ELSEVIER

Journal of Nuclear Materials 307–311 (2002) 299–303

journal of nuclear materials

www.elsevier.com/locate/jnucmat

Void swelling in reduced activation ferritic/martensitic steels under ion-beam irradiation to high fluences

H. Ogiwara [a,*], H. Sakasegawa [a], H. Tanigawa [c], M. Ando [c], Y. Katoh [b], A. Kohyama [b]

[a] *Graduate school of energy science, Kyoto University, Gokasyo, Uji, Kyoto 611-0011, Japan*
[b] *Institute of Advanced Energy, Kyoto University, Gokasyo, Uji, Kyoto 611-0011, Japan*
[c] *Japan Atomic Energy Research Institute, Tokai, Naka, Ibaraki 319-1195, Japan*

Abstract

Swelling behavior of a 9Cr–2.0W–V,Ta reduced activation ferritic/martensitic steel (JLF-1) and a tungsten-enriched 9Cr–2.5W–V,Ta steel (JLS-1) under ion-beam irradiation was studied. A technique of dual-beam ion irradiation utilizing a pair of electrostatic accelerators was employed to achieve high fluence levels and a helium production rate that are relevant to fusion power reactor blanket environments. Under single ion-beam irradiation at 743 K, a cavity structure was formed only in regions of more than 40 dpa. Cavities were in the martensite lath structure. Cavities did not form at the displacement peak about 1400 nm. Under dual ion-beam irradiation at 743 K, a cavity structure was formed at dual ion-beam irradiated regions. A bi-modal size distribution of faceted voids and spherical helium bubbles of diameter up to 2 nm were observed.

1. Introduction

Fe–9Cr–2W–V,Ta reduced activation ferritic/martensitic steels (RAFs) are recognized as leading candidates for the blanket first wall structural material of near-term fusion reactors due mainly to their proven mechanical property retention, microstructural stability and swelling resistance under fission neutron irradiation combined with a maturity of similar alloys as industrial materials [1–3]. However, the effects of irradiation to very high fluence levels and a massive production of gaseous transmutation species, both of which are characteristic to fusion reactor environments, are not sufficiently understood. The primary objective of this work is to explore the microstructural development, swelling behavior in particular, in Fe–9Cr–2W–V,Ta steel (JLF-1) under irradiation to fluence levels as high as 100 dpa at a helium production rate relevant to a typical fusion blanket condition. A technique of dual-beam ion irradiation was employed for this purpose, where a reasonably high displacement damage rate and a desired helium-to-dpa ratio are simultaneously achieved [4].

The disadvantage of conventional RAFs as blanket structural materials primarily lays in their upper service temperature limit that will not provide very attractive power cycle efficiency. Therefore, extensive efforts are being made to improve RAFs by means of either oxide particle dispersion or further compositional tailoring [5,6]. A series of Fe–9Cr–xW–V,Ta ($x = 2.5$–3.5) steels (JLS-series) has been prepared in this direction, based on the technical establishment in JLF-1 [5]. Studies on creep rupture properties, annealed microstructures [5] and impact properties [6] of steels in the JLS-series indicates potential benefits of the increased tungsten contents to over-all material performances depending on prospective conditions of usage. A comparative study on irradiation response in JLF-1 and JLS steels are included to the scope of this work, since irradiated microstructual

* Corresponding author. Address: Institute of Advanced Energy, Kyoto University, Gokasyo, Uji, Kyoto 611-0011, Japan. Tel.: +81-774 38 3466; fax: +81-774 38 3467.
E-mail address: ogi@iae.kyoto-u.ac.jp (H. Ogiwara).

