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MICROSTRUCTURE EVOLUTION AND DEUTERIUM RETENTION IN SS316 STEEL IRRADIATED WITH HEAVY IONS, HELIUM AND HYDROGEN

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Using the methods of ion implantation, electron microscopy and nuclear reactions the data on defect microstructure evolution, accumulation levels and temperature ranges of retention of hydrogen isotope - deuterium in austenitic steel SS316 have been obtained. Implantation of 15 keV deuterium ions to a dose of 1×10^{16} cm^{-2}, 30 keV helium ions to a dose of 5×10^{16} cm^{-2}, as well as steel irradiation with 1.4 MeV argon ions to a dose of 1×10^{17} cm^{-2} were carried out at room and elevated temperatures. The formation of dislocation structure, vacancy and gas-filled pores, and the nature of their size distribution were studied. The dependence of amount of trapped deuterium atoms on the defect structure evolution during annealing has been established. A considerable content of hydrogen in the traps associated with helium and argon bubbles was found, that confirms the data obtained for stainless steels irradiated in a light water reactors.
KEYWORDS: implantation, deuterium, helium, argon, defects, microstructure, distribution, accumulation

ЭВОЛЮЦІЯ МІКРОСТРУКТУРИ ТА УТРИМАННЯ ДЕЙТЕРІЯ В СТАЛІ SS316 ПРИ ОПРОМІНЕННІ ВАЖКИМИ ІОНАМИ, ГЕЛІЄМ І ВОДОРОДОМ

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З використанням методик іонної імплантації, електронної мікроскопії та ядерних реакцій отримано дані щодо еволюції дефектної мікроструктури та рівнів накопичення і температурних інтервалів утримання ізотопу водню – дейтерію в аустенітній сталі SS316. Імплантація іонів дейтерію з енергією 15 кеВ до дози 1×10^{16} см$^{-2}$, гелію з енергією 30 кеВ до дози 5×10^{16} см$^{-2}$, а також опромінення сталі SS316 іонами аргону з енергією 1,4 МеВ до дози 1×10^{17} см$^{-2}$ здійснювались при кімнатній і підвищених температурах. Показано розвиток дислокаційних структур, вакансійних і газонаповнених пор та визначено характер їх розподілу за розмірами. Визначена залежність кількості захоплених атомів дейтерію від розвитку дефектної структури при відпалі. Встановлено істотне утримання водню в пастках, пов'язаних з гелієвими і аргоновими бульбашками, що підтверджує дані отримані для неіржавіючих сталей, опромінених в реакторах на легкій воді.
КЛЮЧОВІ СЛОВА: імплантація, дейтерій, гелій, аргон, дефекти, мікроструктура, розподіл, накопичення

ЭВОЛЮЦИЯ МИКРОСТРУКТУРЫ И УДЕРЖАНИЕ ДЕЙТЕРИЯ В СТАЛИ SS316 ПРИ ОБЛУЧЕНИИ ТЯЖЕЛЫМИ ИОНАМИ, ГЕЛИЕМ И ВОДОРОДОМ

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С использованием методик ионной имплантации, электронной микроскопии и ядерных реакций получены данные об эволюции дефектной микроструктуры, уровнях накопления и температурных интервалов удержания изотопа водорода – дейтерия в аустенитной стали SS316. Имплантация ионов дейтерия с энергией 15 кэВ до дозы 1×10^{16} см$^{-2}$, гелия с энергией 30 кэВ до дозы 5×10^{16} см$^{-2}$, а также облучение стали SS316 ионами аргона с энергией 1,4 МэВ до дозы 1×10^{17} см$^{-2}$ осуществлялись при комнатной и повышенных температурах. Показано развитие дислокационных структур, вакансионных и газонаполненных пор, и определен характер их распределения по размерам. Определена зависимость числа захваченных атомов дейтерия от развития дефектной структуры при отжиге. Установлено существенное содержание водорода в ловушках, связанных с гелиевыми и аргоновими пузырьками, что подтверждает данные, полученные для нержавеющих сталей, облученных в реакторах на легкой воде.
КЛЮЧЕВЫЕ СЛОВА: имплантация, дейтерий, гелий, аргон, дефекты, микроструктура, распределение, накопление

The effect of helium and hydrogen on the properties of structural materials in fission and fusion reactors is currently under consideration as a special topic of radiation damage physics and radiation materials science. The atoms of gas impurities are formed in stainless steels, as well as in other metals and alloys, by neutron-induced transmutation. Hydrogen is also introduced into metals by a variety of other mechanisms (corrosion, recoil injection of protons after neutron-water collisions, radiolytic decomposition of water, etc.). In recent years, the research results indicates the increasing negative role of helium and hydrogen at a joint their introduction into material.

