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Void swelling in ferritic-martensitic steels under high dose ion irradiation: Exploring possible contributions to swelling resistance

Xu Wang [a,b], Qingzhi Yan [c], Gary S. Was [b], Lumin Wang [b,a,*]

[a] College of Energy, Xiamen University, Xiamen 361102, China
[b] Department of Nuclear Engineering and Radiological Science, University of Michigan, Ann Arbor, MI 48109, United States
[c] Institute of Nuclear Materials, University of Science and Technology Beijing, Beijing 100083, China

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ABSTRACT

Ion irradiations were performed on reduced-activation ferritic-martensitic steels CNS (China Nuclear Steel) I and CNS II up to 375 dpa and 450 dpa at 460 °C using 5 MeV Fe^{++} ions with 10 appm and 100 appm pre-implanted helium. The swelling of 9Cr CNS I was 5.7–9%, while the 12Cr CNS II showed 20–300× less swelling than CNS I. Helium effects and possible contributions to swelling resistance in ferritic-martensitic steels were explored based on the results of transmission electron microscopy analysis. Radiation induced high density precipitates $(Fe,Cr)_2C$ were found to account for the superior swelling resistance of CNS II.

Core internal components of advanced reactor concepts and fusion reactors will face high radiation doses of 200 dpa or above [1–3]. Cladding and duct in the traveling wave reactor will experience doses up to 600 dpa [1,2]. Ferritic-martensitic (F-M) steels are candidate structural materials for these high dose reactor applications because of their swelling resistance under irradiation. In addition, reduced-activation F-M (RAFM) steels that eliminate long-lived radionuclides from alloy elements such as Mo, Nb, Ni, Cu and N are being considered to minimize residual activity.

CNS I and CNS II are two types of RAFM steels developed at the Institute of Nuclear Materials, University of Science and Technology Beijing (USTB). Their compositions are given in Table 1. The main difference between them is that there is ~9% Cr in CNS I and ~12% Cr in CNS II. The heat treatment of CNS I and CNS II consisted of 950 °C/30 min/water cooling + 780 °C/90 min/air cooling or 1000 °C/30 min/water cooling + 800 °C/120 min/air cooling, respectively. They have demonstrated favorable mechanical and corrosion properties [4–7]. To study the radiation effects, ion irradiation is often used as a surrogate for neutron irradiation because there is little or no residual activity, it requires short irradiation times, and is conducted under well-controlled experimental conditions and at relatively low cost[1,8,9]. In previous work [10], Fe^{++} irradiation was performed up to 188 dpa (4.6×10^{17} ion/cm^2) on CNS I, CNS II and commercial T91. The CNS I samples exhibited slightly lower swelling than commercial T91, while CNS II showed significantly lower swelling than CNS I and T91. In this work, the irradiation dose of CNS I and II was increased up to 450 dpa to determine the swelling resistance after high dose ion irradiation. Possible contributions to swelling resistance in F-M steels will be explored based on these results.

Prior to irradiation, the sample surface was ground using up to #4000 grit SiC paper, followed by mechanical polishing with up to 0.05 μm alumina polishing solutions. After polishing, samples were pre-implanted with 10 appm or 100 appm helium at room temperature using a 400 kV ion implanter in the Michigan Ion Beam Laboratory (MIBL) at University of Michigan. To produce an approximately flat helium concentration (±10%) through the depth of 300–1000 nm from the surface (shown in Fig. 1), helium was pre-implanted using five different energies (80, 140, 220, 310, 420 keV). After helium implantation, ion irradiations were performed at MIBL using a 1.7 MV tandem accelerator or a 3 MV Pelletron accelerator using 5 MeV Fe^{++} ions in raster-scanned mode at a temperature of 460 °C. The temperature was monitored in 3 areas-of-interest (AOIs) on each sample using a high-resolution two-dimensional infrared thermal imager throughout the irradiation. The combined control of the resistive heater and air cooling loop on the stage was used to maintain the temperature within ±10 °C of the target temperature. Based on the many high dose irradiations conducted at MIBL, 460 °C is the peak swelling temperature of F-M steels such as HT-9 [14], T91 and so on. While the irradiations on CNS were only conducted at 460 °C, the authors do not exclude the

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* Corresponding author at: 2355 Bonisteel Blvd, Ann Arbor, MI, 48109, United States.
E-mail address: lmwang@umich.edu (L. Wang).