0022-3115/02/$ - see front matter
PII: S0022-3115(02)01078-4

Table 1
Chemical composition and heat treatments

	C	Cr	W	V	Ta	Mn	Si	Al	P	S	N
JLF-1	0.097	9.04	1.97	0.19	0.070	0.46	<0.1	0.003	0.0030	0.0020	0.0237
JLS-1	0.094	9.19	2.50	0.081	0.050	0.99	0.050	0.003	0.0020	0.0004	0.0267

Normalized at 1323 K $\times$ 3.6 ks followed by air-cooling: (JLF-1) tempered at 1053 K $\times$ 3.6 ks followed by air-cooling, (JLS-1) tempered at 1033 K $\times$ 3.6 ks followed by air-cooling.

instability is a major concern for tungsten-rich steels for nuclear applications.

2. Experimental procedure

The materials used were JLF-1 (Fe–9Cr–2W–V,Ta) and JLS-1 (Fe–9Cr–2.5W–V,Ta) both in standard normalized and tempered conditions. The chemical compositions and heat treatment conditions are given in Table 1. Conditions of ion irradiation are summarized in Table 2. Irradiation was performed up to 60 dpa at the mid-plane of dual-beam irradiated ranges, where the nominal helium injection ratio was maintained at 15 appm He/dpa. Specimen temperature was monitored by infrared thermography during irradiation to ensure that the incident surfaces of the specimens were maintained at an intended temperature at all time. He ions were passed through a rotating thin foil beam energy degraders specifically designed for the dual-beam experiments, in order to broaden the range of helium implantation. Dual-beam irradiated ranges were determined to be approximately 400–1300 nm from the incident surface for the 6.4 MeV Fe^{3+} case and 500–800 nm for the 3.8 MeV Fe^{3+} case, based on the displacement damage and helium concentration profiles, calculated by SRIM-98 code [7] assuming an average displacement threshold energy of 40 eV.

Thin foils for transmission electron microscope (TEM) examination was prepared with a focused ion-beam microprocessing device equipped with a micro-sampling system. This process provides not only excellent cross-sectional TEM thin foils but also a completely negligible effect of ferromagnetism during TEM examination. Further technical details of the procedure are published elsewhere [8].

Table 2
Irradiation conditions

Ions	6.4 MeV Fe^{3+} + degraded 1.0 MeV He^{+} (743 K)
	3.8 MeV Fe^{3+} + degraded 1.0 MeV He^{+} (823 K)
He injection rate	15 appm He/dpa
Displacement rate	1×10^{-3} dpa/s (at the depth 850 nm (6.4 MeV Fe^{3+}), 700 nm (3.8 MeV Fe^{3+}))
Temperature	743 and 823 K
Irradiated dose	40–100 dpa

3. Results and discussion

3.1. Ion-beam irradiated effects on JLF-1

Fig. 1 shows the cross-sectional TEM images of JLF-1 irradiated at 743 K in irradiated regions, for (a) single ion-beam and (b) dual ion-beam. Fig. 3 shows the dependence of void swelling, cavity number density and mean cavity radius on irradiation fluence at 743 K. Cavity structure was formed only in regions of more than 40 dpa under single ion-beam irradiation at 743 K, and cavities were in the martensite lath structure. Cavity

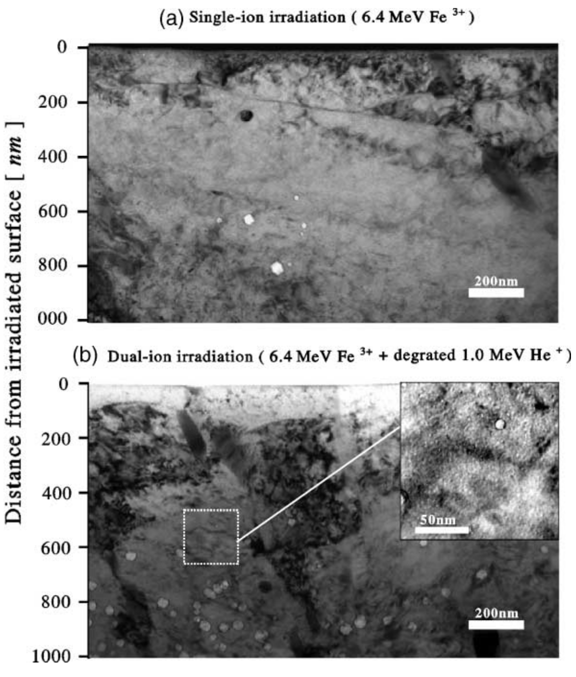

Fig. 1. Cross-sectional TEM images JLF-1 irradiated at 743 K in irradiated regions, (a) single ion-beam and (b) dual ion-beam.

