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Effect of temperature change on microstructural evolution of vanadium alloys under neutron irradiation in JMTR

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Abstract

Recently, it has been pointed out that the effect of varying temperature on microstructural evolution in materials under neutron irradiation can be very complicated. In this paper, evolution of microstructure in vanadium alloys in each step of temperature changes is closely examined in order to analyze the effects after several temperature cycles. Neutron irradiation was performed in JMTR with an upward temperature change. TEM observation, micro-Vickers hardness test and positron annihilation lifetime spectroscopy have been carried out on vanadium alloys. During the irradiation at 220°C, a lot of small defects have nucleated. They grew up after the upward temperature step to 420°C. On the other hand, microstructure remained essentially the same in the irradiation at 340/530°C temperature combination. Vanadium alloys containing titanium show different tendencies; unlike other alloys, a number of radiation induced precipitates were found by TEM in these alloys. © 1999 Published by Elsevier Science B.V. All rights reserved.

1. Introduction

Vanadium alloys have a number of attractive features as candidate structural materials for fusion reactors. In order to clarify the behavior of these alloys under irradiation, numerous neutron irradiation experiments have been performed. According to these experiments with fission reactors, it has been pointed out that there is significant difference in microstructures between the specimens which experienced a low temperature irradiation before reaching the target temperature and those irradiated at the target temperature from the beginning [1–3]. Thus, the effect of varying temperature on microstructural evolution in materials can be very complicated. In the future fusion reactors, structural materials are expected to experience non-steady irradiation temperature, neutron flux and other parameters during reactor start-up and shut-down and at plasma disruption, etc.

In the present work, the effect of temperature change on microstructural evolution during neutron irradiation has been studied with a multi-section, multi-division irradiation rig [3] in Japan Material Testing Reactor (JMTR).

2. Experimental

Pure vanadium and six types of binary vanadium alloys; i.e., V–5%Fe, V–1%Si, V–5%Cr, V–5%Mo, V–5%Ti and V–5%Nb (compositions of these alloys are given in at.%) which cover a wide range of atomic size factor of solutes in vanadium and two candidate alloys: V–4Cr–4Ti–0.1Si and V–3Fe–4Ti–0.1Si.

Neutron irradiation was performed in JMTR with multi-section, multi-division irradiation rig which enables specimen retrieval at any points of fluences during reactor operation. In the present work, specimens were retrieved from the reactor at three different points as illustrated in Fig. 1; i.e. (1) at the end of the lower temperature irradiation period, (2) 3 h after the upward temperature change, and (3) at the end of the higher temperature irradiation. The temperature combinations are 220/420°C and 340/530°C, and the calculated damage is up to 0.25 dpa.

After electro-polishing, the microstructure of alloys was examined by TEM. In order to obtain

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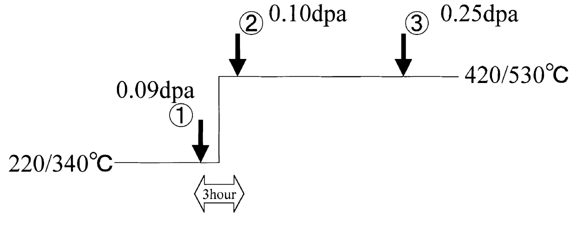

Fig. 1. Neutron irradiation were performed in JMTR. Specimens were retrieved by multi-section multi-division rig.

complementary information relating to invisible small vacancy-type clusters such as microvoids, positron annihilation spectroscopy measurement was carried out. Micro-Vickers hardness test was also carried out in order to examine the correlation between microstructure and mechanical properties.

3. Results and discussion

3.1. Positron annihilation spectroscopy

Fig. 2 shows the change of intensity and lifetime in the selected alloys at each step of temperature varying neutron irradiation. The lifetimes of about 110, 150–200 and 400 ps roughly correspond to annihilation of positrons in the bulk, trapped by dislocations or single vacancies and trapped by microvoids, respectively.

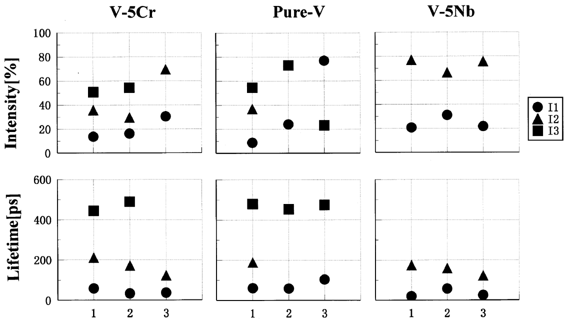

Fig. 2. The behavior of intensity and lifetime in some alloys under 200/420°C temperature varying neutron irradiation.

