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Journal of Nuclear Materials 283–287 (2000) 286–290

journal of nuclear materials

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Microstructure of vanadium alloys during ion irradiation with stepwise change of temperature

H. Watanabe [a,*], T. Arinaga [b], K. Ochiai [b], T. Muroga [c], N. Yoshida [a]

[a] *Research Institute for Applied Mechanics, Kyushu University, 6-1 Kasugakouen, Kasuga, Fukuoka 816-8580, Japan*
[b] *Interdisciplinary Graduate School of Engineering Sciences, Kyushu University, Kasuga, Fukuoka 816-8580, Japan*
[c] *National Institute for Fusion Science, 322-6 Oroshi, Toki, Gifu 509-5292, Japan*

Abstract

To understand the influence of a stepwise change of irradiation temperature on pure vanadium and vanadium alloys, ion irradiation were performed at 473 K to 0.25 dpa and 873 K to 0.5 dpa. In pure vanadium and V–5Cr alloy, shrinkage of interstitial type loops was prominent during irradiation at 873 K. These results were explained by the appearance of a vacancy rich condition during the successive irradiation at 873 K. In Ti-containing alloys (V–5Ti, V–4Cr–4Ti), on the other hand, dissociation of vacancy loops leads to the shrinkage of interstitial type loops and the formation of Ti-enriched precipitates. © 2000 Elsevier Science B.V. All rights reserved.

1. Introduction

An irradiation temperature change during reactor operation can have significant effects on microstructural evolution. Previous studies on Fe–Cr–Ni austenitic stainless steels [1–3] and pure vanadium [4,5] showed that, for particular temperature variations below and above a borderline temperature, pre-irradiation at a lower temperature suppresses interstitial loop formation at a higher temperature. The borderline temperature was estimated to be 573–623 K for Fe–Cr–Ni alloys [1–3] and 673 K for pure vanadium [4]. The phenomena were explained by a vacancy rich condition, which appears temporarily at the beginning of the high temperature irradiation. But the effects of alloying elements on the microstructure and the correlation between loop formation and hardness of the specimens under temperature variation were not known. The present paper summarizes some key findings of a study of microstructure and Micro-Vickers hardness tests of vanadium model alloys under ion irradiation.

2. Experimental procedures

Pure vanadium and three model alloys (V–5Cr, V–5Ti, V–4Cr–4Ti) were used in this study. Disk specimens for microscopy were wrapped with pure zirconium to getter oxygen and annealed for 2 h at 1373 K. The analyzed concentration of oxygen and nitrogen in the V–4Cr–4Ti alloy were 1889 and 12.7 appm, respectively. 2.4 MeV Cu ion irradiation was carried out with the Tandem accelerator at Kyushu University. The combination of irradiation temperature and dose at each temperature was 473 K (0.25 dpa)/873 K (0.5 dpa) and 0.75 dpa for the continuous irradiation at 875 K. After the irradiation, the area near the peak damage region (at around 700 nm) was electro-polished by a back-thinning method. The damage rate and the implanted copper concentration in this region were about 1.7×10^{-4} dpa/s and 10^{-3} at.% (at 0.1 dpa), respectively. Transmission electron microscopy and Micro-Vickers hardness tests were conducted after each step of irradiation and annealing at 873 K. The details of the Micro-Vickers hardness test for ion irradiated samples are described in Ref. [6]. Temperature variation, microcopy and Micro-Vickers hardness tests performed in this study are summarized schematically in Fig. 1.

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[*] Corresponding author. Tel.: +81-92 583 7717; fax: +81-92 583 7690.

E-mail address: watanabe@riam.kyushu-u.ac.jp (H. Watanabe).

