ELSEVIER

Journal of Nuclear Materials 271&272 (1999) 360–364

journal of nuclear materials

Effects of varying temperature irradiation on the neutron irradiation hardening of reduced-activation 9Cr–2W martensitic steels

R. Kasada [a,*], A. Kimura [b], H. Matsui [a], M. Hasegawa [c], M. Narui [c]

[a] Institute for Materials Research, Tohoku University, Katahira, Aoba-ku, Sendai 980-77, Japan
[b] Institute of Advanced Energy, Kyoto University, Gokasho, Uji, Kyoto 611, Japan
[c] The Oarai-Branch of Institute for Materials Research, Tohoku University, Oarai, Ibaraki 311-13, Japan

Abstract

In order to clarify the effects of varying temperature during irradiation on the irradiation hardening of 9Cr–2W steels, tensile tests and positron annihilation lifetime measurements were carried out following the varying temperature irradiation (220/420°C and 340/530°C) utilizing a so called multi-section and multi-division controlled irradiation capsule in JMTR. After the irradiation at 220°C to 0.053 dpa, the steels show irradiation hardening as much as 110 MPa. The hardening was almost completely diminished immediately after the elevation of the irradiation temperature to 420°C. Subsequent irradiation at 420°C up to 0.14 dpa did not cause any hardening. The results of positron annihilation lifetime measurements indicate that microvoids are formed by the irradiation at 220°C but disappear upon elevating the temperature to 420°C and are then formed again by the subsequent irradiation at 420°C up to a total dose of 0.14 dpa. This behavior may be interpreted in terms of decomposition of interstitial loops or migration of small interstitial loops during temperature elevation. There is no good correlation between irradiation hardening and formation of microvoids in neutron-irradiated reduced-activation martensitic steels.

1. Introduction

Superior resistance to irradiation hardening of reduced activation 9% Cr martensitic steels compared to the other structural materials has been shown in a number of recent studies [1–10]. Our previous research indicated that neutron irradiation hardening of reduced-activation martensitic steels depended on the irradiation temperature, and the hardening was attributed to the formation of interstitial dislocation loops, small precipitates and microvoids [11]. However, the role of each structure, especially that of microvoids which are still invisible, has not yet been made clear. Positron annihilation (PA) lifetime measurements have been carried out to investigate the behavior of vacancy in materials, such as vacancy migration and growth of microvoids [12]. It is expected that there will be some correlation between irradiation-induced changes in positron lifetime and hardening if microvoids play a significant role in the hardening. Since irradiation hardening of reduced-activation martensitic steels depends on irradiation temperature, varying temperature irradiation, which is expected to occur in the start-up and shut-down in the operation of fusion reactor, could influence the irradiation-induced evolution of microstructure and hardening of the steels. Varying temperature irradiations have been performed in JMTR, where a so-called multi-section and multi-division controlled irradiation rig is available to irradiate specimens at controlled temperatures up to given fluences [13].

The objective of this research is to investigate the effect of varying temperature irradiation on the irradiation hardening of reduced activation martensitic steels and make clear the contribution of microvoids to the irradiation hardening in the steels.

* Corresponding author. Tel.: +81-774 38 3483; fax: +81-774 38 3483; e-mail: kasada@iae.kyoto-u.ac.jp

PII: S0022-3115(98)00749-1

2. Experimental procedure

Reduced-activation martensitic steels, 9%Cr–2%W base steel (mod.JFL-1) and 9%Cr–2%W–1%Ni low Si/Mn steel (mod.JLF-1/LSM/Ni), were used in this study. The chemical composition and the heat treatment conditions of the steels are shown in Table 1. Disk specimens of 3 mm diameter for positron annihilation study and mini-size tensile specimens which measure $4 \times 16 \times 0.25$ mm^3 were irradiated using the multi-section and multi-division controlled irradiation rig in JMTR, where some of the specimens are removable during reactor operation under temperature control. Two types of stepwise increasing varying temperature irradiations were performed with the temperature combinations of 220/420°C and 340/530°C, as shown in Fig. 1. After irradiation at the lower temperatures (220°C and 340°C) up to a dose of 3.6×10^{19} n/cm^2 (0.053 dpa), one third of the specimens was removed from the reactor. Then the irradiation temperatures were elevated to higher temperatures (420°C and 530°C) within 1 h and another one third of the specimens was removed from the reactor 3.3 h after the first removal, which allowed only a very short irradiation at the higher temperature, namely 3.9×10^{17} n/cm^2 (0.0006 dpa). The final one third of the specimens was irradiated at the higher temperatures up to a dose of 5.6×10^{19} n/cm^2 (0.087 dpa), which resulted in a total dose of 9.0×10^{19} n/cm^2 (0.14 dpa). Following the irradiation, tensile tests were carried out at room temperature at a crosshead speed of 0.2 mm/min. Positron annihilation life time measurements were performed at room temperature. The spectra of PA were decomposed into three components: a matrix component (τ_1, I_1), a vacancy or dislocation component (τ_2, I_2), and a microvoid component (τ_3, I_3), where τ_i and I_i are the lifetime and intensity of i component, respectively.

