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Effect of neutron irradiation on the microstructure and hardness in particle dispersed ultra-fine grained V–Y alloys

Sengo Kobayashi [a,*], Yusuke Tsuruoka [a], Kiyomichi Nakai [a],
Hiroaki Kurishita [b]

[a] *Department of Materials Science and Engineering, Ehime University, 3 Bunkyo-cho, Matsuyama 790-8577, Japan*
[b] *The Oarai Branch, Institute for Materials Research, Tohoku University, Oarai, Ibaraki 311-1313, Japan*

Abstract

Effects of neutron irradiation on the microstructure and hardness in V–1.6 and 2.6Y (mass%) alloys were studied. The alloys consisted of both fine grains of a few hundreds of nanometers in diameter with finely dispersed particles and small amounts of coarse grains of a few micrometers in diameter without dispersed particles. The microstructures after neutron irradiation at 290 and 800 °C to 0.25 and 0.7 dpa, respectively, were examined with transmission electron microscopy. Irradiation did not change the grain size. Interstitial loops and voids were formed in the coarse grains irradiated at 290 °C, while voids were formed only in the fine grains. The number density of voids decreased with decreasing grain size. Interstitial loops and voids were not observed in V–1.6Y alloy irradiated at 800 °C, while thin plates were formed only in the coarse grains. The formation of interstitial loops and voids was efficiently suppressed in the fine grains.

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1. Introduction

Vanadium alloys for structural material of fusion reactor have attractive properties of low induced radioactivity, superior ductility at low temperature and so on [1–5]. However, they are seriously embrittled by neutron irradiation at temperatures below 400 °C [3,6,7]. Since grain boundary and interface boundary act as sinks mainly for point defects, alloys having both ultra-fine grains and fine particles [8–12] are expected to resist irradiation embrittlement. The purpose of this study is to analyze effects of neutron irradiation on the microstructure in vanadium alloys having ultra-fine grains with dispersed particles containing extremely little interstitial impurities [13] after irradiation at 290 and 800 °C up to 0.7 dpa, with particular emphasis on the behavior of radiation damage in fine and coarse grains.

2. Experimental procedures

Both V–1.6Y and V–2.6Y alloys (mass%), which were made by mechanical alloying (MA) with a planetary ball mill in a purified argon atmosphere and hot isostatic pressing at 1273 K and 200 MPa for 3 h, were machined into disks of 3 mm in diameter and 0.1 mm thick. Chemical compositions of the alloys are listed in Table 1. The contents of tungsten and cobalt, which came from the milling pots and balls during MA, and that of argon are suppressed to be very low. Detail procedures for preparation of the alloys have been reported in the previous study [13]. The disks wrapped with Ta foil were encapsulated into evacuated quartz tubes. Dehydrogenation was done at 1000 °C for 3.6 ks. They were irradiated with fluences of 1.3×10^{24} n/m^2 (about 0.25 dpa) at 290 °C and of 3.7×10^{24} n/m^2 (0.7 dpa) at 800 °C in the Japan Materials Testing Reactor (JMTR). Microstructures were analyzed by transmission electron microscopy (TEM). Thin foils for TEM were prepared by means of twin-jet electropolishing using a solution of 5 vol.% H_2SO_4 and 95 vol.% CH_3 OH at

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[*] Corresponding author. Tel./fax: +81-89 927 8524; fax: +81-89 927 9907.
E-mail address: sengo@eng.ehime-u.ac.jp (S. Kobayashi).

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Table 1
Chemical composition of the alloys used in this study (mass%)

	Y	O	N	C	H	W	Co	Ar	V
V–1.6Y	1.56	0.141	0.075	0.022	0.0004	0.09	–	0.0012	Bal.
V–2.6Y	2.57	0.175	0.300	–	–	0.03	<0.003	–	Bal.

around 5 °C at 20 V, and examined in both analytical JEM2000FX microscope operating at 200 kV and JEM4010 microscope operating at 400 kV. Hardness of the alloys was measured by a Vickers microhardness tester with a load of 1.96 N for 20 s.