Helium, having the greatest inertness and extremely low solubility in metals, when introduced into the material by ion implantation, diffuses rapidly via interstitials, easily binds with vacancies forming helium-vacancy complexes and

bubbles, and retained to a high temperatures. Although essentially all of the helium is retained in the steel, it is commonly assumed that most of the hydrogen due to its high mobility cannot be retained at high concentrations and will therefore diffuse out of the steel. Certain fraction of hydrogen can be stored in structural materials in the presence of a sufficient concentration of trapping sites. A review of published data showed that the hydrogen can be captured in the traps both radiation and non-radiation origin. In [1,2] it is shown that radiation-induced Frank loops and dislocation microstructure contribute to the retention of significant amounts of hydrogen. The formation of cavities may increase the trapping, especially if hydrogen located in the cavities in the molecular state. Furthermore, hydrogen retention appears to be accelerated when large amounts of helium are cogenerated [3].

Thus, the contribution to the processes of hydrogen accumulation and retention in metals and alloys provides a spectrum of radiation-induced defects (point defects and their clusters, dislocation loops of vacancy and interstitial types, precipitates of a new phase, vacancy and gas-filled pores). However, the contribution of each defect type in these processes so far not yet completely determined.

Austenitic stainless steel SS316, in addition to its wide practical application as a structural material of II and III generation reactors, ITER, «spallation» neutron sources and others, is a representative object for studying the hydrogen behavior in austenitic steels of 300 series due to the initial virtually defect-free structure.

The aim of this work was an investigation of evolution of SS316 steel microstructure after irradiation with helium or argon ions that simulate displacement damages resulting in structural materials under the influence of high-energy neutrons, and also the impact of radiation-induced defects on the accumulation and spatial distribution of ion-implanted hydrogen isotope – deuterium.

MATERIAL AND EXPERIMENTAL METHODS

Specimens of SS316 steel with dimensions 27x7x0.1 mm, previously solution annealed at 1340 K in a vacuum of 10^{-4} Pa for one hour were used for investigations. Steel composition is shown in Table.

Table.

SS316 steel composition, wt.%

C	Si	Mn	P	S	Cr	Ni	Mo	Ti	Fe
0.056	0.68	1.6	0.034	0.014	16.68	12.03	2.40	<0.01	бал.

The initial microstructure of SS316 steel is shown in Fig. 1. Austenitic grains with a size of about 30 microns were observed in the steel structure. Total dislocation density was ~10^8 cm^{-2}.

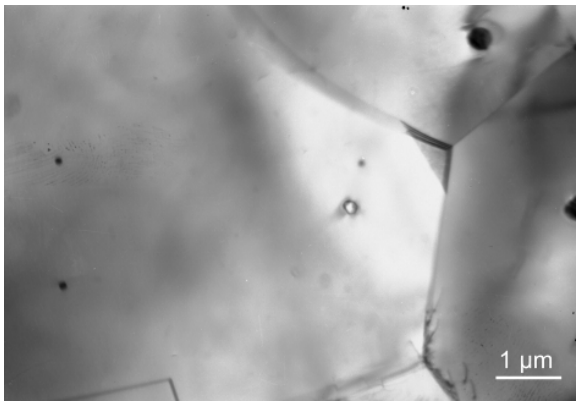


Fig. 1. The initial microstructure of SS316 steel after heat treatment at 1340 K/1 hour.

Implantation of steel with 15 keV deuterium, 30 keV helium and 1.4 MeV argon ions was carried out using accelerating system ESU-2 at room and elevated (780-1050 K) temperatures. The irradiation temperature was controlled by chromel-alumel thermocouple. The annealing of samples in the temperature range of 300-700 K were performed at a rate of temperature increasing and decreasing of 7 Ks^{-1}. After irradiation samples have been in situ examined by means of nuclear reaction analysis or transferred to the EM-125 electron microscope for the microstructure changes observation.

Thinning of specimens to a thickness suitable for TEM studies was conducted by standard jet electro-polishing from un-irradiated surface. Herewith a protective layer of varnish was applied on the irradiated surface of the sample to protect it from etching. Polishing was stopped as soon as the hole appeared in the sample. The sample was removed from the Teflon holder, washed in ethanol and then in acetone to dissolve the protective varnish film. Final washing was carried out in dehydrated ethanol. Analysis of TEM micrographs was performed using image processing software. In some cases samples were thinned from both sides. To remove a specified depth layer of material from irradiated side of the sample the electro-pulse technique was used.

