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Reduction method of DBTT shift due to irradiation for reduced-activation ferritic/martensitic steels

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ABSTRACT

The method for reducing irradiation-induced DBTT shift of reduced-activation ferritic/martensitic steels was examined. F82H-LN (low nitrogen, 20 ppm), F82H+60 ppm11B+200 ppmN and F82H+60 ppm10B+200 ppmN steels tempered at 780 °C for 0.5 h were irradiated at 250 °C to 2 dpa, and the results for Charpy impact tests were analyzed. The upper shelf energy of F82H+^{11}B+N steel was hardly changed by the irradiation, and DBTT shift was very small. From our research, DBTT shift due to irradiation can be reduced by the control of tempered conditions before irradiation, and it is found to be furthermore reduced by impurity doping with 60 ppm^{11}B and 200 ppmN to F82H steel.

1. Introduction

Resistance of irradiation embrittlement of reduced-activation ferritic/martensitic steels is a high priority theme of R&D for the target window materials of spallation targets in an accelerator-driven transmutation system (ADS) and structural materials of fusion DEMO reactors and IFMIF (International Fusion Materials Irradiation Facility) target. Large shifts of DBTT due to irradiation have been reported in several martensitic steels, which had different concentrations in some elements and were also tempered at different temperatures. The improvement of resistances to irradiation embrittlement and hardening will be required. Several researchers [1–7] reported that the increase of yield strength and the shift of DBTT were different in Fe–9Cr alloy and several martensitic steels such as F82H, JLF-1, JLF-1B, ORNL 9Cr–2WVTa, OPTIFER Ia, II, MANET II and Mod.9Cr–1Mo, which had different concentrations in some elements and were tempered at different temperatures. The effects of the normalizing and tempering on tensile and impact behavior in martensitic steels before irradiation were reported by Schafer [8] and Gondi [9]. The mechanisms for the relation between the changes of yield strength and shift of DBTT due to irradiation in these martensitic steels are not clear, and it is necessary to reveal the effects of heat treatment on irradiation hardening and embrittlement [10–12].

The roles of boron addition were investigated to obtain higher strength and superior toughness of weld bond of large heat-input welded steel plane. The effect of additions of B, N and Ti elements on the toughness and microstructures was very effective [13–15]. The toughness of steels is easily affected by the segregation and precipitation of boron and boron nitride [16,17].

The purpose of this study is to examine the mechanism of irradiation embrittlement and hardening. In this study, one of two approaches for increasing the resistance of irradiation embrittlement of reduced-activation ferritic/martensitic steels has been applied for it. The former is the method of modification of heat treatments which are changed with tempered conditions, and the latter is the impurity doping with proper heat treatment which is applied in this study.

2. Experimental procedure

The specimens used in this study are t/2-1/3CVN (Charpy V-notch specimen with half thickness) Charpy impact test specimens of F82H-LN (low nitrogen, 20 ppm), F82H+60 ppm11B+200 ppmN and F82H+60 ppm10B+200 ppmN steels. The chemical compositions of the specimens are given in Table 1. The purities of ^{10}B and ^{11}B isotopes were 95% and 99%, respectively. The shape and size of t/2-1/3CVN specimen is given in Fig. 1. The plate of F82H+B+N steel was also heat-treated under some conditions as below: N&T&N&T treatment (normalized for 0.5 h at 1150 °C, water-quenching and followed by tempering for 2 h at 700 °C), normalizing for 10 min at 950 °C (or 1000 and 1040 °C), water-quenching and followed by tempering for 0.5 h at 780 °C. The objective of the first normalizing at 1150 °C was to solve BN clusters somewhat in matrix and to reduce the size of BN clusters and that of the first tempering was to reduce boron segregation at grain boundaries and to collect boron in high-number density carbides.

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Table 1
The chemical compositions of the specimens used in this study (mass%).

