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Microstructure in vanadium irradiated by simultaneous multi-ion beam of hydrogen, helium and nickel ions

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Abstract

Pure vanadium was irradiated at 500 and 600 °C by either 5 MeV Ni ions (single beam) or Ni + H and He ions simultaneously. The pure vanadium was of nominal 99.8% purity. For the quantitative investigation of damage structure as a function of the depth, we utilized focused ion beam (FIB) microscopy. To preserve the surface of ion-irradiated metals, we deposited tungsten on the irradiated surface. The specimens were electro-polished to remove the damaged region by FIB. When only nickel ions were used, voids formed in the region from the surface to a depth of ~0.5 μm when irradiated at 500 and 600 °C. However, in the region of the damage peak, voids were not observed. Needle-like precipitates of about 100 nm of length were observed for any specimen covering the full ion penetration depth. It is thought that the precipitate is a carbide. Moreover, in the specimen irradiated at 600 °C, the granular precipitates were over the region of the 1.0–1.5 μm depth. Void formation was observed over the whole ion penetration depth when the specimen was subjected to Ni + He simultaneous irradiation. Needle-like precipitates were observed.

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1. Introduction

For fusion reactor applications, there is an interest in vanadium and its alloys. Hydrogen and helium atoms are generated by nuclear transmutation in the fusion environment. These gas atoms play an important role on the evolution of the damage microstructure. It is well known that helium is active in cavity nucleation. Sekimura et al. [1] have carried out multiple beam ion irradiations to study the role of gases. In the present work, quantitative experiments were carried out to study the role of helium and hydrogen on the evolution of damage microstructure in irradiated materials. It is possible to control the concentration of gas atoms in irradiated metals by ion irradiation at high energy. We examined void formation in high energy ion-irradiated, pure vanadium by both single beam (5 MeV Ni) and dual-beam (5 MeV Ni ion and 600 keV He or 260 kV H ion) irradiation. The ion energy was selected so that the projected range of the gas ions in vanadium might coincide with the depth of peak damage (1.3 μm) calculated by the TRIM 95 code. Specimens for TEM cross-sectional observation were prepared by a focused ion beam (FIB) device. The relation between gas atoms and damage structure was derived from experimental results.

2. Experimental procedure

The pure vanadium had a nominal purity of 99.8%. This material was called as-received V (V(AR)). Some specimens were degassed by melting in vacuum at 10^{-5} Pa in a levitation furnace [2]. This specimen was called residual-gas-free V (V(RGF)). Annealed disks 3 mm in diameter and 0.05 mm thick were prepared from

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each material. The irradiation was carried out with the Takasaki Ion Accelerators for Advanced Radiation Application accelerators at the Takasaki-establishment of Japan Atomic Energy Research Institute (JAERI) at 500 and 600 °C. The ion energy was selected so that the projected range of the gas ions in vanadium coincides with the depth of peak damage (1.3 μm) calculated by the TRIM 95 code [3], as shown in Fig. 1. The peak damage rate was 2×10^{-3} dpa/s and total dose at the damage peak was 22 dpa. The ions stop within a depth of 2 μm from the surface and damage was formed up to this depth. For quantitative investigation, the damage structure has to be observed as a function of depth. We utilized FIB microscopy. FIB generates 30 keV Ga ions and illuminates the specimen surface with a glancing angle. To preserve the surface of the ion-irradiated metals, we deposited tungsten on the irradiated surface. In our previous work, it was found that interstitial atoms form near surface clusters throughout FIB-thinned specimens as shown in Fig. 2(a). To overcome this difficulty, we developed a TEM specimen preparation method which is a combination of FIB thinning and electro-polishing [4–6]. To remove regions damaged by the Ga ions, the specimens were electro-polished in a solution of 20% H_2SO_4 and 80% methanol cooled to −30 °C at an applied potential of 12 V for 1 s. The specimens were electro-polished to remove the region damaged by FIB. After electro-polishing, no dot type defects were observed, as shown in Fig. 2(b).