The depth distribution of implanted particles was measured by a nuclear reaction $D(^3He,p)^4He$ with using analyzing beams of ^3He (E = 0.3-1.4 MeV). Measurements were performed in backscattering geometry. ^3He ion beam was incident to the normal of sample surface. Products of nuclear reactions were registered at an angle of 160° with respect to the analyzing beam. The diameter of the beam at the irradiation was 3 mm, during analysis – 2 mm. Depth resolution in the backscattering geometry was 150 nm. Details of method are described elsewhere [4].

RESULTS AND DISCUSSION

Deuterium only. Fig. 2 shows the measured depth distribution profiles of deuterium implanted to a dose of 1×10^{16} cm^{-2} in SS316 steel at room temperature immediately after irradiation and after subsequent annealing at 373 K. Calculated ranges and damage profiles of deuterium with energy of 15 keV in SS316 steel are presented in the insert. All calculations were performed using SRIM 2006 code [5] with the Kinchin-Pease damage energy model and displacement energy of 40 eV for Fe and Cr. Calculated deuterium concentration in this case is about 0.9 at.%, and calculated damage – 0.02 displacement per atom (dpa).

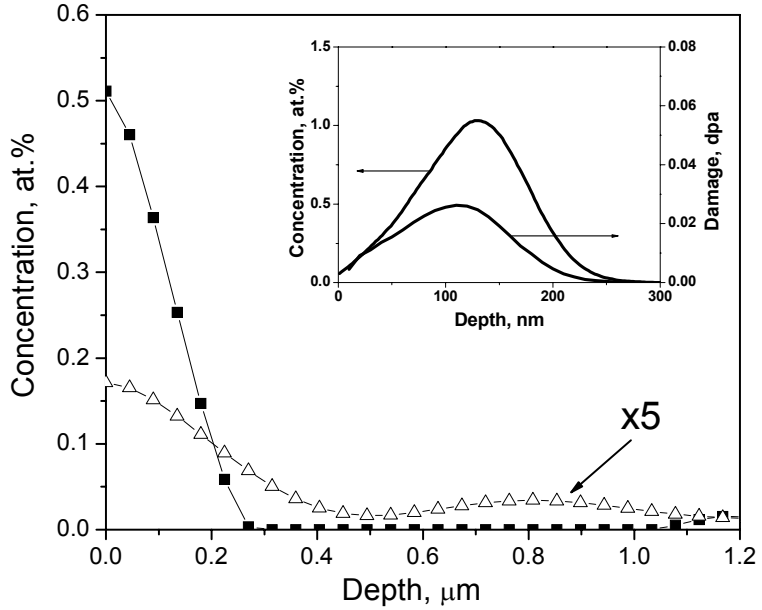


Fig. 2. Depth distribution profiles of deuterium implanted into SS316 steel at room temperature (■) and after annealing up to 373 K (Δ). Insert: Calculated (SRIM 2006) ranges and damage profiles of 15 keV deuterium in SS316 steel.

The microstructure of SS316 steel virtually did not change after irradiation compared to the initial structure. Apparently, non-equilibrium concentration of deuterium-induced defects is insufficient to develop a visible defect microstructure at fluence less than 0.1 dpa. The dose of appearance of smallest visible radiation defects depends on many factors (alloy composition, type and energy of ions, dpa rate, etc.) and estimates as 0.005-0.3 dpa at room temperature irradiation [6,7].

Depth distribution profile of deuterium after irradiation at room temperature has a maximum in the near-surface region (Fig. 2). The profile width corresponds to the calculated value. The amount of deuterium retained in the samples at room temperature is ~80% with respect to the irradiation dose. These data indicate the trapping of the implanted deuterium by radiation-induced traps, because in the absence of such defect-traps hydrogen must leave the implanted area during irradiation due to its high diffusivity in austenitic steels at room temperature $D\sim1\cdot10^{-12}$ $cm^{-2}s^{-1}$ [8].

Annealing at 373 K causes a decreasing of deuterium retention in the near-surface area to 15% with respect to irradiation dose. In addition, the small second peak appears in deuterium distribution at a depths corresponding to the ion range of ^3He analyzing beam with energy of 0.3-1.4 MeV [9]. The observed features of deuterium interaction with SS316 austenitic stainless steel indicate a weak deuterium capturing in traps created at irradiation dose less than 0.1 dpa.