3. Results and discussion

The alloys used in this study contained fine grains of about a few hundred nanometers in diameter with small amounts of coarse grains of about a few micrometers in diameter. These mixed grains were made by an imperfect MA treatment [13]. In this study the fine grain is defined being less than 1 μm in diameter. Mean diameters of the fine grains in V–1.6Y and V–2.6Y alloys were 303 and 170 nm, respectively, which are much smaller than the grains of melting-fabricated vanadium alloys. Size distribution of grains was not changed by irradiation.

Fig. 1(a) and (b) show TEM micrographs of V–1.6Y alloy before irradiation in the regions of coarse and fine grains, respectively. Y_2O_3 particles were formed only in the fine grains as indicated by arrows in (b). Although X-ray diffractometry [13] showed that both Y_2O_3 and YN existed in the alloy, Y_2O_3 were only observed in the TEM [14]. The particles observed in this paper were considered as Y_2O_3. The coarse grains, on the other hand, did not contain any particles, indicating concentrations of yttrium and interstitial impurities such as oxygen were low. Fig. 1(c) and (d) show the microstructures in coarse and fine grain regions of V–1.6Y alloy irradiated at 290 °C, respectively. In coarse grains, self-interstitial loops (I-loops) were formed as shown in Fig. 1(c). Large I-loops were observed as dislocation lines. I-loops were not formed in the vicinity of grain boundaries due to annihilation of interstitials into grain boundaries. The width of I-loop free zone was about 500 nm. Magnified image of the matrix in (c) is inserted on

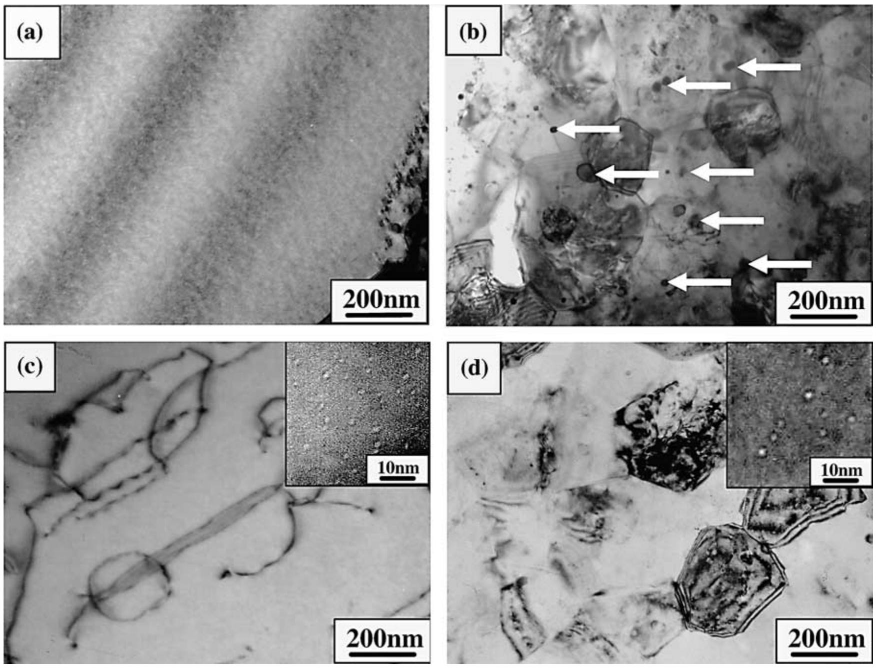

Fig. 1. Bright field images of V–1.6Y alloy showing: (a) coarse grain before irradiation; (b) fine grains before irradiation; (c) coarse grain irradiated at 290 °C; (d) fine grains irradiated at 290 °C, respectively. Magnified images of the matrix in (c) and (d) are inserted on the upper right corner of each figures.

