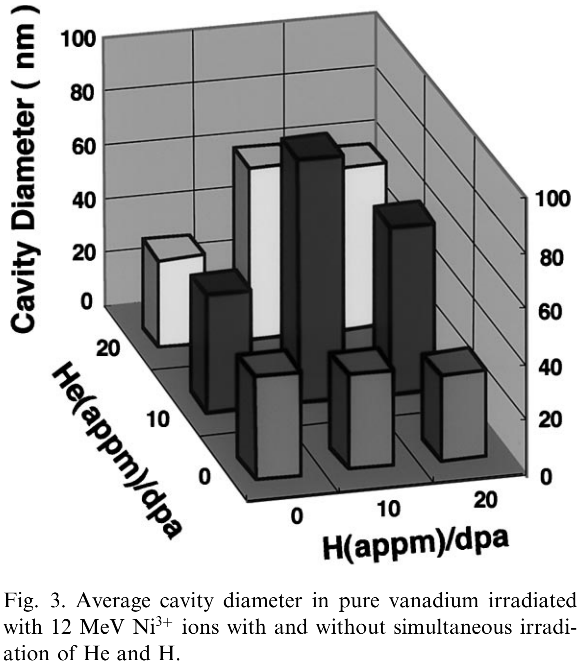

Fig. 3. Average cavity diameter in pure vanadium irradiated with 12 MeV Ni^{3+} ions with and without simultaneous irradiation of He and H.

helium and heavy ions. Detailed TEM observation indicates that fine defect clusters of about 1 nm are formed in the triple-beam irradiated vanadium. Fig. 4 shows measured total dislocation density in the irradiated

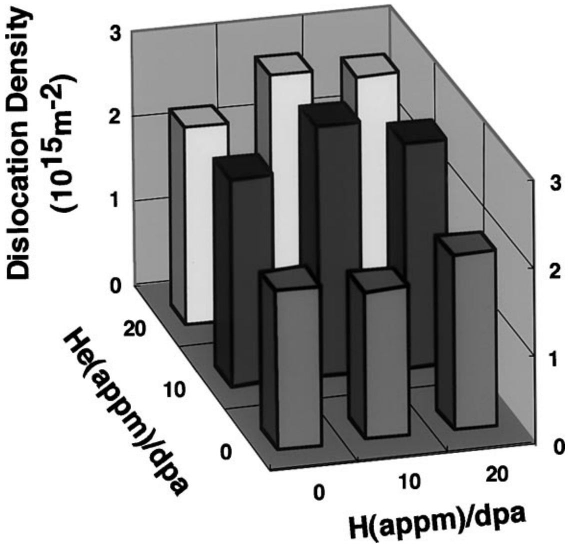

Fig. 4. Dislocation density in pure vanadium irradiated with 12 MeV Ni^{3+} ions to 30 dpa at 873 K with and without simultaneous irradiation of He and H.

specimens at 873 K to 30 dpa for all the nine cases. Total dislocation density seems to have a peak at He (appm)/dpa and H (appm)/dpa ratios of 10. Enhanced evolution of dislocation as biased sink for interstitials is considered to contribute cavity growth and swelling at the dose level of 30 dpa.

Since a high swelling is observed in association with the evolution of high dislocation density, it seems that, in the presence of H and He, vacancies are absorbed by He + H bubbles and the excess interstitials contribute dislocation evolution. Consequently, interstitials to the dislocations are availing excess vacancies for cavity growth. Such feedback seems the swelling mechanism in this case.

3.2. Cavity formation in vanadium alloys

Very few cavities are detected in ternary V–5Cr–5Ti alloy irradiated with single ions to 30 dpa at 873 K. Simultaneous irradiation with hydrogen does not enhance cavity formation in V–5Cr–5Ti. In the recent ion irradiation experiments, V–5Cr–5Ti alloy also shows excellent swelling resistance at other irradiation temperatures even in the dual beam irradiation with heavy ions and hydrogen ions.

