SECTION 1

PHYSICS OF RADIATION DAMAGES AND EFFECTS IN SOLIDS SWELLING OF FERRITIC STRUCTURAL MATERIALS AT HIGH LEVELS OF DAMAGE DOSES AND GAS CONCENTRATIONS

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Investigation results of the swelling behavior of three ferritic steels under conditions of simultaneous irradiation with 1.8 MeV Cr^{3+} ions and gases (helium or hydrogen) at peak swelling temperatures and doses to 50 and 200 dpa are presented. It is shown that the behavior of swelling depends on the concentration of helium or hydrogen. Determined that helium and hydrogen have different influence on the nucleation processes and growth of vacancy voids. Dual irradiation with hydrogen leads to a decrease in voids formed under irradiation. Helium has a stronger effect on voids nucleation, increasing their concentration by almost 10 times with a significant decrease of their size.

PACS: 61.72.Cc, 68.55.Ln

INTRODUCTION

Currently, heat-resistant 9...12% chromium ferriticmartensitic steels are considered promising materials for fusion reactor, 4-th Generation reactors due to their low induced activity, low void swelling and creep, and high resistance to high temperature and helium embrittlement.

There are various international programs pointed at developing promising 4-th Generation reactors, Traveling-Wave Reactors, as well as fusion reactors and accelerator-driven spallation (ADS) systems. These programs are based on the use of ferritic-martensitic steels capable of operating at levels of damage doses in excess of 200 displacements per atom (dpa). Besides it can be used in a case of high gas levels (helium and hydrogen) which have affect not only on the mechanical properties, but also on the increasing of the swelling rate [1-3]. Moreover, ferritic-martensitic steels are potential candidate alloys as structural materials for the Spallation Neutron Source (SNS). In this source, materials are damaged as a result of exposure to strong irradiation, thermal stroke, erosion and corrosion [4, 5].

Ferritic-martensitic steels were chosen for investigation because of its excellent response to neutron irradiation compared with austenitic steels [6]. A lot of data have been generated for this alloys subjected to fission neutron irradiation, but none for the SNS condition. Helium generation in irradiated metals is known to assist void nucleation and thereby accelerates the onset of void swelling at incubation period [7].

The aim of this work was to study the swelling of industrial ferritic-martensitic steels EP-450 and HT-9 - Russian and American production, respectively, and

Japanese-made martensitic steel F82H – while irradiating them with chromium and gas ions (helium and hydrogen) to the doses of 200 dpa and concentrations of helium 100 appm and hydrogen 10000 appm. To study the radiation resistance of these materials, heavy ion irradiation was used. This simulation method is currently the only way to achieve ultra-high doses of radiation and to obtain high concentrations of gases.

1. MATERIALS AND EXPERIMENTAL DETAILS

To study steels, standard disks with a diameter of 3 mm and a thickness of 0.2 mm were used. The chemical compositions and heat treatment conditions of the three alloys are listed in Table.

Dual irradiations $(Cr^{3+}+He^+; Cr^{3+}+H^+)$ were performed using 1.8 MeV Cr^{3+} with 20 keV H^+ and 40 k9B He⁺ up to concentration levels of 0-100 appm helium and 0...10000 appm hydrogen at the peak swelling temperatures as determined for each material during earlier studies not involving gas implantation (430...480 °C) [8].

Irradiation was carried out on the ESUVI electrostatic heavy-ion accelerator located at Institute of SSPMST NSC KIPT. The design and main parameters of the facility have been presented previously [8, 9].

Fig. 1 is a damage deposition, the gas injection profiles and accompanying damage profiles for 40 keV He^+ and 20 keV H^+ , showing that very high but well-defined levels of gas can be deposited in the examined region without inducing significant amounts of additional damage dose.

