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Enhancement of irradiation hardening by nickel addition in the reduced-activation 9Cr–2W martensitic steel

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Abstract

Reduced-activation martensitic (RAM) steels with and without an addition of 1% Ni were irradiated in a so called multisection-multidivision controlled irradiation capsule in the JMTR at 220°C up to 0.15 dpa. The 1/4 power dependence of the irradiation hardening on neutron dose was observed for the specimens irradiated in the controlled capsule. A part of the specimens were simultaneously irradiated in the capsule out of the reactor core where the irradiation temperature was considered to be lower than 170°C. The out of-reactor core irradiation induced a tremendous irradiation hardening as much as 350 MPa in the Ni added RAM steel but only 120 MPa of the hardening in the unadded RAM steel. The tremendous irradiation hardening was never observed following the irradiation at 220°C. As for the results of positron annihilation measurements, no significant effect of the Ni addition was observed in the life time spectrum. Post-irradiation annealing studies indicate that the irradiation hardening observed in the Ni added RAM steel begins to recover at 190°C and diminishes after the annealing at 250°C.

1. Introduction

Reduced-activation martensitic (RAM) steels are the prime candidate for fusion structural materials for DEMO reactor where the concentration of transmutation helium expected to be more than several thousand at ppm. It was shown by previous spectra tailoring experiments that much more remarkable increase in the ductile–brittle transition temperature (DBTT) was observed in the 2% Ni added ferritic steels than in those without the addition after the irradiation in the high flux isotope reactor (HFIR) [1–3], and the larger shift in the DBTT has been interpreted in terms of the effects of transmutation helium from nickel. However, the direct evidence has not been obtained yet. It is well known that the addition of nickel influences the transformation temperature of the martensitic steels, resulting in the changes in the martensitic structure or steel performance [4]. It has been strongly requested to make clear the effects of nickel addition itself on the irradiation embrittlement.

It was also shown by previous helium implantation studies that helium bubble formation at grain boundaries was very scarce in the martensitic steel [5–7], suggesting that most of the helium atoms implanted were trapped in the bulk, such as dislocations and precipitates. Thus, the helium embrittlement in ferritic steel, if it is, is considered to occur as a result of enhanced irradiation hardening by helium.

In this work, the effect of nickel addition on the irradiation hardening of reduced-activation martensitic steel has been investigated utilizing controlled irradiation facility of Japanese materials test reactor (JMTR) where the estimated concentration of transmutation helium is very low.

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Table 1
Chemical composition and heat treatment condition of 9Cr–2W steels (mass%)

	C	Si	Mn	P	S	Ni	Cr	V	Ti	W	Ta	Fe
Low Si/Mn	0.12	*0.01*	*0.01*	0.003	0.0017	*0.02*	8.91	0.26	0.05	2.03	0.069	bal.
1% Ni	0.10	*0.01*	*0.01*	0.004	0.0014	*1.00*	8.92	0.26	0.04	2.02	0.069	bal.

Heat treatment condition: normalized at 1323 K for 30 min and then tempered at 973 K for 1 h, followed by air cooling.

2. Experimental procedure

The materials used were RAM steels with and without nickel addition, and the chemical compositions and the heat treatment conditions of the steels are given in Table 1. Mini-size tensile specimens (4 mm × 16 mm × 0.25 mm) and disk specimens of 3 mm diameter were irradiated in the multisection and multidivision controlled irradiation rig in the JMTR [8,9] at 220°C to neutron fluences of 6.8×10^{18}, 3.2×10^{19} and 1.0×10^{20} n/cm^2, which produced displacement damage of 0.01, 0.05 and 0.15 dpa, respectively. A part of the specimens were irradiated in the capsule but out of the reactor core where the irradiation temperature was lower than 170°C and the total irradiation dose was less than 0.15 dpa. Fig. 1 shows the irradiation histories of each capsule. After the irradiation, tensile tests were carried out at a cross head speed of 0.2 mm/min at room temperature. Micro-Vickers measurements were performed after isochronal annealing (1 h). The lifetime spectra of positron annihilation (PA) were measured and decomposed into two components, that is a matrix component (τ_1, I_1) and a microvoid component (τ_2, I_2), where τ_i and I_i were the lifetime and intensity of *i*th component, respectively.

