Surface modification and deuterium retention in reduced-activation steels under low-energy deuterium plasma exposure

Part II: Steels pre-damaged with 20 MeV W ions and high heat flux

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Abstract

The reduced-activation ferritic/martensitic (RAFM) steels including Eurofer (9Cr) and oxide dispersion strengthened (ODS) steels by the addition of Y_2O_3 particles investigated in **Part I** were pre-damaged either with 20 MeV W ions at room temperature at IPP (Garching) or with high heat flux at FZJ (Juelich) and subsequently exposed to low energy (~20-200 eV per D) deuterium (D) plasma up to a fluence of 2.9×10^{25} D/m^2 in the temperature range from 290 K to 700 K. The pre-irradiation with 20 MeV W ions at room temperature up to 1 displacement per atom (dpa) has no noticeable influence on the steel surface morphology before and after the D plasma exposure. The pre-irradiation with W ions leads to the same concentration of deuterium in all kinds of investigated steels, regardless of the presence of nanoparticles and Cr content. It was found that (i) both kinds of irradiation with W ions and high heat flux increase the D retention in steels compared to undamaged steels and (ii) the D retention in both pre-damaged

and undamaged steels decreases with a formation of surface roughness under the irradiation of steels with deuterium ions with incident energy which exceeds the threshold of sputtering. The increase in the D retention in RAFM steels pre-damaged either with W ions (damage up to ~3 μm) or high heat flux (damage up to ~10 μm) diminishes with increasing the temperature. It is important to mention that the near surface modifications caused by either implantation of high energy ions or a high heat flux load, significantly affect the total D retention at low temperatures or low fluences but have a negligible impact on the total D retention at elevated temperatures and high fluences because, in these cases, the D retention is mainly determined by bulk diffusion.

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1. Introduction

The performance of fusion and advanced fission devices critically depends on the choice of plasma-facing materials and components. Resistance to high heat and particle fluxes, thermomechanical properties, fuel retention as well as the response to neutron (n) damage of the selected materials are critical parameters which need to be understood. Although there still remain several important issues on plasma facing components for ITER, there will be more challenging issues towards a fusion demonstration device, DEMO, because of steady-state operation and high neutron dose. The lifetime of components facing to the plasma is determined by their erosion rate. The erosion rate of high-Z elements, such as tungsten and molybdenum, is several orders of magnitude lower than that of low-Z elements, such as beryllium or carbon, at the low ion energies expected near the plasma-facing materials in confined plasma devices [**1**]. For this reason many DEMO design studies have incorporated the use of thin tungsten covered surfaces for plasma-facing components [**2,3**]. Tungsten remains the leading candidate material for a reactor in the regions of highest heat load, but its tritium retention and mechanical properties after radiation damage can be a concern [**4,5**]. The erosion rate of beryllium is expected to be acceptably small for ITER, but DEMO or a reactor will have a much higher duty cycle and beryllium is not considered viable. In remote regions of the first wall, one possible alternative to use tungsten coating on the surface of a component is to use a bare steel plasma-facing surface. The use of bare reduced-activation ferritic/martensitic (RAFM) steel as first-wall material in a fusion reactor, at least in selected areas of the main chamber, has been proposed in the past [**6,7,8,9**].

In DEMO environment, plasma-facing and structural materials should be capable to withstand the high neutron flux for many years. In general, main differences of DEMO from ITER include the requirement to breed, to extract, to process and to recycle the tritium needed for plasma operation, the higher neutron fluence, which limit remote maintenance options, and the requirement to use low-activation steel for in-

vessel components that must operate at high temperature for efficient energy conversion. In the previous article [**10**], the D retention and surface modification in undamaged RAFM's steels after the exposure to low-energy D plasma were investigated. Despite of a lot of data of modification of steel properties after neutron (n) irradiation, according to our knowledge not much data exist about an influence of n irradiation on deuterium trapping and migration in Eurofer and oxide dispersion strengthened (ODS) steels. As no intense source of 14 MeV neutrons is presently available, it is necessary to simulate such irradiation either in a fast reactor or with ion irradiations. In spite of a significant difference between ion- and n- irradiations, namely, (i) the different displacement rates, (ii) inhomogeneous damage profile with proximity to the surface for the ions versus the flat damage profile for neutrons, and (iii) the difference in the primary knock-on atom, PKA, spectrum, it was shown in [**11**] that heavy ions can be a reasonably good surrogate to simulate n irradiation in W at low dpa (below 10 dpa). In the present article, 20 MeV W ions were used for the simulation of radiation-induced defects produced by n irradiation in steels [**4**]. The choice of W ions to be implanted in steels for generation of radiation-induced defects have been dictated by technical possibility of the present accelerator set-up and the presence of W concentration in initial undamaged steels (1-2% of weight**,** see Ref. [10]). The irradiation of a steel with W ions up to a maximum dose of 6.4×10^{18} W/m^2 (4 dpa) used in the present work, does not lead to a noticeable increase of the originally existing concentration of W in the undamaged steel.

Moreover, the high heat flux applied to the plasma-facing material in a fusion reactor can create the damage. Such damage was simulated using electron (e) beam irradiation in JUDITH-1 [**12**].

After pre-damaging either with 20 MeV W ions or with e-beam, specimens were exposed to low energy (~20 eV-200 eV per D) deuterium plasma up to a fluence of ~2.9×10^{25} D/m^2. The depth profile of deuterium in steels was measured up to 6 μm in depth by Nuclear Reaction

Analysis (NRA) and the total retained amount of D in those materials was determined by Thermal Desorption Spectroscopy (TDS) as described in **[10].**

2. Experimental

We used ODS steels of Fe–(9–16)Cr–2W-0.15Si-0.5Ti–0.35Y_2O_3 alloys manufactured in China and Eurofer steel of Fe–9Cr–1W-0.2V–0.07Ta–0.03N–0.1C produced in the European Union. The composition of steels is given in **[10].** All Eurofer and ODS samples were mirror-like polishing. The size of samples after polishing was 10x10x(0.5-1) mm^3 in the case of ODS and 12x15x(0.5-1) mm^3 in the case of Eurofer. Exceptional size of 10x10x3 mm^3 of samples was used for experiments with high heat loads.

To simulate damage produced by transient events like ELMs, Eurofer specimens were exposed to high thermal heat loads in the e-beam facility JUDITH-1 [**12**]. Four loaded spots of (6x6) mm^2 each were applied on each sample of size of (10x10x3) mm^3 as shown in **Fig. 1**. The base temperature of the specimens was room temperature. The loading conditions for each individual loaded spot were as follows: pulse duration of Δt=1 ms, absorbed power density of 0.71 GW/m^2, heat flux factor of 22.5 $MW/m^2s^{1/2}$, and 10 pulses. Such high heat flux is higher than the melting threshold. A melting layer with a thickness of 10-20 μm on the surface was observed at these conditions (**Fig. 2**). Although, the any scenario producing melt layers would be forbidden in a future fusion device, it can happen and this case should be taken into consideration to account for the worst-case scenario.

