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Abstract

Vanadium alloys, including the highly purified V-4Cr-4Ti alloy called NIFS-Heat2, in the form of sodium-enclosed irradiation capsules, were irradiated up to a damage level of 5dpa in the Joyo reactor at temperatures from 395 to 601◦C.

An increase of the ductile–brittle transition temperature (DBTT) by neutron irradiation and irradiation hardening were observed. No significant loss of ductility was observed even for irradiation at 395 and 450◦C. The addition of titanium to V-Cr alloys was effective for irradiation hardening at high temperature. Hydrogen uptake in the cleaning process during dismantling of the irradiation capsules caused ductility loss of the highly purified V-4Cr-4Ti alloys; the alloys recovered their ductility when they were annealed at 400◦C in vacuum. The uniform formation of Ti(OCN) precipitate was suppressed in highly purified V-4Cr-4Ti alloys irradiated in Joyo in a

liquid-sodium environment.

Introduction

Vanadium alloys are candidate materials for blanket structural materials in fusion reactors, but knowledge about their mechanical properties at high temperatures during neutron irradiation is limited and there are uncertainties, such as the interstitial impurity content of the specimens, that may influence the results. Recently, the technology for material irradiation in a liquid-metal environment has been developed and irradiation experiments in various liquid-metal environments have been performed for vanadium alloys [1,2]. The environmental and irradiation effects on the mechanical properties of the vanadium alloys should be distinguished independently in order to understand the essential behavior of the alloys during irradiation for fusion reactor applications. The objective of this study is to investigate the mechanical properties and microstructural changes of vanadium alloys, including the highly purified V-4Cr-4Ti alloy called NIFS-Heat2, during neutron irradiation. In this study, tensile tests, Charpy impact tests and microstructural observations were carried out for V-4Cr-4Ti alloys and vanadium binary alloys.

Experimental procedure

Various kinds of specimens, including 1.5 mm Charpy V-notched specimens, miniaturized tensile (SSJ) specimens and TEM specimens, were prepared for the Joyo experiments. The materials included unalloyed vanadium, vanadium binary alloys (with Cr, Ti solute atoms up to 5 wt.%), and vanadium ternary alloys (V-4Cr-xTi, x=0.1 to 4). Tables of the chemical components have been given in previous papers [3,4]. The V-4Cr-4Ti alloy used in this study was produced by NIFS and Taiyo Koko Co. and is designated NIFS-Heat2 [5]. The size of the SSJ specimens was 16 x 4 x 0.25 mm. The tensile specimens were prepared from unalloyed vanadium, V-5Cr, V-4Cr-0.1Ti, V-4Cr-1Ti and V-4Cr-4Ti. Charpy specimens were prepared from V-4Cr-4Ti NIFS-Heat alloys. The notch of the Charpy specimens was machined with the plane of crack propagation perpendicular to the rolling direction. The angle of the notch was 30° with a notch depth of 0.45 mm and a root radius of 0.20 mm. The TEM and SSJ tensile specimens were punched and annealed at 1100°C for 2 h after a degassing treatment at 600°C for 0.5 h in a vacuum of ~1×10^{-4} Pa. The 1.5 mm Charpy V-notched specimens were annealed at 1000°C for 2h. The specimens were irradiated in the Joyo reactor in the temperature range 450 to 650ºC with a total neutron dose from 0.47 to 2.1×10^{26} n/m^2. In the previous study, the ratio of the damage level, dpa, to the neutron dose Φ^{tot} in pure vanadium in the Joyo MK-II was given by the relationship 2.5 x 10^{-26}dpa/Φ^{tot} [6]. The estimated damage level ranged from 1.2 to 5.3dpa. Table 1 shows the irradiation conditions in the Joyo experiments Sodium-enclosed irradiation capsules were developed in order to homogenize the temperature on large specimens and prevent the invasion of interstitial impurities from the sodium environment during irradiation. Detailed additional information about the sodium-enclosed capsule installation in the Joyo reactor is given elsewhere [1]. After dismantling the sodium-enclosed capsules and cleaning the specimen surface, tensile and Charpy impact tests were performed at the Oarai Center in IMR/Tohoku University. TEM observations were performed using a JEOL-2010FX in KUR, Kyoto University.

