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Varying temperature effects on mechanical properties of vanadium alloys during neutron irradiation

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Abstract

The varying temperature irradiation experiment in the HFIR was carried out in order to investigate the performance of vanadium alloys subject to temperature variation during operation. No significant differences of irradiation hardening between steady 340 °C irradiation and variable 225/340 °C irradiation could be seen in any alloys. In the case of the irradiation at 520 °C, the temperature variation to 360 °C influenced the formation process of voids in the unalloyed and dilute vanadium alloys compared to isothermal irradiation at 520 °C. It is contributed to the change of irradiation hardening behavior between steady irradiation and temperature variable irradiation at 520 °C. It could not be seen any effect of varying irradiation temperature in vanadium alloys containing >1 wt% titanium in this study. It is caused by the insensitivity of formation process of Ti(OCN) precipitates against the effect of irradiation temperature variation.

1. Introduction

Structural components of fusion reactors will be subject to temperature variation during irradiation under operation. In particular, minor unexpected temperature excursions will occur during reactor shutdown and start-up procedures [1]. Knowing the sensitivity of materials behavior to temperature transients during irradiation will allow us an accurate estimation of material lifetime under fusion reactor operation. Hence, it is important to investigate property changes of materials due to temperature variation under irradiation. The varying temperature irradiation experiment in HFIR was carried out in order to investigate the performance of fusion structural materials subject to temperature variations during operation [2,3]. In the framework of this experiment, vanadium alloys were selected as representative target materials in this study. For a wide knowledge of property changes in vanadium alloys, a wide variety of vanadium alloys were used in this study.

2. Experimental procedure

The varying temperature irradiation experiment in the HFIR was carried out in the framework of the Japan-USA fusion cooperation program (JUPITER). The details of the experiments were reported previously [2–5]. The experiment has four irradiation conditions; steady 520 °C, steady 340 °C, variable 360/520 °C and variable 225/340 °C. In the variable zones, the initial 10% period of each irradiation cycle was at the lower temperature and then elevated for the remaining 90% period. The irradiation was carried out for eight cycles and reached ~4 dpa for vanadium alloys. TEM disks

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and tensile specimens were included in the experiment. The materials included unalloyed vanadium, vanadium binary alloys (with solute atoms of Fe, Cr, Ti, Nb up to 5 wt%), vanadium ternary alloys (V–4Cr–xTi, $x = 0.1$–4 and V–5Fe–yTi, $y = 0.1$–3) and V–4Cr–4Ti–0.1Si. Tables of chemical component have been reported in the previous papers [6,7]. The tensile tests for four irradiation conditions were performed on unalloyed vanadium, V–5Cr, V–5Nb and V–4Cr–4Ti–0.1Si. The tensile tests of the other materials were carried out for the 360/520 and 520 °C conditions. Most of tensile tests were done at room temperature. After tensile tests, some of the tested samples were cut and fabricated into TEM disks by electro-polishing. The microstructural examinations were based on TEM and SEM.

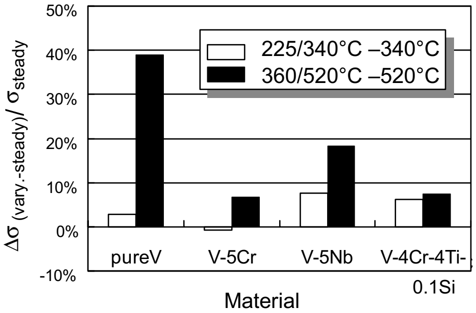

Fig. 1. The yield stress increase for vanadium alloys between steady irradiation and varying temperature irradiation. The data was normalized by each steady state irradiation yield stress.