Alloy	B	N	Cr	W	C	O	Al
F82H	0.0002	0.0023	7.92	1.97	0.099	0.004	0.004
F82H+^{11}B+N	0.0057	0.0200	7.83	2.09	0.099	<0.01	<0.001
F82H+^{10}B+N	0.0059	0.0190	8.09	2.10	0.099	<0.01	<0.001
	Si	P	S	Ti	V	Mn	Ta
F82H	0.11	0.007	0.001	0.006	0.18	0.10	0.05
F82H+B	0.10	0.006	0.001	<0.002	0.30	0.10	0.042
F82H+B+N	0.099	0.006	0.001	<0.002	0.30	0.10	0.039

The second normalizing was performed to reduce grain sizes and to transfer boron atoms from carbides to BN, and the second tempering was also performed to trap free boron atoms in matrix into carbides. The distributions of boron, boron nitride and oxygen were measured by a secondary ion mass spectrometry (SIMS) in a previous work [18,19], and the uniformly distribution of boron was observed in the specimen. The ductile–brittle transition temperature of the specimens was measured by Charpy impact tests.

Irradiation was carried out in the Japan Materials Test Reactor (JMTR) to a displacement damage value of 1.9 dpa, nominally at 250 °C. After the irradiation, Charpy impact tests were performed as a function of test temperatures.

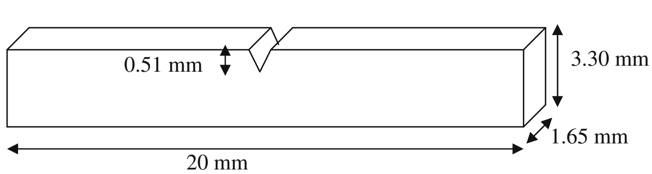

Fig. 1. Shape and size of t/2-1/3CVN Charpy impact specimen.

3. Results and discussion

In previous studies, DBTT shift due to irradiation depended on the tempering conditions [11,22] as shown in Fig. 2. ΔDBTT due to irradiation in the F82H steels irradiated at 250 °C tended to decrease with increasing time from 0.5 to 10 h and temperature of tempering from 750 to 780 °C. This result means that the DBTT shift due to irradiation can be controlled by the heat treatments before irradiation, and the temperature and time of tempering conditions are better in somewhat higher and longer. We would remark in here that the DBTT shift of the standard heat treatment of F82H, i.e., 750 °C for 1 h, was largest, but it was the smallest for the irradiation hardening. The smallest DBTT shift was found in the specimen tempered at 780 °C for 0.5 h.

In order to suppress the irradiation embrittlement of reduced-activation ferritic/martensitic steels, it was applied a combined method of the impurity additions of boron and nitrogen atoms and the proper heat treatment described in above paragraph. Fig. 3a and b shows DBTT before and after the irradiation at about 250 °C to about 2 dpa in F82H-LN, F82H+^{11}B+N and F82H+^{10}B+N steels. The optical micrographs of fracture surface of these specimens after Charpy impact tests are shown in Fig. 4. The upper shelf energy of F82H+^{11}B+N steel was hardly changed by the irradiation. As shown in Fig. 4e and f, brittle fracture surfaces were observed in F82H+^{10}B+N steel tested at 20 °C and −50 °C in the Charpy impact tests, and these specimen have about 300 appm-He which is produced from ^{10}B(n, α)7Li reaction. The fracture properties of F82H-LN and F82H+^{11}B+N tested at about −60 °C were ductile as seen in Fig. 4b and d. The DBTT shift of F82H, F82H+^{11}B+N and F82H+^{10}B+N steels are 70, 25, 145 °C, respectively. The shifts of F82H-LN and F82H+^{11}B+N were induced by irradiation damage, and the shift of F82H+^{10}B+N steel was induced by irradiation damage and helium production. The upper shelf energy of the irradiated F82H+^{11}B+N steel was hardly changed by the irradiation, but these of F82H-LN and F82H+^{11}B+N steels were reduced. The hardness and tensile behaviors of these specimens were previously examined [20,21]. The ratio of DBTT shift to irradiation hardening of F82H, F82H+^{11}B+N and F82H+^{10}B+N steels are about 0.3, 0.2 and 0.6 °C/MPa, respectively, as given in Fig. 5. The ratios could be changed by helium production and impurity doping such as boron and nitrogen atoms.

