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Section 7. Effects of gaseous species on structural materials

Effect of helium production on swelling of F82H irradiated in HFIR

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Abstract

The effects of helium production and heat treatment on the swelling of F82H steel irradiated in the HFIR to 51 dpa have been investigated using ^{10}B, ^{58}Ni and ^{60}Ni-doped specimens. The swelling of tempered F82H-std and F82H doped with ^{10}B irradiated at 400°C ranged from 0.52% to 1.2%, while the swelling of the non-tempered F82H doped with ^{58}Ni or ^{60}Ni was less than 0.02%. At 300°C the swelling in all steels was insignificant. In the F82H + Ni, a high number of density carbides formed in the matrix at these temperatures. The production of helium atoms enhanced the swelling of the F82H steel. However, the non-tempered treatment for the F82H + Ni suppressed remarkably the swelling. The cause of low swelling in the F82H + Ni may be due to the occurrence of the high density of carbides acting as sinks or the decrease of mobility of vacancies interacting with carbon atoms in matrix.

1. Introduction

Low-activation ferritic/martensitic steels are candidate materials for the first wall and blanket structure of fusion reactors. The high-energy neutrons produced in the D–T fusion reaction induce displacement damage and generate gas atoms from (n, p) and (n, α) reactions in the materials. In order to examine the effect of gas atoms on materials properties, one is possible to use the reaction of ^{10}B (n, α)^{7}Li or ^{58}Ni (n, γ)^{57}Ni (n, α)^{56}Fe in steels doped with ^{10}B or ^{58}Ni and irradiating with thermal neutrons in a mixed-spectrum fission reactor, such as the high flux isotope reactor (HFIR). The effect of neutron irradiation and helium production on mechanical properties of F82H steel has been reported [1–3]. Recently, the swelling of F82H and other 7–9Cr low-activation ferritic steels irradiated at 430°C to 67 dpa in FFTF was reported by Morimura et al. [4]; the swelling of F82H was 0.1%, and that of the other steels was between 0.1% and 0.7%. Maziasz and Klueh [5] had investigated the swelling versus helium production rate in 9Cr–1MoVNb (−0, −2Ni) and 12Cr–1MoVW(−0, −2Ni) steels irradiated at 400°C to 47 dpa, using the efficiency of helium production rate for HFIR and FFTF, and they indicated that the swelling of 0.1–0.4% depended on helium production. Purpose of the present study is to investigate the swelling behavior of F82H steel irradiated in the HFIR at different helium-production rates and tempering treatments, using isotope elements of ^{10}B and ^{58}Ni.

2. Experimental procedure

The F82H + natural B and F82H + ^{10}B steels were prepared from F82H doped with 0.0060% natural boron and 0.0058% ^{10}B with 94.37 at.% purity level, respectively. The F82H + ^{58}Ni and F82H + ^{60}Ni steels were prepared from F82H doped with isotopes ^{58}Ni and ^{60}Ni, respectively. The F82H-std and the F82H + B specimens

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were first normalized at 1040°C for 30 min in a vacuum followed by air-cooling. In the F82H + Ni specimens, the normalizing was performed at 1200°C for 2 h. After that, the F82H-std and the F82H + B specimens were tempered at 740°C for 2 and 1.5 h, respectively, in a vacuum followed by air-cooling; however, no tempering was performed on the F82H + Ni steels. The slight difference in tempering times for the F82H-std and F82H + B should not produce significant differences in the microstructures of the steels. However, the lack of a temper for the F82H + Ni steel will have implications on the microstructure, and these will be discussed.

The chemical compositions of the specimens used in this study are given in Table 1. As a comment, the F82H-std, F82H + ^{58}Ni and F82H + ^{60}Ni steels contained boron impurities of 8, 9 and 9 ppm, respectively, and the helium production rates during the irradiation for the F82H-std and F82H + ^{60}Ni steels were very close to each other.

Standard 3-mm diameter TEM disks with 0.25-mm thickness were irradiated in the HFIR target in the HFIR-MFE-JP12 capsule as part of the JAERI/US collaborative program. The exposure was 64904 MWd at 85 MW reactor power and achieved a fluence of 51 dpa for the F82H-std steel [6,7]. The neutron fluence was 1.1×10^{23} n/cm^2 ($E > 0.1$ MeV). The irradiation temperatures were 300°C and 400°C, and He generation rate estimated in these steels during irradiation is given in Table 2.