Chemical	compositions	of	steel	ls

Steel	Chemical composition, wt%							Thermomechanical		
	Cr	С	Mo	V	Mn	Si	Ni	Nb	W	treatment
EP-450	11.2	0.11	1.13	0.20	0.60	0.60	0.08	0.5	_	1050 °C/0.5 h+ 720 °C/1 h
HT-9	12.1	0.2	1.04	0.28	0.57	0.17	0.51	_	0.45	760 °C/0.5 h +33% CD
F82H	7.44	0.10	_	0.20	0.5	0.14	_	_	_	1040 °C/0.5 h +740 °C/2 h



Fig. 1. Profiles of dose distribution and implantation ions fraction at depth dose (----) and injected ion profiles (----) for 1.8 MeV Cr³⁺.
20 keV H ion profiles (----) and 40 keV He (----)

The calculation was made using the SRIM-2006 program. Selecting the depth of the investigated region based on two prerequisites:

1. The investigated layer must be on sufficient depth from irradiated surface to eliminate the surface influence.

2. The number of implanted chromium ions must be minimal.

Note that at these damage doses, the concentration of injected chromium reaches ~ 20%, potentially causing significant alteration in the chemical composition of steel, and at high irradiation doses, excess interstitial chromium atoms have a strong suppressive effect on the void nucleation [10–13]. For microscopy analysis, the irradiated samples were thinned on both sides to 100...200 nm using the pulsed electric polishing technique [14]. This depth also minimizes the influence of the surface on the observable effects, especially at a very high dpa rate in this region $(1\cdot10^{-2} \text{ dpa/s})$.

2. RESULTS OF INVESTIGATIONS 2.1. INITIAL STRUCTURE OF EP-450, HT-9, F82H STEELS

EP-450 is a ferritic-martensitic steel used as a standard structural material for hexagonal fuel assembly wrappers in the BN-600. The microstructure of EP-450 steel before irradiation (Fig. 2,a) was a duplex structure of tempered martensite (referred to sorbite in Russian)

and ferrite in a ratio of ~ 1:1. Large globular $M_{23}C_6$ carbides were observed on ferrite-ferrite and ferrite-tempered martensite grain boundaries, which have a strong contrast in the bright-field image and smaller carbides were found at the boundaries of martensite matrix.

The initial microstructure of HT-9 steel (see Fig. 2,b) has a pronounced deformation structure, typical for a degree of deformation of $\sim 30\%$ CD, and is a dislocation network with a high density of dislocations, sometimes having a cellular structure.

The starting structure of F82H (see Fig. 2,c) is fully martensitic. The dislocation line density is ~ $1 \cdot 10^{14}$ m⁻². $M_{23}C_6$ carbides are observed in the matrix and on grain boundaries, with number density and mean size of $6 \cdot 10^{19}$ m⁻³ and 73 nm, respectively. Only a few MC carbides are observed in the matrix, with number density and mean size of $1 \cdot 10^{20}$ m⁻³ and 14 nm, respectively. The mean width of the lath structure is 440 nm.

2.2. EFFECT OF CHROMIUM IONS IRRADIATION

In this paper the primary focus is on the influence of dual beam irradiation on swelling behavior. In an earlier paper we examined the temperature and dose dependencies of swelling of EP-450 and HT-9 in the absence of helium or hydrogen injection over the range 50...300 dpa [15]. Ferrite grains were observed to begin swelling at a much lower dose than seen in neighboring tempered martensite grains. Voids in ferrite formed as early as 50 dpa with peak swelling at 480 °C.

As shown in Fig. 3 the steady-state swelling rate of the ferrite was ~ 0.2%/dpa even in the absence of gas injection, reached at ~ 150 dpa and still evident at 300 dpa, while the swelling of the tempered martensite phase started only after 200 dpa with the swelling rate still increasing at 300 dpa, but not yet reaching ~ 0.2%/dpa. Irradiation of HT-9 steel to 200 and 300 dpa also led to the formation of vacancy porosity. This dose range for HT-9 steel refers to the transient stage of swelling, when the void system is just beginning to form.



Fig. 2. Initial structure of EP-450 (a), HT-9 (b), and F82H (c) steels



Fig. 3. Swelling of dual-phase EP-450 in absence of gas injection

2.3. EFFECT OF HELIUM ON SWELLING OF FERRITIC-MARTENSITIC STEELS AT DUAL-ION IRRADIATION AT THE STEADY-STATE STAGE

Irradiation of EP-450, HT-9, and F82H steels was carried out at peak swelling temperatures: 480, 450, 430 °C, respectively.

