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Improvement in post-irradiation ductility of neutron irradiated V–Ti–Cr–Si–Al–Y alloy and the role of interstitial impurities

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Abstract

An attempt to maintain the uniform elongation (UE) of vanadium alloys after irradiation is described. Neutron irradiation was carried out at Advanced Test Reactor (ATR) and at Experimental Breeder Reactor II (EBR-II) to the fluence of 11 dpa. The UE was maintained about 6% after irradiation at 388°C. The UE became negligible after irradiation below 300°C for the alloy annealed at 1000°C and 1100°C. The specimens annealed at 900°C and 700°C still showed relatively large UE after such low-temperature irradiation. Comparing with the results in literature, it is proposed that oxygen concentration should be kept below 200 ppm to maintain the UE after irradiation below 400°C. The control of interstitial impurities, especially oxygen in solution, was important to maintain the UE of the alloy.

1. Introduction

Some vanadium alloys show loss of uniform elongation (UE) after neutron irradiation at 300°C or lower [1]. Although vanadium alloys as structural materials for first-wall and blanket of fusion application were considered to be used upto 750°C [2], the components might be exposed to low temperature and low neutron flux irradiation during transient operation such as startup and shutdown period of the reactor. Therefore, irradiation behavior of the alloy at low temperatures between 200° and 400°C is important. Modified V–Ti–Cr alloy series containing Si, Al and Y have been developed for proof-oxidation alloy in Tohoku University [3,4]. It is possible to improve the post-irradiation ductility by suitable thermo-mechanical treatment before irradiation and purification of interstitial impurity and so on. In this paper, an attempt to maintain the UE of the vanadium alloy after irradiation is described.

2. Experimental

2.1. Material preparation and neutron irradiation

Materials examined in this study were V–4.8Ti–4.0Cr–Si–Al–Y and V–3.8Ti–5.9Cr–Si–Al–Y in weight percentage. Chemical analysis of the alloys is shown in Table 1. Detailed procedures for material preparation were described in Ref. [4]. Miniature-size tensile specimens and disks for transmission electron microscopy (TEM) were punched out from 0.25 mm-thick sheets. The tensile specimen had a gauge section of 5 mm long and 1.2 mm wide. Heat treatments were carried out at 700°C for 1.8 ks or at 900°C, 1000°C and 1100°C for 3.6 ks in a vacuum less than 5×10^{-3} Pa. Microstructures showed that the specimen annealed at 700°C was in the stress-relieved condition and that annealed at 900°C was partially in the recrystallized condition. The specimens annealed at 1000°C and 1100°C were fully recrystallized with a grain size of about 13 and 15–20 μm, respectively. Neutron irradiation was carried out at the Advanced Test Reactor (ATR) and at the Experimental Breeder Reactor II (EBR-II). Irradiation conditions are summarized in Table 2 [5,6].

2.2. Post-irradiation experiments

Tensile tests were carried out at ambient temperature and irradiation temperatures with an initial strain rate of

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Table 1
Chemical composition of vanadium alloys (wt%)

	V	Ti	Cr	Si	Al	Y	C	O	N
V–4.8Ti–4.0Cr–Si–Al–Y	Bal.	4.79	4.01	0.85	0.95	0.77	0.0126	0.0140	0.0054
V–3.8Ti–5.9Cr–Si–Al–Y	Bal.	3.77	5.85	0.017	1.07	0.97	0.0056	0.0240	0.0140

Table 2
Summary of irradiation conditions[a]

	Position	Irradiation temp (°C)	Fluence (dpa)	Materials and specimen type
FFTF/MOTA-2A	BC-B-5	374	7.2	A: SSJ
	2D-2	406	50	A: SSJ
EBR-II	B-392-E07	388	10	A: SSJ
ATR	AS10	293	4.6	B: (b), (d) SSJ
	AS11	290	4.7	B: (a) SSJ
	AS12	286	4.5	B: (c) SSJ
				B: TEM
	AS1	141	0.7	B: (a), (d) SSJ
	AS17	207	1.5	B: (b) SSJ
	AS8	250	3.9	B: TEM

