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SINGLE AND DUAL ION IRRADIATION EFFECTS ON SWELLING BEHAVIOR OF EP-450 FERRITIC-MARTENSITIC STEEL

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In spite of all effects of researchers, up to now there is no understanding of the main radiation phenomena advances, in particular, of the swelling at simultaneously effect of damage dose with generated helium and hydrogen. Imperceptions of this problem is due to absence in the world science society of experimental equipment for investigations under super high damages (~ above 200 dpa). In this paper we carried such irradiation and performed results of the swelling behavior in typical ferritic-martensitic steel EP-450 under different irradiation condition: at high irradiation doses up to 300 dpa, and under dual irradiation with gases (helium or hydrogen). Simulation of radiation damage under environment which is typical for future reactors, as for fusion and accelerator driven system (ADS) the irradiation experiments were conducted using Electrostatic Accelerator with External Injector (ESUVI) at NSC KIPT. Swelling of EP-450 ferritic-martensitic steel were studied under irradiation by Cr ions up to the doses 300 dpa at temperature range 430-550^{o}C. Parameters of swelling, incubation state range; dose zone, where the swelling range reaches the steady state were determined. Irradiation under dual beam modes was conducted using 1.8 MeV Cr^{+3}, 40keV He^{+}, and 20keV H^{+}. It is shown that the behavior of radiation swelling depends on the concentration of helium or hydrogen. Helium and hydrogen have different effects on the kinetics and magnitude of swelling. In the incubation period helium increases void nucleation and raises their concentration. On the steady state period helium reduces swelling steels by reducing the voids size; hydrogen is also effective as helium in acceleration of the swelling beginning, but has less effect on voids nucleation that leads to swelling increase on steady-state period due to uniform rise of voids number density.

KEY WORDS: ion irradiation, helium, hydrogen, void swelling, microstructure, ferritic-martensitic steel

ВПЛИВ ОДНО И ДВОХ ПУЧКОВОГО ІОННОГО ОПРОМІНЕННЯ НА ПОВЕДІНКУ РОЗПУХАННЯ В ФЕРИТО-МАРТЕНСИТНІЙ СТАЛІ ЕП-450

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На сьогодні важливим є те, що немає повного розуміння розвитку основних радіаційних явищ, таких як, розпухання при взаємодії пошкоджень з гелієм та воднем, які утворюються під час ядерних реакцій. Відсутність розуміння обумовлена тим, що практично такі експерименти до нас ніхто не проводив, у світової наукової спільноти немає експериментального обладнання для утворення великих пошкоджень (до й вище 200 зна). В роботі представлені результати дослідження поведінки розпухання стандартної ферито-мартенситної сталі ЕП-450 за різних умов опромінення: при високих дозах пошкодження до 300 зна, а також при двох пучковому опроміненні іонами газів (гелій або водень). Вивчено розпухання ферито-мартенситної сталі ЕП-450 при опроміненні іонами хрому до доз 300 зна. В інтервалі температур 430…550^{o}C були отримані результати з параметрів пористості; тривалості інкубаційного періоду; області доз, в якій реалізується перехід до стаціонарної стадії; швидкості розпухання. Показано, що значення розпухання феритної сталі може перевищувати ~20%. Для імітації радіаційних пошкоджень, характерних для реакторів майбутнього покоління опромінення сталі ЕП-450 проводилось на Електростатичному прискорювачі з зовнішнім інжектором (ЕСУВІ), який було розроблено в ННЦ ХФТІ. Двох пучкове іонне опромінення проводилось при 1,8 МеВ Cr^{+3}, 40кеВ He^{+}, и 20кеВ H^{+}. Показано, що поведінка радіаційного розпухання залежить від концентрації гелію та водню; гелій та водень по-різному впливають на кінетику та розмір розпухання. В інкубаційному періоді гелій пришвидшує процес зародження пор, таким чином, збільшує їх концентрацію. На стаціонарній стадії гелій знижує розпухання сталей за рахунок зменшення розміру пор. Водень також ефективен, як і гелій в прискорені початку розпухання, але має менший вплив на зародження пор, це може привести навіть до збільшення розпухання на стаціонарній стадії за рахунок помірного росту концентрації пор.