3. Results and discussion

3.1. Tensile properties

Tensile properties, such as yield stress and uniform elongation, following the controlled irradiations at 220°C are shown in Fig. 2. Each capsule was removed from the reactor during reactor operation so as to enable specimens to be irradiated to desired doses at the same temperature and flux. With increasing dose, yield stress increases monotoneously accompanied by the reduction in the uniform elongation. The irradiation hardening, $\Delta\sigma_y$, is proportional to the $\frac{1}{4}$ power of the neutron fluence as reported by the other researchers [10,11]. No effect of nickel addition was observed in the irradiation hardening. As for the specimens irradiated in capsule #1 shown in Fig. 1, which was irradiated out of the reactor core, a tremendous irradiation hardening as large as 350 MPa, was observed in the steel added with 1% Ni, as shown in Fig. 3. It should be noticed that such a enhanced irradiation hardening was never observed in the steel without the nickel addition. Tensile property data following the irradiation in capsule #1 was plotted in Fig. 4 together with those irradiated in capsules #2, #3

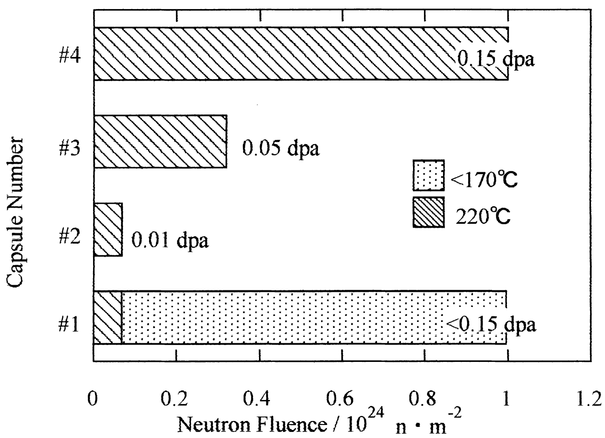

Fig. 1. The irradiation conditions, neutron fluence (>1 MeV) and irradiation temperature, of each irradiation capsule of JMTR.

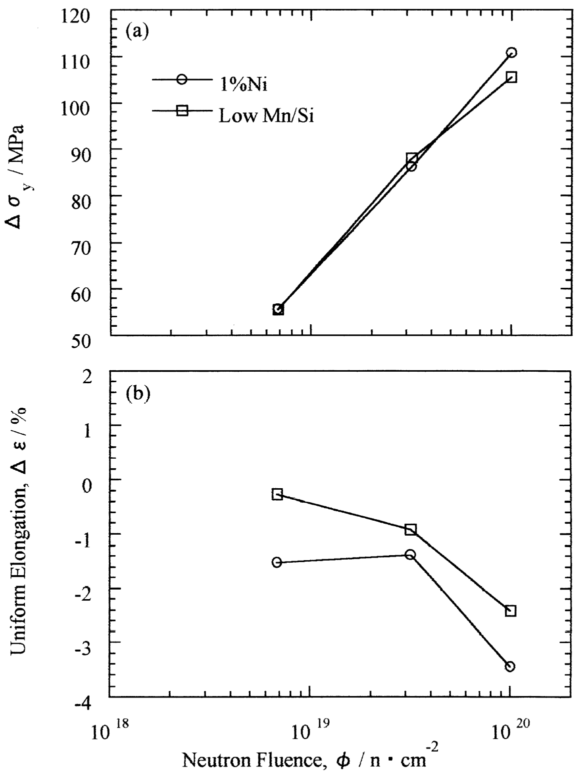

Fig. 2. Dose dependence of tensile properties: (a) yield stress, and (b) uniform elongation, of Low Si/Mn and 1% Ni at 220°C irradiation.

and #4. Since the capsule #1 was irradiated out of the reactor core, the neutron dose is expected to be much smaller than 0.15 dpa, although the capsule was irradiated to the end of reactor operation. Therefore, the enhanced irradiation hardening observed in Ni-added steel can be attributed to the low temperature irradiation.

3.2. Isochronal annealing behavior

In order to investigate the influence of irradiation temperature to the enhanced irradiation hardening, Vickers-hardness measurements were carried out for the 1% Ni steel following post-irradiation isochronal annealings for 1 h at temperatures between 170°C and 400°C. The recovery behavior of irradiation hardening of both the 1% Ni steels irradiated at 220°C and below 170°C is shown in Fig. 5. In the specimen irradiated at 220°C, an observable recovery of the hardening occurred only above 300°C. In contrast, the hardness of the 1% Ni steels irradiated out of reactor core is much higher than that of irradiated at 220°C and the irradiation hardening shows two step recovery; the first stage appears at annealing temperatures from 200°C to 250°C and followed by the second stage above 300°C. The second stage of the recovery seems to be the same with that observed in the steels irradiated at 220°C. This shows that the nickel added martensitic steels has an additional component of irradiation hardening when irradiated below 170°C. It was reported by some of the co-authors of the present paper that irradiation hardening of RAM steels occurs only at irradiation temperatures below 400°C and the hardening is due to small interstitial dislocation loops, precipitates and microvoids [12–14].