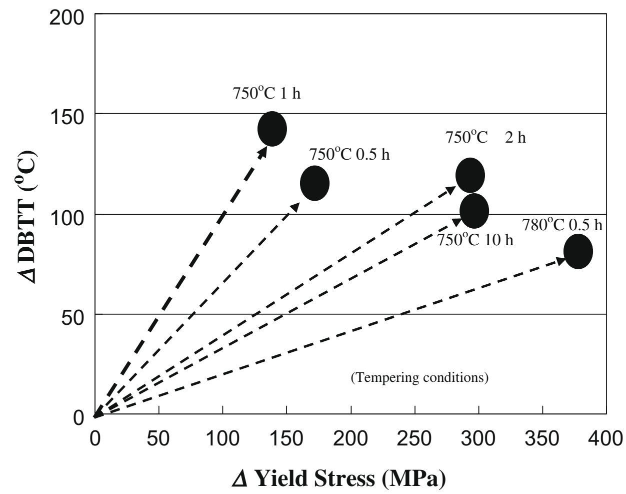

Fig. 2. Relation of ΔDBTT and ΔYS due to irradiation in F82H tempered with different conditions [11].

The effect of helium production on DBTT shift due to 300 appm-He production could be evaluated from the difference between the shift of F82H+^{11}B+N and F82H+^{10}B+N, and it can be estimated as 120 °C, and the ratio of DBTT shift due to helium production is about 0.40 °C/appm-He, and this is nearly the same as our previous data of about 0.33 °C/appm-He, which was obtained by helium implantation experiment by cyclotron [22]. We have to mention some other previous reports; Rieth et al. [3] showed the dependence of the shift in DBTT on helium production using several martensitic steels with different chromium concentrations, and the ratio of the shift on DBTT to helium production was 2.5 °C/appm-He. However the result might be included the effect of chromium concentration on the shift in DBTT and the rate would be thought to be a smaller value; In order to eliminate the chemical effect of doped element of Ni on the DBTT of martensitic steels to produce helium from a reaction of ^{58}Ni(n, γ)59Ni(n, α)56Fe, Kleuh compared the results of martensitic steels between the HFIR and EBR-II experiments, and the ratio of the shift in DBTT to helium production was relatively higher value of about from 1 to 4 °C/appm-He [23]. In our study, the chemical effect of boron and displacement damage on DBTT was eliminated by using isotope dope technique of the comparing the result for the specimens of F82H+60 ppm11B+N and F82H+60 ppm10B+N.

The DBTT shift due to irradiation of F82H+60 ppm11B+N steel, with proper tempering condition at 780 °C for 0.5 h, irradiated at 250 °C to 2 dpa is very small and the upper shelf energy is hardly changed by it. Therefore, it is found that F82H+60 ppm11B+N steel with proper tempered treatment has a very good property and potential for increasing the resistance to irradiation embrittlement. From our studies, it is found that the ratios of DBTT shift to irradiation hardening depend on helium production, impurity doping and tempering conditions before irradiation.

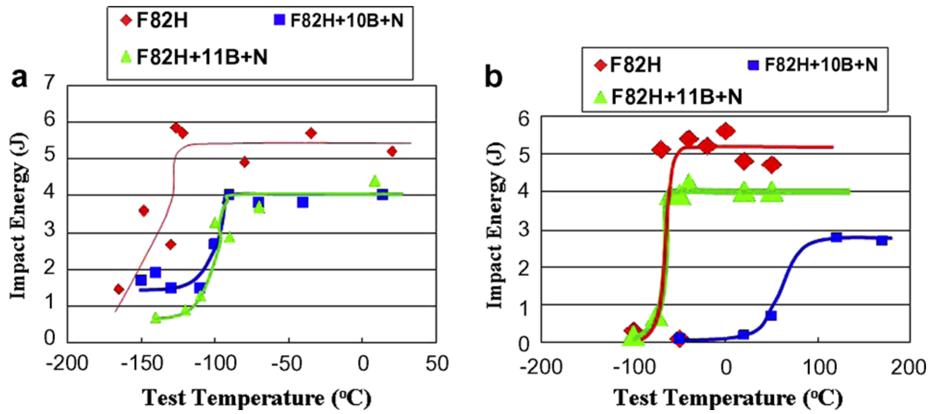

Fig. 3. Impact energies as a function of test temperatures for t/2-1/3CVN type specimens of F82H-LN (low nitrogen, 20 ppm), F82H+60 ppm^{11}B+200 ppmN and F82H+60 ppm^{10}B+200 ppmN steels tempered at 780 °C for 0.5 h (a), and after irradiation about 250 °C to 2 dpa (b).