КЛЮЧОВІ СЛОВА: іонне опромінення, гелій, водень, розпухання, мікроструктура, ферито-мартенситна сталь

ВЛИЯНИЕ ОДНО И ДВУХ ПУЧКОВОГО ИОННОГО ОБЛУЧЕНИЯ НА ПОВЕДЕНИЕ РАСПУХАНИЯ В ФЕРРИТО-МАРТЕНСИТНОЙ СТАЛИ ЭП-450

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Важно отметить, что на сегодня нет полного понимания развития основных радиационных явлений, в частности, распухания при взаимодействии смещающих повреждений с образующимися гелием и водородом. Отсутствие понимания обусловлено тем, что практически такие эксперименты до нас никто не проводил, т.к. в мировой научной общественности нет экспериментального оборудования для создания больших повреждений (до и выше 200сна). В данной работе представлены результаты исследования поведения распухания стандартной феррито-мартенситной стали ЭП-450 при различных условиях облучения: высоких дозах повреждения до 300 сна, а также при двойном облучении ионами газов (гелий или водород). Для имитации радиационных повреждений, характерных для реакторов будущего поколения, для термоядерных ректоров и реакторов, управляемых ускорителями, облучение стали ЭП-450 проводилось на Электростатическом ускорители с внешним инжектором (ЭСУВИ), разработанном в ННЦ ХФТИ исследовано распухание ферритно-мартенситных сталей ЭП-450 при облучении ионами хрома до доз 300 сна. В интервале температур 430…550^{o}C определены параметры пористости; продолжительность инкубационного периода; область доз, в которой происходит переход к стационарной стадии; скорость распухания. Показано, что величина распухания ферритной стали может превышать ~20 %. Двух пучковое ионное облучение проводилось при 1,8 МэВ Cr^{+3}, 40кэВ He^{+}, и 20кэВ H^{+}. Показано, что поведение радиационного распухания зависит от концентрации гелия или водорода; гелий и водород по-разному влияют на кинетику и величину распухания. В инкубационном периоде гелий увеличивает скорость зарождения пор и, следовательно, увеличивает их концентрации. На стационарной стадии гелий снижает распухание сталей за счет уменьшения размера пор. Водород также эффективен как и гелий в ускорении начала распухания, но имеет меньшее влияние на зарождение пор, что может привести даже к увеличению распуханию на стационарной стадии за счет постепенного увеличения концентрации пор.

КЛЮЧЕВЫЕ СЛОВА: ионное облучение, гелий, водород, распухание, микроструктура, феррито-мартенситная сталь

Ferritic-martensitic steels are now the more attractive materials-candidates for claddings and wrappers of nuclear reactors and for the first wall of fusion reactors due to their low induced activity, low void swelling and creep, high resistance to high-temperature and helium embrittlement. There are different international programs of 4 generation reactors development and fusion reactors. These programs are based on the use of ferritic-martensitic steels that will operate in the wide range of temperatures under damage doses of 200 dpa and higher and also under high levels of gases (helium and hydrogen) [1-3]. Also, it is supposed to use ferritic-martensitic steels for reactor TWR claddings; because these steels have the lower swelling under high doses of irradiation. Moreover, ferritic- martensitic steels are potential candidate alloys as structural materials for the Spallation Neutron Source. In the intense spallation neutron sources for the material science as well as the accelerator driven system for transmuting long lived nuclides to shorter ones, materials of target and its vessel are damaged by various influence of strong irradiation, thermal shock, 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]. Until recently, hydrogen was concurrent hydrogen generation can also assist void nucleation and possibly accelerates swelling even in the absence of helium. Hydrogen is known to be strongly captured in helium-nucleated voids or bubbles, thereby contributing to cavity stabilization [8, 9]. Furthermore, in some alloy systems co-injected helium and hydrogen appear to interact synergistically to strongly promote swelling [10, 11].

This paper summarized the effects of different doses (50 -300 dpa) for understanding swelling behavior and actuality of damage and gases (helium and hydrogen) effects on EP-450 ferritic-martensitic steel. To obtain materials performance data in such a severe irradiation environment, experiments were carried out using the ESUVI, because this unique facility could simulate the gas/dpa ratios expected under fussion environment. The objectives of the present study are (1) investigation of swelling of industrial ferritic-martensitic steels EP-450 under irradiation by chromium ions up to the doses of 300 dpa; (2) explore the effects simultaneous displacement damage and gas co-injection on swelling of EP-450 steel in response to allow development and refining of the co-injection techniques needed for future studies.