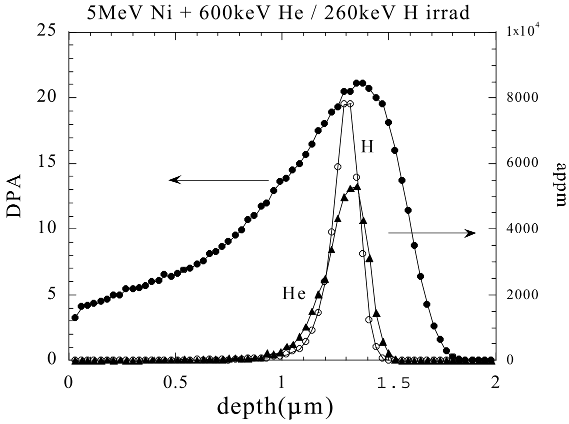

Fig. 1. Depth profile of DPA produced by 5 MeV Ni ions and gas concentrations produced by 600 keV He and 260 keV H ions in pure vanadium, as calculated by the TRIM 95 code [3].

3. Results and discussion

When only nickel ions were used, voids formed in the region from the surface to a depth of about 0.5 μm when irradiated at 500 and 600 °C, as shown in Fig. 3(a) and (b). However, in the region of damage peak, voids were not observed. Needle-like precipitates of about 100 nm in length were observed in all specimens covering the whole penetration depth of the ion. It is thought that the precipitate is some type of carbide, because they are oriented in the ⟨100⟩ direction [7] and the specimen includes a few hundred ppm carbon as a impurity. Moreover, in the specimen irradiated at 600 °C, granular precipitates were observed in the region 1.0–1.5 μm depth. In the case of Ni + H irradiation, the size of the voids was small and the number density was similar compared with the case where only nickel ions were

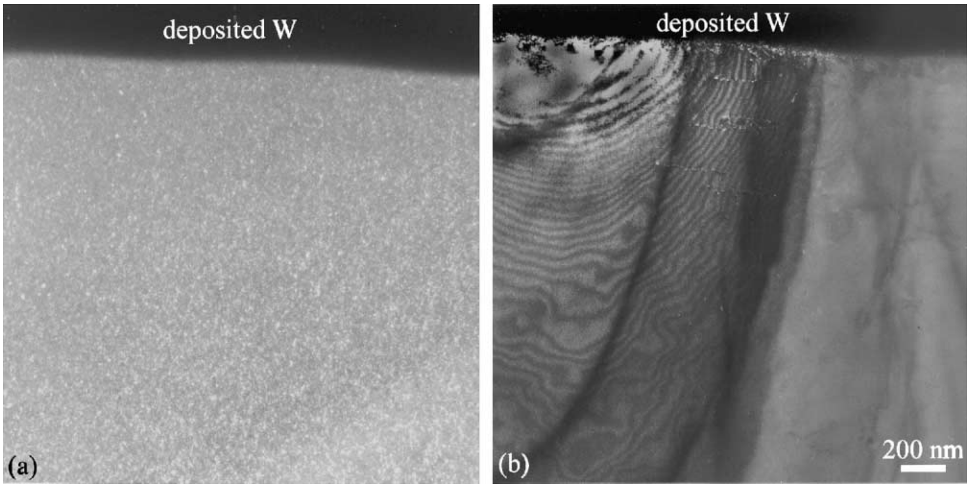

Fig. 2. (a) As-thinned specimen with FIB, many dot clusters were observed. (b) After electro-polishing no dot clusters were observed by weak beam dark field image.

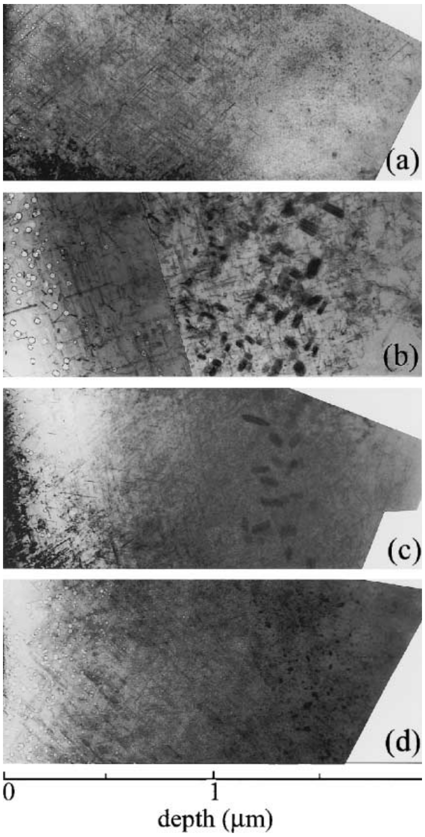

Fig. 3. Damage structure of ion-irradiated, as-received pure vanadium: (a) 5 MeV Ni ion irradiation at 500 °C, (b) 5 MeV Ni ion irradiation at 600 °C and (c) 5 MeV Ni+0.26 MeV H ion irradiation at 600 °C, (d) 5 MeV Ni+0.6 MeV He ion irradiation at 600 °C.

