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Swelling behavior of V–Fe binary and V–Fe–Ti ternary alloys

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Abstract

V–Fe binary alloys with different Fe concentrations, i.e., V–1, 3 and 5 at.% Fe, and V–5% Fe alloy added with 1, 3 and 5 at.% of Ti were irradiated in EBR-II at 380–615°C to about 11 dpa. TEM observation was performed after irradiation. A systematic increase in cavity size was observed with increasing iron concentration in the binary alloys, especially at 510°C and 615°C irradiation. On the other hand, the density of cavities decreased with increasing iron concentration and irradiation temperature. Maximum swelling in V–Fe system occurred between 500°C and 600°C and the amount of swelling was up to 30% at a damage level of 11 dpa. The alloy containing only 1% Fe already showed substantial swelling. The effect of titanium addition to the swelling was very remarkable. One atomic percent of titanium addition to V–5 at.% Fe significantly suppressed cavity formation, and 3 at.% of titanium addition entirely suppressed swelling. There seems to be a threshold titanium concentration for suppression of swelling in V–5 at.% Fe. Radiation-induced precipitation of titanium oxide may be one reason why titanium additions suppress the swelling in vanadium alloys. Homogeneous titanium oxide precipitates were not observed so that the titanium in solution is more likely to be playing an important role for suppression of swelling.

1. Introduction

Vanadium alloys have been identified as an attractive high performance structural material for fusion power plants. Especially, one candidate, V–4Cr–4Ti, shows good resistance to nuclear reactor irradiation [1,2]. On the contrary, It has been reported that vanadium-iron binary alloys show a huge swelling up to 160% under neutron irradiation at a damage level of 50 dpa [3]. It is well known that an addition of undersized atoms relative to matrix atoms enhances void swelling. The mechanism of irradiation-induced swelling also can be well explained by radiation-induced segregation (RIS) [4]. However the cause of the huge swelling cannot be entirely explained by RIS. It is very interesting to determine why huge swelling occurs and what factors cause it.

Titanium additions to vanadium alloys have been reported to be useful for suppression of swelling. In particular, one candidate alloy, V–4Cr–4Ti, contains titanium to prevent from void swelling during neutron irradiation [5]. The suppression mechanism is considered either due to the effect of an oversized atom in solution, or radiation-induced precipitation as suggested by Ref. [6], In this study, it has been found that a small addition of titanium entirely suppressed the swelling of V–5 at.% Fe. It is very important to obtain an insight to the mechanism of swelling suppression for the development vanadium alloys for fusion reactor application.

In the present study, the swelling behavior of V–Fe alloys with different iron concentrations, and the effects of titanium additions to a V–Fe binary alloy have been studied using TEM observation after EBR-II neutron irradiation.

2. Experimental procedure

Vanadium alloys were prepared from 99.9% pure vanadium, 99.999% pure iron and 99.99% pure titanium by argon arc melting. Their nominal compositions are, pure V, V–1Fe, V–5Fe, V–5Fe–1Ti, V–5Fe–3Ti and V–5Fe–5Ti. The alloy was cold rolled to a thickness of 0.25 mm and punched into 3 mm TEM disks and tensile specimens. They were annealed at 1100°C for 2 h. After

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heat treatment, the nitrogen concentration was approximately 25 appm, while the oxygen concentration was approximately 1000 appm. Irradiation was performed in EBR-II reactor, at 380°C, 510°C, 615°C up to the fluence of 1.6×10^{26} n/m^2, which corresponds to the damage level of 11 dpa for pure vanadium calculated by SPECTER-CODE. The specimens were sealed with lithium in TZM capsules for irradiation. After irradiation, specimen capsules were transported to the Irradiation Material Facility at Argonne National Laboratory and disassembled. Unfortunately, half of the irradiated TEM disks are missing in the facility. To replace these specimens, small pieces were punched from irradiated tensile specimens and fabricated into TEM disks. Thin foils for TEM were prepared using a conventional twin-jet electropolishing apparatus. TEM observation was done using JEOL-2010 microscope equipped with EDS and PEELS at IMR, Sendai, Japan.