The binary collision Monte Carlo code SRIM **[13**] was used to calculate the damage distribution produced in a steel with 20 MeV W ions in Kincheen-Pease mode as recommended in **[14].** These calculations used an average of 1000 ions in pure iron and the recommended displacement energy of 40 eV [**15**]. Chromium has the same displacement energy as iron and has minimal influence on the density [**15**]; therefore any differences in these damage calculations

due to chromium content are expected to be minimal. A one-step irradiation using single ion energy of 20 MeV causes a non-monotonously graded damage profile up to ~3 μm with a maximum damage peak, known as a Bragg peak (BP), at ~1.9 μm from the irradiated sample surface. **Fig. 3** shows damage profile in Fe irradiated with 20 MeV W^{6+}.

After pre-irradiation with W ions at IPP (Garching) or pre-irradiation with high heat loads in the e-beam facility JUDITH-1 at FZJ (Juelich), samples were exposed to the low energy D plasma generated by an electron-cyclotron resonance (ECR) plasma source in the experiment PLAQ as described in [**10**]. A d.c. bias of - 60 V and -600 V were applied to the substrate holder to accelerate the ions in the sheath leading to an energy of about 60 eV per ion and 600 eV per ion, respectively. In general, there is a plasma potential of several volts but we do not take it into account because its insignificant contribution. Because the main component of the flux is D_3^+ (97%), the sample bias of -60 eV produces ions with a mean energy of 20 eV per deuteron and the sample bias of -600 V produces ions with a mean energy of 200 eV per deuteron. The plasma exposure was performed with deuteron ion flux of about 10^{20} D m^{-2} s^{-1} at normal incidence. The post-mortem analysis was carried out including a) Scanning Electron Microscopy (SEM) equipped with Energy-Dispersive X-Ray Spectroscopy (EDX) for chemical surface analysis, b) the depth profile of D in steels up to 6 μm depth by NRA and c) the total retained amount of D in those materials was determined by TDS with a linear temperature ramp of 2 K/s. The samples were stored in vacuum between the measurements.

3. Results and discussion

3.1. Surface modification

The surface morphology of Eurofer after exposure to the D plasma with incident ion energy of 200 eV per D at 290 K up to a fluence of $6x10^{24}$ D/m^2 is shown in **Fig. 4** for two cases a) Eurofer pre-damaged with 20 MeV W ions up to 0.56 dpa and b) undamaged Eurofer.

No visible difference in the nano-structured surface modification for undamaged and pre-damaged samples after the plasma exposure was observed. For both cases, the cones with uneven surface were found to be enriched in W. The enrichment in W is due to preferential sputtering (PS) of light elements from steels and radiation-induced segregation (RIS) as reported in [**10**]. We did not observe any noticeable effect of W ions pre-implantation on the surface modification after the plasma exposure because the concentration of W introduced in steels by W ion irradiation up to a dose of $1.6x10^{18}$ W/m^2 (corresponding to 0.56 dpa) is lower than initially existing concentration of W in undamaged steels (1-2% of weight) **[10**]. The same surface modifications were found on the different RAFM's steels. This is consistent with data reported in [10] where an increase of the W concentration in ODS (2% by weight) compared with Eurofer (1% by weight) does not affect the surface roughness formation after irradiation with 200 eV D ions. Moreover, we did not observe any surface modification of steels after pre-irradiation with W ions. Thus, the pre-irradiation with 20 MeV W ions has not any significant influence on the surface morphology of neither Eurofer nor ODS steels before and after the D plasma exposure.

The effect of annealing of a steel pre-damaged with 20 MeV W ions up to 0.56 dpa by TDS with a linear ramp of 2 K/s up to 1200 K after exposure to the D plasma with incident ion energy of 200 eV per D at 290 K on the surface morphology is shown in **Fig. 5**. As in the case of undamaged steels reported in [**10]**, the surface roughness vanishes by annealing up to 1200 K. It is reasonable to suggest that iron and other light elements diffuse towards to the surface and recovery the sputtered layer.

3.2. Deuterium retention in steels pre-irradiated with 20 MeV W ions

Fig. 6 shows depth profiles of D in undamaged steels and steels pre-damaged with 20 MeV W ions up to 0.56 dpa and subsequently irradiated with a) 20 eV D^+ and b) 200 eV D^+ at room temperature. The D concentration in damaged steels is higher by 5-10 times compared to undamaged steels because pre-irradiation with W ions increases the defect density in steels. The D concentration at radiation-induced defects in both ODS and Eurofer steels is similar indicating the similar radiation-induced defect density. The D concentration at radiation-induced defects does not depend on the Cr concentration in pre-damaged steels. According to the present results, nano-scaled ODS particles like Y_2O_3 or $Y_2Ti_2O_7$ are not the centers for the enhanced defect recombination under irradiation with W ions at room temperature and do not reduce the radiation defect formation compared to Eurofer. It seems that, probably, the temperature was too low and the irradiation dose up to 1 dpa was too small to observe an advantage of a reduction of radiation defect formation in ODS steels compared to Eurofer.

In the case of D plasma exposure with 20 eV, the D concentration in damaged steels is higher than in undamaged steels to a depth of ~3.5 μm (**Fig. 6a)** indicating that radiation-induced defects extend on a depth slightly larger than the damaged zone of 3 μm calculated by SRIM. The similar observation was made in the case of the D depth profile in W pre-irradiated with 20 MeV W ions [**4,20**]. The reason can be that the SRIM code does not take into account the mobility of defects. The diffusion of radiation–induced defects beyond the implantation zone may determine the extension of the damaged zone observed by the D depth profile. In the case of D plasma exposure with 200 eV D ions, sputtering and RIS determine the near surface modification and corresponding D depth profile. Because of sputtering, a reduction of the damaged zone after the irradiation of sample with 200 eV D^+ occurs as one can see in **Fig. 6b**. Moreover, the D concentration in damaged steels irradiated with 200 eV D^+ is higher than in undamaged steels to a depth of ~6 μm (**Fig. 6b)** that is far beyond the damaged zone. As the D concentration beyond the damaged zone increased only slightly in the case of 20 eV D^+ exposure

but it is greatly increased in the case of 200 eV D^+ exposure, it is an indication that the enhanced D concentration beyond the damaged zone can be associated with irradiation with 200 eV D^+ but not with a formation of radiation-induced defects far beyond the damaged zone by pre-irradiation with W ions. It is reasonable to suggest that an increase in the desorption flux due to sputtering with 200 eV D ions mentioned in [10] is smaller for damaged steels compared to undamaged ones, resulting to an increase in the mobile D concentration. However, the reason of the reduction of the desorption flux in the case of damaged steel compared to undamaged steel under the sputtering with 200 eV D ions is unclear and the question why the D concentration beyond the damaged zone is higher in the case of damaged steel than in the case of undamaged steel after the irradiation with 200 eV D ions is still open.