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PII: S0022-3115(00)00307-X

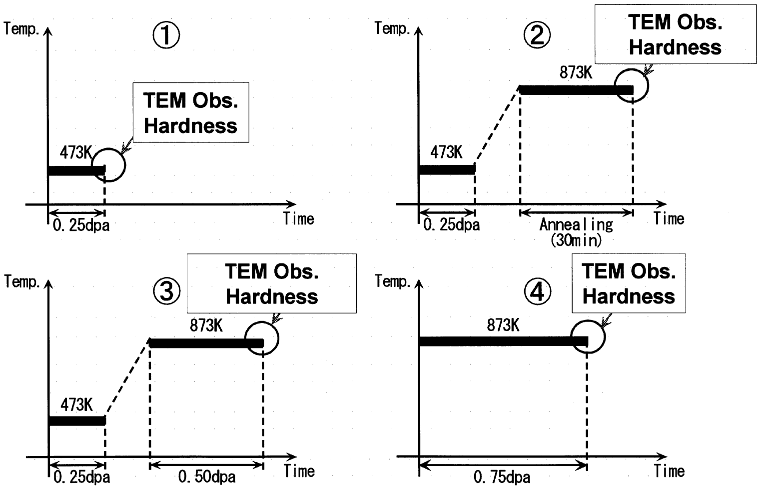

Fig. 1. Schematic view of temperature variation and microcopy (also Micro-Vickers hardness tests).

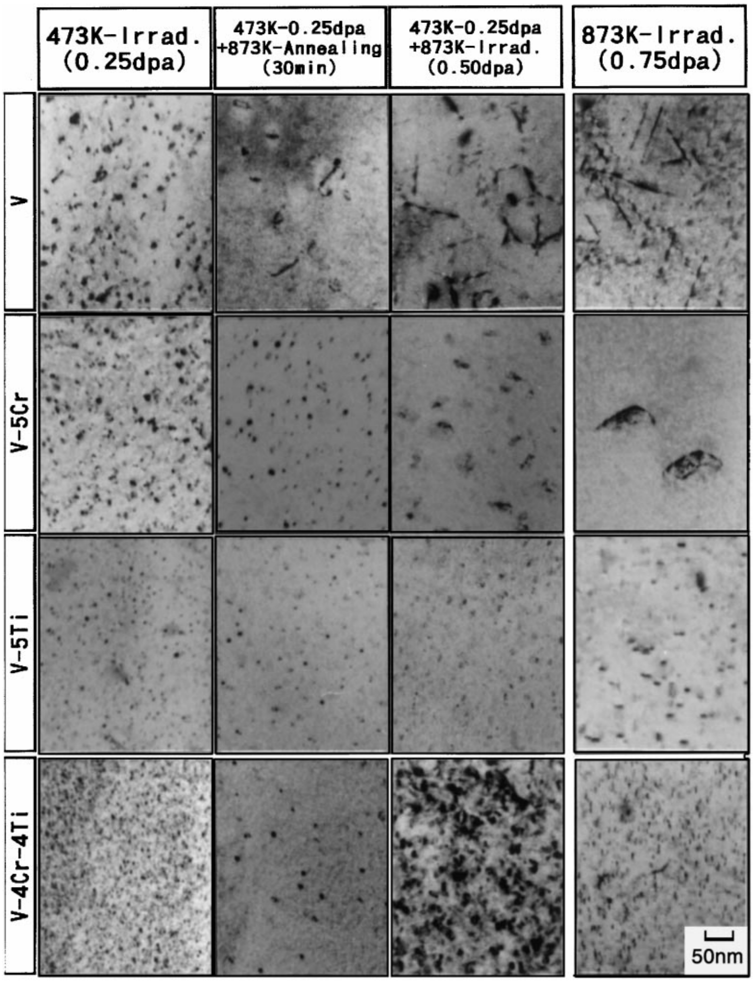

Fig. 2. The microstructure of pure vanadium and three model alloys (V–5Cr, V–5Ti, V–4Cr–4Ti) at each step of irradiation conditions shown in Fig. 1.

3. Results

Fig. 2 shows the microstructure of pure vanadium and three model alloys (V–5Cr, V–5Ti, V–4Cr–4Ti) at each stage of the irradiation history shown in Fig. 1. After constant irradiation at 473 K, a high density of interstitial and vacancy type dislocation loops were formed [4]. The total number density of defects clusters at 473 K was almost the same in all specimens and was about 10^{23} m^{-3}. After both irradiation and annealing at 873 K, however, the interstitial loop density of pure vanadium and V–5Cr alloy decreased significantly.