After the irradiation, the microstructures of these specimens were examined using a JEM-2000FX transmission electron microscope with a LaB_6 gun operated at 200 kV.

3. Results

3.1. Microstructures of the F82H steels before irradiation

In the tempered F82H-std steel, the dislocation density was about 1.0×10^{14} m^{-2}. $M_{23}C_6$ carbides were observed in the matrix and on grain boundaries, and the number density and mean size were 6×10^{19} m^{-3} and about 73 nm, respectively. Only a few MC carbides were observed in the matrix, and the number density and mean size were $<1 \times 10^{20}$ m^{-3} and about 14 nm, respectively. The mean width of the lath structure was about 440 nm. Microstructures of B-doped specimens were very similar to that of the F82H-std steel. On the other hand, the microstructures of the untempered Ni-doped specimens were different from the other specimens. The specimens had MC carbide with a number density of 1×10^{21} m^{-3} and a mean size about 15 nm, a very few M_6C carbides about 20 nm in size formed on lath boundaries, and unidentified precipitates with a rod-shape about 100 nm in length which were sometimes

Table 1
Chemical compositions of the specimens used in this study

	Cr	C	Si	V	Mn	Ta	W	B	Ni (wt%)	^{10}B	^{11}B	^{58}Ni	^{60}Ni (at.%)
F82H-std	7.44	0.1	0.14	0.20	0.49	0.04	2	0.0008	–	0.0008	0.0034	–	–
F82H + natural B	7.49	0.099	0.15	0.20	0.50	0.04	2.1	0.0060	–	0.0059	0.0252	–	–
F82H + ^{10}B	7.23	0.098	0.17	0.22	0.50	0.04	2.1	0.0058	–	0.0305	0.0018	–	–
F82H + ^{58}Ni	7.8	0.04	0.2	0.2	0.4	0.04	2.1	0.0009	1.4	0.0009	0.0038	1.31	0.0045
F82H + ^{60}Ni	7.8	0.04	0.2	0.2	0.4	0.04	2.1	0.0009	1.4	0.0009	0.0038	0.019	1.27

Table 2
Mean helium production rate (appmHe/dpa) in these steels during irradiation (calculation)

	F82H-Std	F82H + nB	F82H + ^{10}B	F82H + ^{58}Ni	F82H + ^{60}Ni
0–51 dpa	0.5	1.5	6.5	9.9	0.5
0–1 dpa	8.7	60	305	9.9	8.3
1–51 dpa	0.3	0.3	0.3	9.9	0.3

observed in the matrix of some grains. The dislocation density in the Ni-doped steels was about 6×10^{14} m^{-2}, and the width of lath boundaries was about 390 nm.

3.2. Swelling of F82H steels irradiated at 400°C to 51 dpa

Figs. 1(a)–(c) show cavities observed in the F82H-std, F82H + natural B (F82H + nB), and F82H + ^{10}B steels, respectively, irradiated at 400°C to 51 dpa in the HFIR. A few lath boundaries and many $M_{23}C_6$ precipitates can be seen in these micrographs. In Fig. 1(a), cavities cannot be seen near the lath boundaries, while in Figs. 1(b) and (c) cavities can be observed even near these lath boundaries. The swelling, the cavity number density and the root mean cube (RMC) of cavity radius for the F82H-std steel were 0.52%, 6.1×10^{20} m^{-3} and 12.7 nm, respectively, in average of all regions. These in the matrix region apart from lath boundaries were about 0.74%, 8×10^{20} m^{-3} and 13 nm, respectively. The average swelling in all regions of F82H + nB and F82H + ^{10}B steels is 1.2% and 1.1%, respectively. The number densities and RMC radius of cavities in these steels were higher and smaller than those in the F82H-std steel, respectively. The growth of $M_{23}C_6$ carbides was observed particularly on lath boundaries at non-bending curves in F82H-std, F82H + nB, and F82H + ^{10}B steels. The dislocation densities in F82H-std, F82H + nB, and F82H + ^{10}B steels are 1.7×10^{14}, 2.5×10^{14}, and 3.1×10^{14} m^{-2}, respectively and these were higher than those before irradiation. Many small M_6C precipitates were formed on the $M_{23}C_6$ carbides, and a few M_6C precipitates were formed in the matrix by the irradiation. A few dislocation loops were observed in the F82H-std steels, and the number density was 6.5×10^{20} m^{-3}, and the density of loops in the F82H + nB and F82H + ^{10}B steels was slightly higher than the F82H-std steel.