Simultaneous irradiation of EP-450 steel with chromium and helium ions to 50 dpa and a helium level of 100 appm leads the swelling of ferrite grains to a value of 0.19%. The swelling during irradiation in absence of He injection is significantly lower (0.02%) [15]. Under simultaneous irradiation at a dose of 50 dpa and He concentration of 500 appm, the void density increases from $2 \cdot 10^{14}$ to $4 \cdot 10^{17}$ cm⁻³ and the swelling increases to 0.32% [16]. Increasing of dose to 200 dpa and a helium level of 100 appm leads to more intense void formation in both ferrite and tempered martensite phases with an average size of 9 and 5 nm, respectively. The average void concentration was $2 \cdot 10^{16}$ cm⁻³, with the swelling value 0.37% (Fig. 4).



Fig. 4. Microstructure observed in ferrite (a) and tempered martensite (b) phases of irradiated EP-450 steel under simultaneous irradiation Cr^{3+} , D = 200 dpa, He - 100 appm, $T_{irr} = 480$ °C

Irradiation of HT-9 steel to 200 dpa with a simultaneous injection of 100 appm helium leads to an increase in void concentration from $4 \cdot 10^{15}$ to

 $3 \cdot 10^{16}$ cm⁻³. In this case, the void size decreases from 18 to 7 nm, which leads to a decrease in the swelling value from 1.5 to 0.5% (Fig. 5)

The same tendency of swelling reduce with simultaneous irradiation with helium is observed in F82H martensitic steel (Fig. 6). After irradiation of Cr+He, the void concentration increases from $5 \cdot 10^{15}$ to $1.5 \cdot 10^{16}$ cm⁻³. In this case, the void size decreases from 14 to 3 nm, which leads to a significant reduction of swelling to 0.1%.



Fig. 5. Microstructure (a) and void size distribution (b) observed in HT-9 steel during simultaneous irradiation Cr^{3+} , D=200 CHa, T_{irr} =450 °C, He-100 appm



Fig. 6. Microstructure (a) and void size distribution (b) observed in F82H steel under simultaneous irradiation Cr^{3+} , D=200 cha, T_{irr} =430 °C, He – 100 appm

The results comparison of the effect of helium at the steady state stage of swelling in EP-450, NT-9, and

F82H steels is presented in Figs. 7, 8. Dual-ion irradiation of Cr+He leads to an increase in number density for all three materials by an order. The void size in all materials reduces significantly with increasing

helium concentration. Since the main contribution to the swelling is the void size, in spite of the increase in void number density under the influence of helium, there is a sharp reduction of swelling in all three materials.



Fig. 7. Effect of helium on number density (a), void size (b) and swelling (c) in EP-450 (\blacksquare), HT-9 (\bullet), and F82H (\blacktriangle) steels under simultaneous irradiation 200 dpa, 0 and 100 appm He

The obtained results suggest that steels with a martensitic structure are exposed to swelling less than steels with a ferritic-martensitic structure.

2.4. EFFECT OF HYDROGEN ON SWELLING OF EP-450 STEEL

Figs. 9, 10 show the effect of dual-ion beam irradiation (Cr+H) on the ferrite and sorbite phases of EP-450 at 480 °C during 50 dpa. The co-injection of 1000 appm H leads to sharp increase of void density both ferrite and sorbite phases. With a further increase in hydrogen concentration to 1 at. % change in void density less sharp (Fig. 10,a).



Fig. 8. Effect of helium on number density (a), void size (b) and swelling (c) in EP-450, HT-9, and F82H steels at 200 dpa and 0 and 100 appm He

Note that hydrogen initially increases swelling significantly, increasing from 0.02 up to 0.93%, but then decreases at levels above ~ 1 atomic percent or 5000 appm to 0.37%. Compared to irradiation with only Cr ions, irradiation with Cr + 5000 appm H increases the swelling by ~ 50 times, while under irradiation with Cr + 10000 appm H the swelling growth factor is 20. The co-injection Cr + H of 1000 appm H leads to dramatic reduction of size from 17 to 8 nm, with a sharp increase of number density from $2 \cdot 10^{14}$ cm⁻³ to $2 \cdot 10^{16}$ cm⁻³. Increasing hydrogen to 10000 appm, the swelling decreases primarily due to decrease of void size.