On the other hand, Ti-containing alloys (V–5Ti and V–4Cr–4Ti), showed a different microstructure after the successive irradiation at 873 K. The dark field weak-beam images of the two Ti-containing alloys are shown in Fig. 3. In comparison with pure vanadium and V–5Cr, the small defects that formed at 473 K were stable. In addition to interstitial and vacancy loops, another kind of small defect, lying on $\{001\}$ habit plane, became visible. The density of the defects after the stepped irradiation was about 10 times higher than that of the density formed during constant irradiation at 873 K (0.75 dpa). Energy dispersive spectroscopy (EDS) analysis showed that these $\{001\}$ defects clusters were enriched in Ti. Defect density changes at each step are summarized in Fig. 4. These Ti-rich $\{001\}$ defect clusters were also formed during annealing above 973 K. But they were not detected after annealing at 873 K.

The change of hardness at each step of irradiation and annealing is shown in Fig. 5. In pure vanadium and the V–5Cr alloy, the increase in hardness due to irradiation at 473 K (0.25 dpa) was recovered by the successive annealing and irradiation at 873 K. In Ti-containing alloys (V–5Ti and V–4Cr–4Ti), on the other hand, there was an increase in hardness after irradiation at 873 K.

4. Discussion

The microstructures of pure vanadium and V–5Cr alloy studied in this experiment showed the typical microstructure, which were observed in previous stepwise (low to high) temperature irradiation [1–4]. Namely, small interstitial loops formed at the lower temperature (473 K for this experiment) were dissociated by successive irradiation at the higher temperature (873 K). The previous results were explained by the appearance of a vacancy rich condition during the successive irradiation at 873 K. A very high vacancy concentration is achieved by the dissociation of vacancy clusters, and this results in the mutual annihilation of vacancies and interstitials [1–4]. On the other hand, recovery of loop density by irradiation at 873 K was not prominent in the Ti-containing alloys. This is presumably due to the suppressed migration of vacancies. Because Ti has a bigger atomic volume than vanadium, the binding energy between a vacancy and

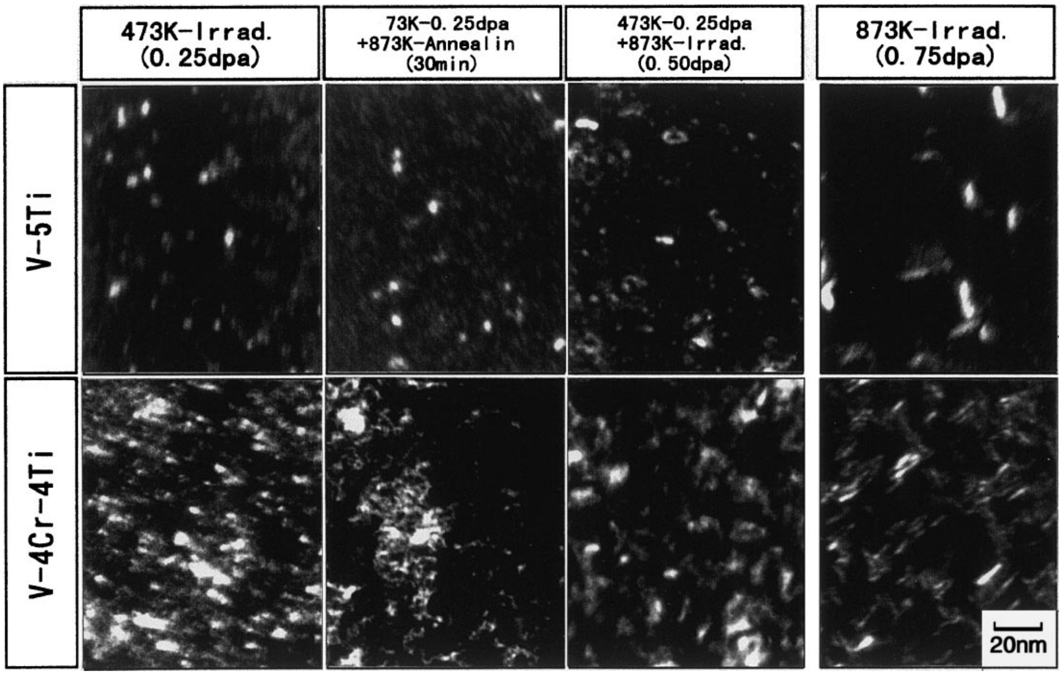

Fig. 3. The dark field weak-beam image of V–5Ti and V–4Cr–4Ti alloys at each step of irradiation conditions shown in Fig. 1.