MATERIAL AND EXPERIMENTAL TECHNIQUE

The EP-450 steel is a typically example of ferritic-martensitic steel used as standard structural material for hexahedral cladding fuel assembly in BN-600 and future fast reactors. In this steel after partial conversion of γ-α and after heat treatment has a duplex structure of tempered martensite (sorbite), and ferrite, in a ratio of 1: 1. For investigation were used standard 3 mm diameter microscopy disks of 0.2 mm thickness. The composition of steel and heat treatment conditions are shown in Table1. The single ion irradiation proceed under 1.8 MeV Cr^{3+} ions with TEM discs irradiated at temperature of swelling maximum 480^{0}C to doses 50 -300 dpa. The dual ion irradiation under helium levels were 0-8000 appm and hydrogen levels - 0-10000 appm. The main irradiation parameters of the specimens are listed in Table 2. Irradiation was carried out on ESUVI electrostatic heavy-ion accelerator located at Kharkov Institute of Physics and Technology. This facility has been used previously to conduct a variety of studies on various alloys [12-13].

Table. 1.

Chemical compositions of EP-450 steel (wt, %).

Steel	Type of precipitates	Chemical composition, wt%								
		C	Cr	Si	Mn	Mo	V	Nb	Ni	Other
EP-450 (13Cr2MoNbVB)	$M_{23}C_6$, MC	0.10-0.15	11.0-13.5	0.6	0.6	1.2-1.8	0.1-0.3	0.25-0.55	0.30	B-0.004

HT: 1050^{0}C/0.5h+720^{0}C/1h

Table. 2.
Irradiation parameters of EP-450 specimens. Displacement dose (D), temperature of swelling maximum (T), helium (He, appm) and hydrogen (H, appm) levels

Specimen	T_{sw}^{max} (^{0}C)	D (dpa)	He, appm	H, appm
EP-450 (13Cr2MoNbVB)	480	50	-	-
			100	-
			500	-
			8000	-
			-	1000
			-	5000
			-	10000
	480	200	-	-
			100	-
			200	2000

dpa rate (k) – $2\cdot10^{-2}$dpa/s.

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. The calculation was made using the Kinchin-Pease option of the SRIM-2006 code. We were based on two reasons then selecting the depth of investigated layer: the investigated layer must be on sufficient depth from irradiated surface to eliminate the surface influence and, on the other hand, the number of implanted chromium ions must be minimal. Note that at the end of range ~20% Cr has been injected, causing significant chemical alteration, as well as acting as injected interstitials that strongly depress void nucleation [14–17]. To minimize the influence of the injected interstitial the irradiated specimens were thinned from both sides, choosing a layer at a depth of 100–200 nm from the ion-incident surface for microscopy analysis. This depth also minimizes the effect of the surface on the examined region, especially at the very high dpa rate of $1\cdot10^{-2}$ dpa/s in this region.

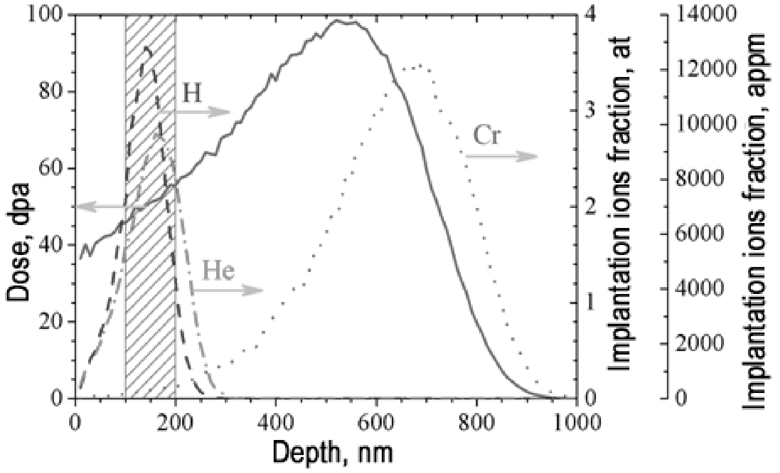


Fig.1. Profiles of dose distribution and implantation ions fraction at depth
dose (—–) and injected ion profiles (⋯⋯)for 1,8 MeV Cr^{+3}. 20 keV H ion profiles (- - -) and 40 keV He (– • –)

Microstructure of irradiated specimens were examined using a JEM-100CX transmission electron microscope.

