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Effects of solid transmutation and helium on microstructural evolution in neutron-irradiated vanadium

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Abstract

Pure V and V alloys with B and Cr irradiated in the HFIR to 10 dpa at 400, 500 and 600 °C are examined by TEM. Nine percent of V is transmuted to Cr during irradiation to 10 dpa, and more than 99% of ^{10}B is transmuted to He and Li during the early stage of irradiation. The Cr generation suppresses cavity nucleation near grain boundaries in pure V. However in V–4.9Cr which contains 13at.%Cr after irradiation, cavities concentrate near grain boundaries, and there are few cavities in the matrix. At 400 and 500 °C, the effect of He on the cavity formation is not clearly observed. At 600 °C, B addition enhancese the cavity nucleation in pure V and V–4.9Cr in the matrix. Growth of cavities is also enhanced in pure V which is converted to V–9.4Cr.

1. Introduction

Vanadium alloys are attractive candidates for fusion blanket structural materials because of their excellent mechanical properties at high temperatures and low induced radioactivities after 14 MeV neutron irradiation [1,2]. Neutron irradiation produces not only displacement damage but also transmutation reactions, resulting in microstructural and macroscopic property change in fusion materials. It is generally known that a high generation rate of helium by transmutation strongly affects microstructural evolution [3,4] and mechanical properties. The effect of helium on microstructural evolution has been investigated by using several techniques such as dynamic helium charging experiments (DHCE) and boron-10 doping [5–7] by neutron irradiation. Recently, the effect of solid transmutation reactions has also been recognized to affect the irradiation behavior of vanadium alloys [8,9], especially during irradiation in mixed spectrum reactors since there exists a large cross-section of the nuclear transmutation reaction from vanadium to chromium by thermal neutrons [9,10]. In this study, pure V and V–Cr alloys with and without B doping are irradiated with mixed spectrum neutrons in the high flux isotope reactor (HFIR) to investigate synergistic effects of the Cr production from V and He on cavity microstructural evolution.

2. Experimental

Specimens were prepared by arc-melting from high purity 99.8% V and 99.9% Cr in an inert gas atmosphere. They were rolled and made into disks of 3 mm in diameter and 0.25 mm in thickness. These specimens were annealed at 900 °C for 3.6 ks in ultra-high vacuum, followed by rapid cooling to insure most of B in solution.

Disk samples of 99.8% pure vanadium, V–100appmB, V–500appmB and V–2500appmB were put in the capsule JP-23 and irradiated at 400, 500 and 600 °C in cycles 322–326 in the HFIR for 1.5×10^7 s. At 600 °C, V–4.9at.%Cr and V–4.9at.%Cr–100appmB alloys were also irradiated in the same capsule. Fast neutron fluences (>0.1 MeV) in cycles 322–326 were ranged from 0.91×10^{26} to 1.04×10^{26} n/m^2, and thermal neutron fluences from 2.3×10^{26} to 2.7×10^{26} n/m^2 [11]. In pure V, the He production is roughly estimated to be 1 appm. It is calculated that during the first 6 days of irradiation,

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Table 1
Irradiation conditions in the HFIR capsule JP-23 during cycles 322–326

Irradiation temperature (°C)	Fast neutron fluence (n/m^2)	Thermal neutron fluence (n/m^2)	Displacement damage (dpa)	Cr content after irradiation (at.%)	Specimens
400	0.91×10^{26}	2.3×10^{26}	8.9	8.1	V
500	1.00×10^{26}	2.5×10^{26}	9.8	9.1	V–100appmB
					V–500appmB
				9.4	V–2500appmB
600	1.04×10^{26}	2.7×10^{26}	10.3	13.8	V–4.9at.%Cr,
					V–4.9at.%Cr –100appmB

more than 99% of ^{10}B was transmuted to ^{4}He. The He contents in V–100appmB, 500appmB and 2500appmB are 20, 100 and 500 appm, respectively. The He (appm)/dpa ratios in irradiated boron-doping alloys were ranged from about 2 to 50. Solid transmutation rates at the midplane of the HFIR were estimated to be 9 at.% for V transmutation to Cr and 0.95% for Cr to V. The irradiation conditions in this study are summarized in Table 1. After irradiation the microstructural changes were examined by 200 kV transmission electron microscopy.

3. Results

3.1. Temperature dependence of cavity density and size in V and V–B

Cavities can be observed in all the irradiated specimens. Cavities in pure V irradiated at 400 and 500 °C are homogeneously distributed in the matrix, while at 600 °C, cavities are inhomogeneously distributed within the matrix as shown in Fig. 1.