formation at the displacement peak of about 1400 nm was not observed, most likely due to well-known injected ion effects. At depths from 600 to 800 nm cavity swelling of single ion-beam irradiated regions was smaller than cavity swelling (3.2%) of the dual ion-beam irradiated regions. Swelling of single ion-beam irradiated JLF-1 was low.

A cavity structure was formed throughout the dual ion-beam irradiated regions. A bi-modal size distribution of faceted voids and spherical helium bubbles of diameter up to 2 nm was observed. The cavities were also in the martensite lath structure. The bubbles were on the lath boundaries, recovered lath boundaries and the matrix surroundings precipitates.

3.2. Difference in swelling of JLF-1 and JLS-1

JLF-1 and JLS-1 were irradiated at 743 and 823 K. Fig. 2 shows (a) TEM images of thin foil from the incident surface to depth of 1.5 μm, (b) Cavity images of JLF-1, JLS-1 specimens irradiated to 60 dpa at 743 K in dual ion-beam irradiated regions, from 600 to 1200 nm. And Fig. 3 shows the dependence of void swelling, cavity number density and mean cavity radius on irradiation fluence at 743 K. At 743 K swelling of JLS-1 was inhibited in comparison with that of JLF-1. Increasing tungsten content to 2.5% from 2.0% has merit not only for creep strength but also for swelling resistance. Cavity images of JLF-1 and JLS-1 specimens irradiated up to 60 dpa at 823 K in dual ion-beam irradiated regions, from 500 to 750 nm, are presented in Fig. 4.

At 743 K swelling was similar between JLF-1(2.0%W) and JLS-1(2.5%W); however, swelling of JLF-1 was lower than that of JLS-1 at 823 K. The incubation periods of swelling in JLS-1 are attributed to the effects of dissolved tungsten. The amount of dissolved tungsten in JLS-1 was larger than in JLF-1 at 823 K. At 823 K JLF-1 is superior in irradiation resistance.

3.3. Comparison with a neutron and nickel ion-beam irradiation data

Fig. 5 shows the dependence of void swelling of Fe–9Cr–xW–TaV ($x = 2.0$–2.5) on irradiation temperature. Under neutron irradiation cavities of low number density formed in lath regions only. The effects of both neutron irradiation and single ion-beam irradiation are comparable. The difference of the peak swelling