[a] A – V–4.8Ti–4.0Cr–0.85Si–0.95Al–0.77Y; B – V–3.8Ti–5.9Cr–0.02Si–1.1Al–1.0Y; SSJ – tensile specimen ($16\times4\times0.25$ mm^3); TEM – disk specimen ($\varnothing 3\times0.25$ mm); (a) – 1100°C, 3.6 ks; (b) – 1000°C, 3.6 ks; (c) – 900°C, 3.6 ks; (d) – 700°C, 1.8 ks.

6.7×10^{-4} s^{-1}. Thin foils were obtained by electro-polishing in a solution of 4 parts of methanol and 1 part of sulfuric acid by volume below −10°C. TEM specimens from a gauge section of tensile specimen were also prepared using a 'sandwich method' [7]. TEM observation was carried out using JEM 2010-S, JEOL operated at 200 kV in the Laboratory of Alpha-Ray Emitters of the Institute of Materials Research.

3. Results

3.1. Tensile property after neutron irradiation in ATR

Typical stress–strain curves of the V–3.8Ti–5.9Cr–Si–Al–Y alloy irradiated in ATR are shown in Fig. 1. Work-hardening capability of the specimens annealed at 1000°C and 1100°C disappears after irradiation at 293°C or lower. Large hardening was observed and the UE becomes almost zero for the all of the specimens except for the one annealed at 900°C. The specimen annealed at 900°C retains work-hardening capability and the UE about 4%. The 0.2% proof stress of the specimen annealed at 700°C is the highest. The proof stress decreases with the annealing temperature increase except for the 900°C. All of them were the ductile fracture mode with dimple pattern. Small precipitate-like particles were observed in the bottom of the dimples. The increase in the proof stress depends on annealing condition before irradiation and on irradiation conditions as shown in Fig. 2. The dependence of the increase of the stress on annealing condition is relatively small for the specimens irradiated at 207°C or lower. Extremely large hardening is shown for the specimens annealed at 1000°C and 1100°C and irradiated at about 290°C. The specimens irradiated about 290°C and annealed at 700°C and 900°C show very small hardening compared to the others.

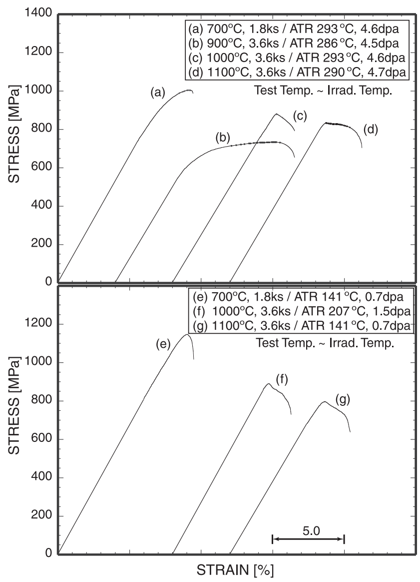

Fig. 1. Typical stress–strain curves of V–3.8Ti–5.9Cr–Si–Al–Y alloys after neutron irradiation in ATR-A1.

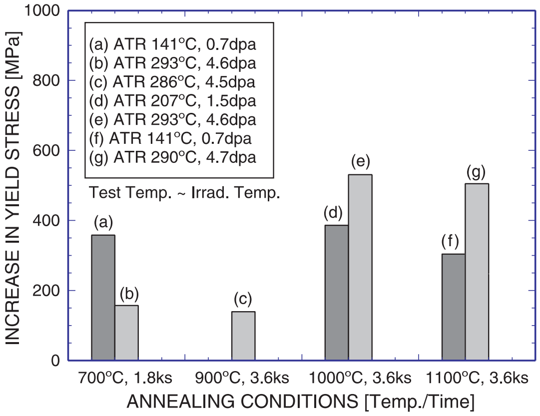

Fig. 2. Dependence of radiation hardening on annealing condition of V–3.8Ti–5.9Cr–Si–Al–Y alloy.