STRUCTURE OF EP-450 STEEL AFTER IRRADIATION BY HEAVY IONS

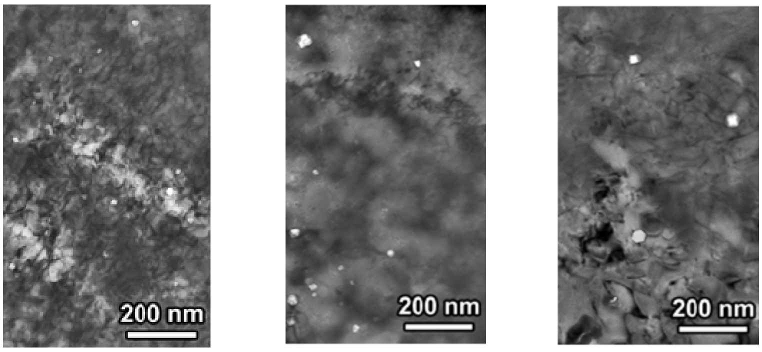
Unirradiated microstructure of steel EP-450 consists of ferrite and sorbite (tempered martensite), in relation to structural components, in the ratio of approximately 1:1. The main redundant phases in steel EP-450 after quenching and tempering are carbides $M_{23}C_6$ (where M is Cr, Fe and Mo) and MC. Mean grain size in steel EP-450 is about 20 μm.

The analysis of microstructure of unirradiated and irradiated specimens was showed previously [18]. The main attention in the present paper is paid to the analysis of swelling behavior in EP-450 steel. The structure changes of EP-450 steel under different irradiation temperature and the dose 100 dpa is shown on Fig.2.

Temperature dependence of main swelling parameters after irradiation under the dose 100 dpa in EP-450 steel is shown on Fig.3-5. Vacancy voids are observed at all studied temperatures under irradiation dose 100 dpa (Fig.3). Reduce of irradiation temperature causes the increase of voids size from 10 to 20 nm and decrease of their number density from $2\cdot10^{14}$ to $8\cdot10^{13}$cm^{-2} (Fig. 3-4). Under this dose void formation is observed only in ferrite.

Maximum of swelling of ferrite phase in steel EP-450 is observed at temperature 480^{o}C (Fig.5) that agrees with previously investigation performed on accelerators and in reactors [19].

Microstructure evolution of steel EP-450 in dependence on irradiation dose at temperature of maximal swelling (480°C) is shown on Fig.6.



a b c

Fig.2. Microstructure of steel EP-450 irradiated to dose 100 dpa at temperature 450 (a); 510 (b); 550°C (c).

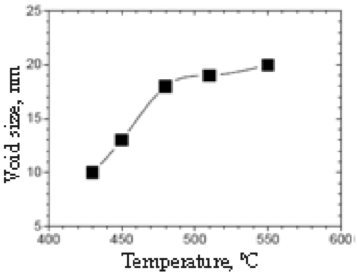


Fig.3. Temperature dependence of void size in irradiated EP-450 steel (D=100 dpa)

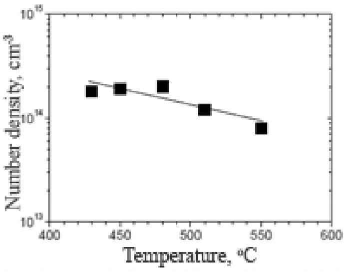


Fig.4. Temperature dependence of number density in irradiated EP-450 steel (D=100 dpa)

