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Influence of structural-phase state of ferritic-martensitic steels on the helium porosity development

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Abstract. Transmission electron microscopy (TEM) has been used to study the effect of the initial structural-phase state (SPhS) of ferritic-martensitic steels EK-181, EP-450 and EP-450-ODS (with 0.5 wt.% nanoparticles of Y_2O_3) on the of helium porosity formation and gas swelling. Different SPhS of steel EK-181 was produced by water quenching, annealing, normalizing plus tempered, intensive plastic deformation by torsion (HPDT). Irradiation was carried out by He^+-40 keV ions at 923 K up to fluence of $5\cdot10^{20}$ He^+/m^2. It is shown that the water quenching causes the formation of uniformly distributed small bubbles ($\bar{d}$ ~ 2 nm) of the highest density (ρ~ 10^{25} m^{-3}). After normalization followed by tempering as well as after annealing bubbles distribution is highly non-uniform both by volume and in size. Very large faceted bubbles (pre-equilibrium gas-filled voids) are formed in ferrite grains resulting in high level of gas swelling of the irradiated layer with S = 4,9 ± 1,2 and 3.8 ± 0.9 % respectively. Nano- and microcrystalline structure created by HPDT completely degenerate at irradiation temperature and ion irradiation formed bubbles of the same parameters as in the annealed steel. Bubbles formed in EP-450-ODS steel are smaller in size and density, which led to a decrease of helium swelling by 4 times (S = 0.8 ± 0.2 %) as compared to the swelling of the matrix steel EP-450 (S = 3.1 ± 0.7 %).

1.Introduction

One reason for degradation of properties of fusion reactor structural materials and shortening of the useful life of its structural components can be helium, accumulating as a result of neutron bombardment and injection from plasma [1]. Many studies have shown that the chemical composition has a significant effect on radiation damage of structural materials under irradiation in reactor [1–3] as well as on the behavior of helium and the development of gas porosity in them [4–11]. At the same time, structural-phase state (SPhS) of materials can also affect the formation of helium porosity and gas swelling. However, this problem has not been studied.

The aim of this study was to determine the gas porosity formation peculiarities in the surface layer of the reactor ferritic-martensitic steels of different initial SPhS implanted by helium ions

2. Experimental procedure

Chemical composition of the investigated steels shown in Table 1. Various SPhS of steel EK-181 create by different thermo-mechanical treatment (TMT): 1) water quenching from 1373 K / 1 hours

(HT-1); 2) normalizing (1373 K / 40 min, air cooling) + tempering (993 K / 3 hours, air cooling) (HT-2); 3) hot rolling + annealing at 1083 K / 1 hours (HT-3); 4) intensive plastic deformation by torsion (p = 6 GPa, T = 673 K, number of revolutions is 10, rotation speed is 0,5 min^{-1}) (IPDT). Steels EP-450 and EP-450-ODS were after normalization at 1423 K / 1 hours, air cooling + tempering at 993 K / 2 hours, air cooling.

Table 1. Chemical composition of steels (wt. %).

Steel	C	Cr	Mo	V	W	Nb	Ta	Others
EK-181	0.1–0.2	10–12	< 0.01	0.2–1.0	1.5	< 0.01	0.17	0.03Ni; 0.0045B; 0.044N
EP-450	0.10–0.15	12–14	1.2–1.8	0.1–0.3	–	0.2–0.4	–	≤ 0.3Ni; 0.004B
EP-450-ODS	0.10–0.15	12–14	1.2–1.8	0.1–0.3	–	0.2–0.4	–	≤ 0.3Ni; 0.004B; $0.5Y_2O_3$

HT-1 used for achieving of supersaturated by alloying elements and impurities solid solution, although this kind of heat treatment is not applicable to ferritic-martensitic steel. HT-2 is the standard heat treatment for these type of steels, but HT-3 is sometimes considered for use to steel EK-181 also. Consideration of HPDT due to the fact that at one time in the scientific community conceived the idea of the creation of nano- and microcrystalline structure could effectively restrain radiation swelling of materials.