It can be assumed that the deuterium trapping occurs on radiation-induced point defects. In considering of interaction of FCC-metals with hydrogen the lowest binding energy, accordingly to the theoretical calculations, has a complex "H – self interstitial atom" (SIA). The binding energies of D-SIA according to experimental data are 0.16 eV for Pd, 0.21 eV for Cu and 0.24 eV for Ni. However, in most publications the interstitials do not considered as probable traps for hydrogen for the reason that the diffusivity of SIAs, especially in FCC- metals, is very high, and they disappear on sinks at temperatures much lower than required for hydrogen de-trapping [10,11]. Analysis of the literature shows that the most probable hydrogen traps in metals are vacancy type defects [12]. The binding energies of D atoms with vacancies are 0.48 eV in α-Fe, 0.43 eV in Ni, 0.41 eV in Cu. The temperatures of corresponding annealing stages of deuterium are very close to that obtained in the present study.

Helium pre-irradiation + deuterium. Irradiation of SS316 steel with 30 keV helium ions to a dose of 5×10^{16} cm^{-2} at room temperature leads to the formation of gas bubbles with an average diameter of 1 nm (Fig. 3). Fig. 4 shows the distribution profile of deuterium after sequent implantation at room temperature into samples pre-irradiated with helium, and after subsequent annealing. As seen from the insert, calculated damage dose and helium concentration are ~0.9 dpa and 5 at.%, respectively.

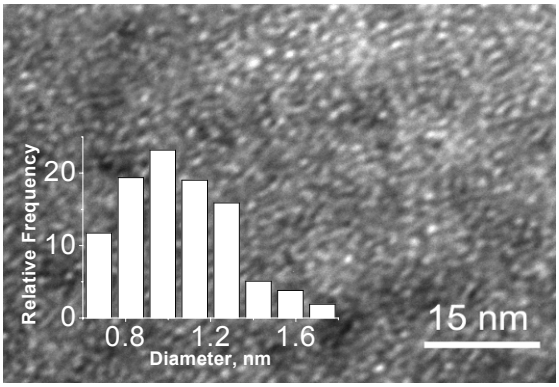


Fig. 3. Microstructure of SS316 steel after irradiation with 30 keV helium ions to a dose of 5×10^{16} cm^{-2} at room temperature.

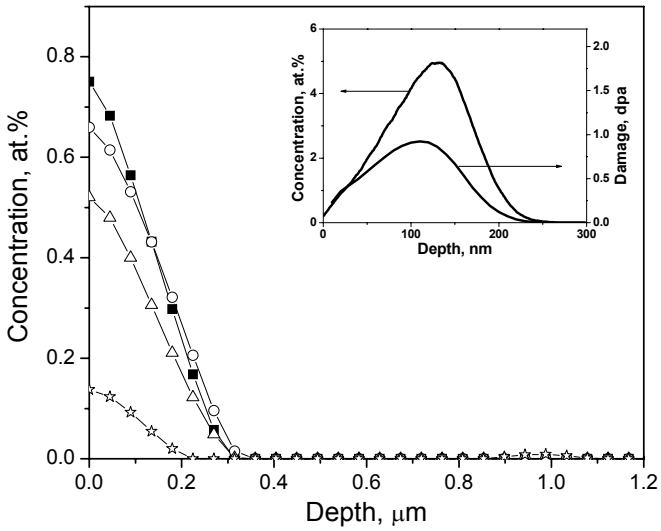
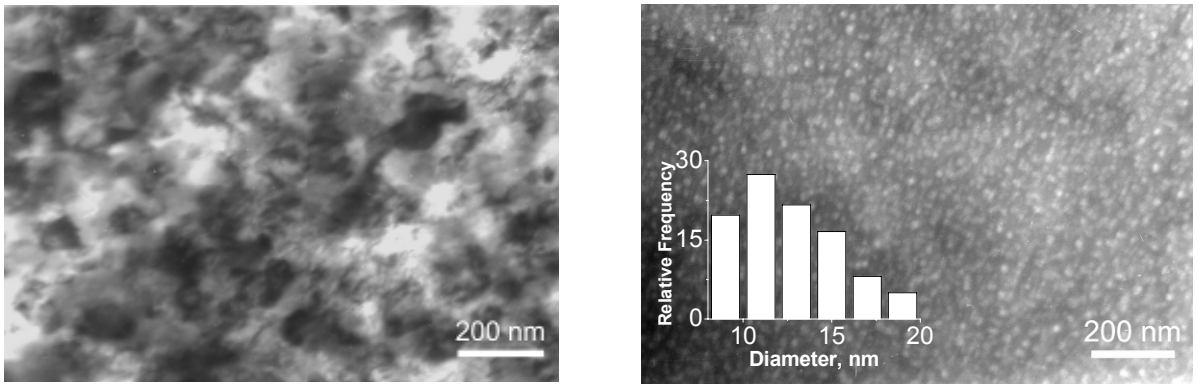


Fig. 4. Distribution profiles of deuterium implanted at room temperature (■) to a dose of 1×10^{16} cm^{-2} in steel SS316, pre-irradiated with helium (30 keV, 5×10^{16} cm^{-2}) and after subsequent annealing 373 (○), 500 (Δ) and 600 K (☆). Calculated (SRIM 2006) ranges and damage profiles of 30 keV helium ions in SS316 steel are shown in the insert.

