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Defect microstructure and deformation behavior of V–Ti–Cr–Si–Al–Y alloy irradiated in ATR

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Abstract

Low-temperature irradiation performance of vanadium alloys, especially after neutron irradiation below 400°C, is a major concern. Defect microstructures and deformation behavior of V–Ti–Cr type alloys containing small amounts of Si, Al and Y after neutron irradiation below 400°C were studied. Neutron irradiation was conducted in the advanced test reactor (ATR) to fluences of 0.7–4.7 displacements per atom (dpa) at temperatures from 141°C to 293°C. A large amount of hardening was observed in the specimens irradiated at 290°C or below, which corresponded to a high density of small dislocation loops. The density and the size of loops depended on the irradiation temperature and the pre-irradiation annealing temperature. The loops in the specimen annealed at 900°C had a lower density and a larger size than those annealed at 1000°C and 1100°C. The specimen annealed at 900°C showed very small hardening and retained good work-hardening capability after irradiation at 290°C.

1. Introduction

Vanadium alloys are considered as promising candidate structural materials for fusion reactors because of their several preferable characteristics, such as low induced radioactivity, high heat-load capability and good compatibility with lithium coolant [1]. Alloys containing 4–5% Cr and 4–5% Ti exhibit good resistance to radiation damage at temperatures higher than 425°C and have been considered as reference materials [2]. Alloys of V–Ti–Cr containing small amounts of Si, Al and Y for oxidation resistance have been developed, and these alloys show good mechanical properties and low swelling behavior after irradiation at temperatures higher than 400°C [3–11]. However, from several irradiation experiments at 80–430°C, it has been reported that V–(4–5)Cr–(4–5)Ti alloys exhibited significant radiation hardening and low or negligible uniform elongation during tensile testing as a result of irradiation-induced loss of work-hardening capability [12–17]. In the alloys that showed the poor work-hardening capability, dislocation channeling occurred extensively [18–20]. This paper describes the microstructural evolution in correlation with deformation behavior of V–Ti–Cr–Si type alloys after neutron irradiation below 400°C.

2. Experimental

V–3.8Ti–5.9Cr–Si–Al–Y was used in this study. Chemical analysis of the alloy is shown in Table 1. Disk specimens 3 mm in diameter and miniature tensile specimens with a gauge section 5 mm in length and 1.2 mm in width were punched out from 0.25 mm thick sheets. The specimens were annealed at 700°C for 1.8 ks as a stress-relief heat treatment and at 900°C, 1000°C, 1100°C for 3.6 ks for recrystallization condition. The specimens annealed at 900°C were partially recrystallized. The grain sizes after annealing at 1000°C and 1100°C were about 13 and 15 μm, respectively.

The specimens were neutron-irradiated in Li-filled capsules inserted in the A1-hole of the advanced test

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Table 1
Chemical composition of vanadium alloy (in wt%)

	V	Ti	Cr	Si	Al	Y	C	N	O
V–3.8Ti–5.9Cr–Si–Al–Y	Bal.	3.77	5.85	0.017	1.07	0.97	0.0056	0.0140	0.024

Table 2
Summary of irradiation conditions and annealing conditions before irradiation

Irradiation	Heat treatment	Irradiation temperature (°C)	Fluence (dpa)
	1100°C/3.6 ks	290	4.7
	1000°C/3.6 ks 700°C/1.8 ks	293	4.6
ATR-A1	900°C/3.6 ks	286	4.5
	1100°C/3.6 ks 700°C/1.8 ks	141	0.7
	1000°C/3.6 ks	207	1.5

reactor (ATR). The irradiation conditions and pre-irradiation annealing conditions are summarized in Table 2 [21]. In this study, data obtained on a V–4.8Ti–4.0Cr–Si–Al–Y alloy was also examined for reference. The specimens of this alloy were irradiated in the material open test assembly of the fast flux test facility (FFTF/MOTA-2A) and the experimental breeder reactor No. 2 (EBR-II) at temperatures around 400°C. The details of the irradiation procedure have been described elsewhere [22].

Tensile tests were carried out using an Instron-type machine in a vacuum of 1×10^{-3} Pa at a strain rate of 6.7×10^{-4} s^{-1}. Test temperatures were ambient temperature and corresponding irradiation temperatures. After tensile tests, specimens were examined by scanning electron microscopy (SEM, JEOL JEM-5300) in order to characterize the fracture mode. The gauge sections of the deformed tensile specimens and the disk specimens were used for microstructural observation by transmission electron microscopy (TEM, JEOL JEM-2010) operating at 200 kV. Thin foils were prepared by twin-jet electropolishing in a solution of 80% CH_3OH and 20% H_2SO_4 below −10°C.