All electropolished steel samples were irradiated under identical conditions by 40 keV He^+ ions at 923 K up to fluence of $5{\cdot}10^{20}$ m^{-2}. Steel EK-181 in the HT-2 and HT-3 conditions was irradiated to the same fluence in the temperature range of 573–903 K.

Samples for TEM were prepared by two techniques: 1) jet electropolishing from unirradiated side in the device Tenupol-5 for the research front microstructure; 2) cutting out of thin foils perpendicular to the irradiated surface by a focused ion beam (FIB technology) for investigation of microstructure along the trajectory of the ion beam. The microstructure of the irradiated samples was studied in a TEM Libra-120 and FEI Tecnai G2 F20 at accelerating voltages of 120 and 200 kV respectively.

3. Results and Discussion

Figure 1 shows typical TEM images of the EK-181 steel irradiated after the HT-1 and HT-2. Parameters of bubbles are shown in Table 2.

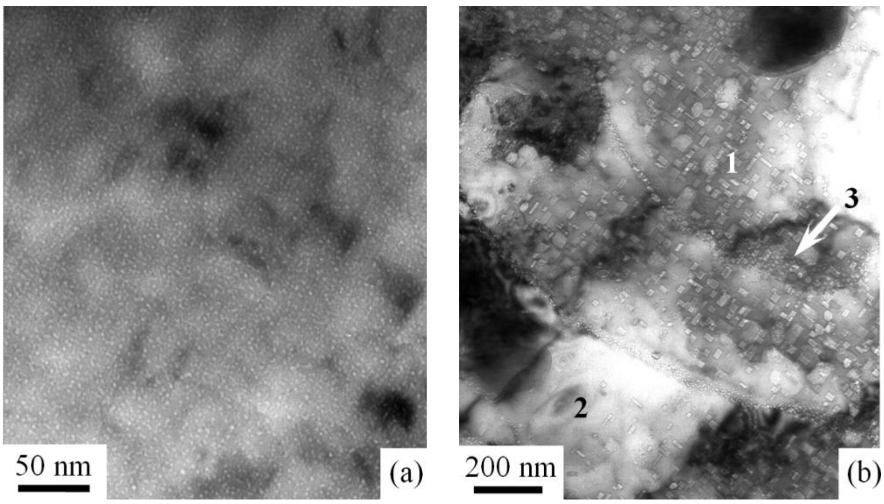

Figure 1. Helium bubbles in steel EK-181 irradiated by He^+ at 923 K: (a) HT-1, (b) HT-2 (Tenupol-5).

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Table 2. Parameters of helium bubbles in irradiated steels: d_{max} and $\bar{d}$ are maximum and average sizes respectively, ρ is bulk density of bubbles, S is gas swelling.

Steel and its TMT	d_{max} (nm)	$\bar{d}$ (nm)	ρ (10^{22} m^{-3})	S (%)
EK-181 (HT-1)	~ 3	~ 2	~ 1000	< 0,03
EK-181 (HT-2)	~ 40	12.1	9 ± 3	4.9 ± 1.2
EK-181 (HT-3)	~ 41	11.4	2.2 ± 0.6	3.8 ± 0.9
EK-181 (IPDT)	~ 30	13.1	1.6 ± 0.4	3,8 ± 0,9
EP-450	~ 22	8.2	2.9 ± 0.7	3.1 ± 0.7
EP-450-ODS	~ 27	6.4	1.0 ± 0.3	0.8 ± 0.2

As can be seen in Figure 1(a) and in Table 2, uniformly distributed in the sample volume smallest (d ~ 2 nm) bubbles of highest density (ρ ~ 10^{25} m^{-3}) formed in water quenched steel. The standard heat treatment (HT-2) resulted in formation of bubbles extremely non-uniformly distributed both by volume and in size. Namely, large faceted bubbles (gas-filled pores) are in the ferrite grains (1), also faceted, but more rare bubbles are in sorbite (2) as well as interlayers of small bubbles (3) are in the grain boundary volumes (see Figure 1(b)).