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Table 1
The composition of CNS I, CNS II and T91 (wt%).

Alloy	Heat Number	Cr	C	Si	Mn	W	Ta	Ti	V	N	Fe
CNS I	1109H4	8.5–9.5	0.09–0.10	0.10–0.15	0.45–0.50	2.0–2.5	~0.05	0.01–0.02	0.04–0.05	0.035–0.045	Bal.
CNS II	1206H2	11.0–12.0	0.12–0.15	0.20–0.25	0.8–1.0	2.3–2.5	~0.08	–	0.01–0.02	0.04–0.05	Bal.

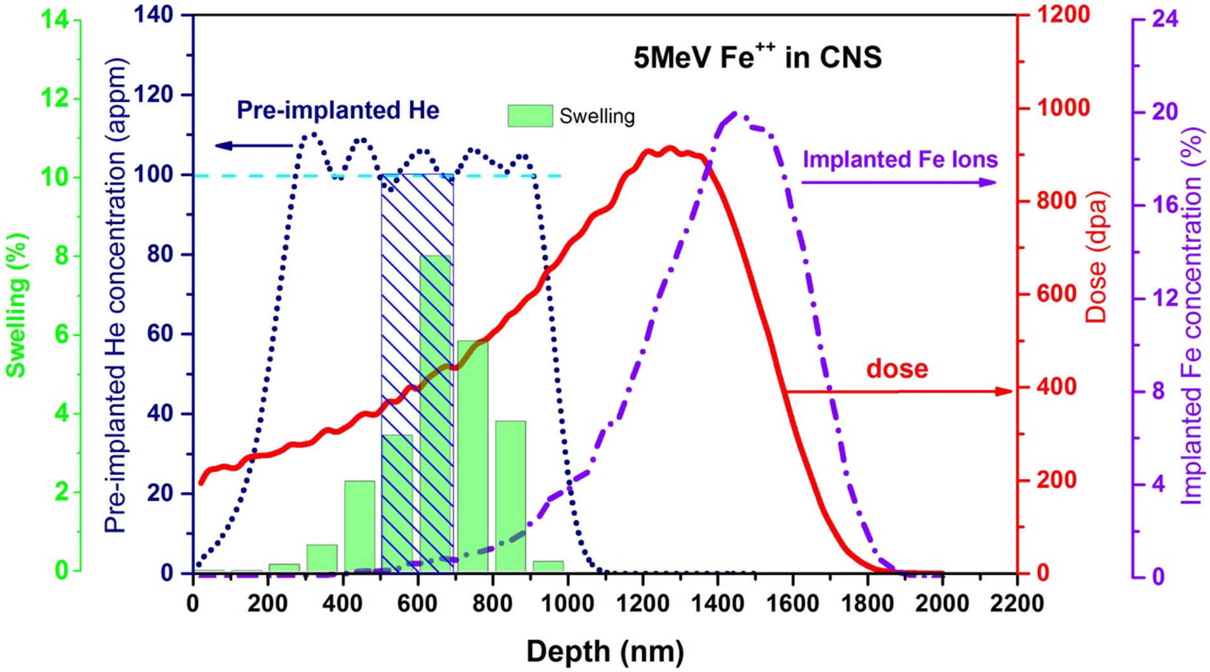

Fig. 1. Profiles of pre-implanted helium distribution, injected Fe^{++} concentration, dose and swelling under 375 dpa Fe^{++} irradiation. 500–700 nm region is selected for swelling analysis and 600 nm is used for the irradiation dose calculation.

possibility that the peak swelling temperature of CNS might not be located at 460 °C.