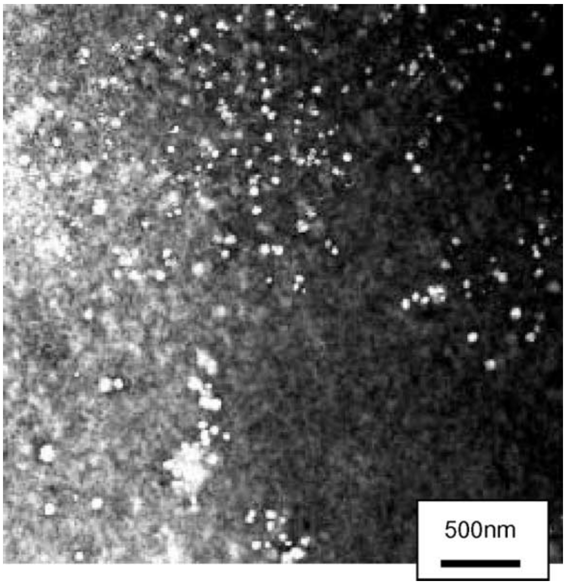

Fig. 1. Cavity microstructure in the matrix of pure V irradiated at 600 °C to 10.3 dpa in HFIR. Pure V converted to V–9.4at.%Cr by transmutation reaction by thermal neutrons. Note that cavities are inhomogeneously distributed in the matrix.

Fig. 2 shows temperature dependence of the cavity density in pure V and V–B alloys. The cavity density decreases monotonically with increasing irradiation temperature, although it cannot be measured quantitatively in V–2500appmB irradiated at 600 °C. Increasing irradiation temperature from 500 to 600 °C strongly decreases the cavity density but increases the cavity size. Average cavity diameter in both V and V–B alloys are about 2 nm at 400 and 500 °C, and about 58 nm at 600 °C. At 400 and 500 °C, the He generation up to 500 appm does not strongly affect the cavity nucleation as shown in Fig. 2.

Fig. 3 shows the cavity size distribution of pure V, V–100appmB and V–500appmB irradiated at 600 °C. The density of tiny cavities increases with B contents indicating that He accelerates the cavity nucleation in V. Cavities larger than 120 nm can also be observed in V–B

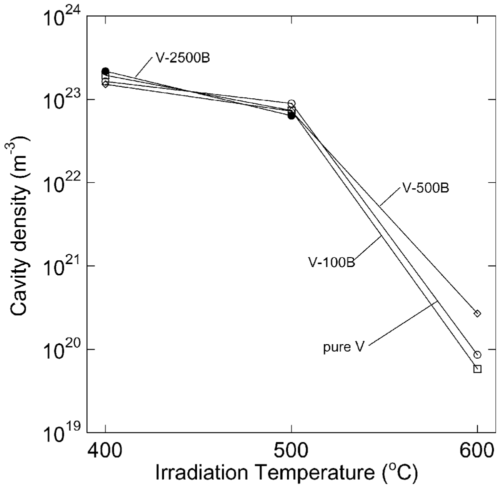

Fig. 2. Irradiation temperature dependence of cavity density in pure V and V–B alloys. Note that the cavity density in the V–2500appmB alloy irradiated at 600 °C cannot be measured because of the extreme inhomogeneous cavity distribution.

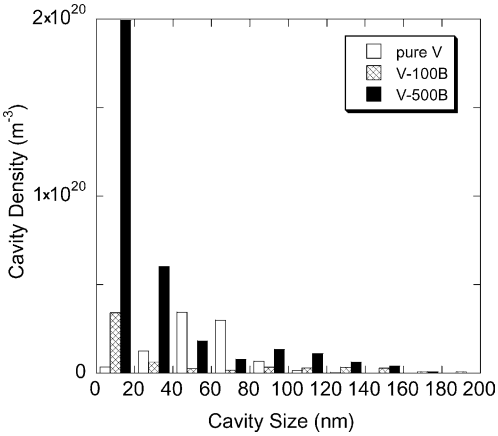

Fig. 3. Cavity size distributions in pure V, V–100appmB and V–500appmB irradiated at 600 °C to 10.3 dpa.

alloys. In V–2500appmB irradiated at 600 °C, almost all cavities are observed on grain boundaries and there are few cavities in the matrix, except for halo rings of cavities around the large V_3B_2 precipitates as is reported in previous papers [6,12–14].

3.2. Effect of transmuted Cr from V

At 600 °C, few cavities are observed on grain boundaries in pure V as shown in Fig. 4(a), which has been converted to V–9at.%Cr during irradiation. However, the high density of cavities are inhomogeneously formed in the matrix as shown in Fig. 1.

In V–4.9at.%Cr, which has become V–13at.%Cr after irradiation, cavities are observed on grain boundaries and very few cavities are detected in the matrix, as shown in Fig. 4(b). The average diameter of grain boundary cavities is about 18 nm. Excess Cr content of 13–14 at.% suppresses nucleation of cavities in the matrix.

