MICROSTRUCTURAL EVOLUTION OF V-4Cr-4Ti DURING ION IRRADIATION AT 200°C*

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OBJECTIVE

The objective of this study is to simulate microstructures produced by irradiation with fusion-energy neutrons in the reference vanadium alloy V-4Cr-4Ti at a relatively low temperature (200°C) utilizing ion irradiation of bulk specimens to understand the concomitant microstructural evolution of the alloy.

SUMMARY

The results of a transmission electron microscopy (TEM) investigation of the microstructural evolution of V-4Cr-4Ti (Heat #832665) that was irradiated with 4.5 MeV $^{58}Ni^{++}$ ions at 200°C are presented. Dose effects were investigated for fluences ranging from 0.5 to 5 dpa. When the irradiation dose was increased, the relative number density of black dots and dislocation loops was nearly constant and accompanied by an increase in the size of the defects. Cavity formation was not observed in any of the specimens, indicating high resistance of the alloy to void swelling at the low temperature of the experiments.

INTRODUCTION

Vanadium-base alloys are the most promising candidate low-activation materials for application in first-wall structures of magnetic fusion reactors. Recently, the V-4Cr-4Ti alloy was identified as a material that exhibited the optimal combination of mechanical and physical properties.[1,2] The virtual immunity to embrittlement and excellent resistance to swelling of several V-Cr-Ti, V-Ti, and V-Ti-Si alloys have been reported previously under both ion and neutron irradiation in the temperature range of 420 - 600°C.[3-6] However, data on the performance of the alloy in the low to moderate temperature range (<400°C) is limited. Confirmation of minimal swelling and demonstration of resistance to embrittlement by displacement damage and helium in this temperature range are particularly important issues.

The customary method for determining the irradiation performance of fusion candidate materials, i.e., tests utilizing fast fission neutrons (E > 0.1 MeV), is now much more difficult to conduct because of reactor shutdowns. Therefore, in this study, we chose an alternative approach to evaluate the irradiation-induced microstructural evolution of vanadium-base alloys at moderate-to-low temperatures. The approach is based on extrapolation of results from ion irradiation experiments. Work consisted of series of ion irradiations at 200°C to obtain doses of 0.5, 2, and 5 dpa in the maximum-damage region of bulk specimens. Microstructural changes in the production-scale (500 kg) heat of V-4Cr-4Ti alloy were generated by $^{58}Ni^{++}$ ion beams produced in the Tandem Ion Accelerator/High Voltage Electron Microscope Facility located at Argonne National Laboratory (ANL). Conventional transmission electron microscopy (TEM) was used to identify the type and number density of produced defects.

MATERIAL AND PROCEDURES

Fabrication of the V-4Cr-4Ti alloy (Heat ID 832665) is described in detail elsewhere.[7] The composition of the extruded plate that was used for the present specimens is given in Table 1. Procedures for preparing of the specimens are described in the previous report,[8] with one modification: discs were ground to a thickness of ≈125 μm before the polishing and annealing steps. The ion irradiations were performed at the Tandem Accelerator facility operated by the ANL Materials Science Division. Beams of 4.5 MeV $^{58}Ni^{++}$ ions were produced by the 2-MV NEC ion accelerator, while the vacuum in the ion chamber used for the irradiations was maintained at 1×10^{-6} Pa. Irradiation temperature was 200 ± 2°C. The procedure for final preparation of foils for TEM observations is described in Ref. 8. Microstructure observations were conducted with a

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Philips CM30 transmission electron microscope. Number density and dislocation loop size were measured from bright field images obtained with **g** = (200) and **g** = $(0\bar{1}1)$diffraction vectors near the [011] foil orientation. Dislocation loop size was measured from prints with a Zeiss particle size analyzer/counter.

Table 1. Composition of production heat of V-4Cr-4Ti alloy [a]

Heat ID	ANL ID	Cr	Ti	Cu	Si	O	N	C	S	P	Ca	Cl	B
832665	BL-71	3.8	3.9	<50	783	310	85	80	<10	<30	<10	<2	<5

[a] Cr and Ti in wt.%, impurities in wppm.