3.3. Positron annihilation lifetime measurements

Fig. 6 shows the PA lifetime spectra of 1% Ni steel before and after the irradiation at 220°C. The spectrum broadened with increasing irradiation dose, which indicates the formation of microvoids where the lifetime is longer than that in the bulk [15]. The results of two component analysis of the PA spectra are shown in Fig. 7 for both the steels. The first component of the lifetime, τ_1, which exists in both the unirradiated and

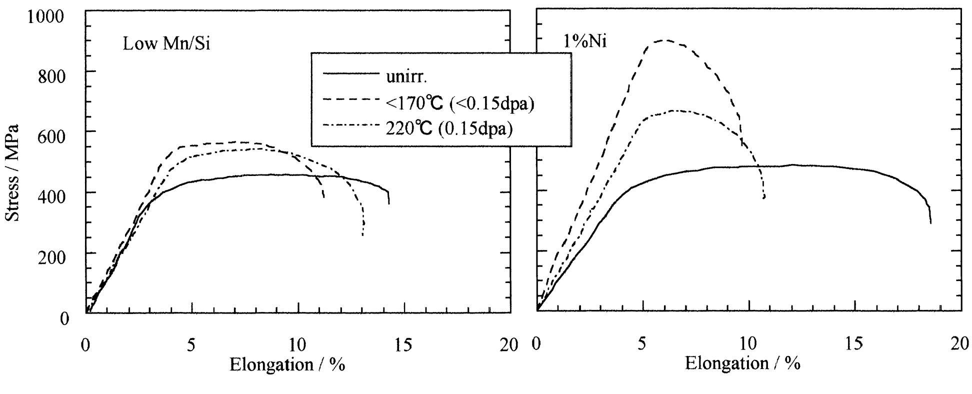

Fig. 3. The Stress-elongation curves in tensile tests of the specimens irradiated at lower 1 70°C (<0.15 dpa), with that of unirradiated and irradiated at 220°C (0.15 dpa) specimens.

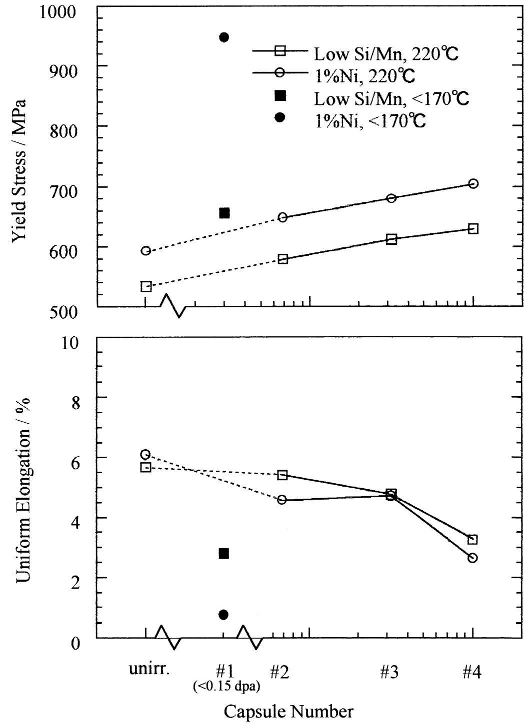

Fig. 4. Tensile properties, yield stress and uniform elongation, of Low Si/Mn and 1% Ni steel at below 170°C irradiation, compared to the 220°C irradiation.

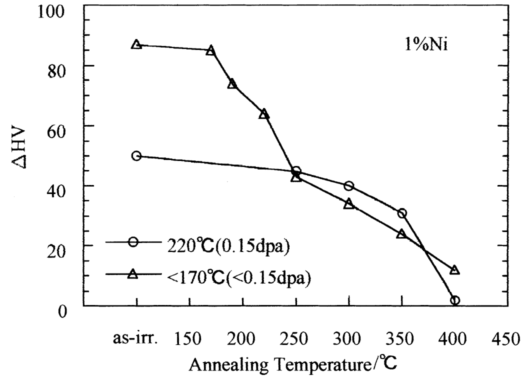

Fig. 5. The recovery of Micro-Vickers hardness for 1% Ni irradiated at 220°C and below 170°C.

