Self-ion Irradiation Damage of F/M and ODS steels

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1. Introduction

Ferritic/martensitic (F/M) steels are the reference structural materials for future nuclear fission and fusion power reactors, as they have achieved the greatest technology maturity [1-3]. The temperature window for use of F/M steels is presently about 300-550 °C, the lower value being limited by irradiation-induced embrittlement effects and the upper value by a strong reduction in mechanical strength [4-7]. Therefore, improving mechanical properties and irradiation resistance are great concern in developing F/M steels.

Oxide dispersion strengthened (ODS) ferritic steels are potential high-temperature materials that are stabilized by dispersed particles at elevated temperatures [8-10]. These dispersed particles improve the tensile strength and creep rupture strength, they are expected to increase the operation temperature up to approximately 650 ºC and also enhance the energy efficiency of the fusion reactor. Moreover, the dispersed oxide particle or nano-cluster generally does the roll as a sink of radiation defects. Some reports described that the nano-clusters are strongly resistant to coarsening by annealing up to 1000ºC, and nanoclusters do not change after ion irradiation up to 0.7 dpa at 300 ºC. ODS steels will be inevitably exposed to neutron irradiation condition; the irradiation damages, creep and swelling are always great concern. The dispersed oxide particles are believed to determine the performance of the steel, even the radiation resistance.

In this study, F/M and ODS model alloys of Korea Atomic Energy Research Institute (KAERI) were irradiated by Fe^{3+} self-ion to emulate the neutron irradiation effect. The different radiation damage behavior between F/M and ODS steels were compared; the contributions of minor elements such as Ti, W and Mo were studied.

2. Methods and Results

Chemical compositions of model alloys are summarized in Table 1. Two F/M steels are listed in the upper part of the table; the major difference in between F/M steel is the Ti content. The F/M-2 specimen was designed to have more Ti content. F/M steels were fabricated by a vacuum induction melting and then formed into a plate by hot rolling at 1150 ºC. After that, normalizing was performed at 1050 ºC for 1h and tempering was carried out at 750 ºC for 2h. Two ODS steels are listed in the bottom part of table; the major difference in between ODS steels were W and Mo contents. It was designed that 2 wt% W of ODS-1 is replaced by 1 wt% Mo in ODS-2. The ODS steels were fabricated by a high energy miller (CM 20) at 240 rpm for 48 hours. The MA powders were placed in an AISI 304L stainless steel container, sealed after a degassing process, and consolidated by a hot isostatic pressing process at 1150 °C under a pressure of 100 MPa for 3 hours. A hot isostatic pressed billet was hot-rolled at 1150 °C into a plate with a reduction ratio of 40%.

All samples were cut into small pieces for ion-irradiation; length, width and thickness were 3, 1.5 and 0.5 mm, respectively. The surface of the samples were mechanically polished with diamond pastes, and then electro-polished by 10% perchloric acid. The electro-polished surface was irradiated by Fe^{3+} ion, the irradiation conditions are shown in table 2. The acceleration voltage was 6.4 MeV, the total amount of accelerated ions was 2.5 X 1020 ions/m^2, radiation temperature was 300 ºC. The resulting damage profiles have been calculated by means of the Montecalro simulation (SRIM 2012) as shown in figure 1. The maximum damage rate in F/M and ODS steels were estimated roughly 6 dpa, the thickness of the damage layer was estimated 2 μm.

	Cr	Ti	Mo	W	Zr	Y_2O_3	Fe
F/M-1	9	0.01		1.1	0.01		Bal.
F/M-2	9	0.02		1.1	0.01		Bal.
ODS-1	15	0.3		2		0.35	Bal.
ODS-2	15	0.3	1			0.35	Bal.

Table. 1. Ion irradiation test samples

Acc. Vol.	**6.4 MeV**
Source	**Fe^{3+}**
Temp.	**300ºC**
# of ions	**$2.5X10^{20}$ ions/m^2**
DPA	**~ 6 DPA**

Table 2. Ion irradiation test conditions

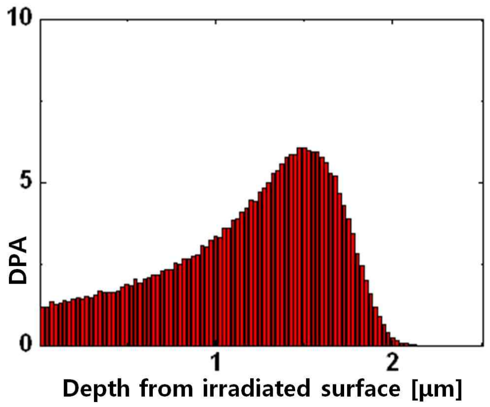

Fig. 1. Estimated depth of the damaged layer using Montecarlo simulation code (SRIM 2012)