Figs. 2(a) and (b) show microstructures of F82H + ^{60}Ni and F82H + ^{58}Ni steels, respectively, irradiated at 400°C to 51 dpa. Small cavities were observed for the F82H + ^{58}Ni steel and the swelling, the number densities, and RMC radius of cavities were 0.002%, 2×10^{21} m^{-3} and 2.9 nm, respectively, but no cavities were observed for the F82H + ^{60}Ni steel. In the Ni-doped F82H + ^{60}Ni and F82H + ^{58}Ni steels, a high density of precipitates about 2×10^{22} m^{-3} were formed by the irradiation as seen in Fig. 2. The precipitates were mainly M_6C carbide, and two unidentified phases were also formed. The detail analysis and result for precipitates formed in the F82H + Ni steels irradiated at 400°C and 300°C will be reported by Ref. [8]. Dislocation loops with an order of 10^{20} m^{-3} and a mean size of a few 10 nm was also observed in these steels. The dislocation density was about 7×10^{14} m^{-2} in the F82H + Ni steels.

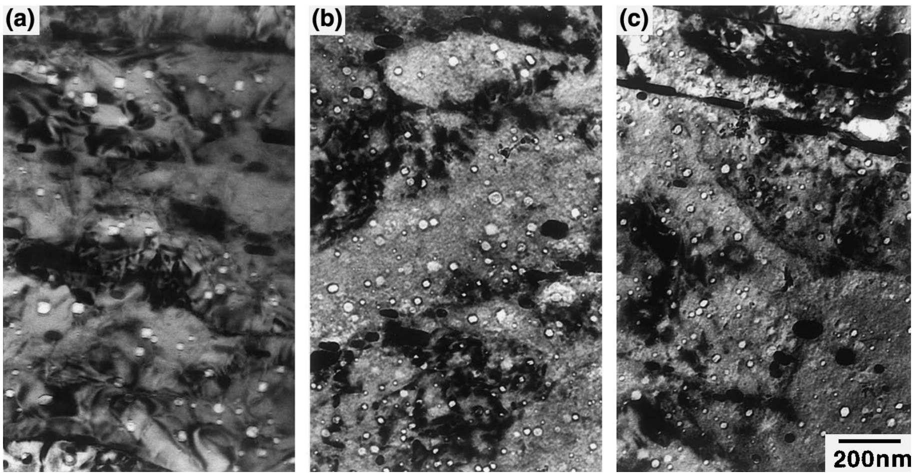

Fig. 1. Cavities formed in the (a) F82H-std (near 100 pole), (b) F82H + natural B (near 110 pole), and (c) F82H + ^{10}B (near 111 pole) steels irradiated at 400°C to 51 dpa. In the F82H-std, cavities cannot be seen near the lath boundaries, while in the F82H + natural B and F82H + ^{10}B steels cavities can be observed near these lath boundaries.

