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SWELLING STUDIES OF VANADIUM AND A VANADIUM-1 wt %
TITANIUM ALLOY USING ION SIMULATION TECHNIQUES*

by

A. T. Santhanam, [†] A. Taylor, B. J. Kestel and C. Steves
Argonne National Laboratory, Argonne, Illinois 60439, USA

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*Work performed under the auspices of the U. S. Atomic Energy Commission.

[†]Present address: Westinghouse Electric Corp., R & D Center, Beulah Road,
Churchill Borough, Pittsburgh, Pa. 15235.

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A. T. Santhanam,[†] A. Taylor, B. J. Kestel and C. Steves
Argonne National Laboratory, Argonne, Illinois 60439, USA

ABSTRACT

The void swelling of HPV and V-1 Ti has been investigated by irradiating the samples side by side with 3-MeV $^{51}\text{V}^+$ ions to damage doses between 2.4 and 54 dpa at a temperature of 700°C. Examination of the irradiated samples by transmission electron microscopy revealed that voids and dislocation networks had formed at 2.4 dpa in HPV and that swelling increased less than linearly with dose. In contrast, V-1 Ti showed negligible swelling even at 54 dpa. The swelling resistance of V-1 Ti to large doses is believed to be, at least in part, due to irradiation-induced precipitation.

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SWELLING STUDIES OF VANADIUM AND A VANADIUM-1 wt % TITANIUM ALLOY USING ION SIMULATION TECHNIQUES

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Argonne National Laboratory, Argonne, Illinois 60439, USA

INTRODUCTION

Investigations of fast-neutron irradiated vanadium and vanadium-titanium alloys, which are candidate materials for the first wall of fusion reactors, have shown that additions of as small as 1 wt % titanium can drastically reduce the swelling of vanadium.¹ The swelling resistance of vanadium-titanium alloys has tentatively been attributed to the presence of a high density of precipitates that act as recombination centers for radiation-produced point defects, perhaps combined with the trapping of point defects by titanium in solution. The purpose of the present study was to determine whether the swelling resistance of vanadium-titanium alloys is retained at larger irradiation doses. The swelling behavior of pure vanadium was also studied at several doses for comparison. Since large damage doses by neutron irradiation require long-time exposures, the specimens were bombarded with energetic self-ions at damage rates ~1000-fold greater than achievable in a reactor. This technique is now well established as a tool for rapid simulation of fast-neutron damage in metals.^{2,3} Tantalum⁴ or nickel ions⁵ have been used in previous investigations to bombard vanadium, whereas self-ions have been used in the present study.

EXPERIMENTAL

High-purity vanadium (HPV) sheet was obtained from the Materials Research Corporation. Vanadium-1 wt % titanium (V-1 Ti) alloy was fabricated at Argonne National Laboratory and rolled to a 0.010-in.-thick sheet. Disks, 3 mm in diameter, were punched from the cold-rolled sheets and given a full recrystallization anneal at 1100°C for 1 hr in a dynamic vacuum of 1×10^{-6} Torr. The interstitial impurities of the two materials are listed in Table I.

TABLE I

Interstitial Impurity Levels in HPV and V-1 Ti

Material	Interstitial Impurities (wt ppm)		
	O	C	N
HPV	15	120	10
V-1 Ti	200	150	30

The surface of the annealed disks were carefully prepared prior to ion bombardment in a manner described elsewhere.⁵ The HPV and V-1 Ti samples were mounted side by side in tungsten holders in square arrays of 16 disks. Both arrays were irradiated with 3-MeV $^{51}\text{V}^+$ ions at 700°C, one to damage levels of 2.5-10 and the other to 10-60 displacements per atom (dpa). The irradiation temperature of 700°C was chosen to coincide with the peak swelling temperature of Ni-ion bombarded vanadium.⁵ The various doses were obtained by the use of a mask to progressively expose the columns of specimens to the ion beam until all were irradiated. The lowest dose specimens were irradiated last. This procedure avoided any postirradiation annealing. The vacuum during the irradiation was 2×10^{-7} Torr. The residual gases were nitrogen, oxygen and water vapor. The intensity profile of the 3-MeV $^{51}\text{V}^+$ defocused and wobbled ion beam was measured before and after the irradiation using a Faraday cage and movable aperture. During the irradiation and beam calibrations, the beam profile and beam current were monitored with an oscillating wire profile monitor; the current was maintained constant to $\pm 5\%$. The mean ion flux at the specimen holder was $7 \times 10^{12} \text{ V}^+ \text{ ions/cm}^2 \text{ sec}$. The distribution of the damage energy as a function of depth in the specimen was calculated from the Brice⁶ computer codes RAS 3 and DAM 2. The total damage energy per atom E_{dam} deposited during the bombardment was converted