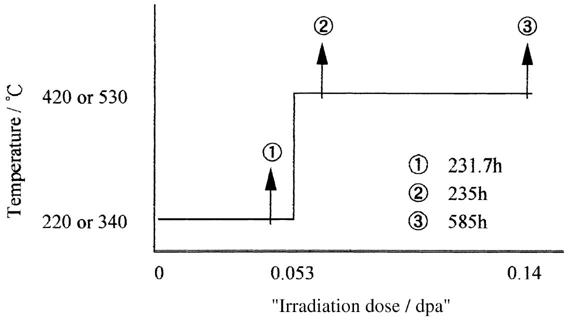

Fig. 1. Irradiation condition, dpa and temperature, of each capsule in JMTR.

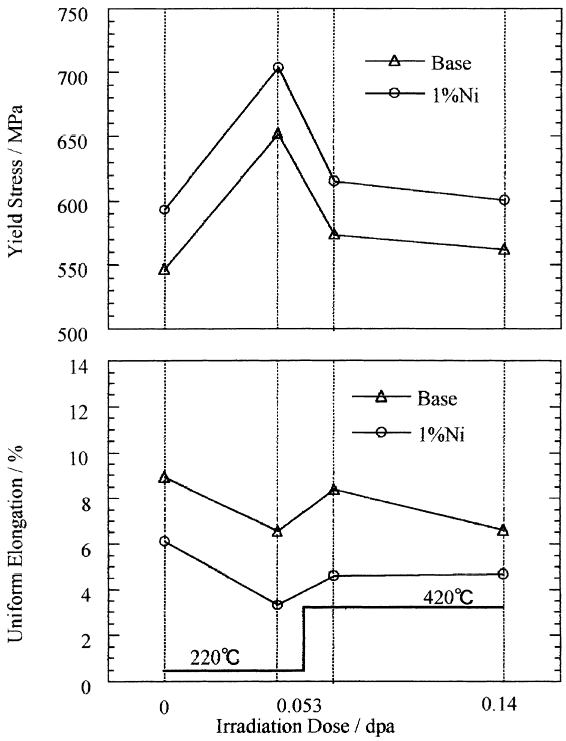

Fig. 2. The tensile properties of each steel irradiated at 220/420°C varying temperature.

3. Results and discussion

3.1. Tensile properties

Fig. 2 shows the changes in yield stress and uniform elongation of each steel caused by 220/420°C varying temperature. The irradiation at 220°C to a dose of 0.053 dpa causes increase in the yield stress of both the steels, indicating almost the same amount of hardening for each steel by as much as 110 MPa. This hardening diminished almost immediately after the elevation of the irradiation temperature to 420°C. The subsequent

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
Base	0.12	0.10	0.52	0.004	0.0017	0.02	8.98	0.26	0.04	2.07	0.070	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.

irradiation at 420°C up to a total dose of 0.14 dpa did not cause any hardening. The change in uniform elongation is similar to that of yield stress. Since our previous research on the effects of FFTF/MOTA irradiation on the same martensitic steels indicated that no hardening was observed after the irradiation at 420°C [14], it is considered that pre-irradiation at 220°C is ineffective to the hardening by subsequent irradiation at 420°C.

Fig. 3 shows the results of 340/530°C varying temperature irradiation. The irradiation at 340°C to 0.053 dpa causes a hardening of about 50 MPa. The elevation of the temperature to 530°C results in the annealing out of the hardening and the yield stress continues to decrease with increasing dose to 0.14 dpa due to recovery of the martensitic structure.