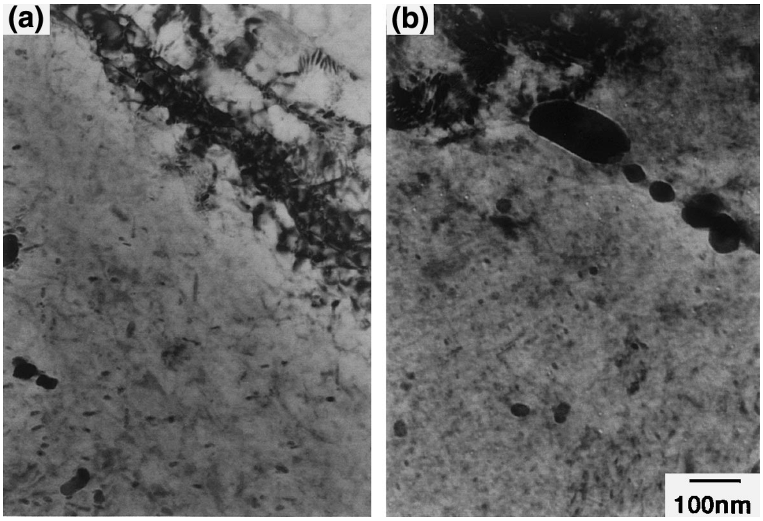

Fig. 2. Microstructure of (a) F82H + ^{60}Ni, and (b) F82H + ^{58}Ni steels irradiated at 400°C to 51 dpa. Cavities were formed in the F82H + ^{58}Ni steel, but no cavities were formed in the F82H + ^{60}Ni steel. A high number density of precipitates was also formed in F82H + Ni steels.

3.3. Swelling of these F82H steels irradiated at 300°C to 51 dpa

Figs. 3(a)–(e) show microstructures of F82H-std, F82H + nB, and F82H + ^{10}B steels, F82H + ^{60}Ni and F82H + ^{58}Ni, respectively, irradiated at 300°C to 51 dpa. Cavities were observed in only the F82H + ^{10}B and F82H + ^{58}Ni steels, and the values of swelling were 0.002% and 0.02%, respectively. The growth of $M_{23}C_6$ carbides was observed on lath boundaries in F82H-std, F82H + nB, and F82H + ^{10}B steels, and many small M_6C precipitates were also formed on the $M_{23}C_6$ carbides. MC carbides with a density of 3×10^{22} m^{-3} and the mean size of 3.7 nm were induced by the irradiation in F82H + Ni steels. Many dislocation loops were formed in all steels. The dislocation density in these steels seems to be unchanged during irradiation.

A summary of the results for the microstructures of these irradiated steels is given in Table 3.

4. Discussion

The present swelling data, Miwa's low damage data [9] for the F82H steels irradiated at 400°C in the HFIR and Morimura's data [4] for the F82H steel irradiated at 430°C to 67 dpa in the FFTF are summarized in Fig. 4. This figure shows the dependence of swelling on the

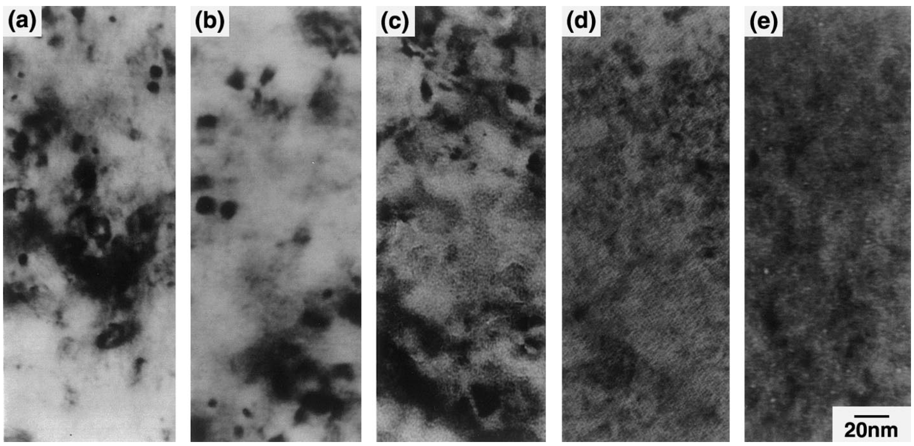

Fig. 3. Microstructure of (a) F82H-std, (b) F82H + nB, (c) F82H + ^{10}B, (d) F82H + ^{60}Ni, and (e) F82H + ^{58}Ni steels irradiated at 300°C to 51 dpa. Small cavities were observed in only the F82H + ^{10}B and F82H + ^{60}Ni steels.