In this case, the distribution profile of deuterium remains unchanged (within experimental error) up to a temperature of 373 K. At annealing temperatures of 500 and 600 K the deuterium concentration reduced to 70 and 15% of the radiation dose, respectively. The observed increasing of deuterium retention can be attributed to the influence of preliminary implanted helium. On the basis of electron microscopic studies (Fig. 3), it is believed that helium bubbles are effective deuterium traps.

Results of the study of helium porosity are well represented in the literature [13]. The formation of microscopic gas bubbles was observed after irradiation of metals and alloys with inert gas ions even at room temperature [14]. At helium implanted concentration of $0.6 < C_{He} < 3$ at.% the dominant microstructure in copper was a high density of small bubbles, and their growth and coalescence was observed at 573 K [15]. The influence of helium porosity on enhancement of hydrogen retention is confirmed by the results of numerous studies. Nevertheless, it still remains unclear the trapping mechanism. Some authors believe that trapping occurs as a result of deuterium atoms chemisorption on the helium bubbles walls, while others believe that traps are the elastic stress fields around the bubbles.

Argon pre-irradiation at room temperature + deuterium. The microstructure of SS316 steel irradiated with high energy argon ions to a dose of 1×10^{17} cm^{-2} is shown in Fig. 5. Fig. 6 shows the evolution of depth profiles of deuterium post-implanted at room temperature to a dose of 1×10^{16} cm^{-2} in steel previously irradiated with argon. It should be noted that the sample temperature during irradiation with argon ions is exceeded the room temperature by 20-40 K.



a b

Fig. 5. The microstructure of SS316 steel at a depth of ~100 (a) and ~450 nm (b) after irradiation of samples with 1.4 MeV argon ions (dose of 1×10^{17} cm^{-2}) at room temperature.