Fig. 4(c) shows the typical cavity microstructure in V–4.9at.%Cr–100appmB irradiated at 600 °C, in which 20 appm He has been generated from B. Cavities are observed on grain boundaries, as is observed in V–4.9at.%Cr without B addition. Cavities are also produced in the matrix in V–4.9at.%Cr–100appmB. The cavity density in the matrix is about 6×10^{18} m^{-3} in V–4.9at.%Cr and about 5×10^{19} m^{-3} in V–4.9at.%Cr–100appmB. He is found to enhance the cavity nucleation in the matrix even in V–Cr alloys which contain 13at.% Cr. However, the large cavity growth as observed in V under high He generation rate is not observed. The average cavity diameter, about 32 nm, in the matrix is slightly larger than that on grain boundaries, about 22 nm.

4. Discussion

4.1. Effects of Cr by transmutation on cavity microstructure in V and V–Cr

The cavity density of pure V irradiated in HFIR to 9.8 dpa at 500 °C is much higher than that of V–5Cr irradiated in JOYO to 13.7 dpa at 500 °C [15]. The cavity diameter in HFIR is much smaller than that in JOYO (about 30–100 nm). High density of small cavities may be caused by gradual Cr generation during irradiation in HFIR.

It is known from previous studies [6,15–19] that void swelling increases with Cr addition in V at 600 °C and decreases or saturates with Cr addition when the Cr content is larger than 10 at.%. One of the characteristic

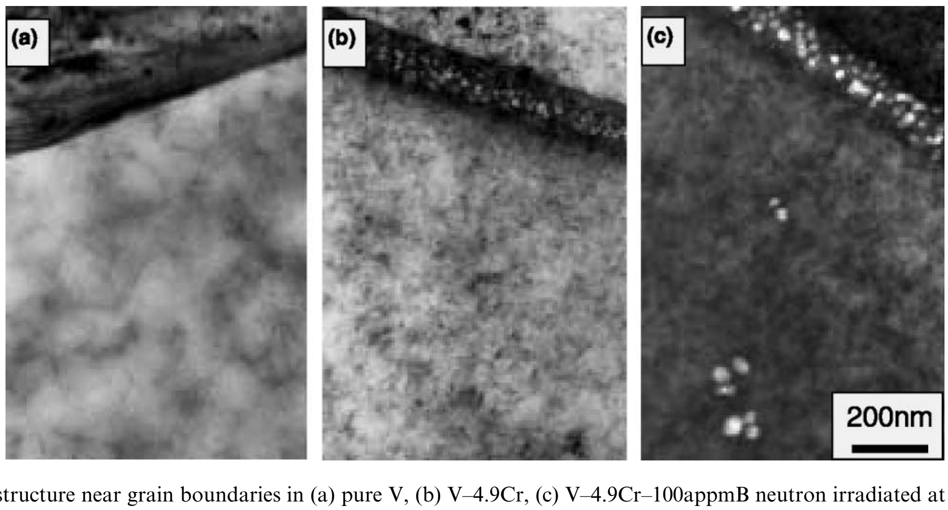

Fig. 4. Microstructure near grain boundaries in (a) pure V, (b) V–4.9Cr, (c) V–4.9Cr–100appmB neutron irradiated at 600 °C to 10.3 dpa.

microstructure is the spatial distribution of cavities depending on the amount of Cr. The content less than 10 at.% in V makes cavities homogeneously distributed in the matrix [5], as is observed in V–4.9Cr irradiated with fast neutrons in fast flux test facility (FFTF) [17]. A similar microstructure is observed in pure V irradiated in HFIR, which converts to 9.4at.%Cr alloy. When the Cr content becomes larger than 10 at.% during irradiation, fine cavities tend to form only on grain boundaries which prevent large swelling.

4.2. Effect of He converted from ^{10}B on the cavity microstructure in V and V–Cr

He generally enhances the cavity nucleation in pure V and V alloys [3,4,6]. However, at 400 and 500 °C, a small effect of He generated from B on cavity density is observed. This may come from the burn-up of ^{10}B at the very early stage of irradiation up to 0.06 dpa.

At 600 °C, cavities can easily nucleate without He in the matrix of pure V which converts to V–9.4Cr. In V–B alloys, the density of cavities increases only by a large amount of He, indicating that the addition of B may suppress the cavity formation. Transmuted Cr is considered to enhance the growth of cavities nucleated by He accumulation at low dose.

Cavity growth is not enhanced in V–4.9Cr–100appmB, as the excess Cr content by transmutation can prevent growth of cavities. He clearly accelerates nucleation of cavities in the matrix, but they cannot grow larger by gradual accumulation of Cr to 13.8 at.% during irradiation to 10.3 dpa in HFIR.

5. Summary

V alloys with several amounts of B addition are irradiated in HFIR, and the effect of Cr and He on the cavity formation is investigated. At 600 °C, the spatial distribution of cavities strongly depends on the Cr content. Cavities in V which is converted to V–9.4Cr exist in the matrix, while cavities in V–4.9Cr which is converted to V–13.8Cr do not exist in the matrix and concentrate on the grain boundaries. He generation enhances the cavity nucleation in the V–4.9Cr matrix. At 400 and 500 °C, small effects of He generation up to 500 appm on cavity formation can be observed.

Acknowledgements

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