3. Results

3.1. Tensile test

Tensile test data for unalloyed vanadium and the V–4Cr–4Ti–0.1Si alloy irradiated for all temperature histories were described in Figs. 2 and 4 in Ref. [4]. In the case of 340 °C, significant irradiation hardening and reduced elongation can be seen under both steady and variable irradiation. The temperature variation for 225 °C does not influence the tensile properties relative to steady temperature irradiation at 340 °C in both alloys. In the case of 520 °C, a significant increase in yield stress and a decease in elongation can be seen in unalloyed vanadium for the 360/520 °C irradiation compared to steady 520 °C irradiation. Fig. 1 shows the changes of yield stress increase at the steady temperature irradiation and the variable temperature irradiation for four materials, unalloyed alloy, V–5Cr, V–5Nb and V–4Cr–4Ti–0.1Si alloys. No significant changes in irradiation hardening at 340 and 225/340 °C irradiations can be seen in all for materials. In the case of 520 °C irradiation, unalloyed vanadium and V–5Nb exhibited an apparent increase of yield stress in temperature-variable irradiation. From the preliminary test, tensile tests for the specimens at 520 °C steady irradiation and 360/520 °C variable irradiation were examined with particular interests. Table 1 shows the tensile test data for the specimens of 520 °C steady irradiation and 360/520 °C variable irradiation. The unalloyed vanadium, V–0.1Fe, V–0.3Fe, V–5Mo and V–5Nb had greater irradiation hardening in the variable-temperature irradiation than steady-temperature irradiation at 520 °C. On the contrary, V–5Fe showed larger increase in yield stress and

Table 1
Tensile data for specimens irradiated at 520 and 360/520 °C

	Yield stress (MPa)			Ultimate tensile stress (MPa)			Uniform elongation (%)		
	Unirradiated	520 °C	360/520 °C	Unirradiated	520 °C	360/520 °C	Unirradiated	520 °C	360/520 °C
Unalloyed V	127	160	223	201	229	325	19	8	7
V–5Cr	278	339	338	349	423	414	14	12	9
V–5Nb	351	328	389	443	426	479	16	11	13
V–5Mo	373	362	404	409	436	514	14	13	9
V–5Ti	266	280	289	328	380	396	13	13	12
V–0.1Fe	174	273	298	197	319	359	10	9	10
V–0.3Fe	178	251	328	200	286	375	12	4	6
V–5Fe	250	328	268	320	825	629	13	12	16
V–5Fe–0.1Ti	257	436	347	320	873	783	13	17	16
V–5Fe–0.3Ti	315	601	559	380	811	815	13	10	10
V–5Fe–1Ti	320	586	630	340	679	711	13	8	8
V–3Fe–4Ti–0.1Si	388	474	484	475	583	593	18	11	12
V–4Cr–0.1Ti	300	452	454	324	547	523	11	11	8
V–4Cr–4Ti–0.1Si	326	394	423	441	489	535	20	11	10

UTS at 520 °C irradiation than 360/520 °C irradiation. With increasing titanium additions to V–5Fe, the changes in yield stress and UTS between steady- and variable-temperature irradiation decreased and no difference could be seen at 3% titanium. In the vanadium alloys containing >3 wt% of titanium as well as V–5Fe–*y*Ti alloys, the change of irradiation hardening due to variable-temperature irradiation could not be seen.

4. Microstructures at steady 520 °C and variable 360/520 °C irradiation

Fig. 2 shows the void microstructures in V–0.3Fe irradiated at 520 and at 360/520 °C. A bimodal distribution of void sizes can be seen in V–0.3Fe due to variable-temperature irradiation in contrast to a unimodal distribution of void size for steady-temperature irradiation. The swelling in both conditions was similar at 0.8%. However the main part (~90%) of the swelling for 360/520 °C irradiation consisted of smaller voids. Similar microstructural changes due to variable irradiation could be seen in V–5Fe, V–4Cr–0.1Ti and V–0.1Fe.

Fig. 3 shows the dislocation microstructures in V–5Ti irradiated at 520 and 360/520 °C. Tangled dislocations and large precipitates (~200 nm) were observed homogeneously in both specimens. The dislocation density and the size and density of precipitates in both specimens were similar. This tendency is found in V–3Fe–4Ti–0.1Si in this study and V–4Cr–4Ti in previous work [5].

The effect of Ti additions on precipitation process can be seen in V–5Fe–*y*Ti ($y = 0.1$–1). The microstructural features in V–5Fe–0.1Ti looked like V–5Fe except for a high density of small precipitates (8×10^{21} ppt/m^3, ~10 nm) in V–5Fe–0.1Ti during steady 520 °C irradiation. With addition of titanium to V–5Fe–Ti alloys, the void density and size distribution were similar for both steady temperature irradiation and variable temperature irradiation. The effect of titanium additions on void swelling suppression controls the void formation process. Above 1 wt% titanium, void formation could not be found for both temperature conditions.