Results

Tensile and Charpy impact tests

Figure 1 shows the results of Charpy impact tests for V-4Cr-4Ti alloys irradiated at 395 and 449 ºC. The absorbed energy was normalized by the ligament size, which is *B* x *b* =1.05 x 1.2 mm^2. The ductile–brittle transition temperature (DBTT) and the upper shelf energy (USE) of unirradiated V-4Cr-4Ti NIFS-Heat2 alloys were <-196ºC and 0.4 J/m^3, respectively [7]. The USE values for V-4Cr-4Ti alloys irradiated in Joyo were lower than for unirradiated ones, and the DBTT increased to -80 and 10ºC for 395 and 449ºC irradiation, respectively.

Figure 2 shows the neutron-dose dependence of the yield stress from the results of tensile tests at room temperature for pure vanadium and V-(4-5)Cr-xTi (x=0 to 4). In pure vanadium, significant irradiation hardening occurred in the low-temperature regime from 400 to 450ºC, but the hardening at 600ºC was small. The addition of chromium was quite effective for increasing the irradiation hardening at low temperatures. The addition of titanium to V-Cr alloys increased the yield stress at 450 and 600ºC irradiation. Irradiation hardening increased significantly at 450ºC by the addition of 0.1% titanium, and the stress level of irradiation hardening might be saturated at a neutron fluence of $7X10^{25}$ n/m^2 (~1.8dpa). Further addition of titanium apparently increased the irradiation hardening at 600ºC and the hardening might be saturated above a neutron fluence of $6X10^{25}$ n/m^2 (~1.5dpa).

A reduction in ductility of the vanadium alloys could be seen for all irradiation conditions. However, it is necessary to handle the ductility-loss data for vanadium alloys carefully because of hydrogen uptake into the specimens during cleaning of the specimen surface after dismantling the sodium-enclosed irradiation capsules. It has been reported that hydrogen absorption reduced the uniform elongation without hardening and caused a sudden drop of flow stress in tensile tests [8]. In order to eliminate the effect of hydrogen uptake, the irradiated V-4Cr-4Ti vanadium alloys were heat treated for 5 h at 400ºC in vacuum. Table 2 shows the changes of the mechanical properties of V-4Cr-4Ti NIFS-Heat2 alloys tested at room temperature before and after heat treatment. Sudden drops after yielding or at flow stress were not observed, and recovery of uniform elongation could be seen in the heat-treated specimens. It is necessary todistinguish between the essential behavior of ductility loss and the effect of hydrogen uptake in V-4Cr-4Ti alloys in liquid-metal environments during neutron irradiation.

Microstructural analysis

Figure 3 shows a typical example of the microstructure of V-4Cr-4Ti alloy and pure vanadium irradiated in the Joyo reactor. Dislocation loops and Ti(OCN) precipitates were the dominant microstructures for V-4Cr-4Ti alloys irradiated at 400 and 450ºC. There were high densities of rafting loops, and tiny precipitates were distributed locally around the loop-rafting area. At 600ºC, dislocation loops and Ti(OCN) precipitates were the dominant microstructures for V-4Cr-4Ti with a neutron dose of 0.61 x 10^{26} n/m^2 . The structure of the dislocation loops evolved and changed into a tangled dislocation structure with increasing neutron dose. However, the precipitates were dissipated in V-4Cr-4Ti alloys irradiated with a neutron dose of 1.3 x 10^{26} n/m^2.

In pure vanadium, voids were observed; their size increased and their density decreased when the irradiation temperature and neutron dose increased. Dislocations were coarsened and the dislocation density of pure vanadium decreased when the irradiation temperature and neutron dose increased. The microstructure of pure vanadium in this study had the same features as pure vanadium irradiated in FFTF reactor at 430 to 600 ºC to a damage level ranging from 15 to 27dpa [9].

