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Effect of Ti addition on microstructural evolution of V–Cr–Ti alloys to balance irradiation hardening with swelling suppression

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ABSTRACT

Transmission electron microscopy studies and nano-indenter tests were used to examine model alloys of V–4Cr–*x*Ti (where *x* is less than 1 wt.%) that were irradiated in a nuclear reactor with neutrons and irradiated in an ion accelerator with He ions. Void formation resulted in V–4Cr–0.1Ti and 0.3Ti from 400 °C to 600 °C with a damage level of 21 dpa in neutron irradiation. No void formation was visible in V–4Cr–1Ti. A threshold concentration of void formation and Ti(CON) precipitate formation occurred for x = 0.3 wt.% to 1 wt.% in the V–4Cr–*x*Ti alloy. Void formation and Ti(CON) precipitation competed in neutron irradiation as caused by vacancy migration depending on the concentration of the added Ti in the V–4Cr–*x*Ti alloy. A study of the He-ion irradiation indicated the same competitive process of void formation and Ti(CON) precipitate formation depending on the added Ti concentration.

Introduction

The V–4Cr–4Ti alloy is believed to be one of the best vanadium alloy candidates for fusion applications. In the design of vanadium alloys as candidates for fusion-reactor applications, Ti addition in vanadium alloys has a significant effect on the suppression of swelling [1,2]. However, the susceptibility of vanadium-based alloys to low-temperature embrittlement during neutron irradiation may limit their application at low temperature (< 400 °C) [3]. This behavior is attributed to the presence of fine Ti(CON) precipitates, and the reduction of these precipitates is expected to improve the radiation resistance of the alloys at low temperature [4]. We used model alloys of V–4Cr–*x*Ti (where x < 1.0 wt.%) to optimize the amount of Ti added to balance low irradiation hardening because of Ti(CON) precipitation and low void swelling that is produced by neutron irradiation. Neutron-irradiation data and an experimental study by He-ion irradiation were used to optimize the effect of Ti addition on the microstructural evolution and dimensional stability during irradiation.

Experimental procedures for neutron irradiation

Transmission electron microscopy (TEM) specimens of V–4Cr–*x*Ti (x = 0.1 wt.%, 0.3 wt.%, 1.0 wt.%) were used for neutron irradiation. The impurity levels were 500–600 wppm for O and < 20 wppm for N and C [5]. The annealing conditions were 1000 °C for 2 h. Irradiations were performed in a JMTR, Joyo, HFIR from 400 °C to 598 °C with a damage level of 0.18–21 dpa [6–8]. Table 1 shows the neutron-irradiation conditions. TEM observations were carried out with an accelerating voltage of 200 keV at room temperature at the Oarai Center of the IMR/Tohoku University, and KUR.

Experimental procedures for He-ion irradiation

Plate specimens of V–4Cr–*x*Ti (x = 0.1 wt.%, 0.3 wt.%, 1.0 wt.%) were used for ion irradiation. Specimen impurity levels and annealing conditions were the same as the TEM specimens in the neutron-irradiation experiments. A He-irradiation experiment was carried out at the Wakasa-Wan Energy Research Center with a tandem accelerator. Specimens were irradiated with He ions at 2 MeV at 500 °C. Crystal grains with the same azimuth were selected by the electron backscatter diffraction measurement for unirradiated and irradiated samples and nano-indenter tests were carried out on the selected grains. The nanoindentation depth was fixed at 500 nm because the depth of the damage profile by ion irradiation was ~2 μm. TEM samples were studied by focused-ion-beam microscopy and Ga ion damage on the specimen surface was removed by Ar ion milling. TEM observations were carried out at the University of Fukui at 200 kV and room temperature.

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Table 1
A list of irradiation condition in a series of neutron irradiation.

Reactor	Capsule no.	Temperature	Damage level (dpa)	V–4Cr–0.1Ti	V–4Cr–0.3Ti	V–4Cr–1Ti
JMTR	98M-5U	500	0.18	○		○
JOYO	JNC40	400[a]	1.5	○		○
JOYO	JNC41	500[a]	1.5			○
JOYO	JNC42	600[a]	1.5			○
JOYO	JNC55	410	19.5	○		○
JOYO	JNC56	508	21	○		○
HFIR	13J	520	4	○		○
HFIR	17J	425	3.7	○	○	○
HFIR	17J	598	3.7	○	○	○

[a] Nominal temperature; no temperature measurement.