Fig. 7 shows a comparison of depth profiles of the D concentration in Eurofer pre-damaged with 20 MeV W ions up to 0.56 dpa and subsequently irradiated with 20 and 200 eV D ions at room temperature. In both cases of 20 eV D^+ and 200 eV D^+, the density of radiation-induced defects and their distribution is the same because the W ion pre-implantation was carried out at the same conditions for all specimens prior the D plasma exposure. Therefore, a decrease in the depth where D decorates the radiation-induced defects after irradiation with 200 eV D indicates the sputtering of this layer. The sputtered layer can be easy estimated from the difference in the D depth profiles for 20 and 200 eV D ion irradiation in the damaged zone in **Fig. 7**. The sputtered layer is ~**1.1** μm. It is possible to estimate the sputtering yield as

$$Y=(\text{Density} \times \text{sputtered layer})/\text{Fluence}= 6.3\times10^{28}\text{x}1.1\text{x}10^{-6}/6\times10^{24} = 1.15\times10^{-2}\ \text{Fe/D} \qquad (1)$$

The estimated sputtering yield of $1.15\text{x}10^{-2}$ Fe/D is about two times lower than the sputtering coefficient for a pure iron of $2.5\text{x}10^{-2}$ Fe/D reported in [**21**] due to preferential sputtering (PS) of light elements [**10**], leaving behind a thin tungsten-rich surface which can decrease the erosion yield [**22**]. The present estimation does not claim to be very accurate. The more precise estimation can be done by widely used weight loss measurements, see, for example [**22].**

However, this new method allows us to roughly estimate the sputtered layer and, consequently, the erosion yield.

We did not observe any significant reduction in the D concentration at radiation-induced defects after storing the samples in vacuum for several months (**Fig. 8**) in contrast to the strong reduction of the D concentration at intrinsic defects in steels reported in [**10**]. **Fig. 8** shows an example of depth profiles of D in ODS-9Cr steel pre-damaged with 20 MeV W ions up to 0.56 dpa and subsequently irradiated with 20 eV D^+ at room temperature up to a fluence of $2.9x10^{25}$ D/m^2. Insignificant change in the D concentration in the damaged zone during four months of storing the sample in vacuum after the plasma exposure was measured. At the same time, the D concentration is strongly reduced in a depth behind the damaged zone and the depth profile measured after four months beyond 3.5 μm is more or less smooth without sharp maximum. This is consistent with the data reported in [**10**] for undamaged steels. This means that weakly bounded D was desorbed or diffused into the bulk far away from the measured depth of 6 μm in order to be de-trapped at defects with high binding energy.

Pre-irradiation at different dpa at room temperature shows that the D concentration increases steeply at low dpa and gradually at high dpa as illustrated in **Fig. 9**. **Fig. 9** shows the D concentration at a peak damage in steels in comparison with that in tungsten (W) [**11**] as a function of dpa. The D concentration at radiation-induced defects saturates at ~0.25 dpa in steels and at ~0.5 dpa in W. A comparison of the D concentration at radiation-induced defects produced in W and RAFM's steels by pre-implantation with W ions at room temperature shows that the D concentration in W is about one order of magnitude higher than in steels because (i) lower binding energy of D with defects in steels compared to tungsten materials [**16,17,18**] and (ii) lower radiation-induced defect density in steels since the radiation-induced defect recovery occurs in steels at much lower temperature than in W **[4]**. The pre-irradiation with 20 MeV W ions was done at 300 K where free migration of vacancies occurs in steels with partial annealing

of radiation defects due to recombination and annihilation as it was reported in [**19]**. This leads to lower radiation-induced defect density **[4]** compared to W where the significant radiation-induced defect recovery occurs only at temperatures above 1100 K [**20]**. **Fig. 10** shows the D concentration in Eurofer at a peak damage in comparison with the D concentration in undamaged Eurofer measured at the same depth. No effect of pre-damaging at 300 K on the D retention after the D plasma exposure at temperatures above 500 K is observed in steels that is in an agreement with data reported in [**4**].

Thermal desorption spectra of D from (a) undamaged Eurofer and (b) Eurofer pre-damaged with 20 MeV W ions up to 0.25 dpa after the D plasma exposure with 20 and 200 eV per D at room temperature are shown **in Fig. 11**. The modification of the surface layer by sputtering with 200 eV D ions results in a significant reduction of the low-temperature TDS peak of 550 K for both undamaged and pre-damaged samples. Consequently, the surface modification due to sputtering under irradiation at 200 eV per D can reduce the total D retention as shown in **Fig. 11** and also in **Ref. [10]** due to an increase of the desorption flux which results in the reduction of the mobile D concentration near the surface, thus, decreasing the diffusion flux into the bulk. The reduction in the D retention after irradiation with 200 eV per D compared to irradiation with 20 eV D per D is ~1.5 and ~3 times for undamaged and damaged Eurofer, respectively.

Thermal desorption spectra of D from Eurofer pre-damaged with 20 MeV W ions at different dpa and subsequently irradiated with 20 eV per D at room temperature are shown in **Fig. 12.** A considerable increase of about 10 times in the primary low-temperature TDS peak of 550 K compared to less significant increase in other peaks of TDS after pre-damaging with W ions is observed. This means that pre-irradiation with W ions produces mainly radiation-induced defects with low binding energy for D. Therefore, at temperatures above 500 K, D is de-populated from defects with low binding energy and, consequently, an effect of pre-damaging on

the D retention is diminished that is consistent with data presented in **Fig. 10.** Already at 0.25 dpa, saturation in the D retention occurs as it was already seen in the case of the saturation in the D concentration at 0.25 dpa in damaged steel in **Fig. 9**. It is interesting to note that the pre-irradiation with W ions seems to enhance the density of already dominant defect from intrinsic traps in steels.

As it was mentioned in **[10],** the total D retention can be presented as the sum of the near surface retention, Ret_surf, and the bulk retention, Ret_bulk,

$$Ret\_total = Ret\_surf + Ret\_bulk \quad (2)$$

In general, the pre-damaging with 20 MeV W ions results in a formation of radiation-induced defects in a depth up to 3-4 μm and, therefore, an increase of Ret_surf. If the surface modification due to sputtering under irradiation with 200 eV per D can reduce the total D retention as shown in **Fig. 11**, the modification in a surface layer due to the formation of radiation-induced defects can considerably increase the total D retention as shown in **Figs. 11 and 12.** However, due to fast diffusion of D in steels, the deuterium saturates the radiation-induced defects fast and the D retention in the bulk, Ret_bulk, mainly contributes to the total D retention at high fluences. Thus, the radiation-induced defects produced to a depth of 3 μm do not significantly affect the overall D retention, Ret_total, in the case of high fluences as well as elevated temperatures in sufficienty thick samples. For this reason, one can see only slight increase in the total D retention in TDS presented in **Fig. 13** due to radiation damage up to 3 μm in depth after the implantation of 20 eV D ions in ODS steels up a fluence of 2.9×10^{25} D/m^2. Slight increase in the D retention in ODS steels in TDS peaks of 550, 980 and 1140 K is observed. No obvious change in the D retention in TDS peak of 780 K is observed. Concerning to neutron irradiation, radiation-induced defects will be distributed over all steel thickness and will influence the D retention at both low and high fluences.