3. Results

3.1. Microstructure after irradiation at 385°C

Fig. 1 shows a typical example of TEM images of specimens irradiated at 385°C in EBR-II. The upper part shows dislocation contrast images, which are near the exact Bragg condition of (1 1 0) reflection. The lower part shows void images, which are taken under kinematical reflection condition. From left to right, pure V, V–1Fe, V–5Fe, V–5Fe–1Ti, V–5Fe–3Ti and V–5Fe–5Ti are placed in a row. The size of void in V–Fe alloys becomes large with increasing iron addition to vanadium. Fig. 2 shows the iron concentration dependence of cavity size in V–x% Fe binary alloys and titanium concentration dependence in V–5%Fe–*y*%Ti ternary alloys at three irradiation temperatures. The average size of void in pure vanadium, V–1Fe and V–5Fe is about 4, 6 and 11 nm, respectively at 385°C. The void growth rate seems to be linearly dependent on the iron concentration. On the contrary, the addition of titanium decreased void size from V–5Fe. Fig. 3 shows the alloying element dependence of cavity density. Voids in V–5Fe–1Ti, formed inhomogeneously with a linear morphology as if the voids formed on dislocations. Increasing the addition of titanium exterminated voids entirely from the matrix and precipitation of titanium oxide appeared. Fig. 4 shows the alloying element dependence of the dislocation density. In this study, dislocation loops were not observed probably because the damage level was too high to create isolated dislocation loops. The dislocation density also increases with the iron concentration, but the addition of titanium did not affect the dislocation density at 385°C.

3.2. Microstructure after irradiation at 510°C and 615°C

Fig. 5 shows a typical example of TEM images of specimens irradiated at 510°C in EBR-II. It can be seen

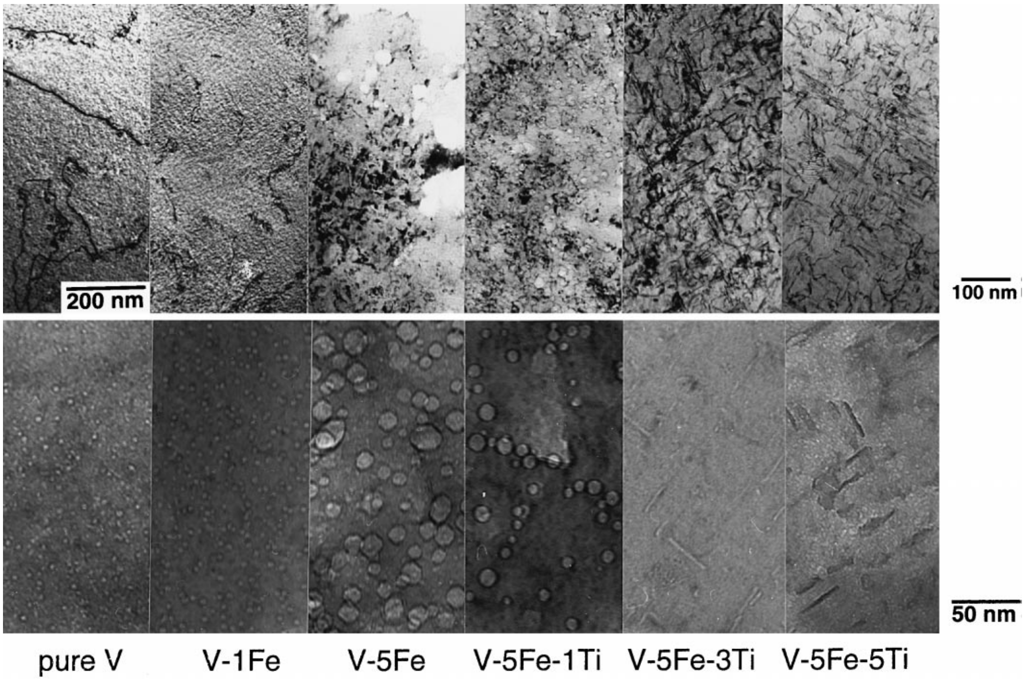

Fig. 1. Typical example of V–Fe and V–Fe–Ti alloys irradiated in EBR-II to 11 dpa at 385°C. Upper micrographs show dislocation images, and lower ones cavity images. The scale indicates 100 nm in the top half and 50 nm in the bottom.