Depth profiles of displacement damage, injected Fe ion and pre-implanted helium are shown in Fig. 1. They were calculated using the Quick Kinchin-Pease Mode in SRIM with displacement energy of 40 eV [11]. Fig. 1 also demonstrates one example of the swelling through depth. The swelling dropped off after 700 nm due to the injected interstitials effects [12–14]. The strong depth dependence of swelling before 700 nm may due to the dose gradient, surface effects, and the void sink strength gradient caused by void distribution. The region of 500–700 nm was chosen for calculation of the nominal swelling value to avoid the proximity to the surface, to be within the flat helium concentration range, to avoid strong injected interstitial effects, and to achieve the highest possible dose and limit the dose variation throughout the examined region [10,14,15]. A depth of 600 nm was used for the nominal irradiation dose calculation, and the doses of the Fe^{++} irradiation in this work were 375 dpa and 450 dpa. The dose rate was 3.5×10^{-4} dpa/s.

TEM sample preparation and electron microscopy were conducted at the University of Michigan Electron Microbeam Analysis Laboratory (EMAL). Cross-section TEM samples were prepared by focused ion beam (FIB) lift-out method using a FEI Helios 650 Nanolab Dualbeam FIB. The transmission electron microscope (TEM) and scanning transmission electron microscope (STEM) observations were conducted in a JEOL 3011 TEM and a JEOL 2100 STEM, respectively.

9Cr-CNS I is fully tempered martensite while 12Cr-CNS II consists of tempered martensite and <5% ferrite. The mean lath/subgrain size of CNS I and CNS II are 0.56 μm, and 0.36 μm, respectively. Regarding the microstructure of F-M steels prior to irradiation, the tempered martensite consist of matrix (including grain boundary), lath/subgrain boundaries, carbides, dislocations and solutes in the matrix; while the ferrite consist of matrix, dislocations and solutes. After irradiation, conventional through-focus TEM and high angle annular dark field (HAADF) STEM and BF STEM imaging were used to identify voids in these samples. Z-contrast technique HAADF was mainly used for void counting since voids would not be hidden under other diffraction contrast.

Fig. 2 shows the swelling results of CNS I and II in the region 500–700 nm irradiated to 375 and 450 dpa. Swelling values, void density and void mean diameter are shown beneath the under-focused TEM images. There are around 20% uncertainties in the swelling data due to the measurement of the void diameter and sample thickness, and the spatial heterogeneity in void distribution. Compared to the 188 dpa data reported earlier [10], the swelling of CNS I increased to 8.0% and 5.7% at 375 dpa for 10 appm and 100 appm helium samples, respectively. At 450 dpa, it increased to 8.9% at 10 appm helium and to 5.7% at 100 appm helium. For CNS I, the 100 appm helium samples exhibited a higher density of smaller voids compared to the 10 appm helium samples. This is presumably because helium increased void nucleation by lowering the void critical radius and the available vacancies were distributed among more voids, decreasing the diameter on average [16]. At 188 dpa, the CNS I with 100 appm helium had higher swelling than that with 10 appm helium sample due to the higher void density. The situation reversed when the dose was increased to 375 dpa and 450 dpa, in which the swelling of CNS I with 10 appm helium was higher than 100 appm helium specimens. This indicates that at lower dose (188 dpa), void nucleation is dominant thus void density contributes more to swelling. While at higher doses (375 dpa and 450 dpa), void growth is dominant and void size contributes more to swelling. Meanwhile, the high density of voids may act as neutral sinks thereby retarding swelling [16,17]. Suppression of void swelling by pre-implanted and simultaneous helium was observed in ion irradiated pure iron [18,19], annealed austenitic alloy [20] and F-M steel [14].

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