To measure permeation through samples, samples were pre-damaged with 20 MeV W ions up to 0.56 dpa on both upstream and downstream sides. The permeation through samples was detected using the NRA measurements of the D concentration on the downstream side. **Fig. 14** shows one example of depth profiles of D on the plasma-facing side and downstream side of Eurofer pre-damaged with 20 MeV W ions up to 0.56 dpa and subsequently irradiated with 20 eV per D at 370 K up to fluences of $7x10^{23}$ D/m^2 and $2.2x10^{25}$ D/m^2. Deuterium occupies the radiation-induced defects on the downstream side already at low fluence such as $7x10^{23}$ D/m^2. At high fluence of $2.2x10^{25}$ D/m^2, the D concentration at radiation-induced defects on the downstream side of Eurofer is the same as on the plasma-facing side indicating that D saturates radiation-induced defects on the downstream side. This means that D penetrates through the sample.

3.3. Deuterium retention in steels pre-damaged with high heat flux

Pre-damaging with high heat flux was performed in Juelich using e-beam loading. An increase in the D retention in Eurofer pre-damaged with high heat flux compared to undamaged Eurofer after the D plasma exposure at temperatures of 290, 470, and 700 K is shown in **Fig. 15**. Although the damaged zone created by e-beam heating (~10 μm) in steels is larger than that created by W ion irradiation (~3 μm), the D retention after the D plasma exposure at 290 K is higher in Eurofer with radiation-induced damage than in Eurofer pre-damaged by high heat flux loading. At the same time, the radiation-induced defects do not affect the D retention after the D plasma exposure at temperatures above 500 K but the damage produced by e-beam loading slightly increases the D retention even after the D plasma exposure at 700 K. It is reasonable to suggest that e-beam heating produces both defects with low binding energy for D (i.e. dislocation, vacancies) and defects with high binding energy for D (i.e. vacancy clusters, pores).

Despite the fact that the density of defects with high binding energy for D produced with e-beam loading is less than the density of defects with low binding energy for D (see comparison of 290 and 700 K data in **Fig. 15**), it seems that it exceeds the intrinsic defect density of vacancy clusters and pores in undamaged steel. For this reason, the trapping of D in defects producing by e-beam can also contribute the D retention at high temperatures in contrast to irradiation with W ions when mainly the radiation-induced defects with low binding energy for D is generated.

4. Conclusions

Neutron-induced damage in RAFM steels was simulated by irradiation with 20 MeV W ions in this work. The high heat flux damage was simulated via e-beam thermal shock loading.

As it was reported in [**10**], the dispersion strengthening of the reduced activation ferrite-martensitic steel with yttrium oxide nanoparticles results in significant increase of the deuterium uptake. However, the pre-irradiation with 20 MeV W ions at room temperature up to 1 dpa results in similar concentration of deuterium in all kinds of investigated steels independent on the presence of nanoparticles and Cr content. The D concentration at radiation-induced defects after the exposure of pre-damaged steels to the D plasma at room temperature is higher by a factor of ten than in undamaged steels. The D concentration increases steeply at low dpa and gradually at high dpa and saturates already at 0.25 dpa. A comparison of the D concentration at radiation-induced defects produced in W and RAFM steels by pre-implantation with W ions at room temperature shows that the D concentration in W is about one order of magnitude higher than in steels because the considerable radiation-induced defect recovery occurs in steels already at room temperature.

The decrease of the D retention in pre-damaged steels with surface roughness formation at incident D ion energy of 200 eV was observed for damaged specimens which is

similar to undamaged steels. No visible difference in the nano-structured surface modification for different RAFM steels and for steels pre-damaged with W ions was observed. Thus, the pre-irradiation with 20 MeV W ions up to 1 dpa has no noticeable influence on the steel surface morphology after the D plasma exposure. This is not surprising. The radiation-induced defects (dislocations, vacancies, vacancy clusters, etc.) are effective trapping sites for D but do not affect the sputtering.

Pre-damaging with high heat flux increases the D retention in steels compared to undamaged steels exposed to D plasma at room temperature but has less pronounced effect compared to an increase in the D retention due to a formation of radiation-induced defects in steels. At the same time, the radiation-induced defects do not affect the D retention after the D plasma exposure at temperatures above 500 K but the damage produced by e-beam loading slightly increases the D retention even after the D plasma exposure at 700 K.

From the discussion above, we can conclude that (i) the formation of surface roughness due to sputtering decreases the D retention in both pre-damaged and undamaged steels, (ii) the formation of melting and cracking on the surface via thermal shock loading up to ~10 μm increases the D retention and (iii) the formation of radiation-induced defects up to ~3 μm increases the total D retention. The effects of above surface modifications diminish at elevated temperatures and high fluences because, in these cases, the D retention is mainly determined by bulk diffusion.

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Figure captions

Fig. 1. Eurofer samples after damaging with e-beam in JUDITH-1. Pulse duration is Δt=1 ms, absorbed power density is 0.71 GW/m^2, and pulse number is 10.

Fig. 2. SEM of Eurofer after damaging with e-beam in JUDITH-1. Pulse duration is Δt=1 ms, absorbed power density is 0.71 GW/m^2, and pulse number is 10.

Fig. 3. SRIM calculations of damage profile in Fe irradiated with 20 MeV W^{6+}.

Fig. 4. SEM images observed at 52° tilting of a) Eurofer pre-damaged with 20 MeV W ions up to 0.56 dpa and b) undamaged Eurofer after exposure to the D plasma with incident ion energy of 200 eV per D at 290 K up to a fluence of $6x10^{24}$ D/m^2.

Fig. 5. SEM image observed at 52° tilting of Eurofer pre-damaged with 20 MeV W ions up to 0.56 dpa, sequentially exposed to the D plasma with incident ion energy of 200 eV per D at 290 K up to a fluence of $6x10^{24}$ D/m^2 (**see Fig. 4a**) and then annealed by TDS with a linear ramp of 2 K/s up to 1200 K.

Fig. 6. Depth profiles of the D concentration in undamaged steels (lines with open symbols) and steels pre-damaged with 20 MeV W ions up to 0.56 dpa (lines with solid symbols). Both undamaged and pre-damaged steels were irradiated with incident ion energy of (a) 20 eV per D and (b) 200 eV per D at 290 K up to a fluence of $6x10^{24}$ D/m^2. Damage profile calculated by SRIM is shown as a dashed line.