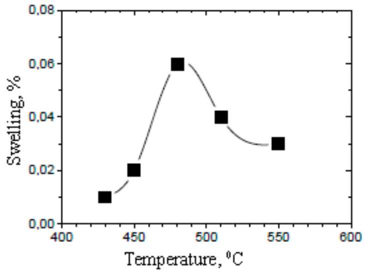
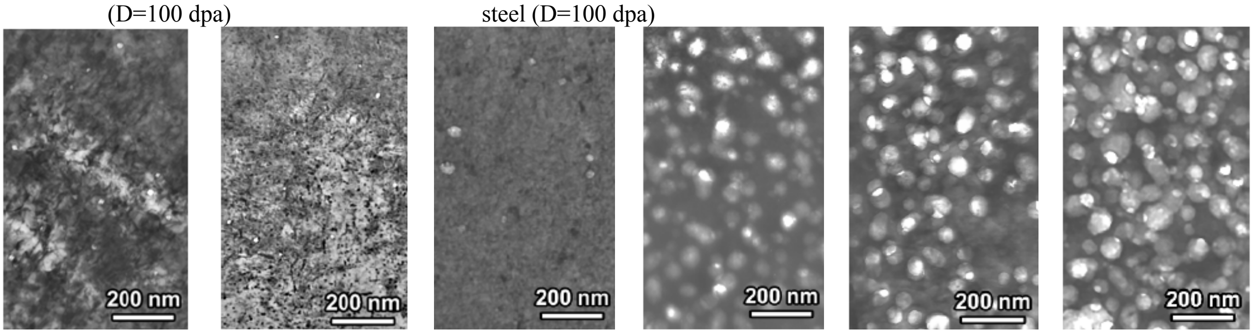


Fig.5. Temperature dependence of swelling in irradiated EP-450 steel (D=100 dpa)



a b c d e f

Fig.6. Microstructure of steel EP-450 irradiated at T_{irr}=480°C to dose 50 (a); 100 (b); 150 (c); 200 (d); 250 (e) and 300 dpa (f).

In previously work, it was shown that only ferrite swelling to dose 200 dpa [13]. The characteristic structure evolution is sharp increasing of swelling under doses higher than 200 dpa. At this dose the sorbite stars to swelling as ferrite. Swelling became more uniform from grain to grain and in sorbite grain although the nonuniformity of swelling is more pronounced and remains even under dose 300 pa (Fig.6f).

The mean size of voids is increased with the dose in steel EP-450 from 15 to 60 nm. Number density of voids typically increases but under irradiation doses ≥200 dpa stabilizes on the level (1…2)$\cdot 10^{15}$cm^{-2}. Dependence of swelling and parameters on irradiation dose are shown on Fig 7 - 9.

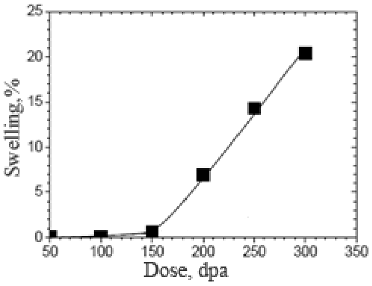


Fig.7. Dose dependence of swelling in irradiated EP-450steel (T_{irr}=480°C)

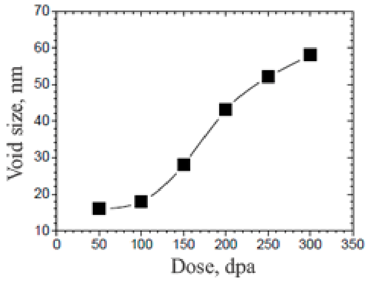


Fig.8. Dose dependence of void size in irradiated EP-450steel (T_{irr}=480°C)

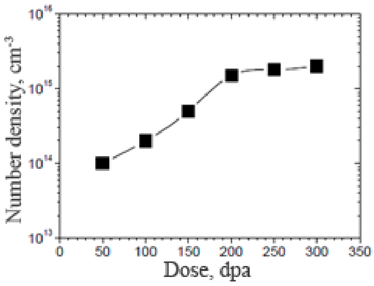


Fig.9. Dose dependence number density in irradiated EP-450steel (T_{irr}=480°C)

As it is seen from Fig.7 the dose dependence of swelling in EP-450 steel has the traditional form characteristic for all metals and alloys, namely, the presence of incubation stage, transient stage with increasing rate of swelling and steady state stages of swelling The high increase of swelling rate in steel EP-450 is related with the high increase of void size (Fig.8 - 9). The mean size of voids under the dose ≥200 dpa makes more than 50 nm and some voids reach the size more than 100 nm and contribute substantially to swelling.