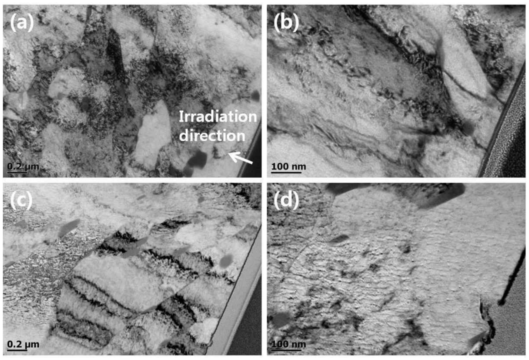

Fig. 2. Radiation-damaged area of the ARAA alloys were shown. The microstructure of the F/M-1, (a) 0-2.5 μm deep from the surface, (b) typical morphology of damaged area. The similar microstructure of the F/M-2 were shown in (c) and (d), respectively. Lots of point and line defects were found, no specific voids were observed.

The irradiation induced hardness change in the damaged layer was evaluated by nano-indentation. Indentation loads in the range from 2 to 100 mN corresponding to a maximum indentation depth from about 100 nm to 2 μm were applied. A TEM sample was manufactured using a focused ion beam (FIB, NOVA200), and the sample was sliced into thin foil to be observed along the perpendicular direction of Fe3+ ion irradiation. The microstructure evolutions in irradiated samples were shown by bright field image of TEM (JSM 2100F).

In figure 2, the ion-irradiated areas of the F/M steels are shown by TEM bright-field image. Ion-irradiated direction is indicated by white arrow, the microstructure of radiation-damaged area (0 - 2.5 μm deep from the surface) is fully shown in figure 2(a) and 2(c). Two different samples commonly showed that the microstructure of un-irradiated and irradiated area is quite similar; it was hard to discriminate one from another. The magnified image of typical morphology is shown in figure 2(b) and 2(d), lots of point or line defects, dislocation cell structures were observed. The effect of Ti addition was hard to be quantified; however, the typical line defects and dislocation loops were more frequently observed in F/M-2 specimen as shown in figure 2(d). The observed microstructure evolution in figure 2(d) seemed to be advantageous for higher toughness of F/M steel, because the ductile brittle transition temperature (DBTT) of the F/M-2 was 20 ºC less than the F/M-1 specimen under the same impact test conditions.

In figure 3, ion-irradiated areas of the ODS steels are shown by TEM bright-field image. Ion-irradiation direction is indicated by black arrow, the microstructure of radiation-damaged area (0 - 2.5 μm deep from the surface) is fully shown in figure 3(a) and 3(c). Contrary to the F/M steel, microstructure of un-irradiated and irradiated area could be roughly distinguished by cavities distributions. The magnified image of typical morphology is shown in figure 3(b) and 3(d), small nano-sized circular cavities were observed. If cavities were generated in the specimen during radiation, the probability of cavities diffusing to sinks or trapping sites increases. The dispersed oxide particle or nano-cluster generally does the roll as a sink of radiation defects, probably the location of cavities are coincident with the location of oxide particles or nano-clusters.

The effect of Mo addition was hard to be quantified; however, the number density of cavities seemed to be reduced in ODS-2 than ODS-1. Instead, ODS-2 showed more dislocation loops and cell structure after radiation, Mo in solid solution is generally effective in reducing dislocation mobility of iron matrix. Although the oxide particles or nano-clusters are the sinks of cavities, the Mo-added matrix suppressed the generation of cavities, because the more amount of radiation damage is stimulated in the Mo-added matrix.

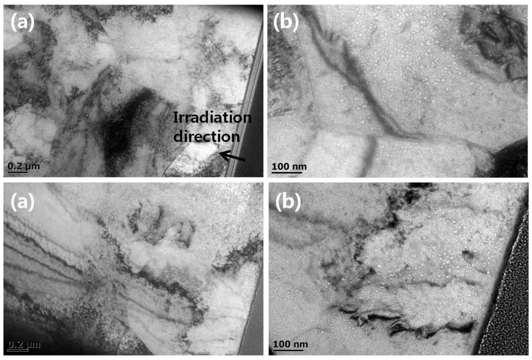

Fig. 3. Radiation damaged area of the ODS alloys were shown. The microstructure of the ODS-1, (a) 0-2.5 μm deep from the surface, (b) typical morphology of damaged area. The similar microstructure of the ODS-2 were shown in (c) and (d), respectively. Small nano-sized circular shapes were observed, they were cavities.