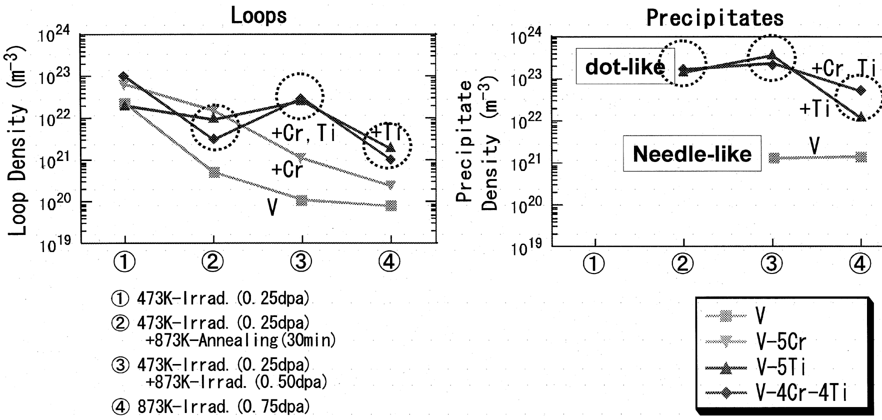

Fig. 4. The change of loop and precipitate density after each step of irradiation.

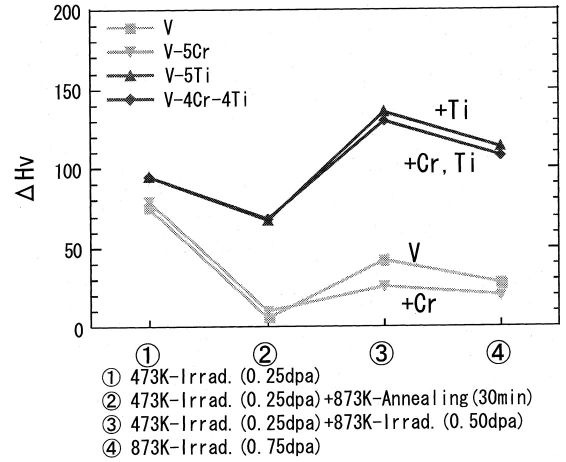

Fig. 5. The change of Micro-Vickers hardness after each step of irradiation.

Ti is large. After the successive irradiation at 873 K, enhanced formation of fine Ti-enriched precipitates was also observed. These Ti-enriched precipitates were also detected in V–4Cr–4Ti–0.1Si and V–3Fe–4Ti–0.1Si alloys under a stepwise temperature experiment using a Japanese Material Testing Reactor (JMTR) [5]. In that study, the precipitates were identified to be radiation-induced titanium oxides (TiO_2) and were shown to promote hardening. Ti-enriched plate-like precipitates lying on {0 0 1} habit plain were also reported in a V–4Cr–4Ti alloy during constant irradiation [7,8] and in tungsten arc welded specimens [9]. In the present study, small defect clusters, which formed during the successive irradiation at 873 K, were not identified. But, judging from the {0 0 1} habit plane, these defect clusters were probably titanium oxides.

It is well known that the magnitude of hardening strongly depends on the density and size of the barriers for the mobile dislocations. The increase in the yield stress, $\Delta\tau$, is given by the summation of a long range ($\Delta\tau_{LR}$) and short range term ($\Delta\tau_{SR}$) [10]. The long and short range forces are due to the interaction of a moving dislocation with the dislocation network and with obstacles (voids, precipitates, loops) lying on the slip plane, respectively. They are given by

$$\Delta\tau = \Delta\tau_{SR} + \Delta\tau_{LR}, \tag{1}$$

$$\Delta\tau_{SR} = \left[\sum \Delta\tau_i^2\right]^{1/2}, \tag{2}$$

$$\Delta_i = \alpha_i Gb(N_i d_i)^{1/2} \quad \text{(for obstacles)}, \tag{3}$$

where α_i is a constant for each type of defect (i), N_i the number of defects of a given type and diameter d_i per unit volume, G the shear modus, and b is the Burgers vector. The long range term is also given by

$$\Delta\tau_{LR} = \alpha Gb(\rho_d)^{1/2} \quad \text{(for dislocation network)}, \tag{4}$$

where ρ_d is the dislocation density of the material.