Discussion

Significant increases of DBTT for V-4Cr-4Ti irradiated at low temperatures have been reported in previous work [10, 11]. Low ductility (less than 1%) and significant hardening up to 800 MPa were reported for irradiated V-4Cr-4Ti alloys, along with a significant increase of DBTT in V-4Cr-4Ti irradiated at 400^{o}C with a damage level of 4–6dpa [11]. This study also showed significant hardening and low ductility of 5–10% in the V-4Cr-4Ti alloy after irradiation to 1dpa at 395^{o}C. Tiny precipitates were formed around rafted loops and were distributed inhomogeneously. Most of the precipitates in V-4Cr-4Ti irradiated at temperatures less than 400^{o}C were formed homogeneously, accompanied by dislocation loops [11, 12]. It was believed that the inhomogeneous distribution of precipitates was caused by redistribution during irradiation: some pre-existing precipitates were dissolved into the matrix during irradiation and the alloy elements diffused and nucleated new precipitates around the pre-existing ones. The NIFS-Heat2 alloys have a smaller impurity level than any of the previous V-4Cr-4Ti alloys and the uniform formation of precipitates in the matrix might be suppressed in this study. The effect of impurity reduction on mechanical- property changes under irradiation can be seen in the redistribution of the irradiation-induced precipitates and the recovery of ductility in V-4Cr-4Ti alloys. Therefore, the purification of these alloys is very important for the improvement of their mechanical properties at low irradiation temperatures.

Summary

Vanadium alloys, including the highly purified V-4Cr-4Ti alloy called NIFS-Heat2, were irradiated up to 5dpa in the Joyo reactor in liquid sodium in the temperature range 395 to 601°C. An increase of the DBTT by neutron irradiation and irradiation hardening were observed. Significant loss of ductility was observed during irradiation at 395 and 450°C. The addition of titanium to the V-Cr alloys was effective for irradiation hardening at high temperatures. Hydrogen uptake in the cleaning process during dismantling of the irradiation capsules caused ductility loss in the highly purified V-4Cr-4Ti alloys; the ductility was recovered by annealing at 400°C in vacuum. The formation of Ti(OCN) precipitates was not uniform, which resulted in a significant loss of ductility in the highly purified V-4Cr-4Ti irradiated in the Joyo reactor in a liquid-sodium environment.

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Figure captions

Fig.1. Test-temperature dependence of absorbed energy for Charpy impact testing of NIFS-Heat2 V-4Cr-4Ti alloys irradiated in the Joyo reactor at 395 and 449ºC.

Fig.2. Dependence of yield stress on neutron dose for pure V, V-5Cr, V-4Cr-0.1Ti, V-4Cr-1Ti and V-4Cr-4Ti alloys in tensile tests.

Fig.3. Microstructures of V-4Cr-4Ti alloy and pure vanadium irradiated in the Joyo reactor. The irradiation temperature and dose in units of 10^{26} n/m^2 are given for each column.

