MICROSTRUCTURAL EVOLUTION OF V-4Cr-4Ti DURING DUAL–ION IRRADIATION AT 350°C*

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OBJECTIVE

The objective of this study is to simulate simultaneous irradiation by fusion–energy neutrons and helium generation in the reference vanadium alloy V–4Cr–4Ti at relatively low temperatures (<400°C) by dual ion irradiation utilizing $^{58}Ni^{++}$ and $^{3}He^{+}$ ions generated in tandem ion accelerator/high voltage electron microscope facility in Argonne National Laboratory and to characterize the concomitant microstructural evaluation in the alloy.

SUMMARY

The preliminary results of TEM investigation of microstructural evolution of V-4Cr-4Ti (Heat #832665) alloy irradiated with 4.5 MeV $^{58}Ni^{++}$ ions at 350°C, with and without simultaneous $^{3}He^{+}$ injection, are presented. This work is the basis of an extensive study designed to evaluate ion irradiation experiments as a tool for simulating and understanding fusion neutron damage and helium generation in V-Cr-Ti alloys. The effects of ion–irradiation damage (at moderate temperatures of <400°C) on mechanical properties of these alloys will be also evaluated in this study. This initial report includes descriptions of specimen preparation techniques, procedures performed during ion irradiation, postirradiation analysis, and results of preliminary transmission electron microscopy (TEM) investigation. Specimens irradiated to ≈10 dpa by $^{58}Ni^{++}$ ions showed a high density of "black–dot" defects and dislocations. Cavity formation in the specimens irradiated simultaneously with $^{3}He^{+}$ ions to a rate of ≈5 appm/dpa He was not observed.

INTRODUCTION

Vanadium–base alloys are the most promising candidate materials for application in fusion reactor first wall structures. Recently, the V–4Cr–4Ti alloy was identified as having the optimal combination of mechanical and physical properties.[1] Previous ion and neutron irradiation experiments have demonstrated that several V–Cr–Ti, V–Ti, and V–Ti–Si alloys retain high ductility, toughness, and strength and undergo minimal irradiation-induced swelling during irradiation in the temperature range of 425 to 600°C.[2–9] However, there is lack of information on the irradiation performance of vanadium–base alloys in the moderate temperature range (200 to 425°C) relevant to, e.g., the projected operational conditions of the International Thermonuclear Experimental Reactor (ITER). Of particular importance are the confirmation of minimal swelling and the demonstration of good resistance to embrittlement by displacement damage and helium in this irradiation temperature range.

The customary method for determining the irradiation performance of fusion candidate materials is to conduct fission–reactor tests utilizing fast neutrons (E > 0. 1 MeV), e.g., in FFTF and EBR–II. Unfortunately, because these fast fission reactors are no longer available, alternate means must be sought. One option is simulation of neutron damage by irradiation with ions, and extrapolation of the results to predict property changes due to neutron irradiation effects. This study follows such an approach in evaluating the irradiation performance of vanadium-base alloys at moderate temperatures. Preliminary work consisted of irradiating specimens at 350°C to 10 dpa. Microstructural changes in the production–scale (500–kg) heat[10] of V–4Cr–4Ti alloy were generated by single ($^{58}Ni^{++}$) and dual ($^{58}Ni^{++}$ and $^{3}He^{+}$) ion beam irradiation in the tandem ion accelerator/high voltage electron microscope facility located at Argonne National Laboratory. Conventional TEM observations were performed to identify the type and number density of produced defects, precipitates, and voids.

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In a previous investigation of a laboratory–scale (30–kg) heat of V–4Cr–4Ti (ANL ID BL–47) irradiated in the Dynamic Helium Charging Experiment (DHCE), contrasting behaviors of irradiation–induced precipitation of Ti_5Si_3 and helium bubbles were observed upon irradiation at 500–600°C and 425°C.[9] That is, Ti_5Si_3 precipitation was significant and helium bubbles were negligible at the high irradiation temperatures, whereas Ti_5Si_3 precipitation was negligible and helium bubble density was relatively higher at the low irradiation temperature. In view of this, special attention was given to characterizing the behavior of Ti_5Si_3 precipitation and He bubble formation at <400°C in the dual–ion–irradiated specimens.