into the number of displacements per atom, d , with the aid of the modified Kinchin-Pease formula, $d = k E_{\text{dam}} / (2 E_d)$, discussed by Norgett et al.⁷ and by Doran et al.⁸ using a threshold displacement energy, E_d , of 40 eV and a k -factor of 0.8.

After irradiation, a 6500-7500-Å-thick layer was removed electrochemically from the bombarded surface of the specimen. The thickness of the removed layer was measured to an accuracy of ~ 500 Å, relative to an unsectioned region of the irradiated surface, using a two-beam interferometer. Foils for transmission electron microscopy examination of the area close to the peak damage region were then produced by thinning from the back surface. The foil thickness was determined from parallax measurements on stereopairs of photomicrographs. The mean displacement rate in the foils examined was 5.5×10^{-3} dpa/sec.

RESULTS

The irradiated HPV samples showed voids and dislocation networks, whereas the irradiated V-1 Ti specimens contained a high density of precipitates. The void parameters (void volume fraction $\Delta V/V$, mean void size, and void density) were determined from photomicrographs at a magnification of 168,000X. The dislocation densities were obtained by the linear intercept method.

Figure 1 illustrates the evolution of void and dislocation microstructure with irradiation dose from 2.4 to 34 dpa. The voids were cubic in shape, which was also observed in vanadium irradiated with $^{58}\text{Ni}^+$ ions.⁵ Slight truncations of the voids on the (111) planes at higher doses required a 2% correction to the void volume.

The data on void volume fraction, void size, void number density and dislocation density as a function of dose are plotted in Fig. 2. The swelling and mean void size increased monotonically with dose, whereas the void concentration decreased

from 5×10^{15} voids/cm³ at 2.4 dpa to 3×10^{14} voids/cm³ at 34 dpa. The dislocation density also decreased with dose. Note that the void volume fraction extrapolated to 60 dpa is consistent with our previous data point at 700°C using Ki^+ ion bombardment.

The irradiated V-1 Ti samples showed no voids at low doses, instead, the microstructure contained a high number density ($>10^{16}$ /cm³) of small precipitates (<50 Å) and dislocation networks, as shown in Fig. 3. The precipitates were more clearly visible in dark-field images, which were produced by the streaks shown in the selected area diffraction pattern in the inset of the 54-dpa micrographs. The unirradiated control samples of V-1 Ti do not show any precipitates. Figure 3 shows that precipitates are formed as a consequence of irradiation even at low doses. Identification of the precipitate was not possible because no extra reflections were present in the diffraction pattern. An earlier study¹ of internal friction in V-1 Ti alloys of different oxygen contents indicated that the precipitates could be a titanium oxide. The TEM investigation of the precipitation phenomenon in vanadium-titanium alloys by Kramer⁹ showed that the precipitates obtained by aging in vanadium-titanium alloys were TiO with a cubic NaCl structure. Kramer also showed that the precipitates do not give extra reflection in the [100] orientation, which is in agreement with the present observation. With increasing irradiation doses, the size of the precipitates coarsened only slightly. At 22 dpa, small voids were observed near grain boundaries in narrow regions that were devoid of precipitates, as shown in Fig. 4b. At 40 dpa and higher doses, the precipitates appeared to be platelets aligned along [100] directions; but an examination of a bright-field micrograph taken at a higher magnification (Fig. 4a) suggests that the precipitates may be individual particles aligned along [100] directions. A few voids appeared

within the grains at 54 dpa; however, their small size ($\sim 70 \text{ \AA}$) and concentration ($\sim 5 \times 10^{14} \text{ voids/cm}^3$) produced negligible ($\sim 0.02\%$) swelling.