Table 3
Summary of radiation-induced defect clusters formed at 300°C and 400°C to 51 dpa in the F82H steels. The number density and mean size of defect clusters denote as N and d, respectively. RMC radius and S denote root mean cube of cavity radius and swelling, respectively

Alloy	300°C–51 dpa			400°C–51 dpa		
	Loop (N, d)	Cavity (N, RMC, S)	Precipitate	Loop (N, d)	Cavity (N, RMC, S)	Precipitate
F82H	4×10^{22} m^{-3}	No	M_6C on $M_{23}C_6$	6.5×10^{20} m^{-3}	6.1×10^{20} m^{-3}	M_6C on $M_{23}C_6$, few M_6C in matrix
	10 nm		$M_{23}C_6$ growth on lath boundary	27 nm	12.7 nm, 0.52%	$M_{23}C_6$ growth
F82H + nB	5×10^{22} m^{-3}	No	M_6C on $M_{23}C_6$	Order of 10^{20} m^{-3}	2.4×10^{21} m^{-3}	M_6C on $M_{23}C_6$, few M_6C in matrix
	11 nm		$M_{23}C_6$ growth on lath boundary	A few 10 nm	10.6 nm, 1.2%	$M_{23}C_6$ growth
F82H + ^{10}B	6×10^{22} m^{-3}	2.4×10^{21} m^{-3}	M_6C on $M_{23}C_6$	Order of 10^{20} m^{-3}	6.1×10^{21} m^{-3}	M_6C on $M_{23}C_6$, few M_6C in matrix
	11 nm	1.3 nm, 0.002%	$M_{23}C_6$ growth on lath boundary	A few 10 nm	7.6 nm, 1.1%	$M_{23}C_6$ growth
F82H + ^{58}Ni	1×10^{23} m^{-3}	4.3×10^{21} m^{-3}	Fine MC in matrix (3×10^{22} m^{-3})	Order of 10^{20} m^{-3}	2.0×10^{21} m^{-3}	Fine M_6C in matrix (2×10^{22} m^{-3})
	12 nm	2.0 nm, 0.02%		A few 10 nm	2.9 nm, 0.02%	Others (low density)
F82H + ^{60}Ni	1×10^{23} m^{-3}	No	Fine MC in matrix (3×10^{22} m^{-3})	Order of 10^{20} m^{-3}	No	Fine M_6C in matrix (2×10^{22} m^{-3})
	10 nm			A few 10 nm	0%	Others (low density)

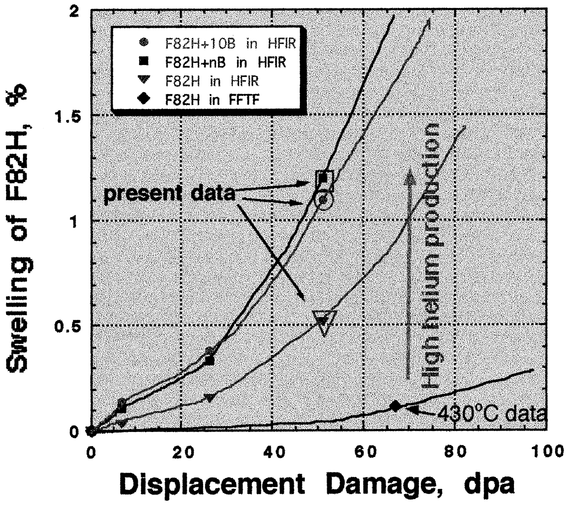

Fig. 4. Swelling behavior of the F82H steel irradiated at 400°C to 51 dpa on helium production and displacement damage. The figure includes Miwa's data in HFIR at 400°C [9] and Morimura's data in FFTF at 430°C [4].

displacement damage and helium production in the F82H-std steels. With increasing displacement damage and helium production, the swelling tended to increase. Swelling behavior [10–12] is affected by the net bias or vacancy supersaturation, and helium production rate. A point defect partitioning factor Q for either vacancies or interstitials is described as below:

$$Q = Z^{\mathrm{d}}\ N_{\mathrm{d}}/4\pi\ rN_{\mathrm{c}}\ Z^{\mathrm{c}}, \quad (1)$$

where the subscripts of d and c represent dislocation including dislocation loops and cavities, respectively; N and Z are the concentration or number density and the capture efficiency of the cluster for interstitials and vacancies, respectively.