3.2. Microstructure evolution after irradiation in various conditions

Microstructure evolutions of the alloys annealed at 1100°C for 3.6 ks after various irradiation conditions are shown in Fig. 3 [8]. Small dislocation loops are typically formed in the microstructure. Neither precipitates nor cavities were detected in the specimens. The number density of the loops in the specimen irradiated at 290°C is the highest at 6.8×10^{22} m^{-3}. The mean size of the loops was 8.5 nm, which was a half of those irradiated at high temperatures and doses. The increase in proof stress after irradiation and the microstructure are summarized in Table 3. The strengthening of the material can be described using a interaction parameter, α, and the following relation:

$$\Delta\sigma_y = \alpha M \mu \mathbf{b} (Nd)^{1/2}, \tag{1}$$

where M is the Taylor factor ($M = 3.1$), μ the shear modulus ($\mu = 4.67\times10^4$ MPa), $\mathbf{b}$ the Burger vector for vanadium ($\mathbf{b} = 0.26$ nm), N the number density and d is the mean diameter of dislocation loops. As compared with the increase of proof stress, the value of the calculation is reasonable agreement for the irradiation at 406°C. In the case of lower temperatures and lower fluence irradiation, the value of calculation is smaller than that observed.

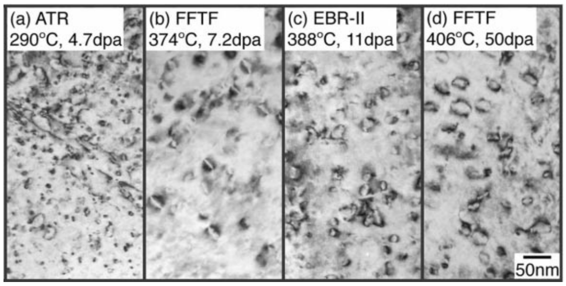

Fig. 3. Microstructure evolution of V–3.8Ti–5.9Cr–Si–Al–Y alloy and V–4.8Ti–4.0Cr–Si–Al–Y alloy irradiated in various conditions.

3.3. Comparison with uniform elongation of V–Ti–Cr type alloys

Fig. 4 shows dependence of the UE on irradiation conditions for various V–Cr–Ti type alloys [9–17]. As described in previous reports [15], V–Cr–Ti type alloys without Si, Al and Y additions shows enough large UE at temperatures higher than 430°C, but little elongation at temperatures lower than 400°C. In the case of V–Ti–Cr alloys containing Si, Al and Y, considerably large elongation was shown around 400°C. The scattering of the data is caused by irradiation conditions [16,17]. The UE at the temperatures ranging from 141°C to 323°C is quite low except for V–Ti–Cr–Si–Al–Y alloy. The alloy

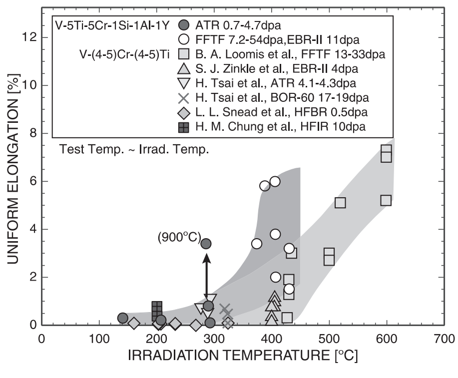

Fig. 4. Dependence of uniform elongation of vanadium alloys on irradiation temperature.