MATERIAL AND PROCEDURES

Fabrication of the V–4Cr–4Ti (Heat ID 832665) alloy is described in Ref. 10. Chemical composition of the extruded alloy plate is given in Table 1. Specimens for ion irradiation were prepared from ≈250–mm–thick cold–worked sheets obtained by cold rolling of 1.02 mm plate without intermediate annealing. The 3–mm–diameter TEM disks were punched out, polished to 0.05 mm surface finish, and annealed in 1×10^{-5} Pa vacuum in an ion–pumped ultrahigh vacuum–furnace for 1 h at 1050°C. The resultant average grain diameter was ≈30 mm. Disks were then electropolished to remove thermal etching and ensure the flat surface necessary for ion irradiation. The ion irradiations were performed at the Argonne Tandem Accelerator facility operated by the Materials Science Division. Beams of 4.5 MeV $^{58}Ni^{++}$ were produced by the 2–MV NEC tandem ion accelerator. The 0.35 MeV $^{3}He^{+}$ ion beams were obtained from the 0.65–MV NEC ion implanter. The ion chambers used for the irradiations allow simultaneous irradiation with both types of ions. Irradiation temperature was 350±2°C, and vacuum was maintained at 1×10^{-6} Pa in the chambers during irradiation.

Table 1. Chemical composition (impurities in wppm) of production heat of V–4Cr–4Ti

Heat ID	ANL ID	Heat Type	Cr	Ti	Cu	Si	O	N	C	S	P	Ca	Cl	Na	K	B
832665	BL–71	production 500 kg	3.8 wt.%	3.9 wt.%	<50	783	310	85	80	<10	<30	<10	<2	–	–	<5

After irradiation, TEM foils were prepared by removing a 800–nm thick section from the irradiated surface and back thinning the specimen to electron transparency. The depth of 800 nm was selected based on the basis of computer simulation (TRIM code[11]) of ion deposition depths and damage profiles. Thin foils prepared at this depth allow observation of the region with maximum He deposition rate and significant irradiation damage rate. In Fig. 1, examples of damage profiles calculated by the TRIM code are given. An optical photomicrograph of typical grain structure of the material is shown in Fig. 2.

RESULTS

TEM investigation of the specimens irradiated to ≈10 dpa with and without implantation of 50 appm He showed a rather simple microstructure. The prevailing features consisted of a high density of "black dot" defects and small dislocations present in the matrix. The observed dislocations were both small loops (<50 nm diameter) and small segments with Burgers vector a_0<100>. No irradiation–induced or –enhanced precipitation was observed. This seems to be consistent with a previous report in which Ti_5Si_3 precipitates, usually present in V–4Cr–4Ti after neutron irradiation at 500–600°C, were absent after neutron irradiation at 425°C.[9] The specimens with injected helium did not show cavity formation in the matrix or near grain boundaries. Figure 3 is a TEM micrograph of nonirradiated V–4Cr–4Ti alloy. Figure 4 shows the microstructure after ≈10 dpa irradiation with 4.5 MeV Ni ions, while Fig. 5 shows the microstructure after ≈10 dpa irradiation and 5 appm/dpa He injection.

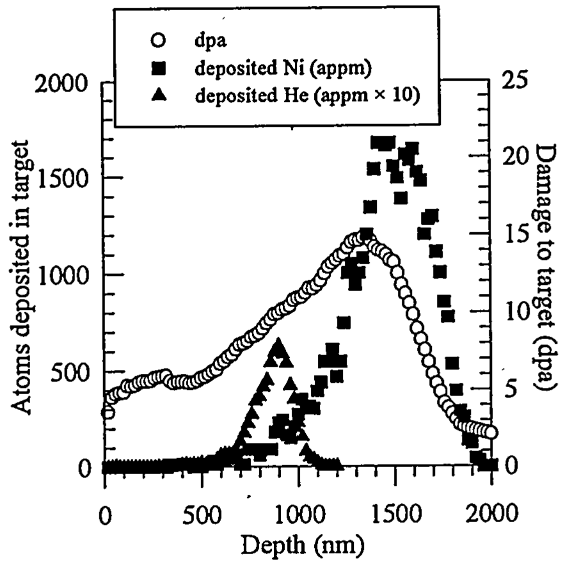

Fig. 1. Damage profile and atom deposition profiles calculated by TRIM Version 92.12 for 4.5 MeV Ni^{++} ions and 0.35 MeV He^{+} ions irradiating V–4Cr–4Ti alloy