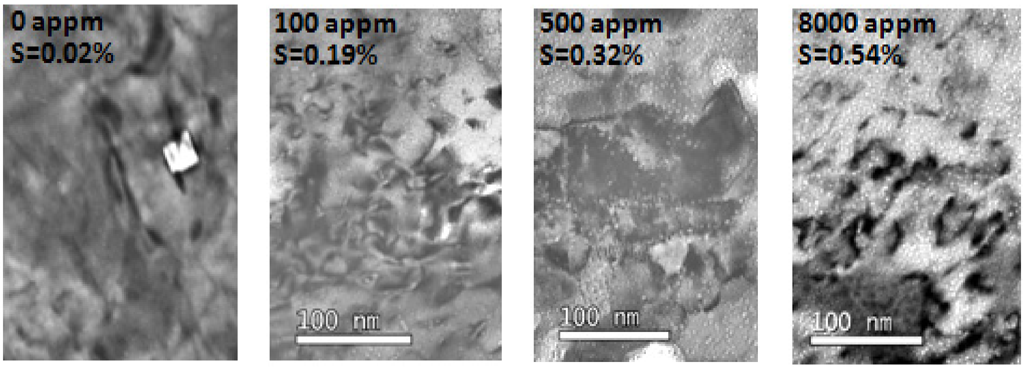
During the steady state stage the rate of swelling of steel EP-450 ~ 0.14%/dpa. Due to the comparatively high rate the swelling of ferritic steel under the dose 300 dpa reaches~20%. Such swelling of ferrite steel is unexpected because ferritic steels were considered as material with low swelling [20].

EFFECTS OF DAMAGES AND DIFFERENT HELIUM LEVELS ON SWELLING OF EP-450 STEEL

Heluim produced by transmutations has long been suspected to play a major role in viod nucleation. Indeed, helium contained in small vacancy clusters can significantly reduce the vacancy reemission rate and thereby increase the nucleation rate.

For investigation of gas effect on swelling in EP-450 steel was chosen doses of 50 dpa (incubation period) and 200 dpa (steady state period) Fig.10.

The results of EP-450 steel swelling obtained under dual irradiation (Cr+He) at 50 dpa and $T_{irr}=480^0C$ and helium levels (0 – 8 000appm) is shown on Fig.10-12. The co-injection of helium leads to reduce of voids size from 17 to 3 nm and to increase of number density from $2 \cdot 10^{14}$ cm^{-3} to $4 \cdot 10^{17} cm^{-3}$ (Fig.11). A characteristic behavior of structure in this case is the sharp increase of swelling from 0.02% up to 0.32 % at helium level 0-500 appm and above 500 appm “slow state” is presented on Fig.12. The increasing of swelling may be due to the increment of gas pressure caused by high helium concentration in voids.



a b c d

Fig. 10. Microstructure of EP-450 steel irradiated at $T_{irr}=480^oC$ and D= 50dpa at different helium levels: 0appm (a); 100 appm (b); 500 appm (c); 8000 (d) appm.

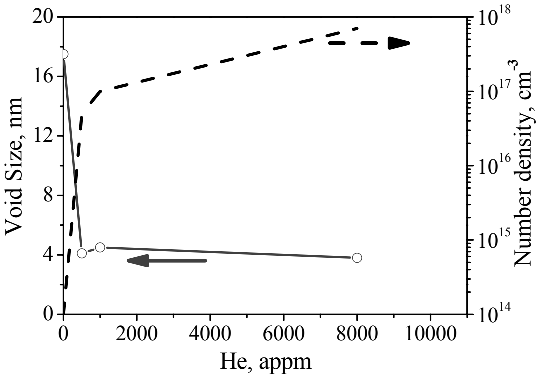


Fig.11. Effect of damage (D=50 dpa) and different helium levels (0-8 000 appm) on void size in EP-450 steel