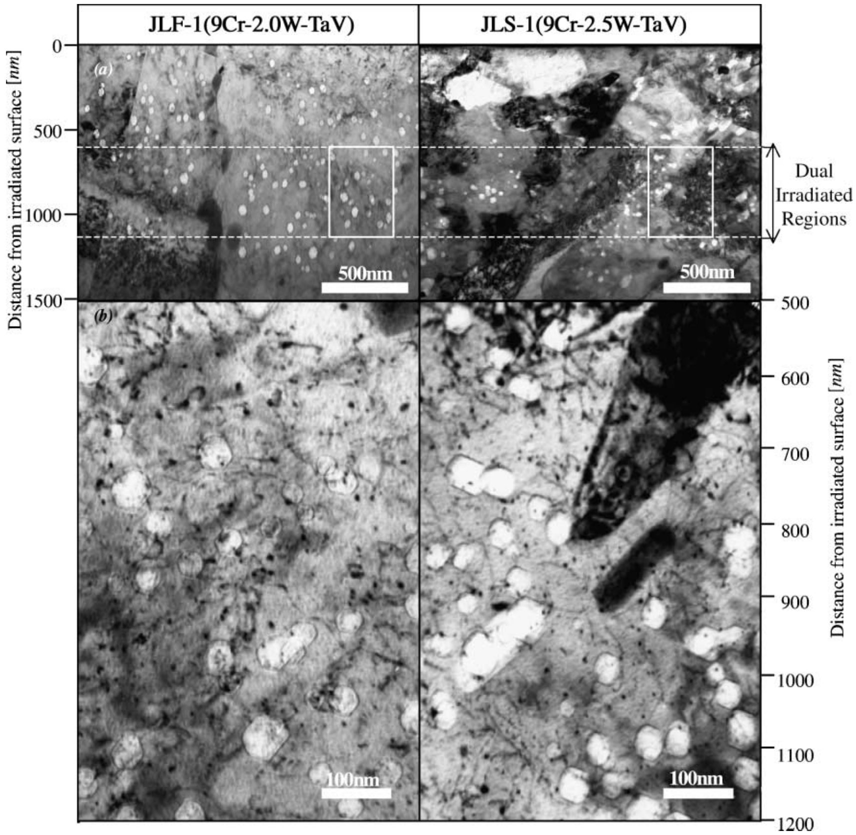

Fig. 2. (a) TEM images of thin foil from the incident surface to depth of 1.5 μm, (b) cavity images of JLF-1, JLS-1 specimens irradiated to 60 dpa at 743 K in dual ion-beam irradiated regions, from 500 to 1200 nm.

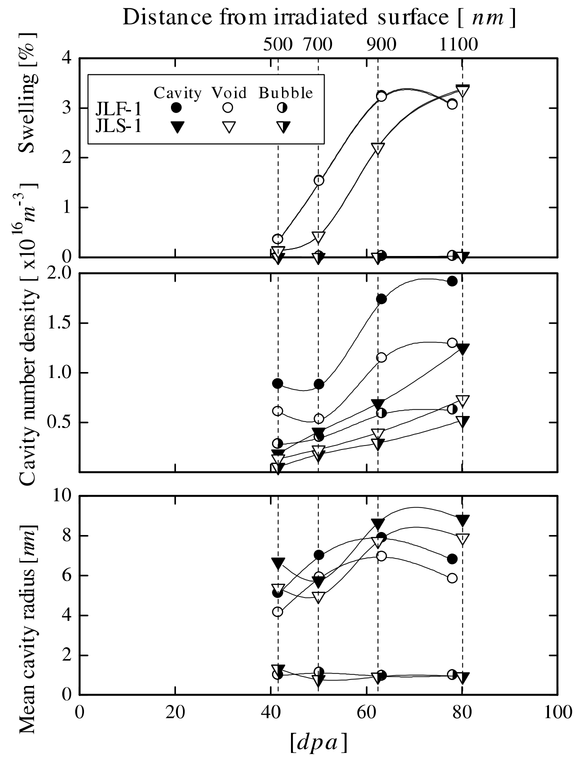

Fig. 3. The dependence of void swelling, cavity number density and mean cavity radius on irradiation fluence at 743 K.

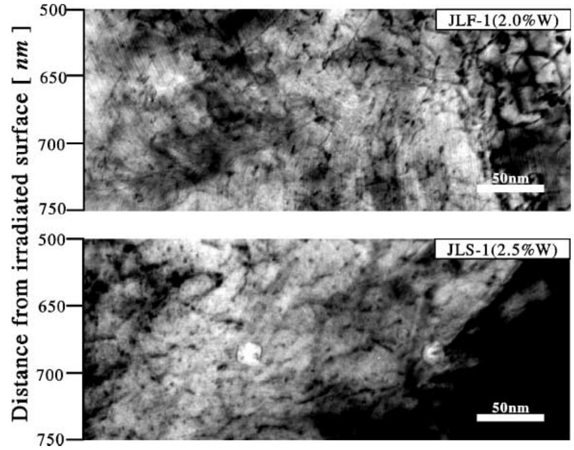

Fig. 4. Cavity images of JLF-1, JLS-1 specimens irradiated up to 60 dpa at 823 K in dual ion-beam irradiated regions, from 500 to 750 nm.

temperature between neutron irradiation and ion-beam irradiation is estimated to be about 50 K. Compared with Ni + He dual ion-beam irradiated data, swelling of Fe + He dual ion-beam irradiation increased greatly. Although Ni + He ion-beam irradiation was performed at a higher displacement damage level (at 3×10^{-3} dpa/s), swelling peak was lower. It is considered that these effects were due to the effects of implanted nickel [9].