Fig. 7. Depth profiles of the D concentration in Eurofer samples pre-damaged with 20 MeV W ions up to 0.56 dpa and subsequently exposed to the D plasma with incident ion energy of either 20 or 200 eV per D at 290 K up to a fluence of $6x10^{24}$ D/m^2. Damage profile calculated by SRIM is shown as a dashed line.

Fig. 8. Depth profiles of D in ODS-9Cr steel pre-damaged with 20 MeV W ions up to 0.56 dpa and subsequently exposed to the D plasma with incident ion energy of 20 eV per D at 290 K up

to a fluence of 2.9×10^{25} D/m^2. The measurements were performed in two weeks and four months after the D plasma exposure.

Fig. 9. The D concentration at a peak damage in steel and tungsten samples pre-irradiated with 20 MeV W ions at 290 K as a function of dpa.

Fig. 10. The D concentration at a peak damage in Eurofer pre-damaged with 20 MeV W ions up to 0.56 dpa in comparison with that for undamaged Eurofer (0 dpa) as a function of the temperature under the D plasma exposure. NRA measurements were done after the D plasma exposure with 20 eV per D up to a fluence of 6×10^{24} D/m^2.

Fig. 11. Thermal desorption spectra of D from Eurofer samples after exposure to the D plasma with incident ion energy of 20 and 200 eV per D at 290 K up to a fluence of 6×10^{24} D/m^2. (a) undamaged Eurofer and (b) Eurofer pre-damaged with 20 MeV W ions up to 0.25 dpa.

Fig. 12. Thermal desorption spectra of D from Eurofer samples pre-damaged with 20 MeV W ions at different dpa and subsequently irradiated with 20 eV per D at room temperature up to a fluence of 6×10^{24} D/m^2.

Fig. 13. Comparison of thermal desorption spectra of D in undamaged ODS steels and pre-damaged with 20 MeV W ions up to 0.56 dpa (marked as ODS-dam) and subsequently irradiated with 20 eV per D at 290 K up to a fluence of 2.9×10^{25} D/m^2.

Fig. 14. Depth profiles of the D concentration on plasma-facing and downstream sides of Eurofer pre-damaged with 20 MeV W ions up to 0.56 dpa and subsequently exposed to the D plasma with incident ion energy of 20 eV per D at 370 K up to fluences of 7×10^{23} D/m^2 and 2.2×10^{25} D/m^2. The thickness of Eurofer samples was 0.5 mm.

Fig. 15. A comparison of the D retention in undamaged Eurofer with Eurofer pre-damaged with W ions and high heat flux and sequentially exposed to the D plasma with 20 eV per D up to a fluence of 6×10^{24} D/m^2. The thickness of Eurofer samples was 1 mm.

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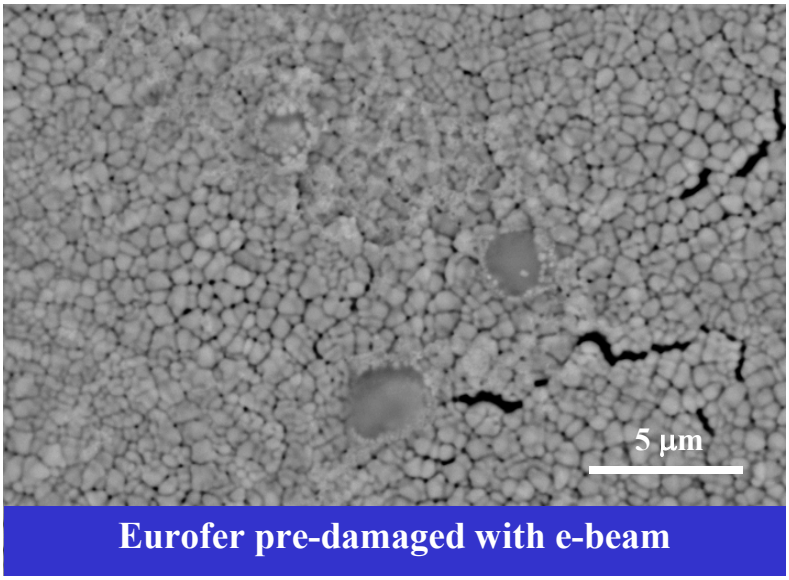

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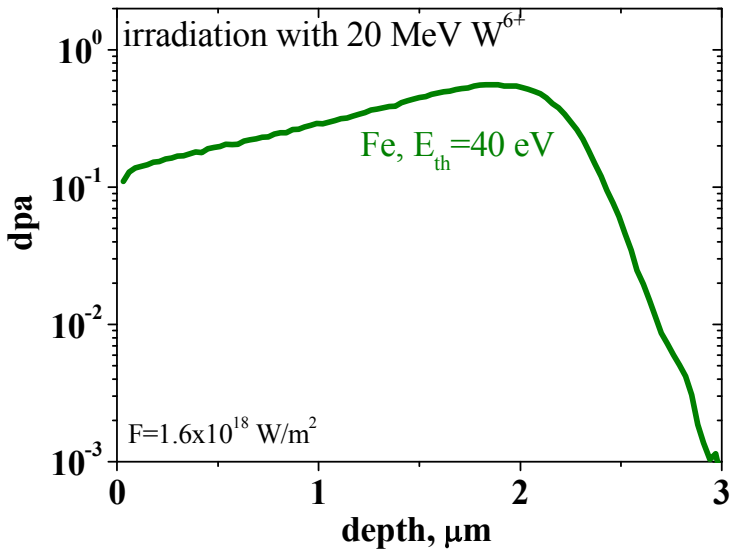

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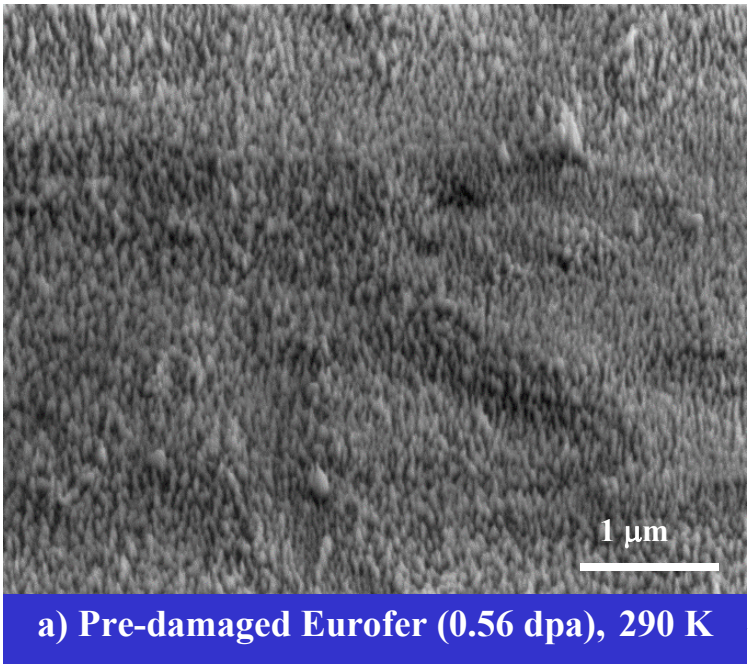