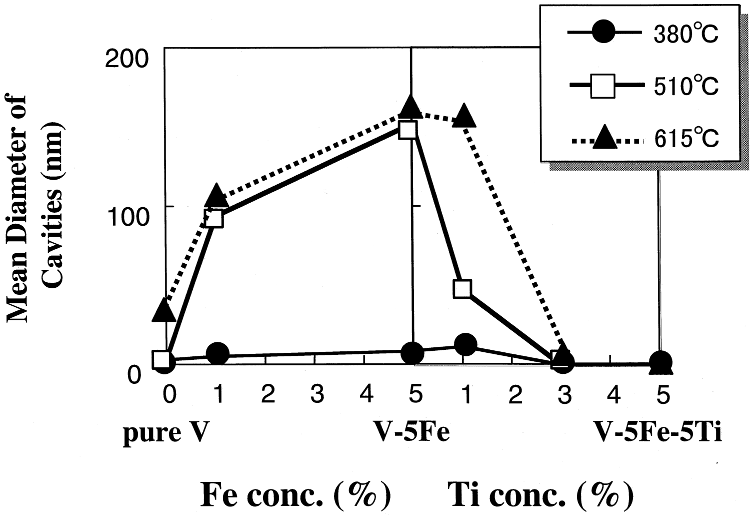

Fig. 2. Mean Size of cavities as a function of iron concentration in V–Fe binary alloys and on titanium concentration in V–5Fe–Ti ternary alloys.

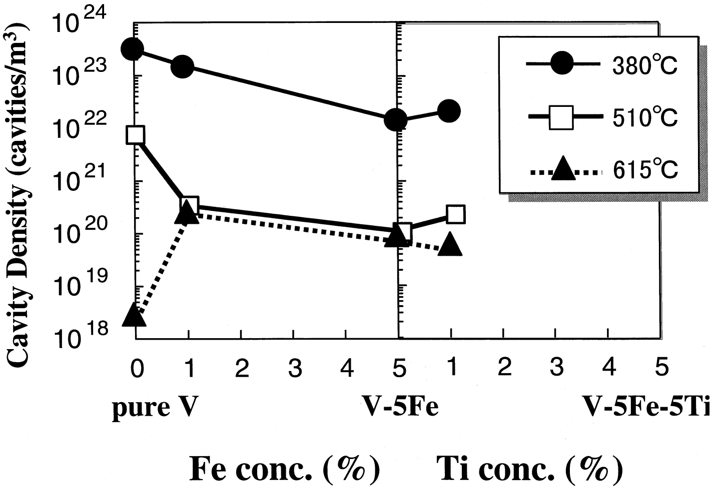

Fig. 3. Cavity density as a function of iron concentration in vanadium and titanium concentration in V–5Fe.

that V–Fe binary alloys exhibit huge swelling or the formation of extremely large voids beyond 100 nm even though the damage level is only 11 dpa. It is interesting to note that rather huge swelling occurred also in V–1Fe, despite the fact that it contains much smaller iron concentration than V–5Fe; huge swelling has been reported in the latter alloy. The density of voids decreased with iron concentration, while the void size increased with iron concentration, as shown in Figs. 2 and 3. The swelling determined from the density and size of cavity reached to 30% in V–5Fe at a damage level of 11 dpa. Dislocation density also increased with iron concentration. The distribution of dislocations is rather homogeneous.