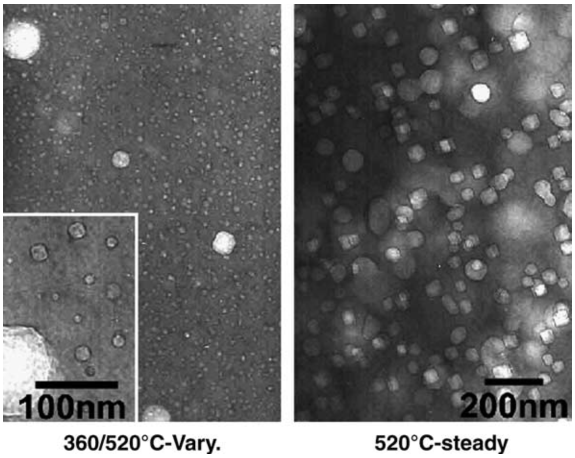

Fig. 2. TEM microstructures of V–0.3Fe for the 360/520 and 520 °C cases.

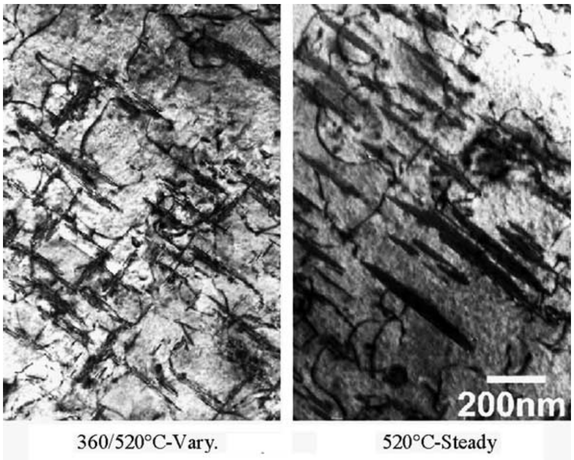

Fig. 3. TEM microstructures of V–5Ti for the 360/520 and 520 °C cases.

5. Discussion

The microstructures of unalloyed vanadium and V–4Cr–4Ti–0.1Si have been reported in previous work [5]. In the case of unalloyed vanadium, a bimodal distribution of void sizes for 360/520 °C irradiation as in Fig. 2 has not been reported. There are differences in microstructural evolution between unalloyed vanadium and V–0.3Fe alloy. Void nucleation in V–0.3Fe was more important than void growth for the 360 °C period of 520 °C variable irradiation. The different bimodal and unimodal distribution found in unalloyed vanadium and V–0.3Fe is expected to be determined by several factors like the peak shifts of both void nucleation and void growth. It is expected that peak shift also related to differences in vacancy diffusion rate or void sink strength relative to additions of undersized solute elements, such as Fe. Therefore, the temperature variation which overlaps different stages of defect cluster formation will influence the microstructural evolution.

Irradiation hardening results from formation of tiny voids in unalloyed vanadium and V–0.3Fe as mentioned above. On the other hand, irradiation hardening in V–5Fe and V–5Fe–0.1Ti at steady 520 °C irradiation was larger for variable 360/520 °C irradiation. In order to understand what factors control the irradiation hardening in V–5Fe–Ti alloy, an estimation of the yield stress increase from the microstructural observation was done using the well known dispersed barrier hardening equation

$$\Delta\sigma_{\text{tot}} = \left(\sum(\Delta\sigma_{\text{SR},i})^2\right)^{0.5} + \Delta\sigma_{\text{LR}}, \qquad (1)$$

where $\Delta\sigma_{\text{tot}}$ is the change in total yield stress, $\Delta\sigma_{\text{LR}}$ is the change due to long-range obstacles, such as dislocation networks, and is the change due to the ith short-range obstacle [9]. Each $\Delta\sigma$ is described by