In 220/420°C irradiation, the lifetime for microvoid (third component τ 3) was detected at irradiation steps (1) and (2) in several binary alloys. Between the values of (1) and (2), there is no significant difference. It follows that thermal stability of V-cluster is well maintained throughout this temperature change. After the upward temperature change (3), the intensity of microvoid component disappeared or became negligibly small. This indicates that microvoids grow, and its number density became too low to be detected by means of positron annihilation spectroscopy. This agrees with the results that voids were observed by TEM in these specimens. Alloys including titanium: V–5Ti, V–4Cr–4Ti–0.1Si and V–3Fe–4Ti–0.1Si have different tendencies where no microvoid was observed. It is suggested that trapping of vacancy by solute atoms is responsible for this behavior. Since titanium has bigger atomic size than vanadium, the binding energy between solute atoms and vacancies is large enough to suppress the migration of vacancies.

In 340/530°C irradiation, the lifetime corresponding to microvoids was never detected in all irradiation conditions in all alloys except in pure vanadium. There is no significant difference by irradiation conditions. In this temperature region, microvoid grows and its density becomes too low to be detected. Also in this temperature condition, alloys containing titanium have no microvoid.

3.2. Micro-Vickers hardness test

Fig. 3 shows the yield stress change by irradiation calculated from micro-Vickers hardness using the following formula obtained experimentally [4]:

$$\Delta\sigma \approx 3\Delta Hv. \qquad (1)$$

In 220/420°C irradiation, significant irradiation-induced hardening occurred during low temperature irradiation in alloys containing undersized solute atoms

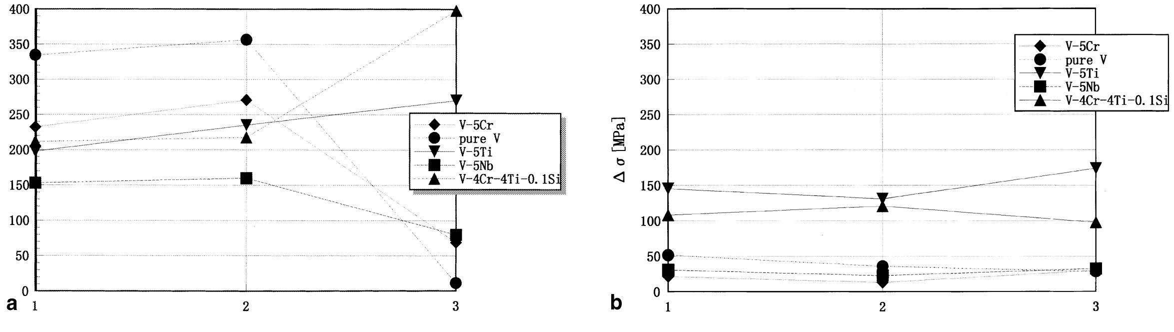

Fig. 3. (a) The yield stress change under 220/420°C irradiation calculated from micro-Vickers hardness; (b) the yield stress change under 340/530°C irradiation calculated from micro-Vickers hardness.