Table 3
Summary of irradiation hardening and microstructures in V–Ti–Cr–Si–Al–Y alloy after neutron irradiation

Irradiation conditions	$\Delta\sigma_{y,\,\mathrm{meas.}}$ (MPa)	N (m^{-3})	d (nm)	$\Delta\sigma_{y,\,\mathrm{cal.}}$; $\alpha = 0.28$	$\alpha = 0.4$ (MPa)
ATR; 290°C, 4.7 dpa	505.0	6.8×10^{22}	8.5	253	362
FFTF; 374°C, 7.2 dpa	242.2	1.1×10^{22}	18.9	152	217
EBR-II; 388°C, 10 dpa	247.9	1.8×10^{22}	17.0	184	263
FFTF; 406°C, 50 dpa	167.9	8.2×10^{21}	20.5	137	195

which showed large UE was annealed at 900°C before irradiation and the others were annealed at 950°C or higher. The UE of the V–Cr–Ti type alloys after irradiation at 400°C or lower are summarized in Fig. 5 as a function of interstitial-impurity level. The UE depends on the concentration of C, N and O, especially oxygen concentration. In order to maintain the UE of the alloys, it seems necessary to have less than 200 weight ppm of oxygen.

4. Discussion

4.1. Role of interstitial impurities for irradiation performance of V–Cr–Ti alloy

Disagreement between calculation and observation of hardening is thought to be due to the difference of interaction parameter, α [18]. Interstitial impurities could be migrated to the dislocation loops during irradiation [19]. Bajaj and Wechsler [20] suggested that strengthening of defect cluster by interstitial-impurity trapping for vanadium. It is possible that deformation dislocations drag the impurities at the temperature in the alloy [18]. Therefore, it is important to verify the interaction parameter with mechanistic understanding of the interaction between deformation dislocation and loops decorated with such impurities. Comparing to the V–Cr–Ti alloy without Si, Al and Y, the alloy of V–Ti–Cr–Si–Al–Y had a lower concentration of interstitial impurities. The microstructure of the V–Ti–Cr–Si–Al–Y alloy showed larger size and smaller number density of loops than the V–Cr–Ti alloy without Si, Al and Y. Interstitial impurities and Si, Al and Y could affect nucleation behavior of the defect clusters.

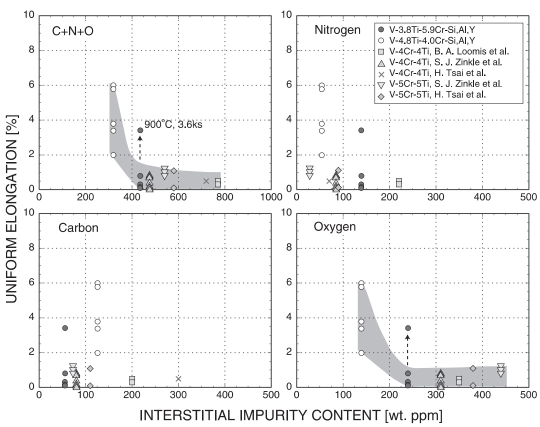

Fig. 5. Dependence of uniform elongation of vanadium alloys on interstitial impurity levels.

4.2. Consideration of optimal compositions of V–Ti–Cr type alloy

Purification of the alloy is thought to be effective to keep the UE after relatively low-temperature irradiation [7]. The addition of Y and Al could be useful to reduce the oxygen concentration during fabrication process [7,21]. On the basis of tensile data obtained from this experiment, purification utilizing small amounts of Y and Al is reasonable way for the advanced alloy development. The Charpy impact properties of the alloy do not always agree with tensile properties [22–24]. Precipitates in the dimples were observed in fracture surface after tensile tests and Charpy impact tests [23,24]. The precipitates contained Y and Ti. The formation of the small precipitates might be related to the degradation of toughness of the alloy. Although the precipitates in the dimple observed in the specimen had relatively high impurity levels, that is, about 500 weight ppm C + N + O, more work is needed to seek for an optimal composition of the additions.

5. Summary

Modified V–Ti–Cr alloy series containing Si, Al and Y were examined after neutron irradiation at 300°C or lower. Purification of the interstitial impurity was effective to reduce the extremely large radiation hardening observed in V–4Cr–4Ti type alloy. It is proposed that oxygen concentration should be kept below less than 200 weight ppm to maintain the UE after irradiation below 400°C.

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