Co-implantation of helium at 873 K, however, produces cavities in V–5Cr–5Ti. Fig. 5 compares observed cavity volume fraction in pure vanadium, V–5Cr and V–5Cr–5Ti irradiated with 12 MeV Ni^{3+} ions to 30 dpa at 873 K at 10 appm He/dpa with and without simultaneous irradiation of hydrogen. It should be noted that co-implanted hydrogen with helium also enhances

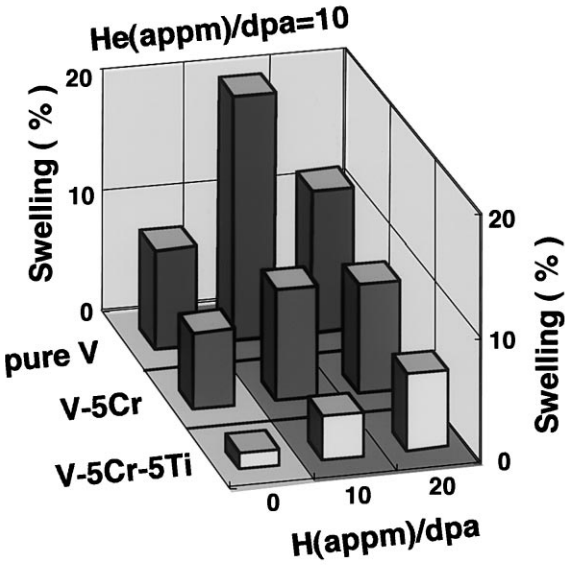

Fig. 5. Cavity volume fraction in pure vanadium, V–5Cr and V–5Cr–5Ti irradiated with 12 MeV Ni^{3+} ions to 30 dpa at 873 K at He(appm)/dpa ratio of 10 with and without simultaneous irradiation of H.

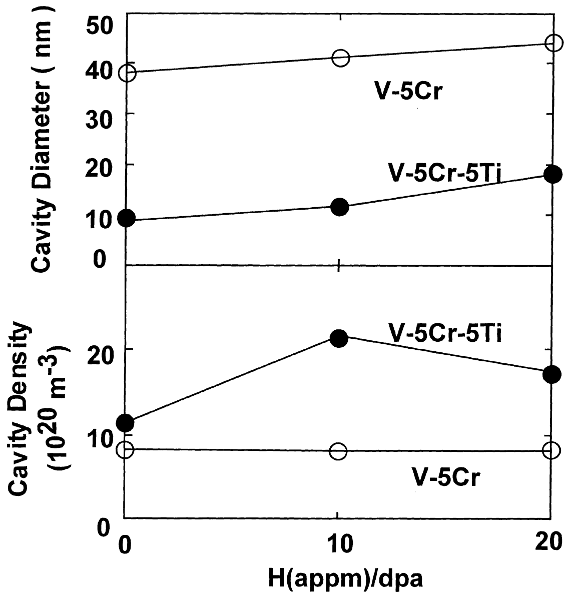

Fig. 6. Cavity density and average diameter in V–5Cr and V–5Cr–5Ti irradiated with 12 MeV Ni^{3+} ions to 30 dpa at 873 K with simultaneous irradiation of He at He(appm)/dpa ratio of 10.

swelling in vanadium alloys as well as pure vanadium. Fig. 6 shows cavity density and average cavity diameter in V–5Cr and V–5Cr–5Ti irradiated with 12 MeV Ni^{3+} ions to 30 dpa at 873 K at 10 appm He/dpa as a function of H appm/dpa ratio. In the cases of triple beam irradiation with simultaneous implantation of hydrogen and helium, cavity size and number density increase with hydrogen implantation rate, whereas cavity density in V–5Cr binary alloy is not strongly influenced by hydrogen. At the H appm/dpa ratio of 20, cavity density in V–5Cr–5Ti is larger than that in V–5Cr, and cavities also grow larger in V–5Cr–5Ti.

Titanium atoms appear to play an important role in cavity nucleation in V–5Cr–5Ti alloy. Titanium atoms behave as trapping sites for interstitial impurity atoms such as oxygen and nitrogen. Hydrogen may stabilize trapped helium clusters at Ti atoms. Titanium atoms were found to promote evolution of dislocation microstructure and to segregate around grain boundaries in V–5Cr–5Ti alloys irradiated at 873 and 973 K [8]. It is also found that co-implantation of helium at 873 K increases dislocation densities in both V–5Cr–5Ti and V–5Cr alloys.

4. Conclusion

Series of ion irradiation experiments in pure V and V alloys are performed using triple ion beam irradiation using the facility in the TIARA.

The highest swelling is observed in vanadium irradiated with triple ion beams. The cavities are stabilized by both helium and hydrogen, and excess interstitials are considered to enhance dislocation microstructure evolution resulting in excess vacancies for cavity growth.

V–5Cr–5Ti is very resistant to cavity formation in the absence of He. However, fine cavities are nucleated when helium is injected. Cavity nucleation and growth are further amplified when hydrogen is injected together.

Acknowledgements

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