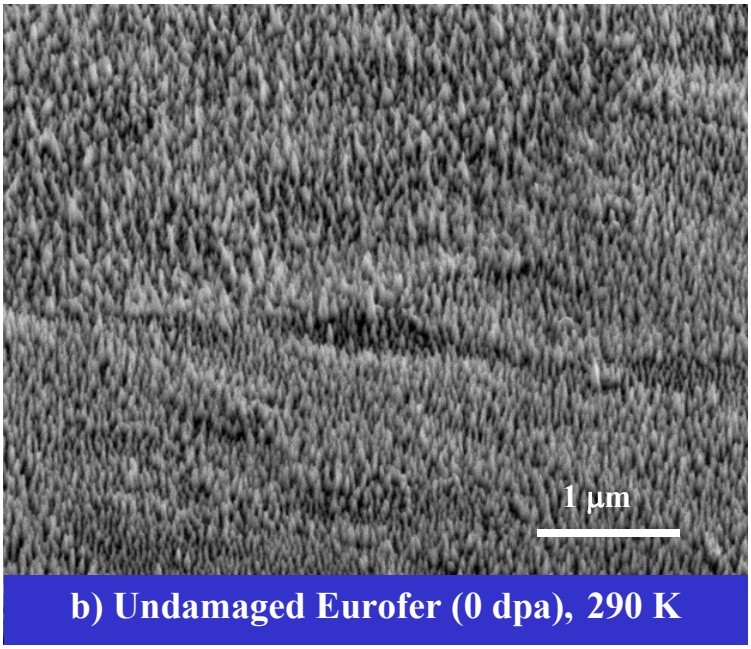

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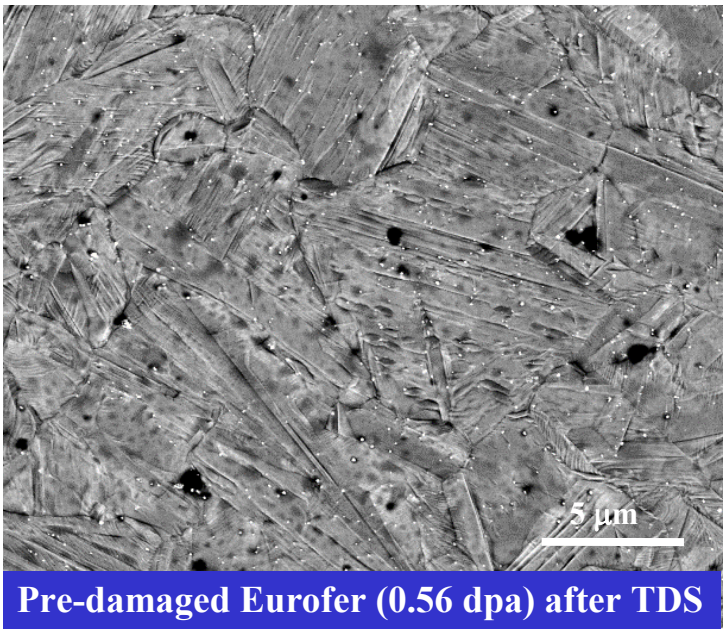

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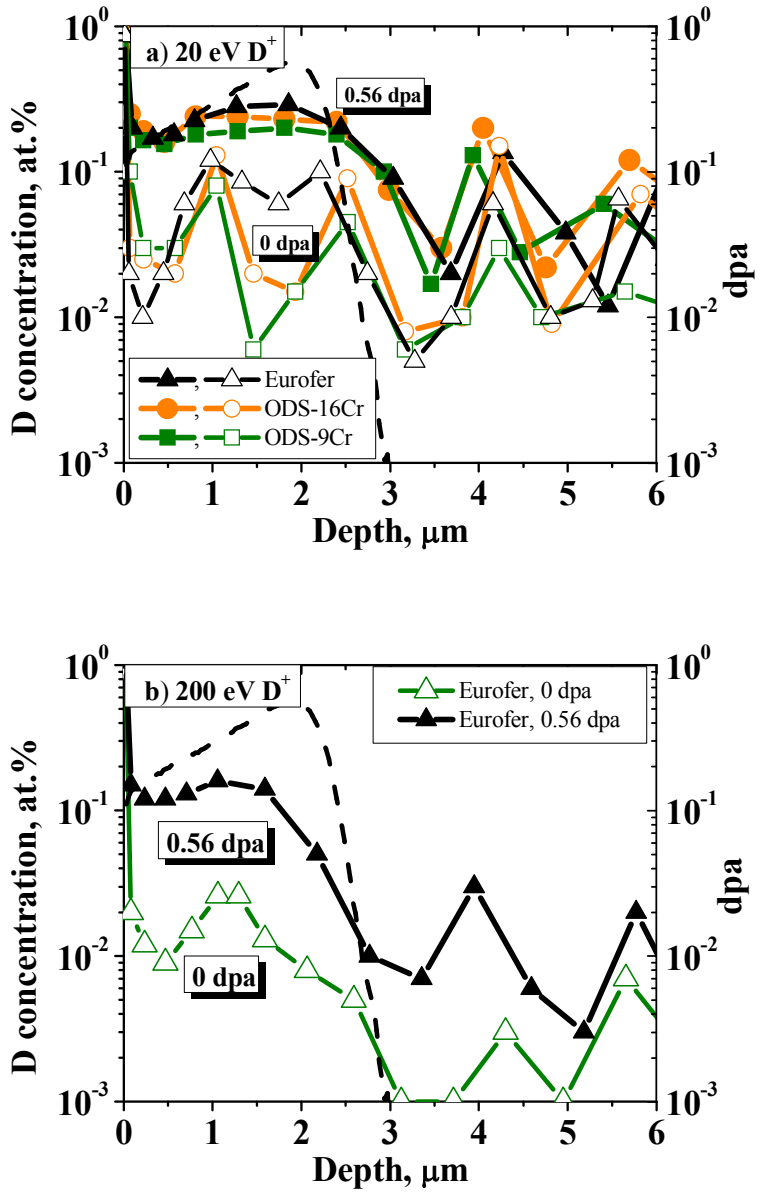