Fig. 9. Micrographs showing the effect of hydrogen co-injection to very high levels on swelling of ferrite phase in EP-450, D = 50 dpa, $T_{irr} = 480$ °C



Fig. 10. Eeffect of hydrogen on number density (a), void size (b) and swelling (c) in EP-450 steel under simultaneous irradiation $Cr^{3+}+H^+$ at $T_{irr} = 480 \ ^{\circ}C$, hydrogen levels of 0...10000 appm (\blacksquare -ferrite, \bullet - sorbite)

3. DISCUSSION

Analyzing the presented results in Figs. 3–10 and published earlier in [16], where the authors described the effect of co-irradiation of steel with a triple-beam (Cr+H+He), it is arguable that helium and hydrogen individually significantly affect the parameters of radiation porosity. Under implantation of helium and hydrogen, the void size is significantly reduced. The void density in this case increases by an order. The dependences obtained in the case of Cr+H and Cr+He co-irradiations, having different quantitative parameters, correspond to the general laws (of void density increasing and void size decreasing).

In addition to general trends, there are differences in the nature of the influence of helium and hydrogen on the parameters of porosity and swelling. The difference in the evolution of swelling processes with the simultaneous injection of helium or hydrogen can be explained by the following: helium influences in two directions: it increases the equilibrium concentration of vacancies and stabilizes small vacancy clusters.

Since in the presence of helium the critical void radius is significantly reduced, helium stabilizes small voids well. So, a complex of two vacancies plus helium can be stable at operating temperatures of the material. The helium atom can push its own atom of the matrix from the equilibrium position in the lattice and take its place in the lattice. Two interstitial helium atoms in the lattice are push out their own atom and form a complex of two helium – vacancy more probably.

Hydrogen has a binding energy with defects much less than helium due to its energy characteristics. Hydrogen does not have the ability to push its own atom from the equilibrium position in the lattice, therefore, creates less void nuclei. The critical radius of the void, when stabilized by hydrogen, is smaller than during the evolution of vacancy porosity, but much larger than in the case of helium. Therefore, the addition of hydrogen leads to the evolution of porosity with large void sizes, compared with helium, but with a lower density. During double irradiation of EP-450 steel (Cr+H, Cr+He), voids are observed in both structural components during the incubation period, which is not typical for irradiation only with heavy ions.

Note that when steels are irradiated only with chromium ions, the porosity is uniform, however, void-free boundary zones and local zones with increased porosity are observed [15]. After dual beam irradiation with both Cr+H and Cr+He, the efficiency of the effect of grain boundaries and surface on the evolution of radiation porosity decreased. Void-free zones along grain boundaries and the surface of the sample are generally absent. Probably, this is due to the fact that helium and hydrogen contribute to the homogeneous nucleation of voids, and also that gas bubbles nucleate

and grow at grain boundaries, reducing the role of boundaries as sinks.

The diffusion rate of both hydrogen and helium in a defect-free lattice when H or He atoms are in the interstitial positions of the crystal lattice is very high even at room temperature. At elevated temperatures (430...480 °C) this speed is huge, so the gas after birth in nuclear reactions or implantation very quickly finds itself in traps, which can be intrinsic lattice vacancies, voids, dislocations, grain boundaries and other extended defects. The output energy of helium atom from the traps is usually higher than the output energy of hydrogen atom, therefore, helium is stored in the bulk of the material at higher temperatures than hydrogen. It can be expected that the effect of helium and hydrogen on the swelling has similar mechanisms, but manifests itself in different temperature ranges. The temperature limit of this effect for hydrogen is significantly lower than for helium. It seems that at the considered temperature of 480 °C, atomic hydrogen has little effect on swelling.

Getting into subcritical vacancy voids, helium atoms stabilize them, transforming them into supercritical ones. Thus, helium reduces the critical size of vacancy voids (as a rule, voids with a size already of several atomic volumes are stable in the presence of a helium atom), significantly accelerating the void nucleation. Due to this, the incubation period of swelling in the presence of helium is less than in absence of gas injection. This is confirmed by experimental observation of void density. In absence of helium, the average sizes of the observed voids are significantly larger than in the presence of helium.

A recently published paper [17] shows that coimplantation of helium with iron enhances cavity nucleation of alpha-Cr and co-implantation of hydrogen with iron augments cavity growth. Under triple beam irradiation, hydrogen also accelerates cavity nucleation via stabilizing initial embryos.