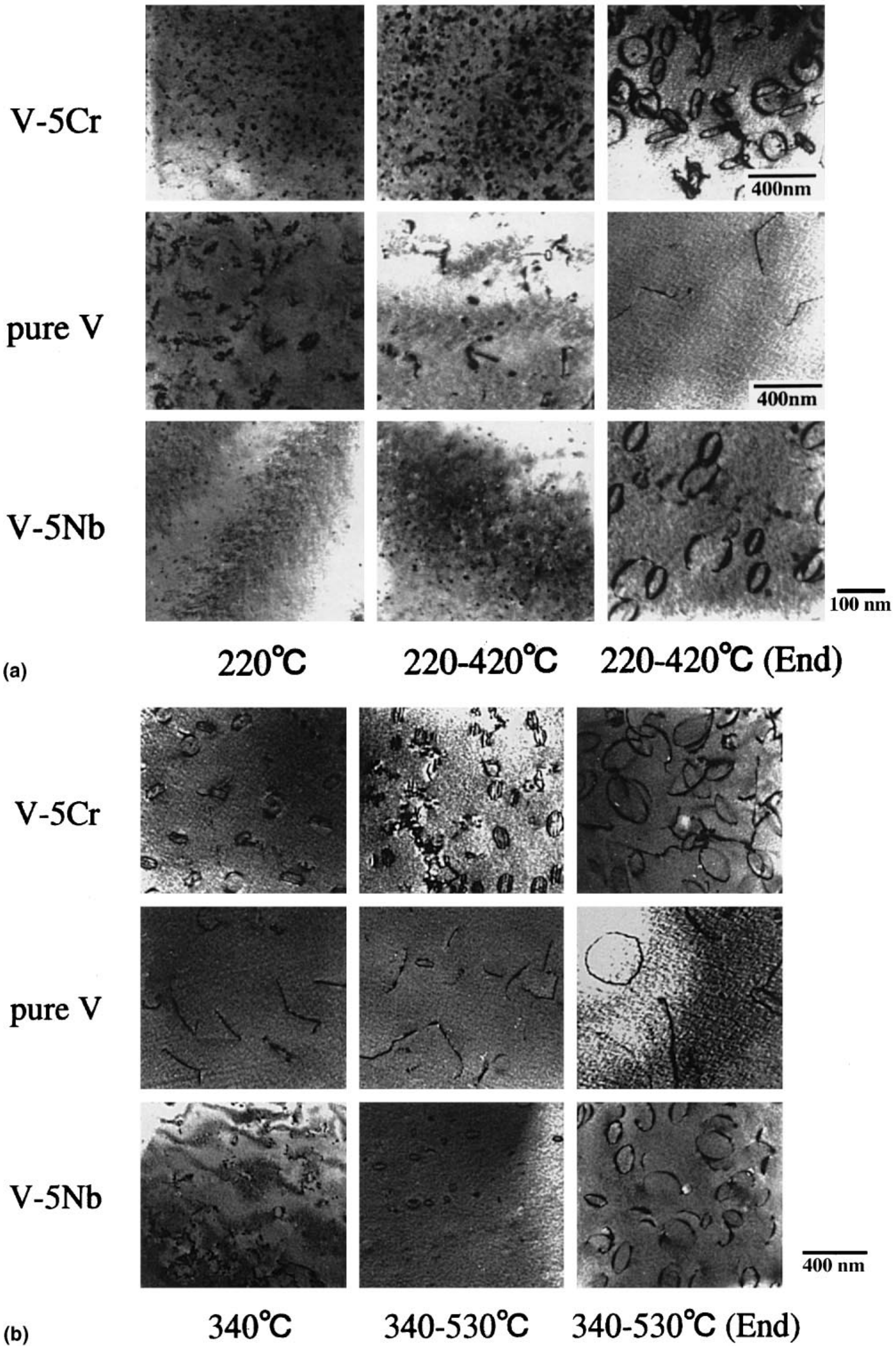

Fig. 4. (a) Variation of microstructure of V–5Cr, pure-V, V–5Nb under 200/420°C temperature change; (b) variation of microstructure of V–5Cr, pure-V, V–5Nb under 340/530-°C temperature change.

except for V–5Fe. This hardening recovered after the high temperature irradiation (condition (3)). There were no significant differences between conditions (1) and (2). This means that defect clusters are thermally stable during this temperature change. In V–5Fe, the hardening was not significant under the low temperature irradiation and the change of hardening by the temperature change during irradiation was smaller than that of other alloys. It is suggested that each alloy has a specific temperature region where defects can nucleate or grow. It is likely that in V–5Fe, 220°C is a high enough temperature for defect growth. In alloys containing titanium, the hardening did not recover but increased after high temperature irradiation.

On the other hand in 340/530°C irradiation, the hardening under low temperature irradiation was not very significant. The hardness value did not show remarkable change on temperature change. There was, however, significant hardening in the alloys containing titanium for the same temperature changes.