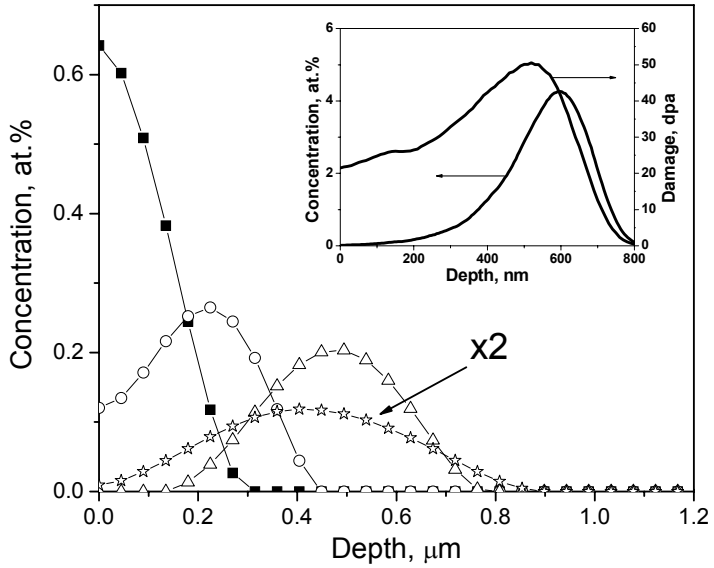


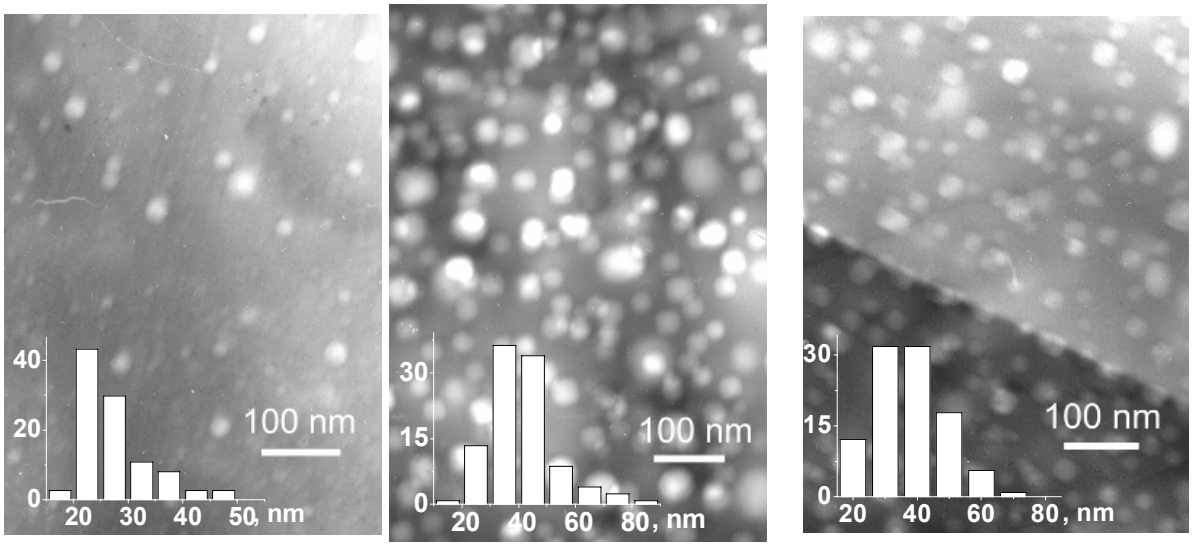
Fig. 6. Distribution profiles of deuterium implanted to a dose of 1×10^{16} cm^{-2} at T_{room} (■) in steel SS316 pre-irradiated with argon ions (1.4 MeV, 1×10^{17} cm^{-2}), and after subsequent annealing at 373 (○), 500 (Δ) and 600 K (☆). In the insert: calculated (SRIM 2006) ranges and damage profiles of 1.4 MeV argon in SS316 steel.

Irradiation of steel with high-energy gas ions having a relatively large average projected range causes the formation of depth-layers with different combinations of damage and concentrations of implanted particles. In present case, for simplicity, it seems appropriate to consider two virtual zones. In the first zone (0-200 nm), accordingly to SRIM 2006 calculations (see insert in Fig.6), there are mainly displacement damages at a level of 25 dpa and negligible amount of implanted argon atoms. The second zone (400-600 nm) is characterized by damage level up to 50 dpa and the argon concentration reaching 4 at.%. TEM examinations of the first zone have shown the formation of dislocation tangles (Fig. 5a). Despite the extremely high dislocation density ($\rho = 5\times10^{10}$ cm^{-2}), separate dislocation loops have been observed, indicating the prolongation of processes of loops nucleation and growth. Meanwhile, the bubble structure with a density of 8×10^{15} cm^{-3} and an average bubble diameter of 12 nm has been formed in a layer of 400-600 nm (Fig. 5b).

During irradiation at room temperature deuterium stops in the sample at the depths corresponding to the ranges of 15 keV/D^+ ions and within the region of defect structure previously created by argon irradiation (Fig.6, curve with a marker ■). At subsequent annealing one portion of deuterium has moved deeper into the specimen towards the zone of bubble structure (Fig.6, curve with a marker Δ), while another part has desorbed from surface. With increasing of annealing temperature the deuterium retention gradually decreases, and after annealing at 600 K in the sample remains approximately 30% of deuterium from the implant dose.

Argon pre-irradiation at elevated temperatures + deuterium. In experiments with cold-deformed and porous (in particular, electro-deposited) metals it was established surely that hydrogen is efficiently captured and strongly retained in cavities [16]. Microcavities can be formed in irradiated metals by combining of non-equilibrium radiation-induced vacancies both during irradiation and subsequent annealing.

In present study, a porous structure was formed by irradiation of SS316 steel with 1.4 MeV argon ions at elevated temperatures (Fig. 7a-c). It was found that the voids grow in the near surface region (0-200 nm).



a b c

Fig. 7. Void microstructure in SS316 irradiated with argon ions to a dose of 1×10^{17} cm^{-2} at temperatures of 950 (a), 970 (b) and 1000 K (c).

Temperature dependence of the swelling, presented in Fig. 8, has a typical bell-shaped form. An increasing of swelling occurs in the temperature range of 910-940 K. With rising irradiation temperature up to 1000 K the swelling decreases. Swelling curve maximum is located at 940 K and shifted towards higher temperatures by ~20 K in comparison with the irradiation of austenitic steels with metallic ions [17]. Apparently, this may be due to the presence of argon atoms in voids. It is known that the atoms of inert gases are stabilizers of very small vacancy clusters, which have a very short lifetime without such stabilization. On the other hand, at a low temperature irradiation the void formation can be substantially suppressed by a high concentration of ultrafine bubbles [18]. In addition, parameters of steels swelling depend on the mode of an inert gas introducing. At simultaneous irradiation with heavy ions and inert gas implantation a shift of swelling curve to higher temperatures is occurred.