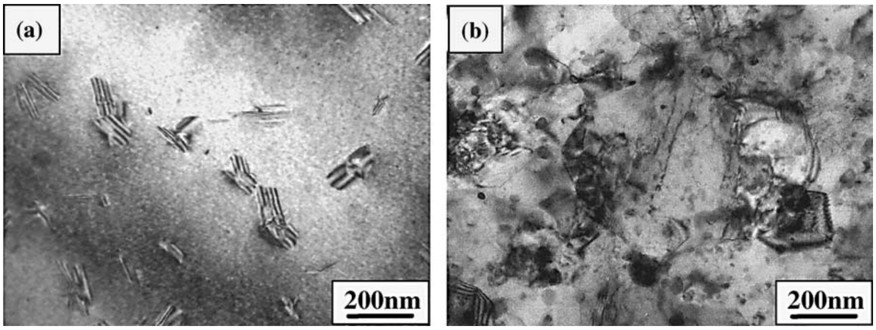

Fig. 2. Bright field images of V–1.6Y alloy irradiated at 800 °C showing: (a) plates having fringes formed in the coarse grain and (b) fine grains with dispersed Y_2O_3 particles.

the upper right corner of (c), showing homogeneous formation of voids, in contrast with void formation being scarcely in melting-fabricated vanadium alloys irradiated at temperatures below 350 °C [15]. It indicates that vacancies are mobile at this temperature in the alloy. It suggests that impurity level of the alloy used is lower than that in the melting-fabricated alloys. In fine grains I-loops were not observed. Magnified image of the matrix in (d) is inserted on the upper right corner of (d), showing void formation. Since average grain radius of fine grains was smaller than the width of I-loop free zone, most interstitials in fine grain region annihilate into grain boundaries, resulting in no I-loop formation.

Fig. 2(a) and (b) show the microstructures in coarse and fine grain regions, respectively, in V–1.6Y alloy irradiated at 800 °C. Only in fine grain region, Y_2O_3 particles were formed. Since the volume fraction of particles increased after irradiation, additional nucleation of particles occurred. Since free yttrium is expected to exist in the fine grain before irradiation [14], oxygen introduced from the surrounding during irradiation would combine with yttrium to nucleate Y_2O_3 particles. Voids were not observed in both grain regions. In coarse grain region, however, plates with fringes were observed, as shown in Fig. 2(a). Enrichment of yttrium into the plates was not detected by a composition analysis using energy dispersive X-ray spectroscopy equipped with TEM. Extra spots on diffraction pattern could not be detected. Since yttrium might not be contained in coarse grain region, impurities such as oxygen could not form yttria precipitate. Therefore, when vacancy loops (V-loops) were formed in coarse grain region during irradiation, impurities might accumulate to V-loops. The stacking fault energy would decrease with the impurity accumulation, resulting in dominant formation of V-loops with stacking fault. Plate free zone of about 500 nm in width was observed in the vicinity of grain boundaries. The suppression of the plate formation in the vicinity of grain boundaries suggests vacancy annihilation to grain boundary, corresponding to the fact that these plates were not observed in fine grain region.

Fig. 3(a) and (b) show TEM micrographs of V–2.6Y alloy before irradiation in coarse and fine grain regions, respectively. Y_2O_3 particles were formed only in fine grain region. The amount of particles in V–2.6Y alloy was larger than that in V–1.6Y alloy. This is probably due to the fact that the content of nitrogen in V–2.6Y alloy is much higher than that in V–1.6Y alloy. The average grain size of fine grains in V–2.6Y alloy is much smaller than that in V–1.6Y alloy. The microstructures in coarse and fine grain regions of V–2.6Y alloy irradiated at 290 °C are shown in Fig. 3(c) and (d), respectively. In coarse grain, I-loops of about 100 nm in diameter were formed in the matrix as shown in Fig. 3(c). I-loops were not formed in the vicinity of grain boundaries. The width of I-loop free zone was about 500 nm. Magnified images of the matrix in (c) and (d) are inserted on the upper right corner of each figures, showing void formation. In fine grain region, very small amounts of voids indicated by an arrow without I-loops were observed. Since average grain size of V–2.6Y alloy is much smaller than that of V–1.6Y alloy, most vacancies in V–2.6Y alloy might annihilate at grain boundaries, resulting in small amounts of voids. It suggests that grain boundaries suppress the formation of not only I-loops but also voids.