3.3. TEM observation

Fig. 4 shows microstructure of alloys after irradiation. In 220/420°C irradiation, a number of small dislocation loops were observed under low temperature irradiation and also after the temperature change (conditions (1) and (2)). Between these two conditions, there is no significant difference except in V–5Fe. The thermal stability of defect clusters in the alloys throughout the temperature change was well maintained again. In the irradiation condition (3), the loops that nucleated during the low temperature irradiation grew up and the microstructure of alloys became coarse. Estimated changes of yield stress are shown in Fig. 5. These values have been derived from TEM microstructure with Orowan's equation [5]:

$$\Delta\sigma = \left(\frac{\alpha\mu b}{k}\right)\sqrt{\sum \rho_i d_i}, \qquad (2)$$

where σ is the yield stress, α is the coefficient representing the strength of the defect as a barrier against dislocation motion, μ is the shear modulus, b is the Burgers vector, k is the Taylor factor, ρ is the number density of defects and d is the size of defects. In the present paper, α is taken as 0.2 for network dislocation, 0.3 for cavities and dislocation loops and 0.6 for precipitates. These results for almost all alloys are in poor agreement with the data from Vickers hardness test. It is considered that defects are too small to be observed by TEM, e.g., micro-voids, contribute to the hardness to some extent. In alloys containing titanium, a lot of precipitates were observed which contribute greatly to hardness. Precipitates grew after the temperature change, while, the hardening did not recover but increased. Cavities were observed in under-size alloys and pure vanadium after the temperature change; i.e., condition (3). This is in good agreement with the results obtained using positron annihilation spectroscopy; i.e. micro-voids which were detected by positron, grew up to the size which can be observed by TEM.

In 340/530°C irradiation, microstructure coarsening occurred as the temperature increased but the changes were not so drastic compared to the ones under 220/420°C irradiation. Cavities were observed in under-size alloys and pure vanadium during the irradiation even after the low temperature irradiation. No cavity was observed in alloys containing titanium in these temperature regions.

4. Conclusion

There is quite a significant difference between the 220/420°C and the 340/530°C irradiation. In the former case, significant change of microstructure was observed by the temperature change but in the latter case, the change is not so large. It can be said that there are temperature regions where the nucleation of the defects mainly occurs and where the growth of defects mainly occurs. The

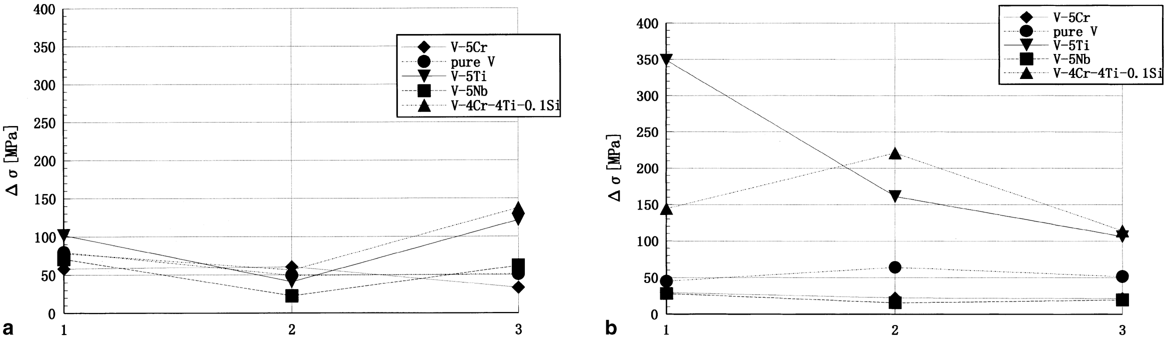

Fig. 5. (a) The yield stress change under 220/420°C irradiation calculated from TEM micrograph; (b) the yield stress change under 340/530°C irradiation calculated from TEM micrograph.

relative position of these temperature regions and the two temperatures of the temperature-varying irradiation has decisive effects on the evolution of microstructure under temperature-varying irradiation. The resulting microstructure, in turn, reflected on the mechanical properties of the material.

Microstructure in the specimen that is retrieved soon after the temperature change did not show significant difference to the one that irradiated at the low temperature. Temperature change itself in this case does not have a strong effect on the microstructure of alloys. In V-5Fe, however, there is significant growth of defects even in the specimen which was retrieved soon after temperature change to 420°C. There must be certain nucleation and growth temperature regions characteristic of each alloy.

Radiation-induced precipitates were observed in alloys containing titanium, which promote the hardening significantly. In a previous study, it has been confirmed that these precipitates are titanium oxides (TiO_2). The density and size of precipitates seem to be controlled by the amount of oxygen in the specimen, which is temperature dependent. No cavity, not even microvoid, was observed in the alloys containing titanium because this solute atom, which is bigger than vanadium, restricts the nucleation of cavity by suppressing vacancy mobility.

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