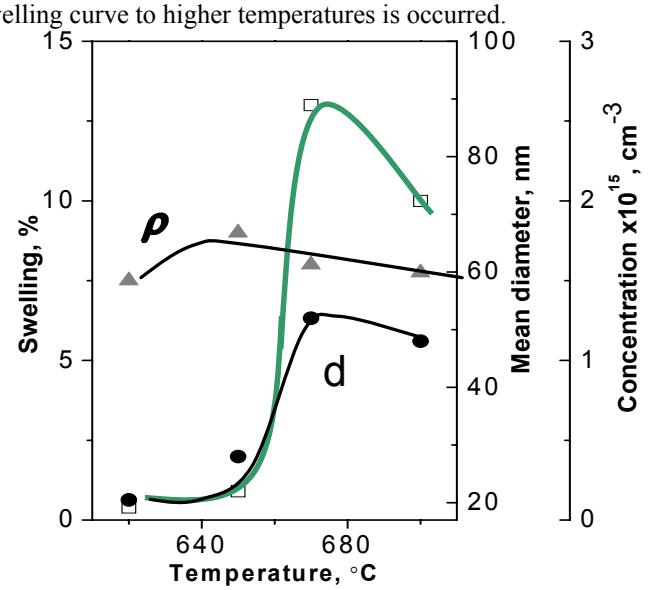


Fig. 8.Temperature dependences of swelling, void concentration (ρ) and mean void diameter (d).

Typical depth profiles of deuterium implanted to a dose of 1×10^{16} cm^{-2} at room temperature and after annealing of steel with previously created void structure are shown in Fig. 9. The behavior of deuterium in this case are qualitatively similar to its behavior after implantation into the structure created by argon pre-irradiation at room temperature: with temperature increasing the deuterium is redistributed from the zone of introduction and location of void structure towards the zone of larger damages and argon concentration, where TEM observations showed the formation of bubble structure.

Fig. 10 shows the temperature dependences of retained deuterium concentration for all above mentioned irradiation conditions. Data was obtained by processing of depth profiles. Fig. 10a represents the total amount of deuterium throughout the probing depth, and Fig. 10b – deuterium content (normalized per unit) in two layers 0-200 nm and 400-600 nm.

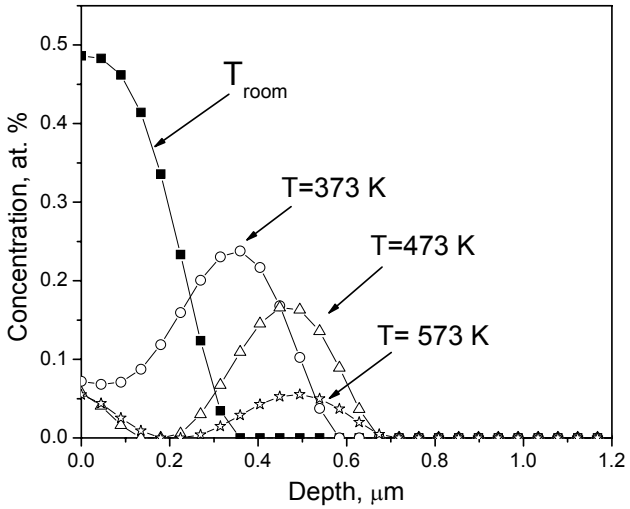
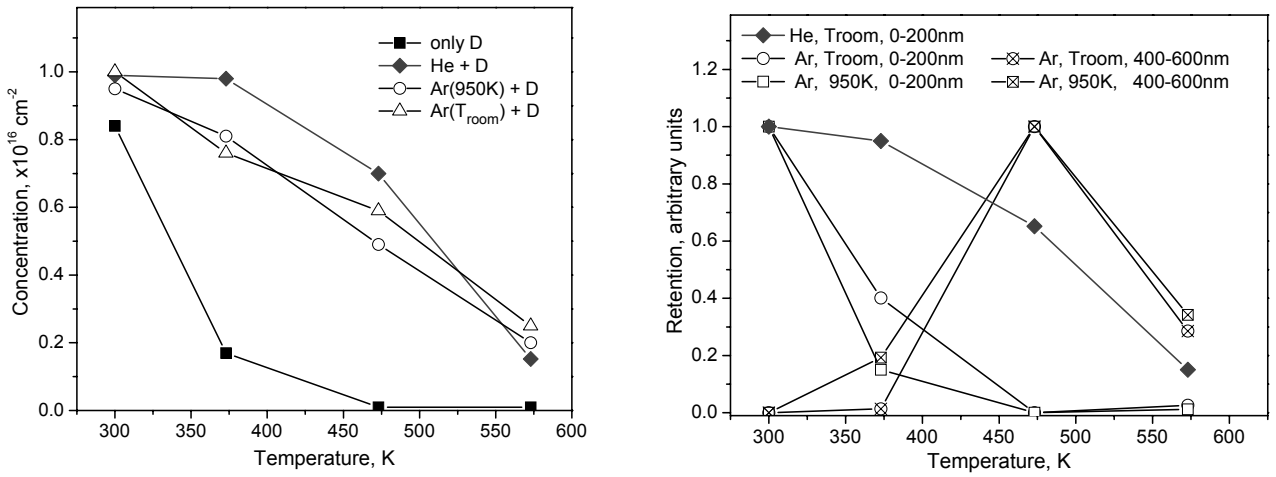


