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# The study of temperature variation during HFIR irradiation on vanadium

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**Abstract**

The effects of temperature variation on pure vanadium and vanadium model alloys under neutron irradiation were studied in high flux isotope reactor (HFIR). Pure vanadium and four vanadium model alloys (V–5Cr, V–5Ti, V–4Cr–4Ti, V–4Cr–4Ti–0.1Si) were irradiated under variable temperature conditions at 633/793 K for a total 8 of irradiation cycles. After the irradiation, the microstructure of the samples was compared to those of the same alloys, which were continuously irradiated at 793 K.


## 1. Introduction

The effects of varying temperature irradiations on pure vanadium and vanadium model alloys have been studied under fission neutron [1–3] and also ion [4–6] irradiation. To understand the effects of temperature variation at relatively higher dose levels, the varying temperature irradiation experiment using the high flux isotope reactor (HFIR) was selected as one of the major tasks in the Japan–USA Fusion Cooperation Program (JUPITER Project) [7]. In the HFIR 13J varying temperature experiment, four irradiation zones were set, low-temperature (613 K) constant, high temperature (793 K) constant, high temperature variable (633/793 K) and low-temperature variable (498/613 K). In the variable zones, the temperatures were reduced for the initial 10% period of each irradiation cycle, and elevated for the remaining 90% period. Our previous study of HFIR 13J varying temperature (498/613 K) irradiation [2] showed that was very sensitive to the irradiation conditions. Namely, long platelet shaped carbides were only observed in the sample which was irradiated under varying temperature. In addition, the carbide formation resulted in smaller void size. On the other hand, the effects of low-temperature irradiation at 498 K on vanadium alloys were relatively small, except for V–4Cr–4Ti alloy. In V–4Cr–4Ti, a high density of small voids was only detected under 498/613 K irradiation condition. The present paper summarizes some key findings of pure vanadium and vanadium alloys under constant temperature (793 K) and variable temperature (633/793 K) irradiation conditions. Microstructural information obtained from the HFIR 13J experiment were also compared to our previous results obtained by using a multi-section, multi-division irradiation rig in the Japan Materials Testing Reactor (JMTR) which was designed and developed by Kiritani [8].

## 2. Experimental procedure

Pure vanadium and four model vanadium alloys (namely, V–5Cr, V–5Ti, V–4Cr–4Ti, V–4Cr–4Ti–0.1Si) were used in this study. The detailed specimen compositions are reported in Ref. [9]. The oxygen concentration in each vanadium alloy ranged between 13–1889 appm. Disk specimens for electron microscopy were wrapped with pure zirconium as a getter of oxygen and

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annealed 2 h at 1373 K. The irradiation was conducted for a total 8 cycles using in europium shield at the RB* position of HFIR, which resulted in a fast fluence ($E > 0.1$ MeV) of $8 \times 10^{21}$ n/m$^2$. This corresponds to 4 dpa in pure vanadium. The experimental design, fabrication of the capsule and a summary of the HFIR operation were reported in Ref. [10,11].

## 3. Results

### 3.1. Pure vanadium

In Fig. 1, the microstructure of pure vanadium at constant temperature irradiation (793 K) and varying temperature irradiation (633/793 K) were compared. At the constant temperature irradiation, relatively larger voids were formed. The measured void size and density were 16 nm and $2.1 \times 10^{23}$ m$^{-3}$, respectively. However, at varying temperature, very small void of about 5 nm and larger voids of about 9 nm were observed simultaneously. The average void density of smaller voids and larger voids were $4.2 \times 10^{23}$ m$^{-3}$ and $5.4 \times 10^{23}$ m$^{-3}$, respectively. In both samples, as shown by arrows in the figure, small density of carbides which oriented ⟨1 0 0⟩ directions, were also observed.

### 3.2. Vanadium alloys (V–5Cr, V–5Ti, V–4Cr–4Ti, V–4Cr–4Ti–0.1Si alloys)

In V–5Cr, as shown in the Fig. 2, the constant and varying temperature irradiation produced a tangled dislocation structure with almost same dislocation density in both cases. The measured dislocation densities under constant and varying temperature irradiation were $4.4 \times 10^{13}$ m$^{-2}$ and $2.1 \times 10^{13}$ m$^{-2}$, respectively. In comparison with pure vanadium, the voids detected under both irradiation condition were larger in size and lower in number density. In addition, small voids of about 7.5 nm diameter were also observed in the sample which was irradiated under variable temperature conditions.

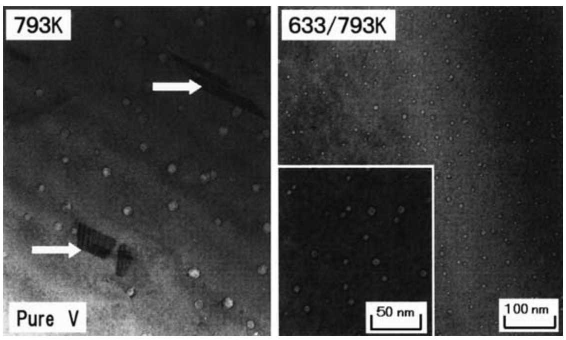


Fig. 1. Comparison of voids observed in pure vanadium at 793 and 633/793 K. The arrows in the figure show carbides formed in the sample.