Figure 2(a) shows the depth distribution of helium bubbles in steel EK-181 irradiated by He^+ ions in a HT-3 state. It can be seen that the largest bubbles develop in the layer of a high concentration of radiation vacancies instead of maximum implanted concentration of helium. There is a high density of small bubbles in some volumes of samples at great depths (more than 300 nm) where no radiation vacancies and should be virtually no helium. Probably, this is due to helium permeation through a ion mixing and/or channeling in certain crystallographic directions of the BCC lattice of steel.

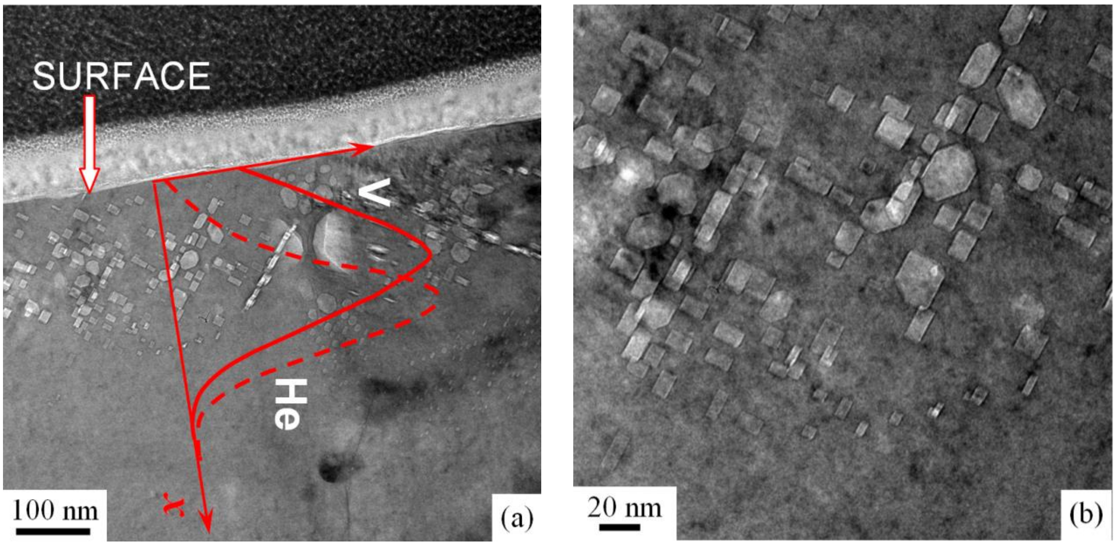

Figure 2. Depth distribution of bubbles in EK-181 steel irradiated by He^+ ions at 923 K in a state of HT-3 (FIB); (b) is an enlarged view of bubbles shown in (a). The depth distribution of radiation vacancies and helium calculated by the program SRIM.

Character of the porosity development in the steel with HT-3 (Figure 2(b)) almost the same as the development of porosity in steel irradiated in a state of HT-2 (see Figure 1 (b)). Bubble parameters and helium swelling of annealed steel (HT-3) also differ slightly from the samples irradiated in the state of HT-2 (see Table 2).

Once there was the idea of creating materials with nano- and microcrystalline structure for suppression a radiation swelling. Figure 3 shows the character of bubbles development in the EK-181 steel irradiated by He^+ ions in the SPhS created using IPDT. As seen in Figure 3, the type of porosity does not differ from the nature of the porosity of the steel irradiated after HT-3 (see Figure 2(b)). As this takes place, the bubble parameters and level of gas swelling are practically identical (see Table 2). This is due to a complete degeneration of nano- and micro-crystalline structure created by IPDT even at an annealing temperature of 873 K (Figure 4 (c)), especially at more higher irradiation temperature of 923 K.

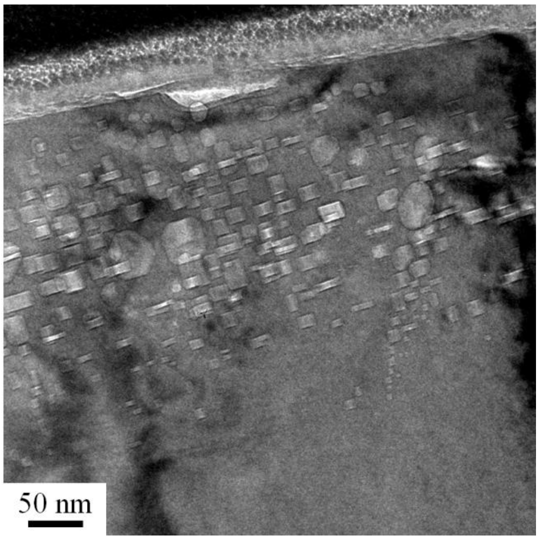

Figure 3. Helium bubbles in EK-181 steel irradiated by He^+ ions at 923 K in IPDT condition (FIB).