Using Eq. (1), the F82H-std irradiated at 400°C to 51 dpa in the HFIR must have $Q \sim 1.4$ which is close to unity using following parameters: dislocation density is 1.7×10^{14} m^{-2}; number density and mean size of dislocation loops: 6.5×10^{20} m^{-3} and 27 nm; the number density and RMC radius of cavities apart from lath boundaries are 8×10^{20} m^{-3} and 13 nm, respectively; and Z^{d} and Z^{c} are approximately one. The Q values of the F82H + nB and F82H + ^{10}B steels irradiated at 400°C to 51 dpa are about 1.0 and 0.8, respectively, and they are nearly unity. Therefore, the increase of swelling in these steels can be expected to continue [10] as shown in Fig. 4. At 300°C to 51 dpa, cavities were observed in the F82H + ^{10}B steels but they could not be observed in the F82H-std and F82H + nB steels. The value Q is estimated at about 60 in the F82H + ^{10}B steels irradiated at 300°C to 51 dpa, so the dislocation sink density clearly dominates. For the F82H-std and F82H + nB steels irradiated at 300°C, the situation would be further dominated by the dislocation sink density, because of the difficulty for the formation and growth of cavities due to a low production of helium atoms in these steels.

In the F82H + ^{58}Ni steel irradiated at 400°C to 51 dpa, the Q is about 9 which is larger than that for the F82H-std, F82H + nB and F82H + ^{10}B steels, and this value might arise from control due to show dislocation sink density. The swelling of F82H + ^{58}Ni steel at 400°C and 300°C was very low, despite having the highest helium production in this study, nor were cavities observed in the F82H + ^{60}Ni steels. All of these F82H-std, F82H + nB, F82H + ^{10}B, F82H + ^{58}Ni and F82H + ^{60}Ni steels were irradiated near each other in the same capsule of HFIR-JP12. Moreover, the helium production rates for the F82H-std and F82H + ^{60}Ni steels were very similar. Therefore, the difference in swelling behavior between these untempered F82H + Ni steels and the series of tempered F82H-std, andF82H + B steels would be very difficult to explain by only based on the difference in production rate of helium and the Q parameter. We must take into consideration other factors such as the formation and growth of precipitates during irradiation and the difference of the initial microstructures in these steels before irradiation. For example, in the F82H + Ni steels irradiated at 400°C to 51 dpa in the HFIR, a high density fine M_6C carbides were formed. The formation of M_6C carbides at 400°C was also reported in similar ferritic steels of Fe–9Cr–Mo–Nb and Fe–9Cr–Mo–Nb–2Ni alloys [5], but the number density of the carbides was lower than in present work. The formation of fine carbides at a high density may be suggested because carbon atoms remained in the matrix due to the non-tempered treatment before irradiation for the F82H + Ni steels, and also the increase in neutral sinks due to the high-density of carbides formed would enhance mutual recombination on these carbides. Carbon atoms in the matrix would interact strongly with vacancies, so that the mutual recombination could be increased or vacancy mobility might be decreased during the irradiation. Therefore, these microstructural changes are thought to be the cause of low swelling in the F82H + Ni steels.

5. Conclusion

Swelling of tempered F82H-std, F82H steels doped with natural boron (311 appm), isotope ^{10}B (305 appm) and non-tempered F82H steels doped with 1.31 at.% ^{58}Ni, and 1.27 at.% ^{60}Ni irradiated at 300°C and 400°C to 51 dpa in the HFIR have been examined by TEM.

1. Swelling at 400°C ranged from 0% in the F82H + ^{60}Ni steel to 1.2% in the F82H + nB steel.
2. Swelling at 300°C ranged from 0% in the F82H-std, F82h + nB, and F82H + ^{60}Ni steels to 0.02% in F82H + ^{58}Ni steel.

3. The production of helium atoms enhanced the cavity formation of the F82H steel.
4. The non-tempered treatment for the F82H + Ni suppressed remarkably the swelling.
5. The cause of low swelling in the F82H + Ni may be due to the occurrence of high density carbides acting as sink or the decrease of mobility of vacancies interacted with carbon atoms in matrix.

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