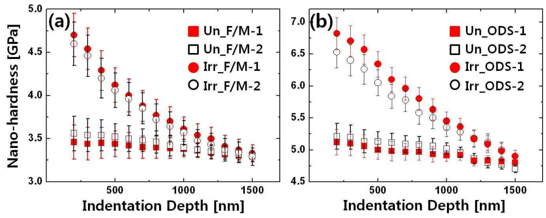

Fig. 4. Nano-hardness were measured on the surfaces of the un-irradiated and the irradiated specimens. Nano-hardness of (a) F/M steel, (b) ODS steel were measured by the differentiating indentation depth, respectively.

In figure 4, nano-hardness of F/M and ODS steel were measured on the surfaces of the un-irradiated and the irradiated specimens. Nano-hardness of specimens were measured by the differentiating indentation depth, nano-hardness profile of F/M and ODS steels were shown in figure 4(a) and 4(b), respectively. First, it was estimated that the hardness of the ODS steel was generally higher than the F/M steel. The un-irradiated specimen of F/M-1 showed nano-hardness value of 3.48 GPa while the ODS-1 showed 5.2 GPa. Second, the F/M and ODS commonly exhibit a radiation hardening; however, the irradiation hardening was more active in the F/M steel than ODS steel. The nano-hardness variations of F/M and ODS steels were roughly 35%, 25%, respectively. Last, the 0.02 wt% Ti-added F/M steel (F/M-2) and the 1 wt% Mo-added ODS steel (ODS-2) showed smaller radiation hardening rate. Ti in F/M steel and Mo in ODS steels were effective to increase the toughness as well as the radiation resistance. During radiation, materials inevitably stimulate the mechanical stress from bombardment of atoms. In those reactions, the stress capacity is dependent on the way of microstructural behavior. F/M and ODS steels gave different microstructural response to the external stress, corresponding irradiation hardening rate were different.

3. Conclusions

In this study, Fe^{3+} self-ion irradiation is used as means of introducing radiation damage in F/M steel and ODS steel. The ion accelerator named DuET (in Kyoto University, Japan) was used for irradiation of Fe^{3+} ion by 6.4 MeV at 300 ºC. The maximum damage rate in F/M and ODS steels were estimated roughly 6 dpa. After radiation, point or line defects were dominantly observed in F/M steel, on the other hands, small circular cavities were typically observed in ODS steel. Nano-indentation is a useful tool to determine the irradiation-induced hardness change in the damage layer of ion-irradiated iron base alloys. The F/M steel and ODS steel commonly exhibit a radiation hardening; the irradiation hardening was more active in the F/M steel than ODS steel. This difference is likely to be caused by the presence of oxide particles and minor elements in F/M and ODS steels.

REFERENCES

[1] M. Ando, H. Tanigawa, S. Jitsukawa, T. Sawai, Y. Katoh, A. Kohyama, K. Nakamura, H. Takeuchi, J. Nucl. Mater. 307–311, 260 (2002).
[2] H.-H. Jin, C. Shin, W.W. Kim, Solid State Phenom. 135, 119 (2008).
[3] W.C. Oliver, G.M. Pharr, J. Mater. Res. 19, 3 (2004).
[4] C. Pokor, Y. Brechet, P. Dubuisson, J.-P. Massoud, A. Barbu, J. Nucl. Mater. 326, 19 (2004).
[5] M. Matijasevic, E. Lucon, A. Almazouzi, J. Nucl. Mater. 377, 101 (2008).
[6] R. Coppola, R. Lindau, M. Magnani, R.P. May, A. Möslang, J.W. Rensman, B. vander Schaaf, M. Valli, Fusion Eng. Des. 75–79, 985 (2005).
[7] M. Matijasevic, A. Almazouzi, J. Nucl. Mater. 377, 147 (2008).
[8] A. Alamo, V. Lambard, X. Averty, and M.H. Mathon, J. Nucl. Mater., 329-333, 333 (2004).
[9] T. Yoshitake, Y. Abe, N. Akasaka, S. Ohtsuka, S. Ukai, and A. Kimura, J. Nucl. Mater., 329-333, 342 (2004).
[10] S. Yamashita, N. Akasaka, and S. Ohnuki, J. Nucl. Mater., 329-333, 377 (2004).