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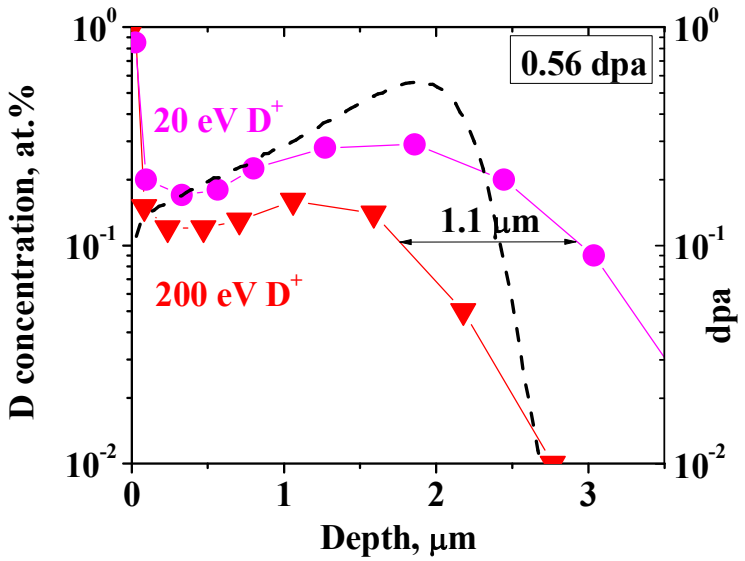

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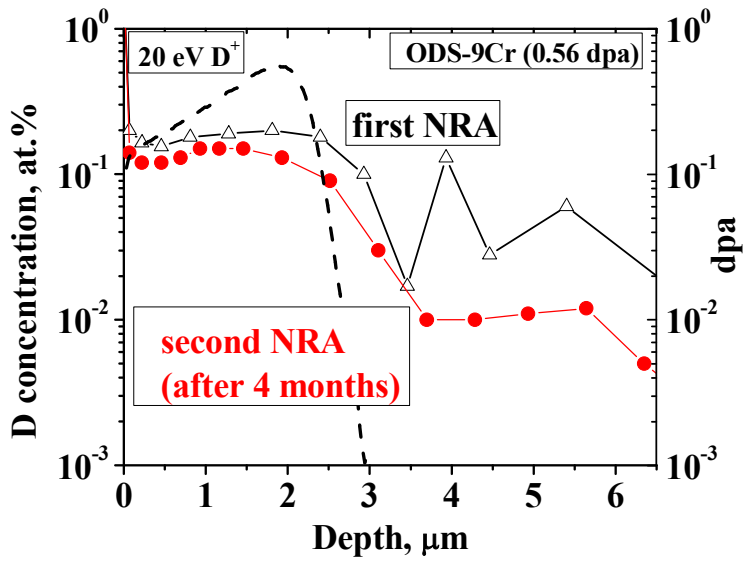

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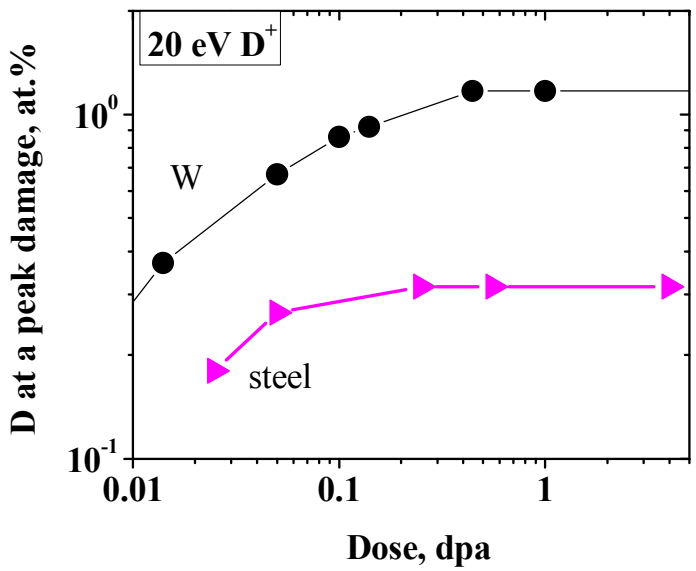

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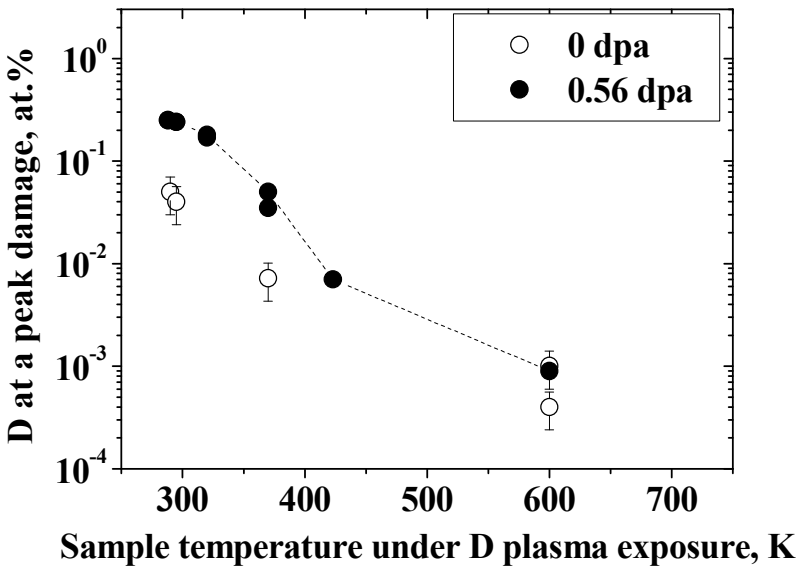

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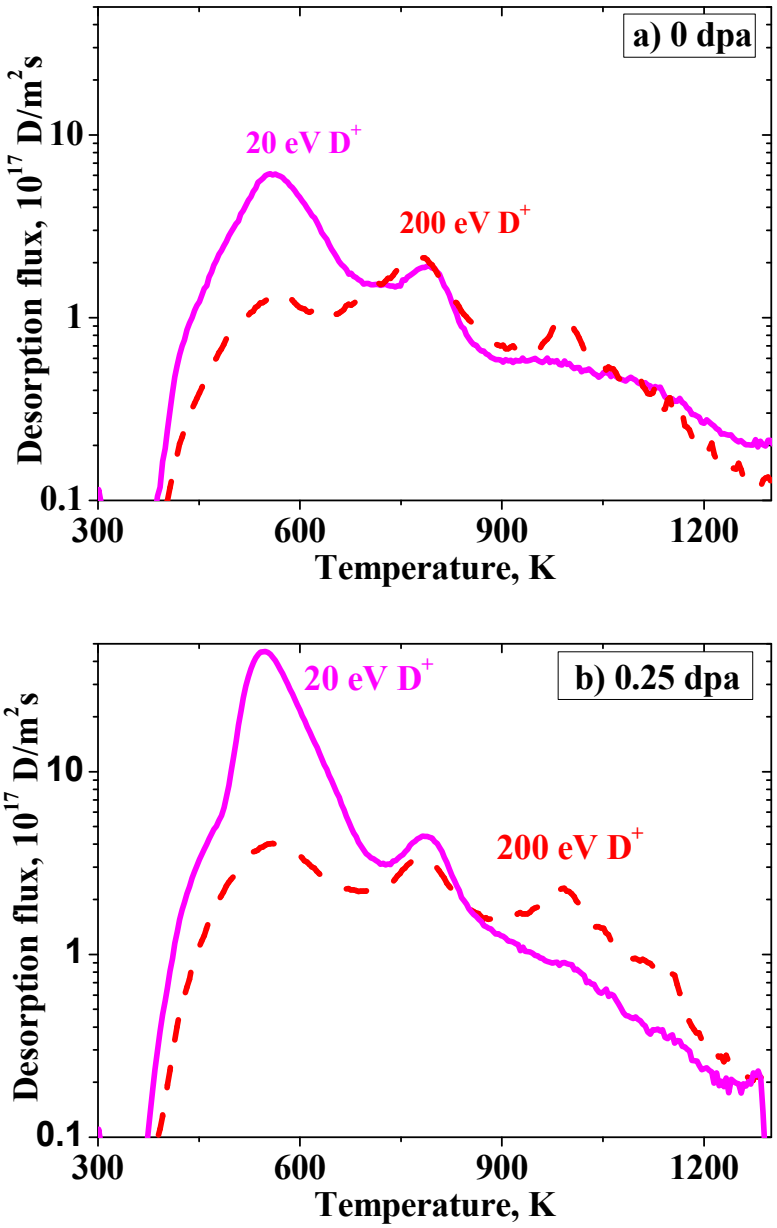