RESULTS AND DISCUSSION

After irradiation up to 5 dpa at 200°C, the microstructure consisted of "black dot" defects (BD) and dislocation loops. Line dislocations were not observed in any of the specimens at this temperature and in this dose range. Two types of dislocation Burgers vectors, $a_0\langle 100\rangle$ and $a_0/2\langle 111\rangle$, were found to coexist in all of the specimens. The combined number density of defects and dislocations was compared among specimens that had received different irradiation doses. These comparisons are presented in Fig. 1. The plots given in Fig. 1 are based on the relative number density of defects that resulted from counting visible defects in photomicrographs with g = $(0\bar{1}1)$. The diffraction conditions in foils with such orientation allow only for visibility of 1/2 of the total number of $a_0/2\langle 111\rangle$ type loops, and 2/3 of the total number of $a_0\langle 100\rangle$ type loops in the body centererd cubic vanadium alloys. However, because the evidence for formation of equal numbers of each type of loop was not obtained, these proportions cannot be easily applied to the numbers obtained from the measurements. Therefore, relative numbers obtained under consistent diffraction conditions are quoted.

Examples of typical microstructures of specimens irradiated to 0.5, 2, and 5 dpa at 200°C are shown in Fig. 1. At 0.5 dpa, the microstructure is dominated by BDs, with only a limited number of dislocation

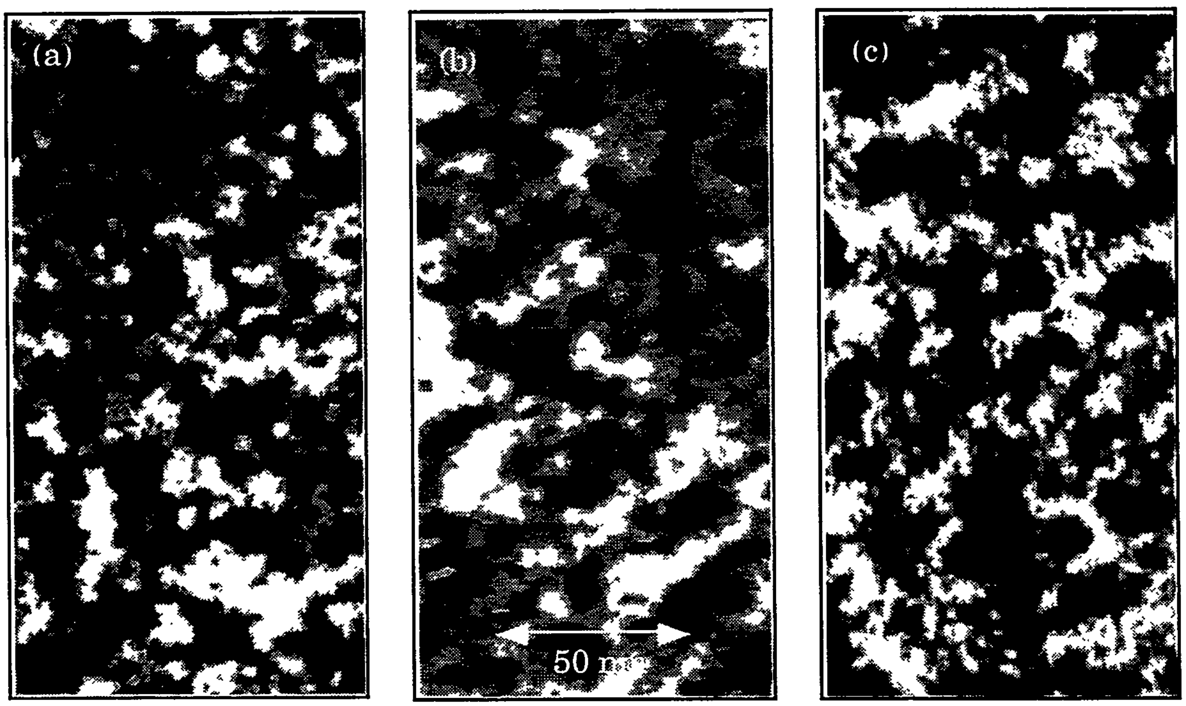

Figure 1. Microstructure of V-4Cr-4Ti alloy after $^{58}Ni^{++}$ ion irradiation at 200°C to: (a) 0.5, (b) 2, and (c) 5 dpa. Kinematical two-beam conditions g = (0-11), near [011] foil orientation.