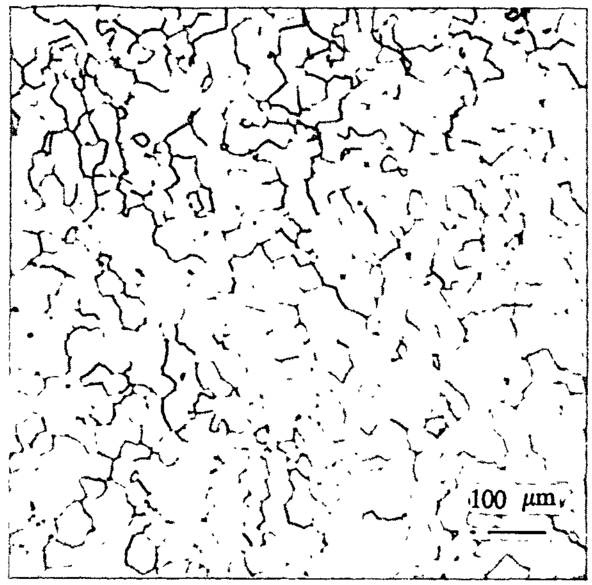

Fig. 2. Optical photomicrograph of grain structure of V–4Cr–4Ti after annealing at 1050°C for 1 h

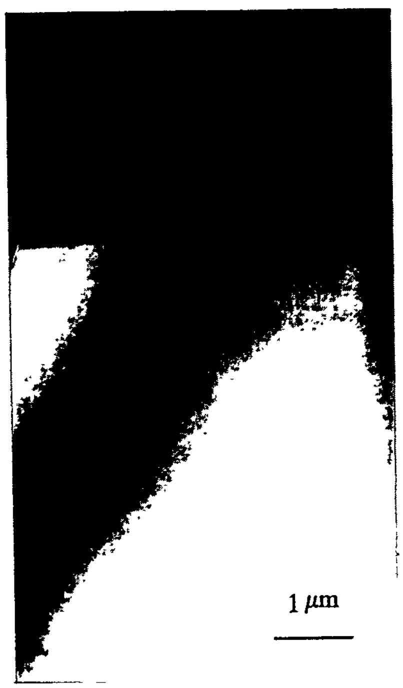

Fig. 3. TEM micrograph of unirradiated V–4Cr–4Ti

Fig. 4. TEM micrograph of V–4Cr–4Ti irradiated at 350°C with 4.5 MeV Ni^{++} ions to ≈10 dpa

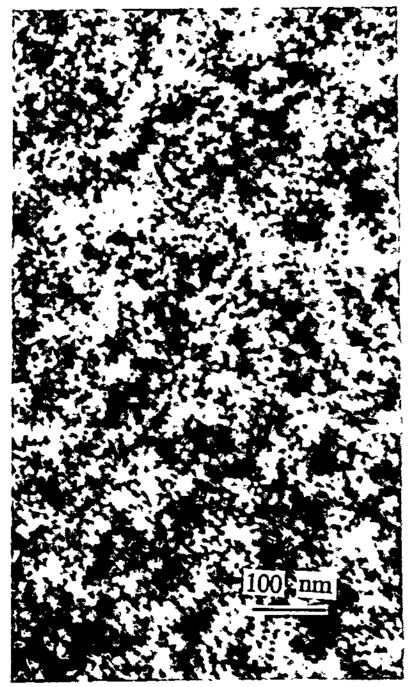

Fig. 5. TEM micrograph of V–4Cr–4Ti irradiated at 350°C with 4.5 MeV Ni^{++} ions to ≈10 dpa with ≈50 appm He^{+} implanted

FUTURE WORK

Future work on this project will include a variety of Ni ion irradiations with and without He injection. Two series of experiments are planned: (a) exploring alloy performance under various irradiation doses at 200°C and (b) investigating temperature effects within the range of 25 to 425°C at 5 dpa. Results of these investigations will be compared to results from fast neutron irradiation of the same production–scale heat of V–4Cr–4Ti in the EBR–II X-530 experiment (≈5 dpa at 375°C).

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