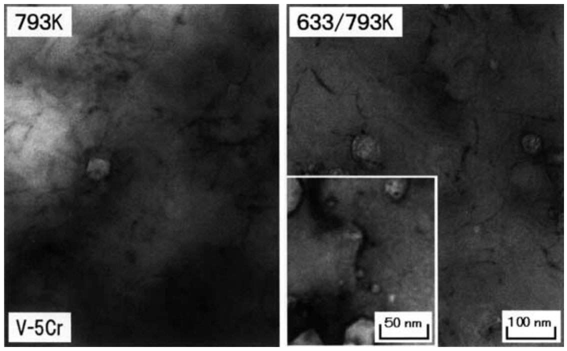


Fig. 2. Comparison of voids observed in V–5Cr at 793 and 633/793 K.

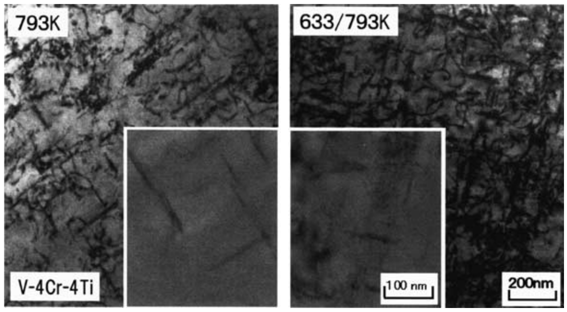


Fig. 3. Comparison of voids observed in V–4Cr–4Ti at 793 and 633/793 K.

The microstructure of V–4Cr–4Ti irradiated under constant (793 K) and varying temperature condition (633/793 K) are compared in Fig. 3. In the figure, the corresponding void contrast images are also inserted. The figure shows that the radiation induced formation of titanium oxides on ⟨1 0 0⟩ habit planes and tangled dislocations in both samples. The average density of oxides and size were $5.3 \times 10^{20}$ m$^{-3}$ and 95 nm, respectively. However, varying temperature irradiation produced smaller oxides with mean size of 52 nm and a higher density of $1.5 \times 10^{21}$ m$^{-3}$. Formation of voids, which were observed previously following low temperature irradiation (498/613 K) [2], was not detected in either irradiation condition investigated here. In V–5Ti and V–4Cr–4Ti–0.1Si alloys, oxides were also found and the microstructures were similar for the two irradiation conditions.

## 4. Discussion

The present study shows that void formation in pure vanadium and V–5Cr alloy was enhanced due to varying

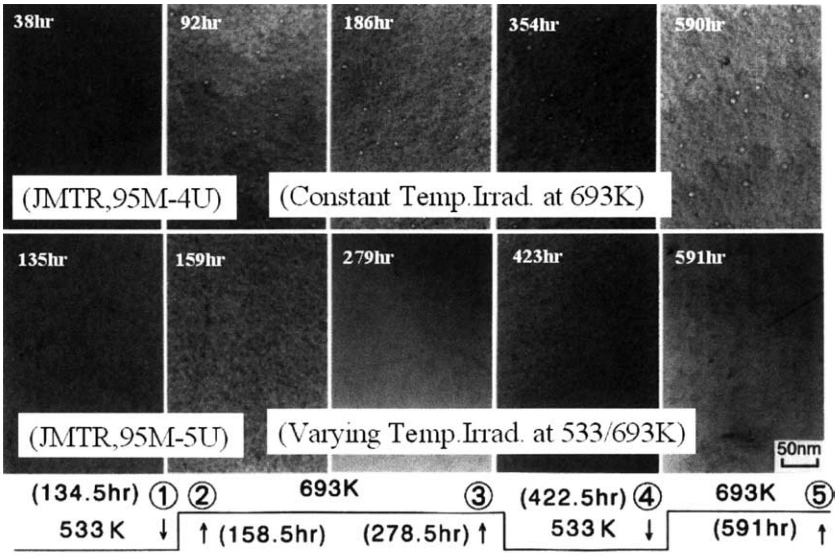


Fig. 4. Comparison of voids observed in pure vanadium at 693 K (upper photos) and 533/693 K (lower photos) using the using a multi-section, multi-division irradiation rig in JMTR.