From V–5Fe to V–5Fe–5Ti, the void size was drastically reduced with increasing titanium concentration. In V–5Fe–1Ti, the size of cavity is one third smaller than that of V–5Fe. In particular, no voids appeared at all in V–5Fe–3Ti. Also the dislocation density decreased and titanium oxide precipitates appeared with increasing titanium concentration. These features also can be seen in the microstructures of vanadium alloys irradiated at 615°C. Fig. 6 shows a typical example of TEM images of specimens irradiated at 615°C in EBR-II. The microstructural tendency of each alloy is similar to that of the 510°C specimens. The swelling behavior of specimens irradiated at 510°C and 615°C is quite similar to each other in V–Fe alloys. The swelling level was up to

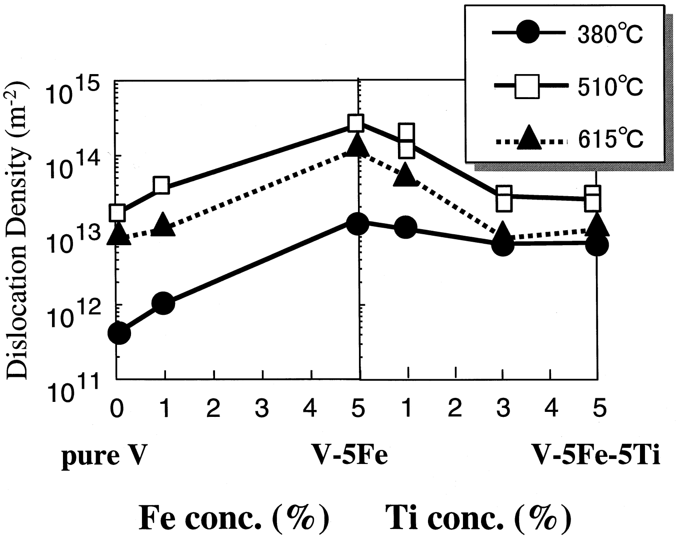

Fig. 4. Dislocation density as a function of the iron concentration in vanadium and titanium concentration in V–5Fe.