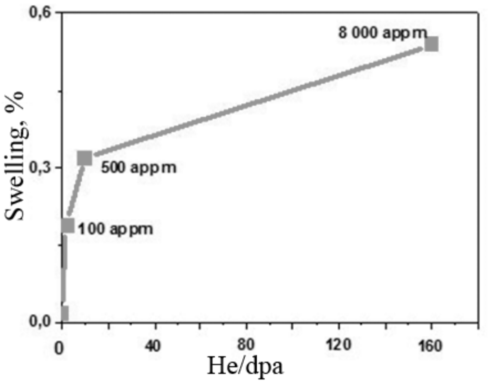


Fig.12. Effect of damage (D=50 dpa) and different helium levels (0-8 000 appm) on swelling in EP-450 steel

Fig. 13-15 show that on the stage of voids formation at 50 dpa (incubation period) 100appm of helium increases the swelling due to increase of number density. At 200 dpa (steady-state) period 100 appm of helium decreases the swelling of EP-450 steel due to reduce of voids size.

The results for EP-450 steel obtained under dual irradiation (Cr+H) at 50 dpa and $T_{irr}=480^0C$ and different hydrogen levels (0 – 10 000appm) are presented on Fig.16-18. The co-injection of hydrogen leads to reduce of voids size from 17 to 6 nm and to increase of number density from $2 \cdot 10^{14}$ cm^{-3} to $2 \cdot 10^{16} cm^{-3}$. A characteristic behavior of structure is the increase of swelling from 0.02% up to 0.93 % at hydrogen range 0-5000 appm and above 5000 appm swelling decrease from 0.93% to 0.37%. At 0-1,000 appm H increase of swelling due to reduce of number density. At 10,000 appm swelling decreases due to voids size (Fig.17-18).

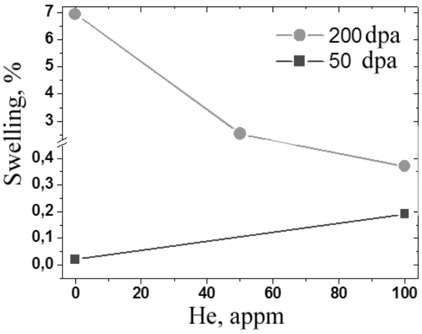


Fig.13. Effect of helium on swelling in EP-450 steel at different dose ranges (50 and 200 dpa).

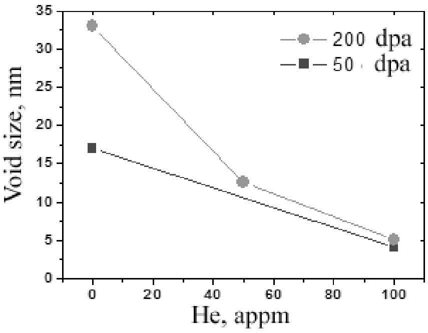


Fig.14. Effect of helium on voids size in EP-450 steel at different dose ranges (50 and 200 dpa).

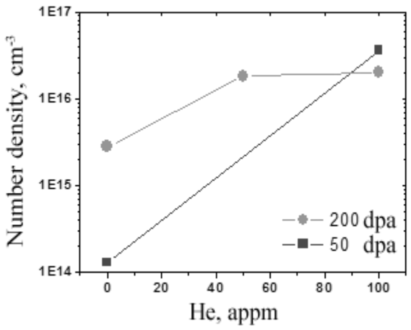
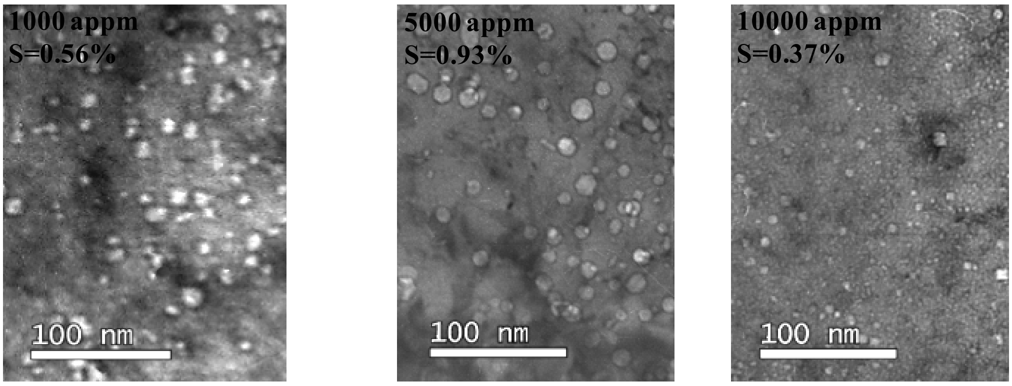