3. Results

3.1. Dependence of tensile behavior and microstructures on irradiation temperature

Typical stress–strain curves of the V–Ti–Cr–Si–Al–Y type alloys after neutron irradiation are shown in Fig. 1. These specimens were annealed at 1000°C or 1100°C before irradiation. At an irradiation temperature around 400°C, the V–4.8Ti–4.0Cr–Si–Al–Y alloy had apprecia-

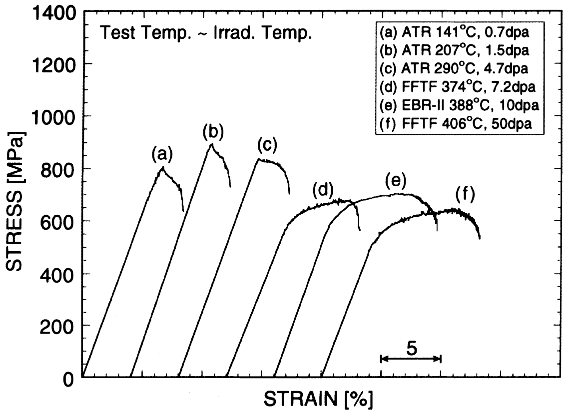

Fig. 1. Typical stress–strain curves of the V–Ti–Cr–Si–Al–Y type alloys after neutron irradiation below 400°C. (a)–(c): V–3.8Ti–5.9Cr–Si–Al–Y; (d)–(f): V–4.8Ti–4.0Cr–Si–Al–Y.

ble work-hardening capability, and the uniform elongation was about 5%. At temperatures below 300°C, the V–3.8Ti–5.9Cr–Si–Al–Y alloy exhibited significant radiation hardening and loss of work-hardening capability.

Fig. 2 shows microstructures in the deformed tensile specimens of the neutron-irradiated V–Ti–Cr–Si–Al–Y type alloys after tensile tests at the irradiation temperature. Extremely dense and small dislocation loops were observed in the specimens after neutron irradiation below 300°C. The average size of these loops was about 8 nm. The average sizes of loops observed in the specimens irradiated at around 400°C were about 20 nm. Cavities and precipitates were not observed in any specimen. Evidence of dislocation channeling was not observed in TEM of the necked region of the deformed tensile specimens.

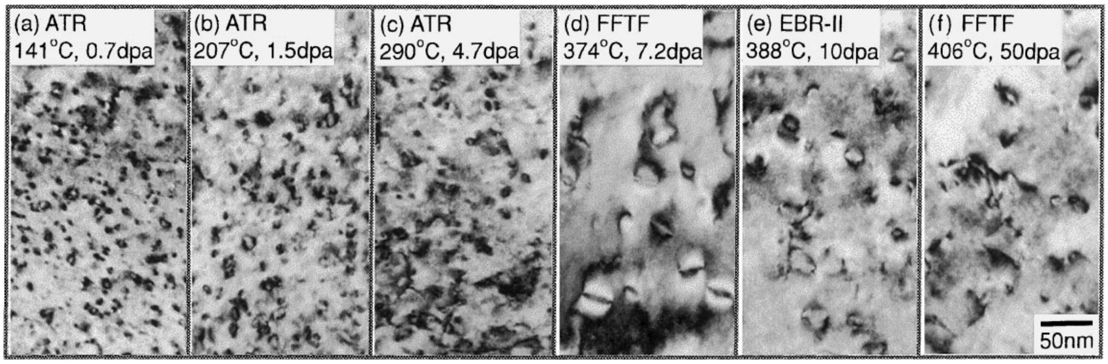

Fig. 2. Microstructures in the deformed tensile specimens of the irradiated V–Ti–Cr–Si–Al–Y type alloys. (a)–(c): V–3.8Ti–5.9Cr–Si–Al–Y; (d)–(f): V–4.8Ti–4.0Cr–Si–Al–Y.

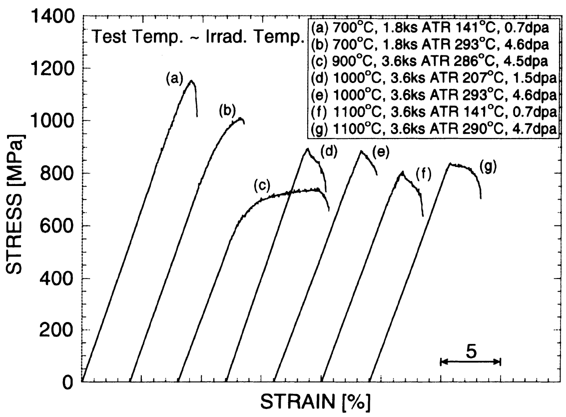

Fig. 3. Dependence of stress–strain curves on pre-irradiation annealing for the V–3.8Ti–5.9Cr–Si–Al–Y alloy after neutron irradiation in the ATR.