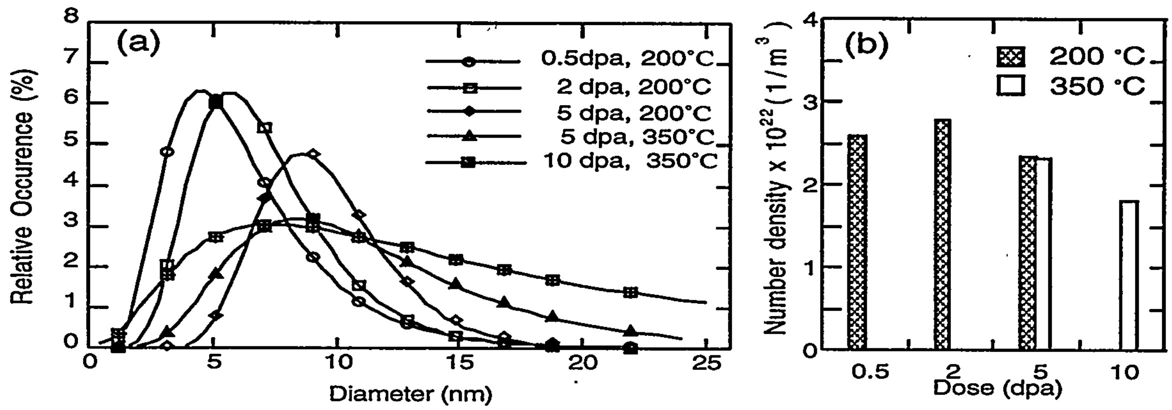

Figure 2. Evolution of microstructure of V-4Cr-4Ti under ion irradiation at 200°C and 350°C: (a) distribution of BDs and loop diameters; (b) relative number density of loops and BDs.

loops resolvable, their diameters reaching at most 20 nm. Similar features were observed in specimens irradiated to 2 dpa, but the number of resolvable loops increased. After 5 dpa, the loops became the prevailing feature of the microstructure, with their diameters reaching up to 50 nm. However, a significant number of BDs still remained after this damage dose. Figure 2a gives the observed distribution of BDs and loop diameter sizes. At all doses, the combined number density of BDs and loops remained approximately the same, as shown in Fig. 2b. The microstructure at 200°C was compared with one described in Ref. 8 that was obtained after 4.5 MeV $^{58}Ni^{++}$ ion irradiation to 5 and 10 dpa at 350°C. The number density of defect was approximately the same, whereas specimens irradiated to 10 dpa also showed formation of dislocation segments not observed at 200°C.

The data indicate early formation of a high density of defect clusters that act as point defect sinks during continuing irradiation. Growth of dislocation loops is sustained by production of freely migrating point defects from the remains of cascades. The other defect sinks present in the specimens include grain boundaries and interfaces between the matrix and Ti(CNO) thermal precipitates formed during fabrication of the alloy. The high density of sinks shortens the diffusion paths of point defects and efficiently removes them from solution. Dislocation segments in specimens irradiated to 10 dpa at 350°C could be explained by unfolding of dislocation loops when they reach a larger size.

Cavity formation was not observed in any of the specimens, including those irradiated to 5 dpa. In a previous investigation[8] with 4.5 MeV $^{58}Ni^{++}$ ion irradiation at 350°C to ~5 and ~10 dpa, cavity formation was also not observed. Likewise, a similar heat of V4Cr4Ti (BL47) was reported[6] to be resistant to cavity formation during fast neutron irradiation at ~420°C. Thus, the alloys of V-4Cr-4Ti composition appear to be resistant to void swelling, even at low temperatures, down to 200°C.

CONCLUSIONS

1. Consistent with the results from void swelling characterization of V-4Cr-4Ti (Heat 832655) irradiated at 350°C to ~10 dpa by $^{58}Ni^{++}$ ions and V-4Cr-4Ti (Heat BL-47) irradiated at ~420°C to ~34 dpa by fast neutrons (E > 0.1 MeV), no cavities were observed in V-4Cr-4Ti (Heat 832655) specimens irradiated by $^{58}Ni^{++}$ ions at 200°C to ~5 dpa. This indicates that the alloy class is resistant to void swelling at low temperatures, down to 200°C.

2. After irradiation with 4.5 MeV $^{58}Ni^{++}$ at 200°C: a) the predominant features of microstructure were small (<50 nm) dislocation loops and "black dot" defects; b) the size of dislocation loops increased with irradiation dose, whereas the combined number density of black dots and dislocation loops remained constant up to a fluence of 5 dpa. These findings indicate early formation of a large density

of defect clusters that act as point defect sinks and grow into dislocation loops during continuing irradiation.

3. The size and combined defect and loop number density after ion irradiation at 200°C were similar to those resulting from ion irradiation at 350°C.

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