used, as shown in Fig. 3(b) and (c). Void swelling was suppressed by simultaneous hydrogen irradiation. Void formation was observed within the whole ion penetration depth in the specimen irradiated simultaneously with Ni + He ions, as shown in Fig. 3(d). A needle-like precipitate was observed. Needle-like precipitation formed with any combination of Ni, Ni + H, Ni + He irradiation, but void formation depended on gas implantation. In pure vanadium, helium atoms promote void nucleation for irradiation at 500 and 600 °C in the low dose region. In the case of Ni + H irradiation at 500 °C, small voids were observed within the whole ion penetration depth. While hydrogen atoms suppressed void formation irradiated at 600 °C. These results show that hydrogen atoms trap small vacancy clusters at 500 °C and vacancy clusters and H atoms or molecules dissolved with increasing temperature. Fig. 4 shows the effect of melting in vacuum, where void contrast images of depth in 0.1–0.5 μm for V(AR) and V(RGF), respectively. In Fig. 4(b) the needle-like precipitates decreased compared with Fig. 4(a). It is thought that precipitates acting as a sink were decreased by melting in vacuum.

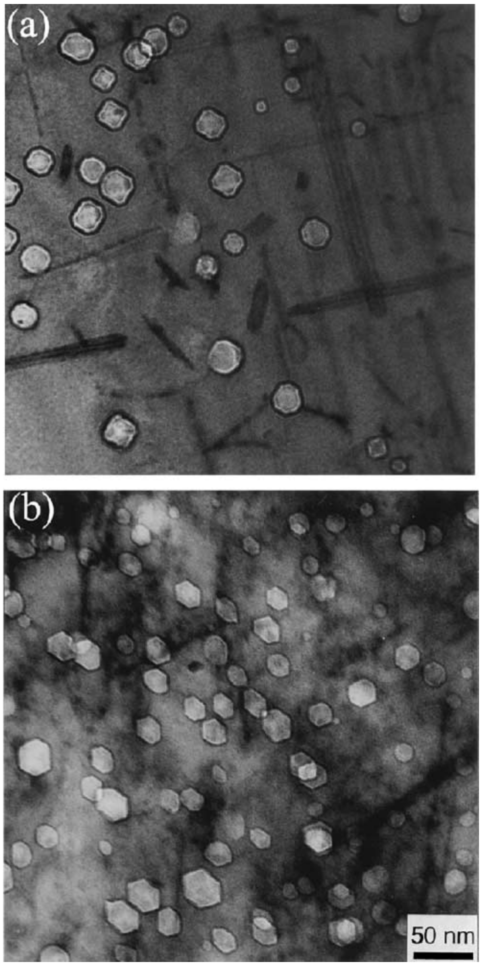

Fig. 4. Void formation in 0.1–0.5 μm region irradiated with only nickel at 600 °C: (a) V(AR) and (b) V(RGF).

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References

[1] N. Sekimura, T. Iwai, Y. Arai, S. Yonamine, A. Naito, Y. Miwa, S. Hamada, J. Nucl. Mater. 283–287 (2000) 224.
[2] K. Sugio, Y. Shimomura, I. Mukouda, M. Kiritani, Radiat. Eff. Def. Sol. 157 (2002) 43.
[3] J.F. Ziegler, J.P. Biersack, The Stopping and Range of Ions in Solids, Pergamon, New York, 1985.

[4] I. Mukouda, Y. Shimomura, T. Iiyama, Y. Katano, D. Yamaki, T. Nakazawa, K. Noda, in: Material Research Society Symposium Proceedings, vol. 540, 1999, p. 549.
[5] Y. Shimomura, I. Mukouda, in: Material Research Society Symposium Proceedings, vol. 540, 1999, p. 527.
[6] I. Mukouda, Y. Shimomura, T. Iiyama, Y. Harada, Y. Katano, T. Nakazawa, D. Yamaki, K. Noda, J. Nucl. Mater. 283–287 (2000) 302.
[7] K. Ochiai, H. Watanabe, T. Muroga, N. Yoshida, H. Matsui, J. Nucl. Mater. 271&272 (1999) 376.