3.2. Effects of pre-irradiation annealing on tensile behavior and microstructures

Fig. 3 shows stress–strain curves of the irradiated V–3.8Ti–5.9Cr–Si–Al–Y alloy annealed at four different conditions before neutron irradiation. The specimens annealed at 1000°C and 1100°C show complete loss of work-hardening capability, and their uniform elongations were less than 1%. The specimens annealed at 900°C still had some work-hardening capability. The uniform elongation of the specimen annealed at 900°C remained at about 4%.

Fig. 4 shows microstructures in the disk specimens of the V–3.8Ti–5.9Cr–Si–Al–Y alloy after neutron irradiation in the ATR at 286°C to 4.5 dpa. The density and the size of the loops were almost constant in the specimen annealed at 1000°C and 1100°C. The loops in the specimen annealed at 900°C had a lower density and a larger size than those annealed at 1000°C and 1100°C. In

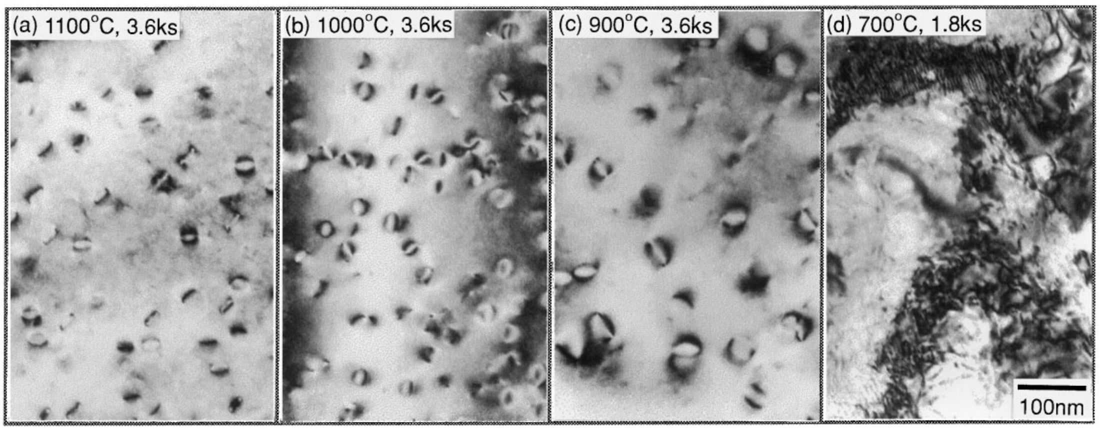

Fig. 4. Defect microstructures in the disk specimens of the V–3.8Ti–5.9Cr–Si–Al–Y alloy after irradiation at 286°C to 4.5 dpa. (a)–(d): Annealing conditions before irradiation.

the specimen annealed at 700°C for 1.8 ks, the dense network dislocation structure introduced in the fabrication process was retained.

4. Discussion

4.1. Correlation between radiation hardening and defect microstructures

It is known that small loops act as barriers to dislocation motion and thereby harden the material. The dislocation barrier strength, α, can be related to other parameters by using the following equation [23]:

$$\Delta\sigma_y = 3\alpha\mu b\sqrt{Nd},$$

where μ is the shear modulus and b is the Burgers vector for vanadium with values of 4.67×10^4 MPa and 0.26 nm, respectively. N is the number density and d is the mean diameter of dislocation loops. Using the measured values of N and d for the deformed tensile specimens by microstructural observations, the calculated values of the dislocation barrier strength are summarized in Table 3.

Fig. 5 shows the dependence of the dislocation barrier strength, α, on irradiation temperature. The values of α varied from 0.34 to 0.56 depending on the irradiation temperature, and show a maximum value at 290°C. At an irradiation temperature around 290°C, a high density of small dislocation loops were formed, and this temperature is high enough for the interstitial impurity atoms to migrate to the loops [24]. So, it is believed that the loops were considerably strengthened and the value of α became large at this temperature. The change in the dislocation barrier strength calculated in this study shows the same tendency as that observed in the V–4Cr–4Ti alloy after neutron irradiation in the HFBR [25].