irradiated specimens, is longer than the lifetime in the bulk, which is due to the other component induced by dislocations of the martensitic structure. The neutron irradiation (0.01 dpa) caused the appearance of the second component, τ_2, as large as 400 or 500 ps, indicating that microvoids were formed by the irradiation. In Low Si/Mn steel, τ_2 increases with dose accompanied by a reduction of intensity, indicating that microvoids grow and the density becomes smaller. On the other hand, the lifetime of 1% Ni is already about 500 ps, which is considered to be a saturated level of positron annihilation lifetime in iron [15]. But no visible void was observed by TEM in this steel. There is no significant difference in the positron spectra between the 1% Ni steel irradiated at 220°C and below 170°C, which clearly indicates that the anomalous irradiation hardening is considered to be not due to microvoids but interstitial dislocation loops involving nickel. The second recovery stage is considered to be due to shrinkage of dislocation loops by absorption of vacancies decomposed from microvoids at these temperatures.

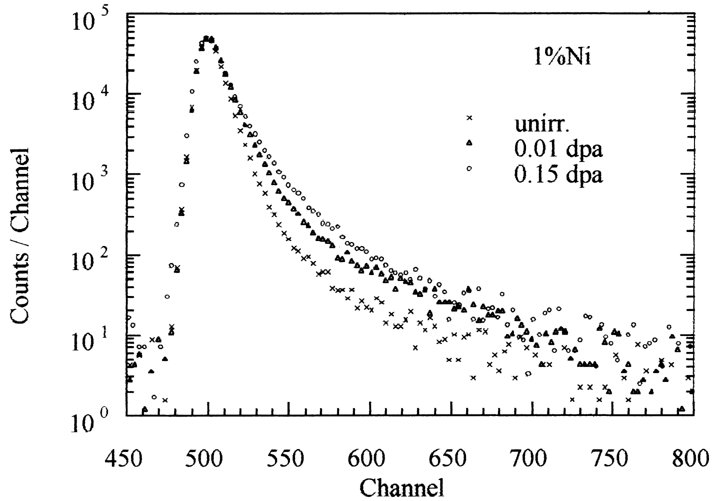

Fig. 6. Positron annihilation lifetime spectrum for 1% Ni specimens irradiated at 220°C to 0.01 dpa and 0.15 dpa in JMTR.

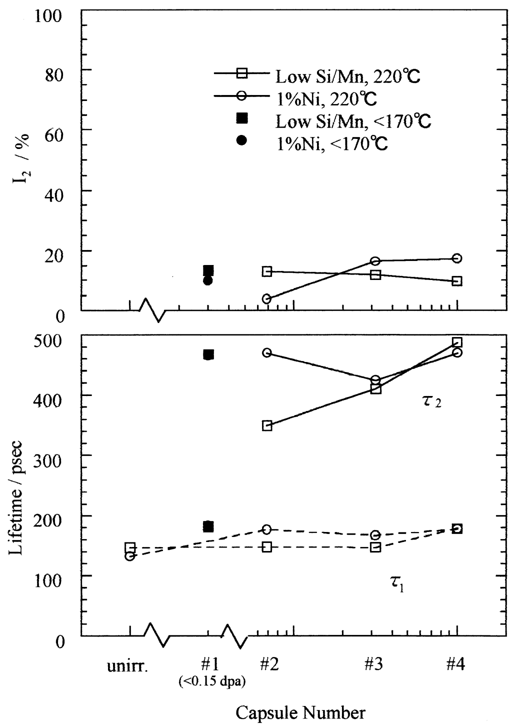

Fig. 7. The two component analysis of positron annihilation spectrometry of Low Si/Mn and 1% Ni below 170°C irradiation, compared to the 220°C irradiation.

4. Conclusion

The tensile properties, Vickers-hardness and PA lifetime were measured for the reduced-activation martensitic steels with and without an addition of 1% Ni following controlled irradiation at 220°C and uncontrolled irradiation below 170°C. The main results are:

1. After the controlled irradiation at 220°C, there is no difference in the irradiation hardening behavior between the steels, and the amount of hardening monotonously increased with increasing the dose, resulting in the hardening as large as 100 MPa at a dose of 0.15 dpa.
2. A tremendous irradiation hardening as large as 350 MPa was observed in Ni added steel irradiated out of the reactor core below 170°C.
3. The tremendous irradiation hardening observed in the Ni added steel showed two-step recovery: the first stage is around 200°C and followed by the second stage above 300°C.
4. Microvoids are considered to be formed by both the irradiation at 220°C and 170°C, but there is no significant difference in the size and density of microvoids. The enhanced irradiation hardening is considered to be due to small interstitial loops containing nickel, which is thermally unstable at temperatures above 200°C.

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