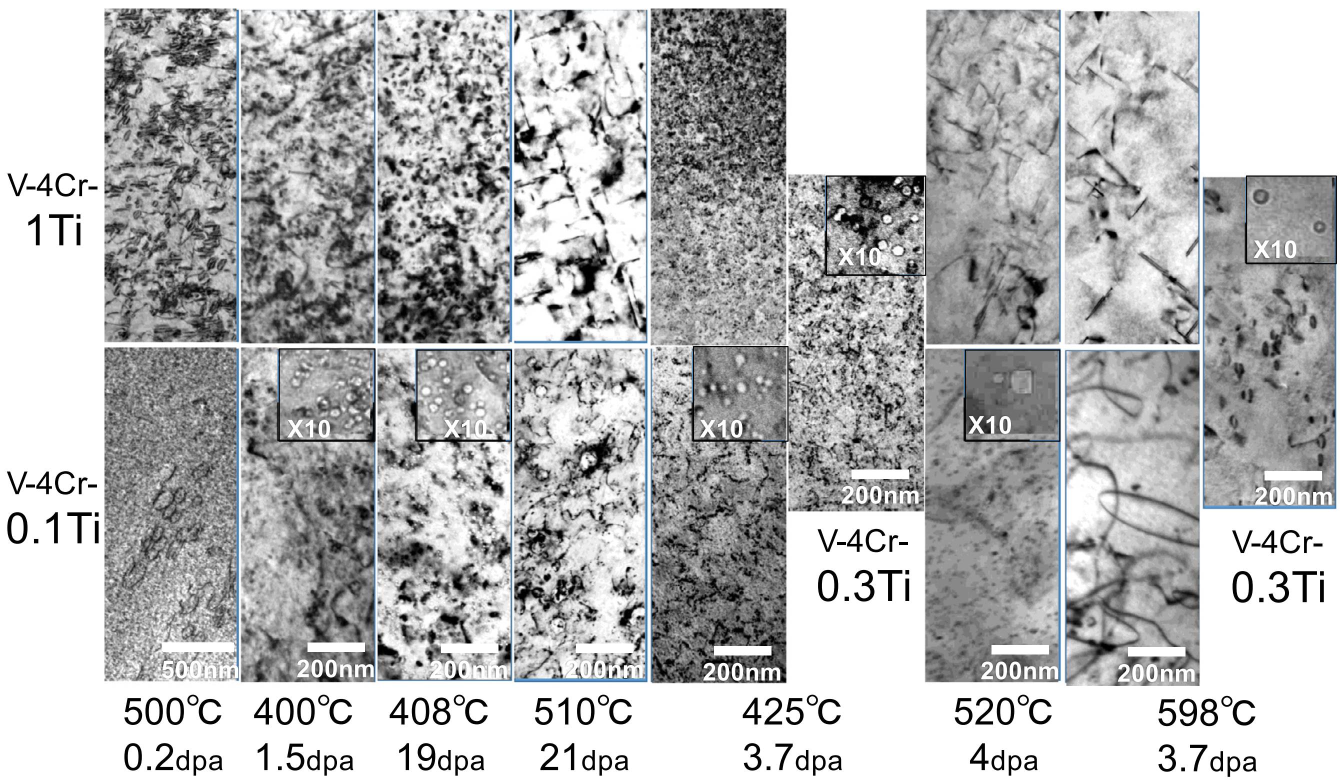

Fig. 1. Series of microstructures of V–4Cr–*x*Ti alloys (*x* = 0.1 wt.%, 0.3 wt.% and 1.0 wt.%) irradiated with neutrons in JMTR, Joyo and HFIR reactors.

Results of neutron irradiation

Fig. 1 shows a series of microstructures of V–4Cr–*x*Ti alloys that were irradiated with neutrons in the JMTR [6], Joyo [7] and HFIR [5,8] reactors. Using TEM observations of the neutron-irradiated V–4Cr–*x*Ti, data on the microstructural evolution were obtained and are shown in Table 2. From the experimental results, the effect of Ti addition on the swelling was remarkable. For the V–4Cr–0.1Ti, with a low neutron-irradiation dose below 0.2 dpa, rafts of dislocation loops and voids were formed at the center of the dislocation loops, but the amount of void swelling was less than 0.1%. With increasing damage levels, the void density and void size increased and the swelling reached approximately 5% at 500 °C and 21 dpa. At 600 °C, a coarsened dislocation structure was dominant in the matrix and minimal void formation was visible. This tendency has been reported previously for the temperature dependence of void swelling in V–5Cr [9]. For V–4Cr–1Ti, no void formation resulted in the experimental conditions of this work from 400 °C to 600 °C with damage levels from 0.18 to 21 dpa. Void formation was suppressed completely in V–4Cr–*x*Ti in ternary alloys with 1% Ti addition. The formation of Ti(CON) precipitate in the V–4Cr–1Ti alloy occurred for all conditions in this study. Fig. 2 summarizes the schematic features of microstructures in V–4Cr–0.1Ti and V–4Cr–1Ti alloys produced by neutron irradiation as functions of irradiation temperature and damage level. Because no Ti(CON) formation resulted in the V–4Cr–0.1Ti alloys under all conditions, void formation and Ti (CON) formation reacted competitively in the V–4Cr–*x*Ti, depending on the amount of Ti added.

Results of He-ion irradiation

He-ion irradiation was conducted to investigate the effect of Ti addition on void formation in V–4Cr–*x*Ti. Fig. 3 shows a series of cross-sectional TEM microstructures in V–4Cr–*x*Ti alloys (*x* = 0.1 wt.%, 0.3 wt.% and 1.0 wt.%) irradiated with 2 MeV He ions at 500 °C. The damage profile of the dislocations and cavity structures in Fig. 3 shows that the microstructures of the V–4Cr–0.1Ti and V–4Cr–0.3Ti alloys were nearly identical. Tangled dislocation networks formed at a depth of 2 μm and large dislocation loops were distributed from 1 to 2 μm. Highly dense cavities formed at a depth of ~2 μm within a 0.2-μm band. In the V–4Cr–1Ti alloy, a high density of defect clusters was distributed within the 0.2-μm-band depth and the damage profile of the ion range was the same as that of the V–4Cr–0.1Ti and V–4Cr–0.3Ti