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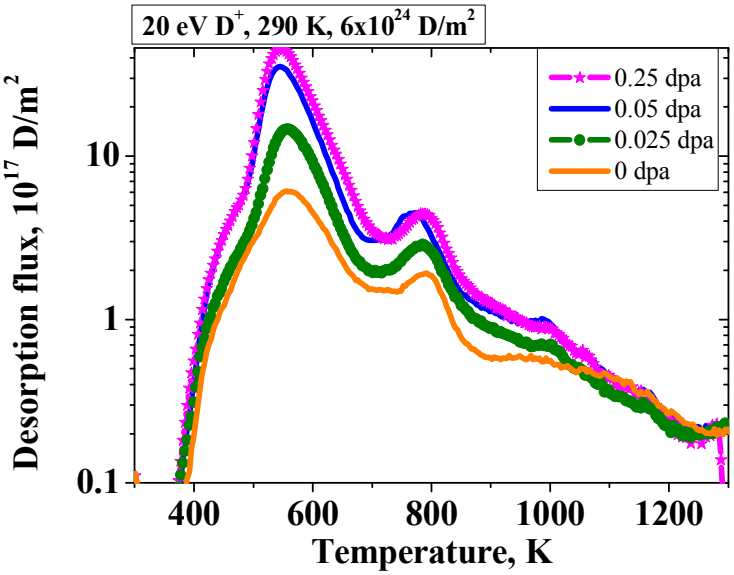

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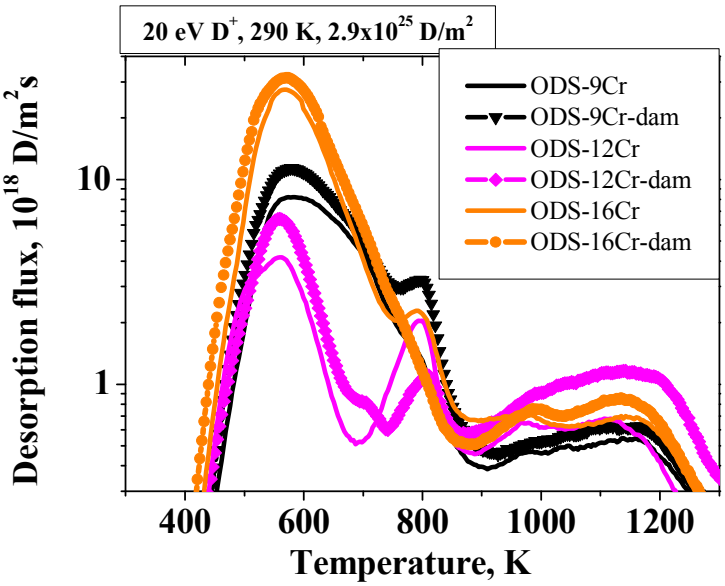

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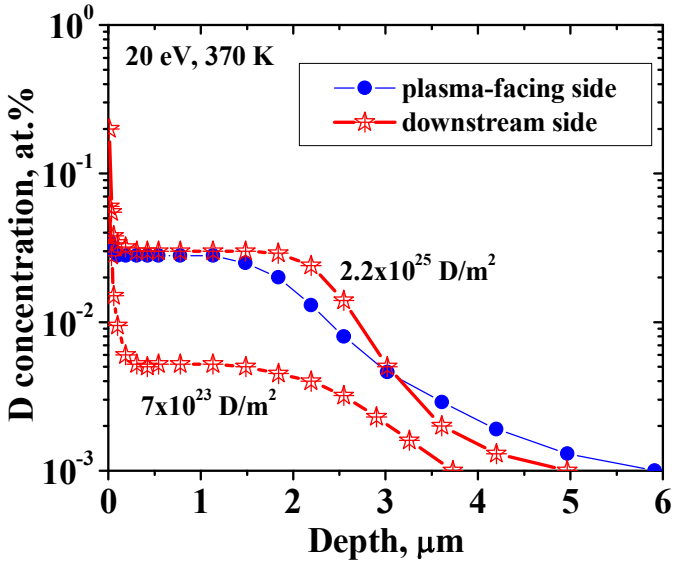

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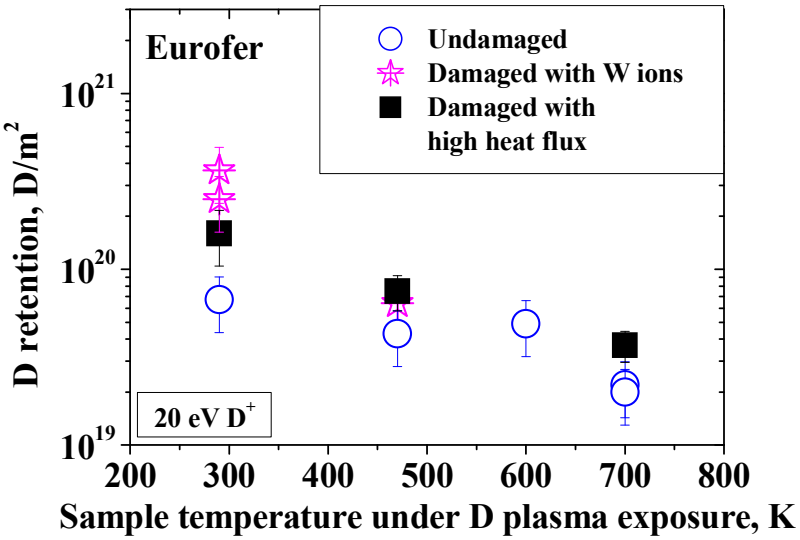

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