temperature (633/793 K) irradiation. Fig. 4 shows voids observed in pure vanadium at 693 K (upper photos) and at 533/693 K (lower photos) using a multi-section, multi-division irradiation rig in JMTR. Using the rig, which allows the removal of some samples from the reactor at the scheduled irradiation time (e.g. 134.5 h in the figure), microstructural evolution of the samples at a certain dose level was obtained. The total neutron dose for a full cycle of irradiation (591 h for the case of JMTR 95M-5U irradiation cycle) was $1.5\times10^{24}$ $m^{-2}$ (>1.0 MeV), which corresponds to 0.22 dpa for pure vanadium. Due to varying temperature irradiation, enhanced void formation is also confirmed. The same enhanced formation of voids in stainless steels and its ternary model alloy (Fe–16Cr–17Ni) was also detected in the previous studies irradiated in HFIR 13J [12] and JMTR [13] experiments. For the case of stainless steels and its model alloys, which have lower stacking fault energy, conversion of stacking

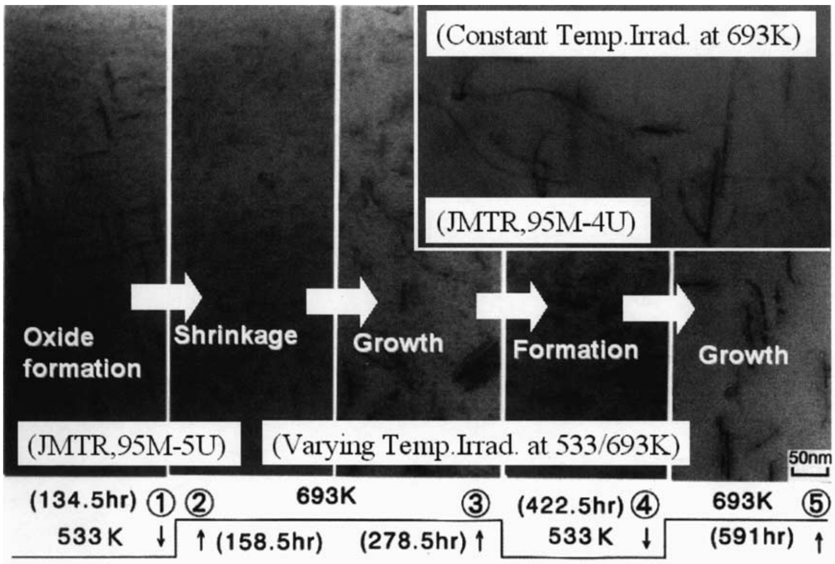


Fig. 5. Microstructural evolution of V–4Cr–4Ti under varying temperature irradiation at 533/693 K using JMTR (JMTR, 95M-5U). The inserted image is the microstructure of the same sample after the irradiation at constant temperature (693 K) and the end of the irradiation cycle.

fault tetrahedra (SFT) to voids was one of the main reasons for the enhanced formation of voids due to the temperature variation. For the case of pure vanadium and vanadium base model alloys, nucleation (at lower temperatures) and growth (at higher temperatures) of voids are occurred successively, which resulted in the higher void density for the samples irradiated under variable temperatures. Further analysis of void nucleation (and growth) is needed to investigate the quantitative influence of varying temperature irradiation.

Fig. 5 shows the microstructural evolution of V–4Cr–4Ti alloy during varying temperature irradiation conducted in JMTR. The irradiation cycle (namely JMTR 95M-5U) is as same as the one shown in Fig. 4. The inserted photo in the upper right corner of the figure shows the microstructure of V–4Cr–4Ti alloy irradiated at 693 K (constant temperature irradiation) after end of the irradiation cycle. As shown in the figure, titanium oxides formed early in the irradiation. Shrinkage and disappearance of oxides occurred during irradiation at 693 K (see ② in the figure). Continued irradiation at 693 K, caused oxide nucleation and growth to occur again. Compared with the microstructure produced at 533 K (①), the oxides formed (as shown in ③) were larger and of lower density. By repeating these processes, in comparison with the oxide density produced after constant temperature irradiation at 693 K, a higher density of oxides was observed at the end of the varying irradiation cycle (namely, ⑤). Enhanced oxide formation due to varying temperature irradiation was also observed by Zinkle et al. [3]. The detailed mechanism of enhanced oxide formation in V–4Cr–4Ti alloy (shown in the present study) and carbide formation in pure vanadium (shown by previous study [2]) due to temperature variation by 469/613 and 633/793 K, respectively, is not known. It is well established that radiation induced precipitation is controlled by the migration of solute atom-point defect (vacancy and/or interstitial) complexes under irradiation. Therefore, enhanced oxide (or carbide) formation is also related to very high concentration of vacancies and/or interstitials caused by the temperature variation.

## 5. Conclusions

Varying temperature irradiation at 633/793 K enhanced void formation in pure vanadium and also V–5Cr alloy. In V–4Cr–4Ti alloy, on the other hand, a higher density of titanium oxides was detected under varying temperature (633/793 K) irradiation conditions. In comparison with previous results obtained in a JMTR varying temperature irradiation, it was confirmed that titanium oxide stability depends sensitively on irradiation temperature. This is the reason for the enhanced oxide formation observed. On the other hand, the effects of low-temperature irradiation on vanadium alloys were relatively small for V–4Cr–4Ti–0.1Si and V–5Ti alloys.

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