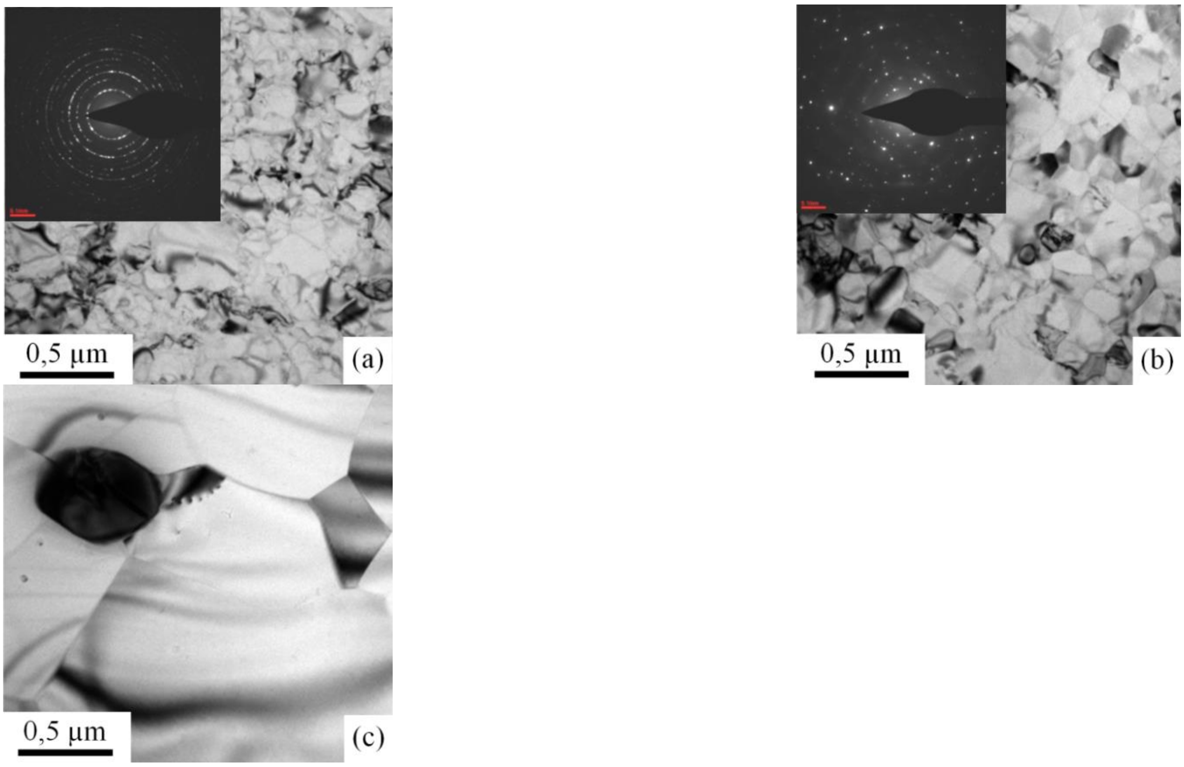

Figure 4. Change of structural-phase states of EK-181 steel under annealings: (a) after IPDT, mean grain size is d_g = 150 nm; (b) annealing at 723 K / 1 hours, mean grain size is d_g = 170 nm; (c) annealing at 873 K / 1 hours, mean grain size is d_g = 1,2 μm (SEM).