Fig. 9. Depth profiles of deuterium implanted to a dose of 1×10^{16} cm^{-2} at room temperature and after annealing in the samples with a previously created void structure by 1.4 MeV Ar irradiation to a dose of 1×10^{17} cm^{-2} at 950 K.



a b

Fig. 10. The amount of retained deuterium implanted to a dose of 1×10^{16} cm^{-2} in SS316 steel having various irradiation pre-treatments.

Changes of the total deuterium amount at annealing (Fig. 10a) indicates almost identical deuterium retention in the defect structures induced by helium or argon ions pre-irradiation. However, the temperature ranges of actual retention of each trap system are differ (Fig. 10b). In the case of argon pre-irradiation the reducing of concentration of deuterium that has been primarily trapped in the void-type or dislocation-type defects occurs at 373 K (Fig. 6, Fig. 9). At the same time, the de-trapped deuterium migrates into the bulk where the re-trapping of deuterium by argon gas bubbles takes place. Besides, at this stage a part of deuterium is desorbed out of specimen. Note, that at the same temperature (373 K) the deuterium retention in helium pre-irradiated steel remains unchanged.

All above implies that the inert (helium or argon) gas-filled bubbles appear to be the strongest traps of implanted deuterium in stainless steel. The de-trapping of deuterium from these bubble-type defects observed during annealing to ~600 K. What is the reason for deuterium de-trapping from these traps?

Analysis of the published data indicates that in the temperature range of 500-700 K in structural steels a significant increase of vacancies mobility occurs. In [19] the positron annihilation method was used to study the formation and annealing of vacancy clusters in austenitic steels and Fe-36% Ni alloy. Defects were induced by electron (5 MeV) irradiation at room temperature and subsequent stepwise annealing. It has been shown that the small vacancy clusters are formed in these materials. Vacancy clusters were thermally stable up to 450 K. For solution annealed samples, it was found that the stage associated with dissociation of vacancy type clusters is observed in the temperature range of 500-650 K.

It can be assumed that similar as it occurs at the high temperature implantation [14], the processes of growth and coalescence of inert gas bubbles are also take place during annealing of implanted samples. Transformation of bubbles during annealing assists the deuterium de-trapping from them.

CONCLUSIONS

Using the methods of ion implantation, transmission electron microscopy and nuclear reactions the examination of microstructure evolution and the influence of radiation-induced defects on the accumulation and the spatial distribution of ion-implanted deuterium in the SS316 austenitic stainless steel have been performed.

Data obtained in this paper show that irradiation of 316 steel in the dose range from 0.02 to 50 dpa at temperatures 300-1050 K depending on the irradiation mode is results in the formation of dislocation structure (loops, dislocation tangles), voids and/or gas-filled bubbles. The characteristics of trapping and the temperature range of deuterium retention in traps formed by prior implantation of helium or argon depends on the type of developed defects.

Ion implanted deuterium is weakly trapped by vacancy-type defects produced in 15 keV D^+ displacement cascades. Deuterium retention is reduced five times after annealing up to 373 K.

Radiation-induced dislocation and porous structures promotes the deuterium retention in the range 300-450 K.

Inert gas bubbles are the strongest traps of implanted deuterium in stainless steel. The bubble structure can retain the deuterium up to 600 K. Trapping of significant amounts of hydrogen can influence the onset and amount of void swelling, development of hardening and possibly on corrosion and cracking.

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