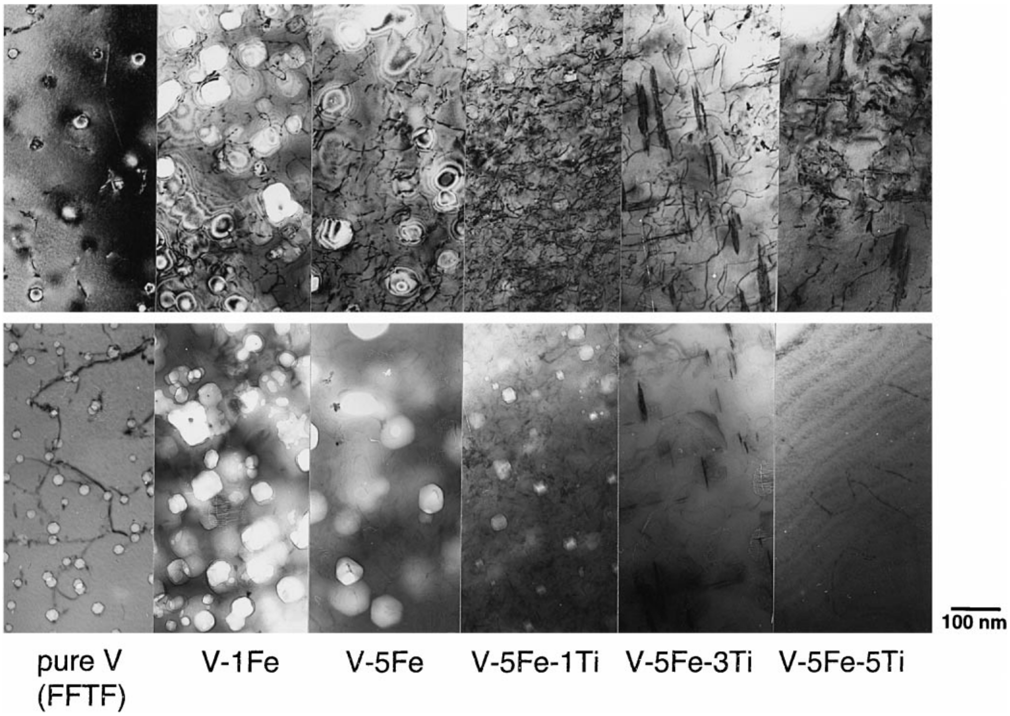

Fig. 5. Typical example of V–Fe and V–Fe–Ti alloys irradiated in EBR-II to 11 dpa at 510°C. The arrangements of the figure is the same as in Fig. 1.

30% in V–5Fe. A large swelling occurred in V–5Fe–1Ti, but was entirely suppressed in V–5Fe–3Ti. On the other hand, their precipitation behavior is different. No precipitates were seen at any location in any of the V–Fe–Ti alloys irradiated at 615°C and the dislocation density is also very low.

4. Discussion

Fig. 7 shows the result of swelling obtained in V–Fe and V–Fe–Ti alloys irradiated at 385°C, 510°C and 615°C. From these results, it is evident that swelling is very clearly correlated with addition of iron and titani-

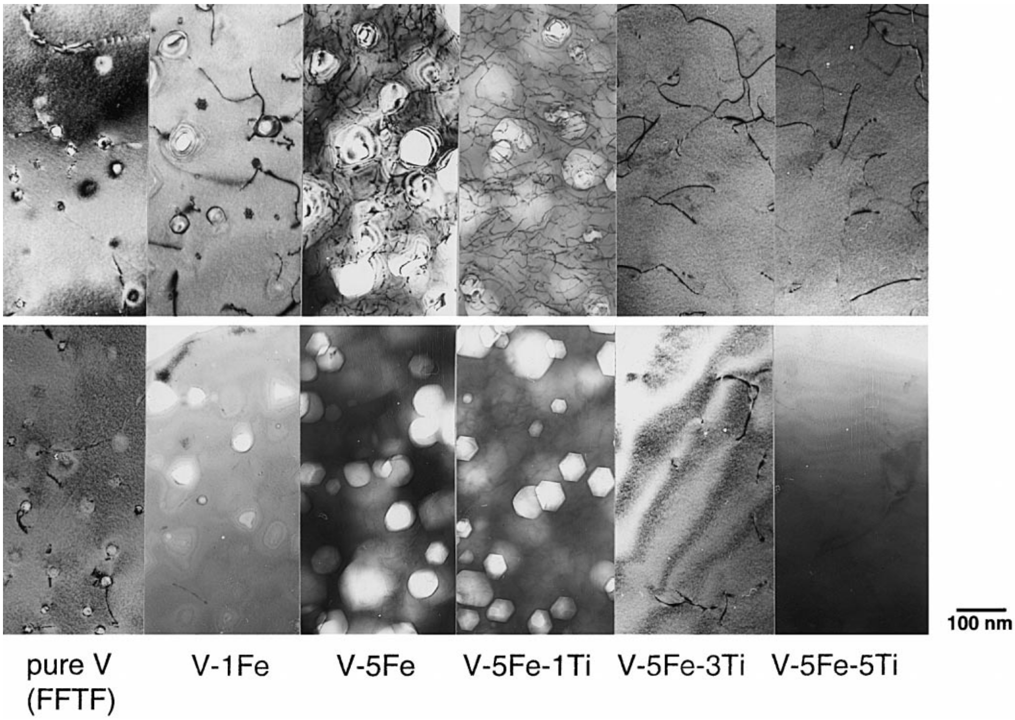

Fig. 6. Typical example of V–Fe and V–Fe–Ti alloys irradiated in EBR-II to 11 dpa at 615°C. The arrangements of the figure is the same as in Fig. 1.