3.2. Positron annihilation lifetime measurement

The effect of irradiation on positron annihilation lifetime spectra is shown in Fig. 4 for the base steel. Neutron irradiation causes the broadening of the spectrum, indicating the formation of microvoids where the lifetime is longer than in bulk [15]. The spectrum of the specimen irradiated at 220°C was the broadest, and the elevation of irradiation temperature to 420°C resulted in closing

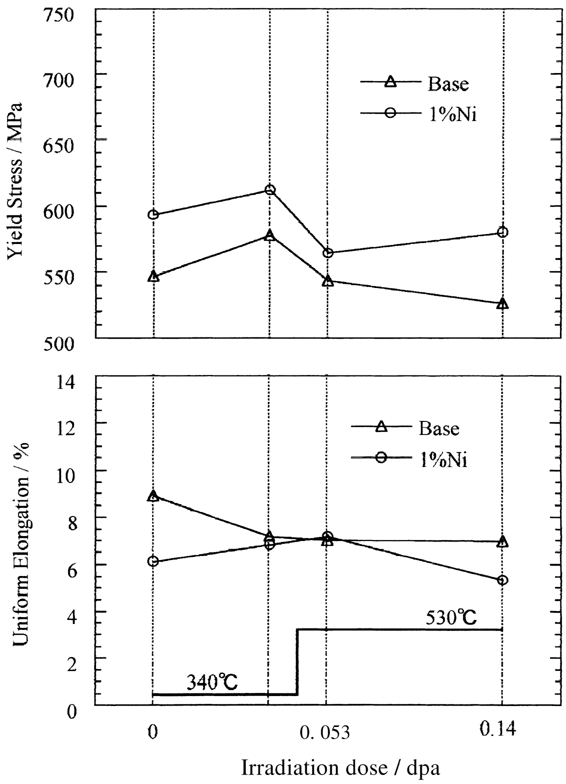

Fig. 3. The tensile properties of each steel irradiated at 340/530°C varying temperature.

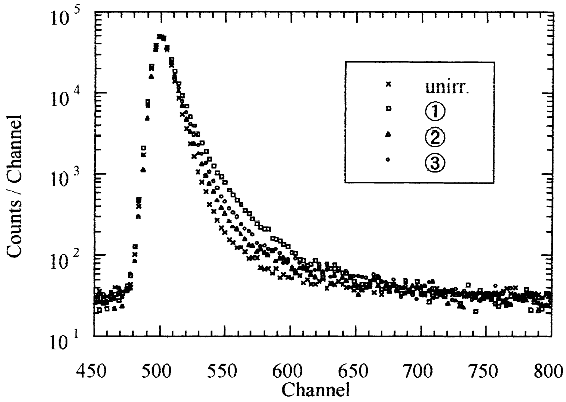

Fig. 4. The positron annihilation lifetime spectrum of 1% Ni steel irradiated at 220/420°C.

the spectrum to that of the unirradiated specimen. The subsequent irradiation at 420°C up to 0.14 dpa broadened the spectrum but the broadening was still smaller than that of the specimen irradiated at 220°C up to 0.053 dpa.The results of the three component analysis of PA spectra is shown in Fig. 5 for both steels irradiated at 220/420°C. The first and second component of the lifetime, which are also observed for unirradiated specimens, are considered to be due to annihilation in matrix and at dislocations of martensitic structure. It is shown in Fig. 6(a) that following the irradiation at 220°C the third component of lifetime, τ_3, appeared with a significant intensity, I_3, suggesting the formation of microvoids composed of about twenty vacancies by the irradiation. The microvoids, however, disappeared after the elevation of irradiation temperature to 420°C, which could be interpreted in terms of (1) growth of microvoids, (2) dissolution into vacancies or (3) annihilation with small interstitial loops. Since observation by transmission electron microscopy (TEM) revealed that there were no visible voids in the specimen after the temperature elevation, it seems that the microvoids formed by the irradiation at 220°C disappeared by either dissolution or annihilation. This will be discussed in the next session. Following the subsequent irradiation at 420°C up to 0.14 dpa, PA shows that new microvoids composed of a few vacancies (τ_3 = 300 ps) were formed again by the irradiation. It is interesting that the microvoids formed at 420°C are smaller than at 220°C. A similar tendency was observed for the 1% Ni steel.

3.3. Comparison with constant temperature irradiation

Fig. 6 shows the comparison between varying temperature (220/420°C) irradiation and constant temperature (220°C) irradiation [16] of the lifetime and its intensity of the third component (a) which reflects the size and density of microvoids, respectively, and the