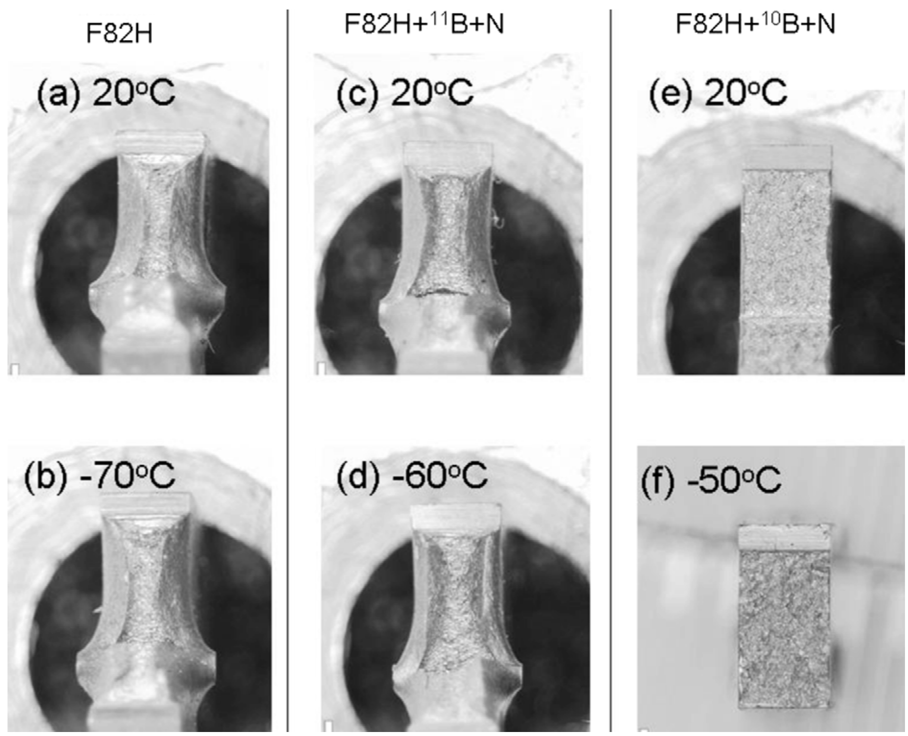

Fig. 4. The optical micrographs of (a) and (b) F82H-LN (low nitrogen, 20 ppm), (c) and (d) F82H+60 ppm^{11}B+200 ppmN and (e) and (f) F82H+60 ppm^{10}B+200 ppmN steels.

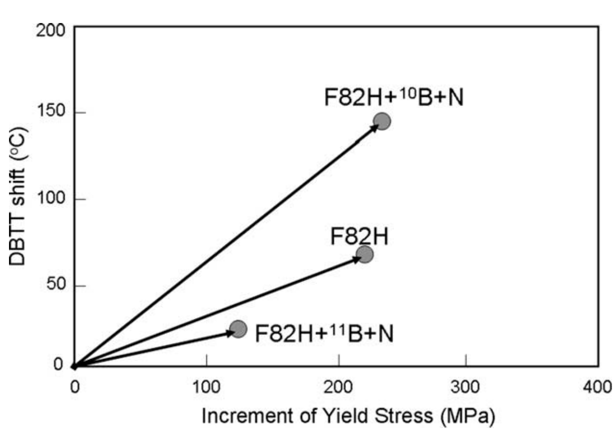

Fig. 5. The relationship between DBTT shift due to irradiation and increment of yield stress.

4. Conclusion

The method for increasing the resistance of irradiation embrittlement of reduced-activation ferritic/martensitic steels was examined. Irradiation experiments of F82H-LN (low nitrogen, 20 ppm), F82H+60 ppm11B+200 ppmN and F82H+60 ppm10B+200 ppmN steels tempered at 780 °C for 0.5 h were performed at 250 °C to 2 dpa using t/2-1/3CVN specimens, and the results for Charpy impact tests were analyzed. The main contents of this study are described as below:

(1) The upper shelf energy of F82H+^{11}B+N steel was not evidently changed by the irradiation.
(2) DBTT shift of F82H+^{11}B+N steel was very small after the irradiation.
(3) DBTT shift due to irradiation can be reduced by the control of tempering conditions before irradiation, and it is found to be furthermore reduced by impurity doping with 60 ppm^{11}B and 200 ppmN.

(4) The ratios of DBTT shift to irradiation hardening depended on helium production, impurity doping and tempering conditions before irradiation.

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