Why does helium suppress swelling at a high dose of 200 dpa? The reason is the high concentration of small voids, which reduce the rate of swelling. The swelling rate at the steady state is proportional to the value:

$$\frac{dS}{dK_0 t} \approx \frac{\delta B / \rho_v}{\left(1 + n / \rho_i + B / \rho_i\right)^2}; \qquad (1)$$

$$\rho_{i,v} = 2\pi\rho_d / \ln(R_e / r_{0i,v}) \approx \rho_d;$$
(2)

$$B = 4\pi b R_b; \tag{3}$$

$$\delta = (\rho_i - \rho_v) / \rho_i$$
(4)

Here ρ_d – dislocation density; R_e – radius of the area of dislocations influence; b – void density (number of voids per unit volume); B – void density as a sink of point defects; R_b – average void radius; n – neutral sinks density; $K_0 t$ – dose in dpa [18].

Right side (1) reaches its maximum at $B = n + \rho_v$. That is, for the fastest swelling, the void density *B* as sinks of point defects should be approximately equal to the density of dislocations.

With high void density, when $B/\rho_d >>1$, the swelling rate turns out to be low:

$$\frac{dS}{dK_0 t} \to \delta \frac{\rho_d}{B}, B \gg \rho_d .$$
⁽⁵⁾

Just as, at a high dislocation density, when $B/\rho_d \ll 1$, swelling rate is also low:

$$\frac{dS}{dK_0 t} \to \delta \frac{B}{\rho_d}, \ \rho_d >> B \,. \tag{6}$$

CONCLUSION

The study of swelling of ferritic – martensitic steels EP-450, NT-9 and martensitic steel F82H after dual irradiation with Cr^{3+} ions (1.8 MeV) and H⁺ (20 keV), He⁺ (40 keV) up to doses of 200 dpa at the swelling maximum.

It has been determined that hydrogen up to a concentration of 5000 appm increases the swelling of EP-450 steel, and at higher concentrations the swelling becomes lower. The swelling growth factor is 50 for 5000 appm and, accordingly, 20 for 10000 appm.

The effect of helium under dual irradiation is similar to the effect of hydrogen. However, He shows a stronger effect on the void nucleation, increasing their concentration by an order with a significant decrease in their size.

The simultaneous injection of helium under irradiation with heavy ions reduces the swelling of ferritic-martersite steels EP-450, NT-9 and martensitic steel F82H at high radiation doses (200 dpa).

At 50 dpa, hydrogen has a greater effect on the swelling of EP-450 steel than helium.

The authors are grateful to N.P. Lazarev for participating in the discussion of the obtained results, and to Y.E. Kupriyanova for help in carrying out of electron microscopic studies.

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Article received 09.07.2020

РАСПУХАНИЕ КОНСТРУКЦИОННЫХ МАТЕРИАЛОВ ФЕРРИТНОГО КЛАССА ПРИ ВЫСОКИХ УРОВНЯХ ПОВРЕЖДАЮЩИХ ДОЗ И КОНЦЕНТРАЦИЙ ГАЗОВ

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Представлены результаты исследования поведения распухания трех сталей ферритного класса в условиях одновременного облучения ионами Cr^{3+} с энергий 1,8 МэВ и газов (гелия или водорода) при температурах максимального распухания и дозах 50 и 200 сна. Показано, что поведение радиационного распухания во многом зависит от концентрации гелия или водорода. Установлено, что гелий и водород поразному влияют на процессы зарождения и роста вакансионных пор. Дуальное облучение с водородом приводит к уменьшению размера пор, образующихся при облучении. Гелий проявляет более сильное влияние на зарождение пор, увеличивая их концентрацию на порядок при значительном уменьшении их размера.

РОЗПУХАННЯ КОНСТРУКЦІЙНИХ МАТЕРІАЛІВ ФЕРИТНОГО КЛАСУ ПРИ ВИСОКИХ РІВНЯХ ПОШКОДЖУЮЧИХ ДОЗ І КОНЦЕНТРАЦІЙ ГАЗІВ

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Представлено результати дослідження поведінки розпухання трьох сталей феритного класу в умовах одночасного опромінення іонами Cr³⁺ з енергією 1,8 MeB і газів (гелію або водню) при температурах максимального розпухання і дозах 50 і 200 зна. Показано, що поведінка радіаційного розпухання багато в

чому залежить від концентрації гелію або водню. Встановлено, що гелій і водень по-різному впливають на процеси зародження і зростання вакансійних пор. Подвійне опромінення з воднем призводить до зменшення розміру пор, що утворюються при опроміненні. Гелій проявляє більш сильний вплив на зародження пор, збільшуючи їх концентрацію на порядок при значному зменшенні їх розміру.