The values of Vickers hardness before and after irradiation are tabulated in Table 2. Increments of hardness due to irradiation are 3–70 Hv, which are much smaller than that in melting-fabricated vanadium alloys, 97–117 Hv [16]. Since mobility of interstitials and vacancies in the alloys containing low impurities is expected to be high, interstitials and vacancies are recombined and annihilated at grain boundaries. Furthermore, grain miniaturization would promote the annihilation of interstitials and vacancies. Thus, defect clusters such as I-loops and voids in the alloys were largely suppressed, resulting in small increment of hardness by irradiation.

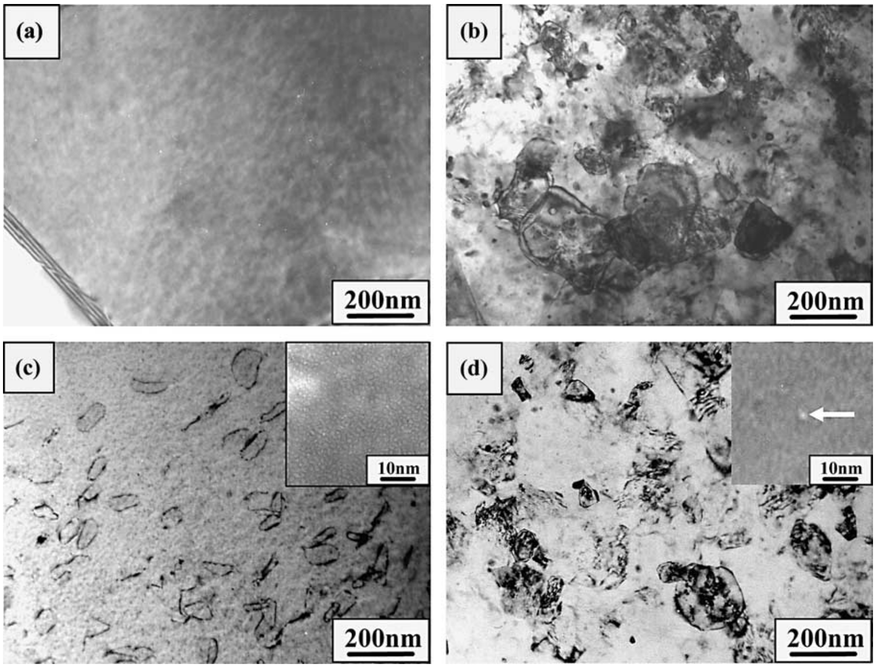

Fig. 3. Bright field images of V–2.6Y alloy showing: (a) coarse grain before irradiation; (b) fine grains before irradiation; (c) coarse grain irradiated at 290 °C; (d) fine grains irradiated at 290 °C, respectively. Magnified images of the matrix in (c) and (d) are inserted on the upper right corner of each figures.

Table 2
Vickers hardness numbers of the alloys before and after irradiation

	Before irradiation	Irradiation at 290 °C	Irradiation at 800 °C
V–1.6Y	185	255	249
V–2.6Y	278	281	–

4. Conclusions

Interstitials annihilate at grain boundaries and interface boundaries of Y_2O_3 particles, resulting in suppression of I-loop formation in V–1.6Y alloy irradiated at 290 °C. Interstitials and most vacancies in V–2.6Y alloy annihilate into grain boundaries and interface boundaries of particles. Formation of I-loop and void was well suppressed in V–2.6Y alloy. In V–1.6Y alloy irradiated at 800 °C interstitials and vacancies migrate easily to flow into grain and interface boundaries and recombine with each other. Both grain miniaturization and particle dispersion efficiently suppress the formation of radiation-induced defect clusters such as I-loop and void, resulting in small increment of hardness by irradiation.

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