DISCUSSION

HPV

The present investigation of the dose dependence of void swelling in vanadium irradiated with 3 MeV at 700°C showed that stable voids were formed at dosages $< 2.4 \text{ dpa}$ and grew as the dosage increased. The void volume fraction extrapolated to 60 dpa is consistent with data obtained with Ni^+ ion bombardment.⁵ The strong decrease in the void number density with dose at comparatively small void volume fractions, at which void impingement and coalescence is unlikely, warrants further investigation. Recently, decreases in void number density with an increase in dosage have been observed in ion-irradiated pure nickel specimens; the effect may be associated with changes in the dislocation structure.¹⁰ Void nucleation in vanadium is sensitive to interstitial impurities such as C, N, and O. The variation in the void number density could be related to impurity pick up by the samples prior to and during ion bombardment. However, the observed number densities obtained from the two sets of the present experiments does not seem to support this explanation.

V-1 Ti

Perhaps the most significant result of the present study is the demonstration of the beneficial role of titanium in suppressing void swelling in vanadium. The previous neutron irradiation study of vanadium-titanium alloys was limited to a dose of $\sim 1 \times 10^{22} \text{ n/cm}^2$, $E > 0.1 \text{ MeV}$,¹ corresponding to $\sim 6 \text{ dpa}$.^{8,11,12} In the present investigation, it has been shown that the beneficial effect of titanium persists at least up to 54 dpa, approximately an order of magnitude higher damage level. As in the neutron study, the

predominant microstructural feature in the irradiated V-1 Ti was found to be fine precipitates. The precipitates appear to be relatively stable under prolonged irradiation. A dislocation network was visible only at low doses. Either the fine-scale precipitate dispersion or the lack of dislocations in the structure, or both, could be responsible for the swelling resistance of vanadium-titanium alloys. Support for the above argument can be derived from the microstructure of V-1 Ti at 22 dpa. Figure 5(b) shows narrow regions along grain boundaries that are devoid of precipitates and voids preferentially nucleated in the precipitate-denuded region. This observation indicates that the finely dispersed second phase suppresses void formation in this alloy, which is in contrast to the solid solution effect observed by Johnson et al.¹³ in the Fe-Ni-Cr system.

The results suggest that vanadium-titanium alloys could retain dimensional stability to target fluences expected in future fusion reactors.

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FIGURE CAPTIONS

- FIG. 1. Void and dislocation microstructure of HPV irradiated with 3-MeV V^+ ions at 700°C to the doses indicated.
- FIG. 2. (a) Void volume fraction and average void size measured across the cube edges vs dosage and (b) dislocation and void number density vs dosage.
- FIG. 3. Microstructure of V-1 Ti irradiated with 3-MeV V^+ ions at 700°C to the doses indicated. The dark-field photomicrographs showing the precipitation were imaged using a [100] streak shown in the inset selected area electron diffraction pattern.
- FIG. 4. Precipitate structure in irradiated V-1 Ti. (a) 54-dpa specimen oriented to give maximum contrast to the precipitates, and (b) voids formed in a zone adjacent to a grain boundary that was denuded of precipitates at 22 dpa.

VOID MICROSTRUCTURE

DISLOCATION MICROSTRUCTURE

2.4

4.3

22

34 dpa

FIG. 1.

Void and dislocation microstructure of HPV irradiated with 3-MeV V^+ ions at 700°C to the doses indicated.

FIG. 2. (a) Void volume fraction and average void size measured across the cube edges vs dosage and (b) dislocation and void number density vs dosage.

BRIGHT FIELD

DARK FIELD

2.4

4.3

34

54 dpa

FIG. 3.

Microstructure of V-1 Ti irradiated with 3-MeV V^+ ions at 700°C to the doses indicated. The dark-field photomicrographs showing the precipitation were imaged using a [100] streak shown in the inset selected area electron diffraction pattern.

(a)

(b)

FIG. 4. Precipitate structure in irradiated V-1 Ti. (a) 54-dpa specimen oriented to give maximum contrast to the precipitates, and (b) voids formed in a zone adjacent to a grain boundary that was denuded of precipitates at 22 dpa.