Fig.15. Effect of helium on number density in EP-450 steel at different dose ranges (50 and 200 dpa).



a b c

Fig.16. Microstructure of EP-450 steel irradiated at T_{irr}=480^{o}C and D= 50 dpa at different hydrogen levels 1000 appm (a), 5000 appm (b) and 10000appm (c).

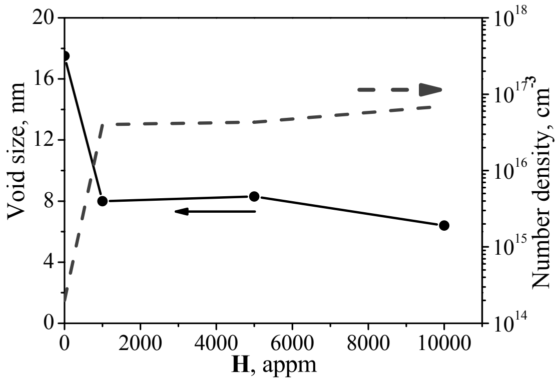


Fig.17. Effect of damage (D=50 dpa) and different hydrogen levels (0-10 000 appm) on void size in EP-450 steel

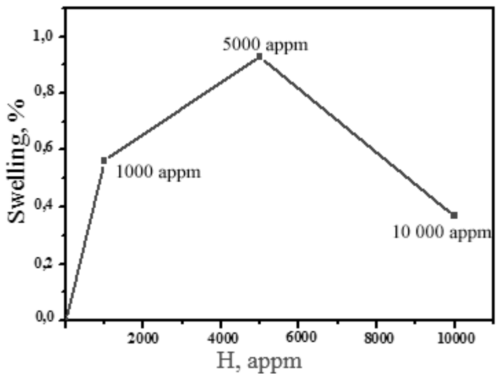


Fig.18. Effect of damage (D=50 dpa) and different hydrogen levels (0-10 000 appm) on swelling in EP-450 steel

CONCLUSION

Ion bombardment -now may be unique chance to explore radiation behaviour of advanced steels to high dpa levels and different levels of gases (helium and hydrogen) which are typical for new reactor’s generations.

Firstly the results on the swelling behaviour of structural materials at very high radiation doses and ultra-high levels gases - hydrogen and helium were obtained. Speciments of ferritic-martensitic steel EP-450 were irradiated in ESUVI under single, dual beam irradiation to dose 50 and 300 dpa at temperature of swelling maximumand at helium and hydrogen levels 0 – 8 000 appm and 0- 10 000 appm, respectively. The results of TEM observation demonstrate:

(a)The maximum of steel EP-450 swelling is situated in the region ~480^{o}C. After extended incubation period ~150 dpa the transition to steady state stage of swelling with the rate 0,14%/dpa is observed.

(b)It is shown that swelling of steel with BCC may reach the value higher 20%.

(a) Ferritic –martensitic steels are sensitive to co-injection of helium and hydrogen.

(b) At incubation period helium increases the swelling. Increase of swelling due to number density increase. Helium exhibits a strong influence on the nucleation of voids, increasing their concentration.

(c) At incubation period hydrogen is also effective as helium in acceleration of the swelling beginning, but has less effect on voids formation that leads to swelling increase on steady-state period due to uniform rise of voids number density.

(d) At steady - state period helium co-injection decreases the swelling of EP-450 steel.

The obtained results refuse the existing opinion that steels of this class have considerably high resistance to swelling; so the additional investigations of this phenomenon are necessary, also with simultaneously injection of gas ions (He, H).

These results are needed to expand the understanding of the fundamental radiation effects and associated rradiation consequences, such as swelling, phase instability, as well as the application of this knowledge for the development of radiation-resistant alloys for future generations of reactors.

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