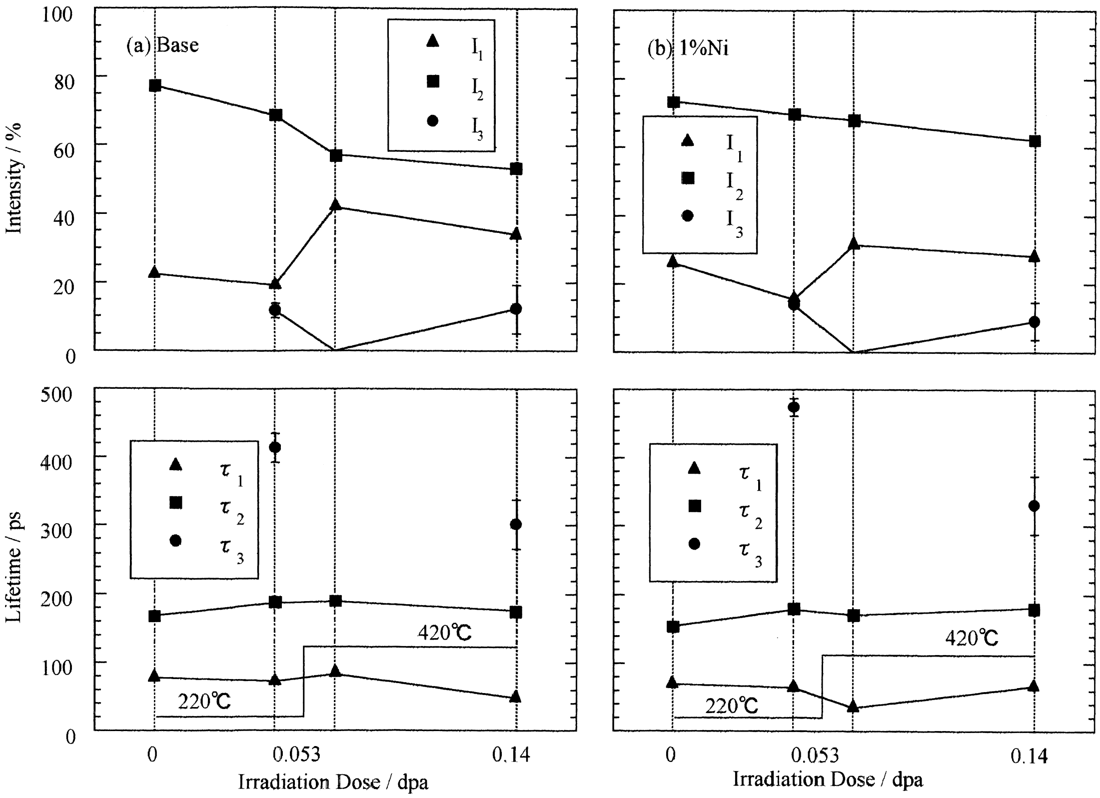

Fig. 5. Three component analysis for positron annihilation lifetime spectrum of (a) Base and (b) 1% Ni at 220/420°C irradiation.

yield stress (b) of 1% Ni steel. The irradiation at 220°C up to a dose of about 0.053 dpa induces irradiation hardening by as much as 70 MPa, and further irradiation at 220°C causes an increase in the hardening. Positron lifetime and its intensity also increase with increasing dose. From these results, there appears to be a good correlation between irradiation hardening and the formation of microvoids. Varying temperature irradiation, however, revealed that there is no good correlation between those, because microvoids observed by PA measurements still exist after the subsequent irradiation at 420°C up to a total dose of 0.14 dpa, at which condition no irradiation hardening is observed. Therefore, it is concluded that the microvoids do not cause irradiation hardening in the martensitic steels.

Previous work showed that the hardening caused by FFTF/MOTA irradiation was mainly due to formation of dislocation loops [14]. Since the neutron irradiation dose in this work is much smaller than the previous work, the hardening observed in this study is considered to be due to small interstitial loops which are invisible by TEM. Although both the small interstitial loops and microvoids are formed by the irradiation at 220°C, only the interstitial loops are considered to cause the hardening. Further hardening by the irradiation at 220°C could be due to an increase in the density of interstitial

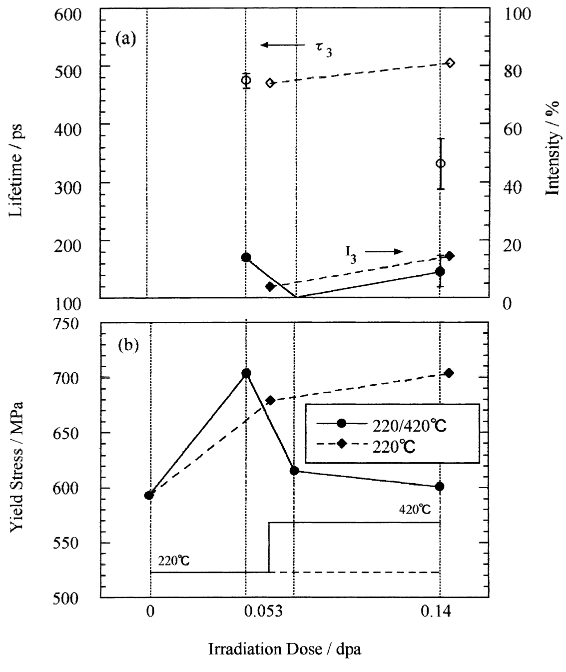

Fig. 6. The comparison of constant and varying temperature irradiations for (a) PA and (b) yield stress of 9Cr–2W–1Ni.