The uniform elongations of the V–4.8Ti–4.0Cr–Si–Al–Y alloy were larger than those of the V–(4–5)Cr–(4–5)Ti alloy after neutron irradiation at around 400°C [12,13]. However, the V–3.8Ti–5.9Cr–Si–Al–Y alloy showed negligible uniform elongation after irradiation at 290°C or below, except for the specimen annealed at 900°C before irradiation, similar to the V–(4–5)Cr–(4–5)Ti alloys [14–17]. The V–3.8Ti–5.9Cr–Si–Al–Y alloy (C = 56, N = 140, O = 240 wppm) contained larger amounts of interstitial impurity atoms than the V–4.8Ti–4.0Cr–Si–Al–Y alloy (C = 126, N = 54, O = 140 wppm). These interstitial atoms should affect microstructural evolution [26]. When the concentration of interstitial impurities is reduced, the amount of interstitial impurity atoms that can freely migrate to and decorate the loops becomes smaller. In addition, the density of the dislocation loops may be lower, since these interstitial impurity atoms are considered to act as nucleation sites of the defect clusters. Then, the values of α may be smaller than those calculated in this study, and significant radiation hardening at temperatures below 290°C may be suppressed.

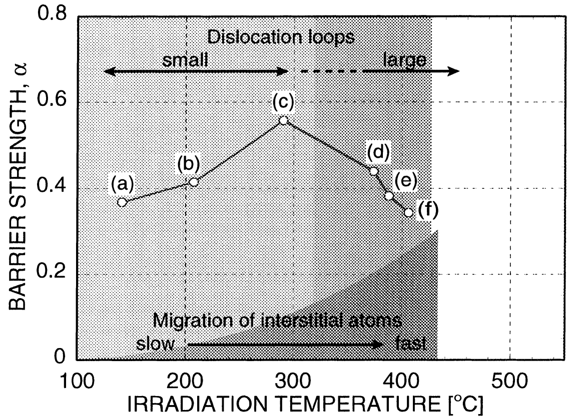

Fig. 5. Dependence of the dislocation barrier strength on irradiation temperature. (a)–(c): V–3.8Ti–5.9Cr–Si–Al–Y; (d)–(f): V–4.8Ti–4.0Cr–Si–Al–Y.

4.2. Effects of pre-irradiation annealing on tensile behavior and microstructure evolution

Microstructures in the specimen annealed at 900°C for 3.6 ks before irradiation showed a partially recrystallized condition. In the specimen annealed at 700°C for 1.8 ks, the high density of network dislocations

Table 3
Radiation hardening and microstructure of the V–Ti–Cr–Si–Al–Y type alloys after neutron irradiation[a]

Irradiation conditions	$\Delta\sigma_y$ (MPa)	N (m^{-3})	d (nm)	α
(a) ATR 141°C, 0.7 dpa	402	1.2×10^{23}	7.0	0.37
(b) ATR 207°C, 1.5 dpa	480	1.1×10^{23}	8.6	0.42
(c) ATR 290°C, 4.7 dpa	505	6.8×10^{22}	8.5	0.56
(d) FFTF 374°C, 7.2 dpa	242	1.1×10^{22}	18.9	0.44
(e) EBR-II 388°C, 10 dpa	248	1.8×10^{22}	17.0	0.38
(f) FFTF 406°C, 50 dpa	168	8.2×10^{21}	20.5	0.34

[a] (a)–(c): V–3.8Ti–5.9Cr–Si–Al–Y; (d)–(f): V–4.8Ti–4.0Cr–Si–Al–Y.

introduced in the fabrication process was retained as shown in Fig. 4(d). It is believed that the grain boundaries and network dislocations could act as sinks for point defects introduced under irradiation. This might lead to a lower density of the dislocation loops than those specimens annealed at 1000°C and 1100°C, which resulted in less radiation hardening. Hence, appropriate heat treatment before irradiation is considered to be effective in retaining the work-hardening capability of the V–Ti–Cr–Si–Al–Y alloys after irradiation at 300°C or below.

In several studies of the tensile behavior of the V–4Cr–4Ti alloy, it has been reported that dislocation channeling occurred extensively in the alloy after neutron or ion irradiation at 390°C or below [18–20]. In this study, there was no evidence of dislocation channeling in all specimens irradiated below 400°C. On the other hand, the microstructure was different between the disk specimens and the deformed tensile specimens irradiated in the ATR at similar conditions; that is, the size of dislocation loops in the disk specimens was about three times larger than that in the deformed tensile specimens. This difference might have occurred, because these specimens were irradiated in different subcapsules. Otherwise, the size of the loops might be reduced during the tensile test by the motion of glissile dislocations, and this might lead to a loss of work-hardening capability.

5. Summary

A V–Ti–Cr type alloy containing small amounts of Si, Al and Y retained good work-hardening capability after neutron irradiation at 290°C when the alloy was annealed at 900°C before irradiation. Therefore, appropriate heat treatment before irradiation appears to be effective in retaining work-hardening capability.

Extremely dense and small dislocation loops contributed to the large hardening in the specimens irradiated at 290°C or below. The loops in the specimen annealed at 900°C had a lower density and a larger size than those annealed at 1000°C and 1100°C. This could be attributed to its smaller grain size.

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