Owing of the low irradiation dose, a dislocation network was not observed in this study (as shown in Figs. 2 and 3), and the long range term due to the dislocation network should be negligible. Therefore,

$$\Delta\tau = \Delta\tau_{SR} + \Delta\tau_{LR} \fallingdotseq \Delta\tau_{SR}, \tag{1$'$}$$

$$\Delta\sigma_y = M\Delta\tau, \tag{5}$$

$$\Delta H_v \fallingdotseq (1/3)\Delta\sigma_y, \tag{6}$$

where M is the Taylor factor to relate the shear stress to applied tensile stress necessary to activate slip in polycrystal. There is still some discussion of correct value of M in vanadium base alloys [7]. M has been assumed to have values as low as $(3)^{1/2}$ and as high as 3. ΔH_v is the change of Micro-Vickers hardness due to irradiation.

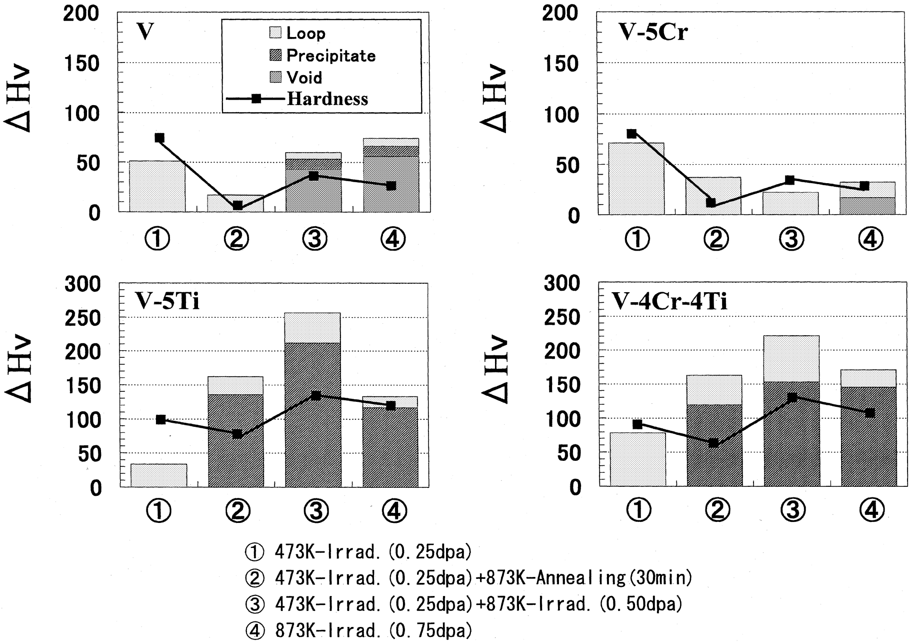

Fig. 6. The estimated change of Micro-Vickers hardness using the values of defect density and size observed in this study.

The results of Micro-Vickers hardness tests of the annealed samples in this study revealed that the value of α (for Ti precipitates) was close to 0.8. The estimated change of Micro-Vickers hardness using the values of defect density and size observed in this study is shown in Fig. 6. Fig. 6 shows an example of the case with using α (for void) $= 0.1$, α (loop) $= 0.3$ and $M = 3$. The estimation revealed that the increase of hardness after the successive irradiation at 873 K is presumably due to the formation of fine Ti-enriched precipitates. In many previous studies on V–4Cr–4Ti alloy [7,11], mechanical property changes were mainly attributed to dislocation loops as barriers for the motion of dislocations. The present study showed that the fine Ti-enriched precipitates are also important, especially in conditions where the irradiation temperature is changing.

5. Conclusions

To understand the influence of alloying elements (Cr, Ti) on the microstructure and hardness of vanadium during temperature variations, ion irradiation experiments were carried out in the temperature range 473 and 873 K. The main results are summarized as follows:

1. The microstructure and hardness behavior of model alloys were different for Ti-containing alloys and Ti-free alloys
2. The formation of Ti-enriched precipitates in V–5Ti and V–4Cr–4Ti alloys were enhanced by the successive irradiation at 873 K.
3. The enhanced formation of the precipitates results in hardening of the damaged region.

Acknowledgements

The authors would like to cordially thank Professor H. Matsui of Tohoku University for providing high purity vanadium base alloy used in this study.

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