loops. The elevation of irradiation temperature to 420°C might cause the thermal dissolution of microvoids to vacancies which can move to interstitial loops and annihilate to reduce the density of the loops, resulting in the recovery of irradiation hardening. Although microvoids are formed by the subsequent irradiation at 420°C, no interstitial loop is considered to be formed, since the irradiation induces no hardening. This interpretation, however, makes it difficult to explain the formation of microvoids during further irradiation at 420°C. Previous work indicated that void swelling was a maximum at around 420°C [17], suggesting that voids are thermally stable at the temperature. Thus, the disappearance of microvoids can be interpreted in terms of annihilation with interstitial atoms from decomposed small interstitial loops or by the interacition with the interstitial loops, which are immobile at 220°C but become mobile at 420°C. Thus, both the small loops and microvoids are annihilated during the temperature elevation.

4. Conclusions

The tensile properties and PA lifetime were measured for two reduced-activation martensitic steels following the varying temperature irradiation of 220/420°C and 340/530°C. The main results are as follows.

(1) The hardening caused by the irradiation at lower temperatures recovered immediately after the elevation of the irradiation temperature, and no hardening was observed after the subsequent irradiation at higher temperatures.

(2) The microvoids formed by the irradiation at 220°C disappeared once the irradiation temperature was elevated to 420°C and appeared again after the subsequent irradiation. This suggests that the disappearance of microvoids is not due to their thermal dissolution but to the annihilation with interstitial atoms decomposed from small interstitial loops or small interstitial loops which are immobile at 220°C and becomes mobile at 420°C.

(3) Neutron irradiation hardening of reduced-activation martensitic steels is considered to be mainly due to small interstitial loops, and the contribution of microvoids to the hardening is small.

Acknowledgements

This work was supported by Japan–US collaborative JUPITER program on fusion materials research.

References

[1] A. Kohyama, A. Hishinuma, D.S. Gelles, R.L. Klueh, W. Dietz, K. Ehrlich, J. Nucl. Mater. 233–237 (1996) 138.
[2] R.L. Klueh, D.J. Alexander, E.A. Kenik, J. Nucl. Mater. 227 (1995) 11.
[3] J.J. Kai, R. L Klueh, J. Nucl. Mater. 230 (1996) 116.
[4] T. Shibayama, A. Kimura, H. Kayano, J. Nucl. Mater. 233–237 (1996) 270.
[5] F. Abe, T. Noda, H. Araki, M. Narui, H. Kayano, J. Nucl. Mater. 191–194 (1992) 845.
[6] A. Kimura, H. Kayano, T. Misawa, H. Matsui, J. Nucl. Mater. 212–215 (1994) 690.
[7] H. Kayano, H. Kurishita, A. Kimura et al., J. Nucl. Mater. 179–181 (1991) 425.
[8] H. Kayano, A. Kimura, M. Narui et al., J. Nucl. Mater. 155–157 (1988) 978.
[9] A. Kohyama, Y. Kohno, K. Asakura et al., J. Nucl. Mater. 212–215 (1994) 684.
[10] A. Kimura, H. Kayano, M. Narui et al., J. Nucl. Mater. 179–181 (1991) 737.
[11] A. Kimura, H. Matsui, J. Nucl. Mater. 212–215 (1994) 701.
[12] M. Hasegawa, Materia Japan 35 (1996) 93.
[13] M. Kiritani, T. Yoshiie, S. Kojima, Y. Satoh, K. Hamada, J. Nucl. Mater. 174 (1990) 327.
[14] A. Kimura, T. Morimura, M. Narui, H. Matsui, J. Nucl. Mater. 233–237 (1996) 319.
[15] E. Kuramoto, Y. Takano, N. Kikuchi, M. Takenaka, J. Nucl. Mater. 141–143 (1986) 829.
[16] R. Kasada, A. Kimura, H. Matsui, M. Narui, to be published.
[17] T. Morimura, A. Kimura, H. Matsui, J. Nucl. Mater. 239 (1996) 118.