$$\Delta\sigma = M\alpha\mu b(Nd)^{1/2}, \qquad (2)$$

where $\Delta\sigma$ is the increase in strength above the unirradiated value, μ is the shear modulus, b is the magnitude of Burgers vector of the dislocation. N and d are the density and size of the defect clusters, respectively. M is the Taylor factor, which is set to $M = 3$ [10,11]. α is a barrier factor that depends on defect species as obstacles against dislocation motion. From previous work [11], values of α for loops and voids in unalloyed vanadium were 0.1 and 0.35, respectively. In the case of $\alpha = 0.35$ for voids for the previous study, the mean size of voids was 20 nm due to low temperature irradiation at 340 °C. When the value of $\alpha = 0.35$ was used, no good fitting could be obtained the change of yield stress due to temperature variation at 520 °C in V–0.3Fe and V–5Fe. To solve the problem, a void size dependence on α was adopted. Fig. 4 shows the void size distribution for V–0.3Fe and V–5Fe for variable temperature with 520 °C. From the analysis to fit the measured and calculated data, the best value for $\alpha = 0.35$ was obtained for void sizes less than 30 nm in both alloys. The value of α for void sizes more than 30 nm was 0.6 and 3 for V–0.3Fe and V–5Fe, respectively. A comparison of yield stress increases for the measured data and the calculated data with best fit is listed in Table 2. From the fit analyses, a unrealistic large value of $\alpha = 3$ was obtained for large voids in V–5Fe, even though a value for α approaching one has been reported in the case of void hardening in a previous paper [9]. In order to explain the deficiency of yield stress increase from microstructural analysis, it is considered that there are a lot of invisible defects by TEM with high barrier factor strengthened against dislocation propagation in V–5Fe.

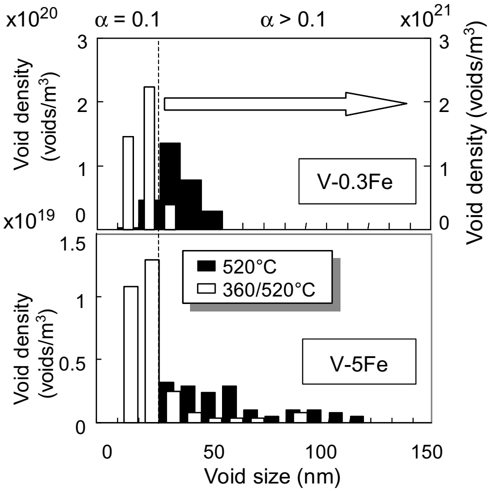

Fig. 4. Size distributions for void sizes in V–0.3Fe and V–5Fe for the 360/520 and 520 °C cases. Note the scale on the secondary axis for V–0.3Fe in the 360/520 °C case.

Table 2
Comparison of the increase of yield stress between the measured and calculated data of V–0.3Fe and V–5Fe at 520 °C and 360/520 °C in HFIR-13J irradiation

	Measured data		Fitted data	
	520 °C	360/520 °C	520 °C	360/520 °C
V–0.3Fe	77	154	107	147
V–5Fe	78	18	69	20

No differences in tensile behavior for temperature variation can be seen in alloys V–5Ti and V–4Cr–4Ti0.1Si [4]. A principle factor controlling the microstructural evolution in vanadium alloys containing more than 1 wt% of titanium is radiation-induced precipitation of Ti(OCN). Despite variation in temperature at 340 and 520 °C, no changes in the size distribution and densities of precipitates were observed. Therefore, the nucleation of precipitates is completed during the initial stage of irradiation at 520 °C and the steady growth of precipitates proceeded through the 520 °C irradiation. When the nucleation of precipitates occurred at 360 °C, the small precipitates formed at 360 °C were absorbed into the large precipitates by Ostwald ripening at 520 °C during irradiation. It has been found that the volume fraction of precipitates in V–5Ti irradiated at 500 °C with damage level of 0.1 dpa is one order larger than at 400 °C with a same damage level [8]. From the point of nucleation and growth of precipitates, the growth rate is much faster at 520 °C than at 360 °C in the case of the 360/520 °C temperature variation. In the other varying temperature experiments in JMTR, vanadium alloys containing titanium contained microstructural features that did not vary very much for the irradiation histories studied in the temperature regime from 300 to 600 °C [8]. It is suggested that Ti(OCN) precipitation due to Ti additions in vanadium is insensitive to temperature variation and vanadium alloys containing >1 wt% of titanium is stable to unexpected temperature variations during irradiation.

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