Figure 5 shows the temperature dependence of the helium swelling of EK-181 steel irradiated in two initial states. Visible in TEM helium bubbles appear already at a irradiation temperature of T_{irrad} = 573 K in annealed at 1083 K / 1 hours (HT-3) EK-181 steel. Bubble sizes and gas swelling are increasing continuously with increasing of T_{irrad} and swelling is about 0.6% at T_{irrad} = 903 K (see Figure 5). In contrast to the HT-3, the standard heat treatment of steel (HT-2) restrains the bubbles formation up to T_{irrad} = 773 K, but swelling increases significantly and reaches to 1,3 % at high irradiation temperature (T_{irrad} = 903 K) due to the formation in ferrite grains larger bubbles with d_{max} ~ 20 nm.

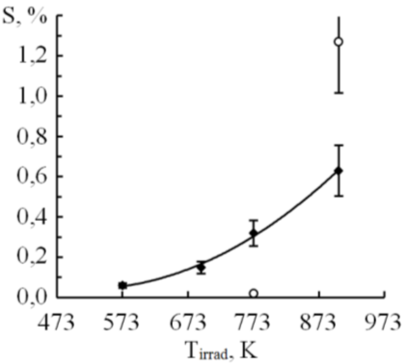

Figure 5. Temperature dependence of the gas swelling in steel EK-181 irradiated by He^+ ions in the two initial states: after the HT-2 (○) and HT-3 (●).

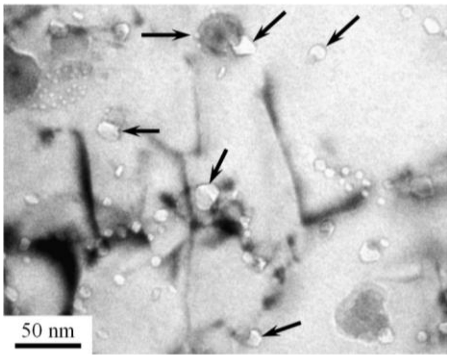

Figure 6. A typical view of helium bubbles in steel EP-450-ODS irradiated by He^+ ions at 923 K (Tenupol-5).

For all the investigated ferritic-martensitic steels irradiated by He^+ ions at a high temperature up to fluence of $5{\cdot}10^{20}$ m^{-2} [12–15], helium bubbles formed about the same character, similar to shown in Figure 1(b). Formed in EP-450-ODS steel bubbles are smaller in size and lower in density, which led to a decrease the swelling 4 times as compared with swelling of the matrix steel EP-450 (see Table 2). Features of gas bubbles development in the steel EP-450-ODS are: 1) bubbles formed at the boundaries of almost all dispersed particles of Y_2O_3, 2) large volumes of the sample does not contain bubbles (Figure 6). Having a high mobility in the metals, the atoms of helium accumulate to the incoherent or semicoherent interfaces of "strengthening particles – matrix", as the particles of Y_2O_3 can not be coherent fully with the matrix due to nonisomorphism of lattices of steel and yttrium oxide (ferrite has a BCC structure with lattice parameter a = 0.287 nm and Y_2O_3 has cubic structure of C-type with a = 1,059 nm). This development of bubbles in the ODS steel restrains the formation of large gas bubbles in the matrix and reduces a gas swelling. At the same time the bubbles on the boundaries of dispersed particles have a high binding energy to them causing the appearance of additional high temperature peaks of gas release in thermal desorption studies [15].

4. Conclusions

Based on the obtained experimental data about the influence of the initial SPhS of reactor ferritic-martensitic steels on the helium porosity formation peculiliarities it can make the following conclusions.

1. It was shown that the state of a supersaturated solid solution of alloying elements and impurities created by quenching of steel EK-181, restrains the formation of helium porosity and gas swelling effectively.
2. It was established that under standart heat treatment (normalization + high temperature tempering) or annealing, high gas swelling observed because of large bubbles formation (gas-filled pores) in the ferrite grains of ferritic-martensitic steel EK-181.
3. It was found that nano- and microcrystalline structure created by IPDT degenerates fully with heating temperature of 873 K, and steel EK-181 swells in a similar manner as in the annealed condition under irradiation by He^+ ions at 923 K.
4. It was shown that in the steel EP-450-ODS bubbles formed smaller in size and in density, which resulted to a decrease in the helium swelling of the 4 times as compared with swelling of the matrix steel EP-450.

Acknowledgements
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