um elements. Small solute, i.e. iron, yields large swelling, even though the amount of iron addition is as small as 1 at.%. The mechanism for the large swelling observed in alloys with undersized solute has been shown to be due to the high dislocation bias. The large dislocation bias in alloys with undersized solutes has been deduced from a molecular dynamics calculation of self-interstitial atoms in the vicinity of an edge dislocation [7]. From 510°C and 615°C, swelling seems to be already saturated at 1 at.% iron addition as well as rapid cavity growth. From ion irradiation experiments, it has been observed that rapid cavity growth occurred from 1 to 3 at.% iron concentration [8]. Although it is difficult to compare neutron irradiation experiments and ion irradiation ones directly, there is considered to be good agreement on the cavity formation and growth of both neutron and ion irradiation.

On the other hand, addition of titanium solute to vanadium suppresses swelling. The mechanism of suppression has been explained by considering the size of

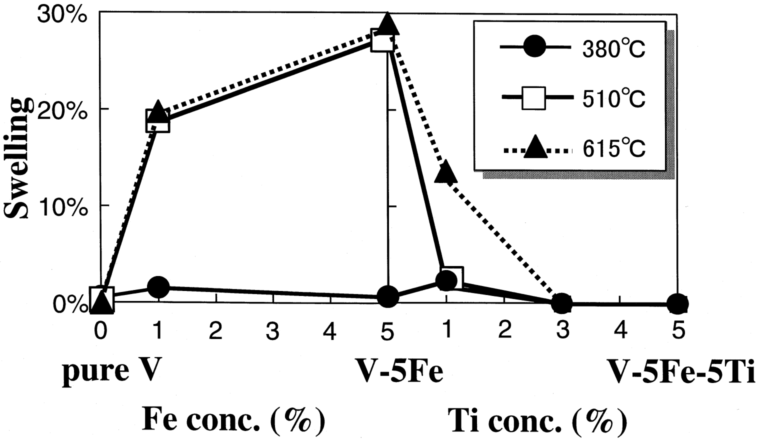

Fig. 7. Void swelling as a function of iron concentration in vanadium and titanium concentration in V–5Fe.

the solute atom. Oversize solutes trap excess vacancies and increase the vacancy concentration leading to the increase in the V-I recombination fraction. The increase in the V-I recombination and slow migration of vacancy due to oversized solute trap make nucleation of cavity suppressed. It has been reported that radiation-induced precipitation of titanium oxide or titanium silicate play an important role in cavity formation and swelling suppression [6]. At 510°C in this study, a lot of radiation-induced titanium oxide precipitates were formed in V–Fe–Ti alloys. No precipitates or cavities, however, were observed in V–Fe–Ti alloys irradiated at 615°C. It is suggested therefore that radiation-induced precipitates with titanium is not essential for suppression of cavity formation. It will be shown later that titanium in solution is more effective for suppression of cavity formation and swelling. In a previous study, it has been observed that large swelling occurred in V–5Nb irradiated at 800°C in FFTF. Niobium is an oversized solute atom in vanadium and its atomic size factor is even greater than titanium. Under the same irradiation condition as this V–Nb alloy, V–5Ti and V–3Ti–1Si did not show any cavity formation [8]. Thus, it is difficult to understand these results in terms of the correlation of atomic size factor and swelling. This discrepancy is considered to be caused by some different bonding system between vanadium atoms and solute atoms from atomic size factor. However, it is exceptional phenomena at higher temperature than this issue, and atomic size factor significantly influences microstructural evolution in vanadium alloys up to 600°C.

5. Conclusion

Huge swelling appeared in vanadium alloys with >1 at.% Fe irradiated at 510°C and 615°C to 11 dpa. This huge swelling is found to be caused by enormous cavity growth. On the other hand, addition of titanium to V–5Fe remarkably reduced the swelling and three atomic percent addition of titanium in completely exterminated cavity formation. The dominant factor of swelling suppression is not radiation-induced precipitates but the presence of an oversized atoms in solution. Addition of titanium is effective for reducing swelling in vanadium even small amounts.

Acknowledgements

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