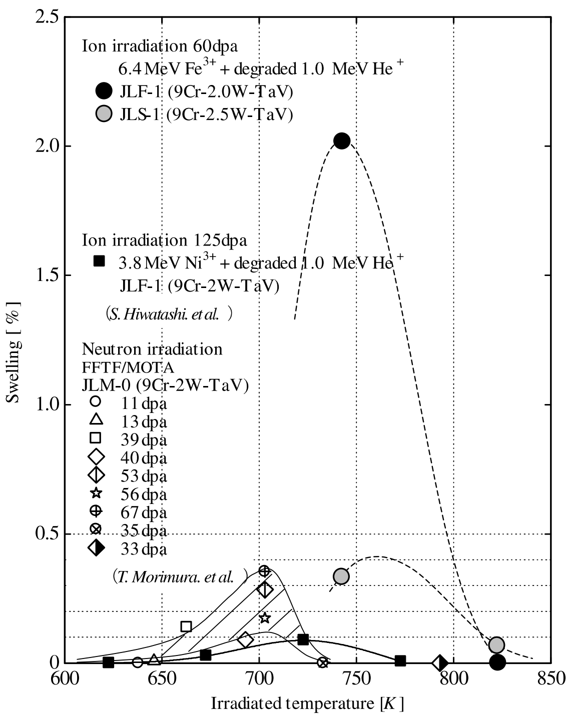

Fig. 5. The dependence of void swelling of Fe–9Cr–xW–Ta,V ($x = 2.0$–2.5) on irradiation temperature.

4. Summary

Under single ion-beam irradiation at 743 K a cavity structure was formed at doses above 40 dpa only. Swelling of single ion-beam irradiated JLF-1 was low.

Swelling of JLF-1 was lower than JLS-1 at 823 K. The incubation periods of swelling in JLS-1 were attributed to the effects of dissolved tungsten.

The difference of the peak swelling temperature between neutron irradiation and ion-beam irradiation is estimated to be about 50 K.

References

[1] A. Kohyama, A. Hishinuma, D.S. Gelles, R.L. Klueh, W. Dietz, K. Ehrlich, J. Nucl. Mater. 233–237 (1996) 138.
[2] Y. Kohno, A. Kohyama, M. Yoshino, K. Asakura, J. Nucl. Mater. 212–215 (1994) 707.
[3] K. Shiba, A. Hishinuma, J. Plasma Fus. Res. 74 (1998) 436.
[4] A. Kohyama, Y. Katoh, M. Ando, K. Jimbo, J. Nucl. Mater. 51&52 (2000) 789.
[5] H. Sakasegawa, T. Hirose, A. Kohyama, Y. Katoh, T. Harada, T. Hasegawa, ASTM STP 1405, (2001) p. 546.

[6] H. Sakasegawa, T. Hirose, A. Kohyama, Y. Katoh, T. Harada, K. Asakura, T. Kumagai, ICFRM-10, (2001) 14–19 October.
[7] J.F. Ziegler, Ion beam interactions with matter. Available from <http://www.srim.org/>.
[8] H. Tanigawa, M. Ando, Y. Katoh, T. Hirose, H. Sakasegawa, S. Jitsukawa, T. Iwai, J. Nucl. Mater. 279–284 (2001) 279.
[9] S. Hiwatashi, Y. Kohno, K. Asakura, K. kohyama, J. Nucl. Mater. 179–181 (1991) 709.