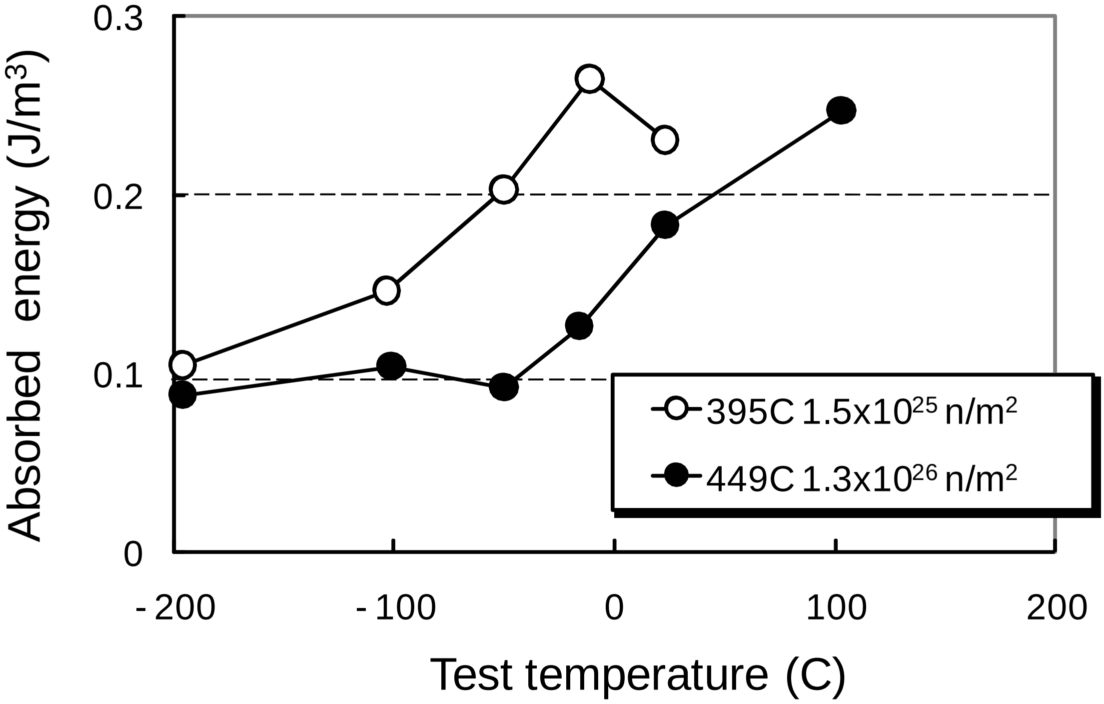

Fig.1 one column

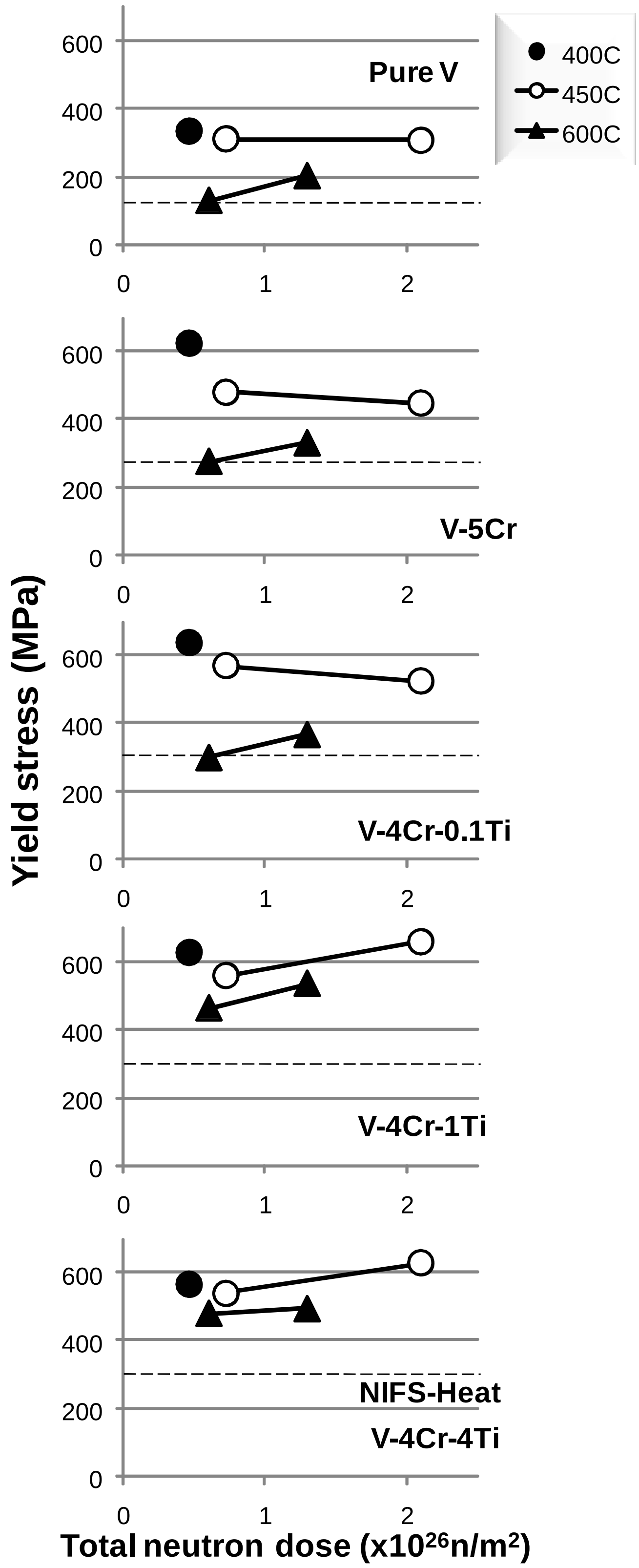

Fig.2. one column

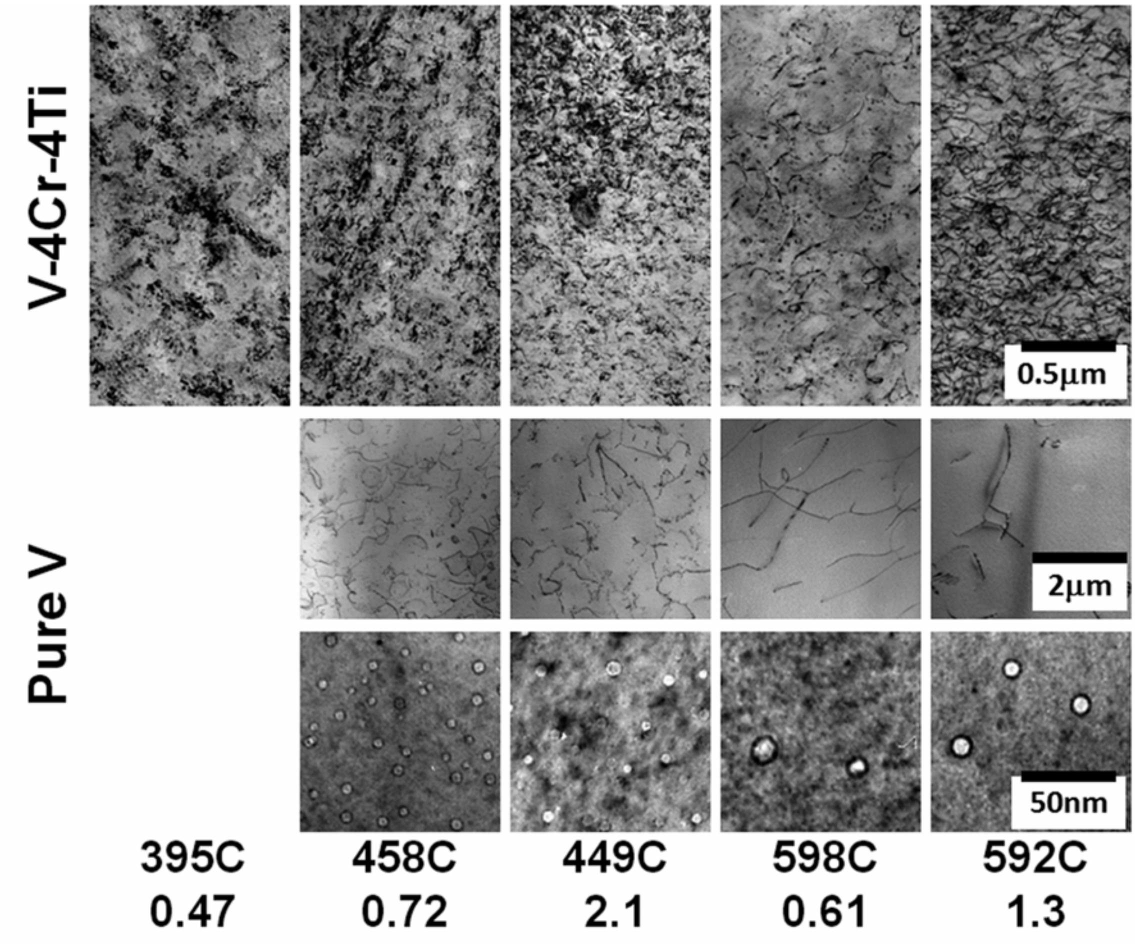

Fig.3. two column

Table 1 Irradiation conditions in the Joyo reactor.

Irradiation temperature (ºC)	Neutron dose (E>0.1MeV) ($x10^{26} n/m^2$)	Total neutron dose ($x10^{26} n/m^2$)	Estimated damage level (dpa)
395	0.15	0.47	1.2
458	0.24	0.72	1.8
449	1.3	2.1	5.3
598	0.32	0.61	1.5
592	0.67	1.3	3.3
748	0.78	1.5	3.8

Table 2 Changes of mechanical properties for NIFS-Heat alloys after hydrogen-removing treatment

		As -irradiated			H-removed			Change after H-removed		
T (°C)	Total neutron dose ($x10^{26} n/m^2$)	σ_y	σ_{UTS}	ε_U	σ_y	σ_{UTS}	ε_U	$\Delta\sigma_y$	$\Delta\sigma_{UTS}$	$\Delta\varepsilon_U$
RT		320	416	15.9						
395	0.47	566	615	7.5	550	586	2.8	-16	-29	-4.7
458	0.72	540	609	2.6	518	576	2.0	-22	-33	-0.6
449	2.1	628	655	0.8	569	609	4.3	-59	-46	3.5
598	0.61	478	529	0.6	398	552	2.6	-80	23	2.0
592	1.3	495	556	1.0	425	517	1.3	-70	-39	0.3
748	1.5	527	559	1.1	427	527	9.7	-100	-32	8.6