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The first study of deuterium retention in tungsten simultaneously damaged by high energy W ions and loaded by D

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Abstract

The first study of deuterium retention in tungsten simultaneously irradiated by W ions and exposed to D atom was performed at five different temperatures from 450 K to 1000 K. Samples were additionally exposed to D atoms at 600 K to populate the created defects in order to obtain the information on the defect concentration. The results were compared to different sequential damaging/exposure experiments. Synergistic effects were observed, namely, higher maximum D concentrations were found in the case of simultaneous damaging and D exposure as compared to damaging at elevated temperatures without offering D. Therefore part of the defects that would annihilate at high temperatures do not due to the presence of solute deuterium atoms in the bulk, that stabilize the defects. However, the deuterium retention is still lower as compared to sequential damaging at room temperature and post defect annealing. We have also observed the effect of exposure temperature on the D retention and depth range. At low temperatures the penetration depth is smaller for a given time i.e. fluence but the concentration is higher. On the other hand the maximum concentration is lower at high temperatures but the penetration is deeper due to faster diffusion. Results of simultaneous damaging and D exposure at elevated temperatures were also compared to the sequential experiment of damaging at room temperature and exposure to D at high temperatures showing that thermal D de-trapping is the dominant process at high temperatures.

Keywords: tungsten, deuterium retention, displacement damage, neutral atoms, NRA

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1. Introduction

Tungsten or advanced tungsten alloys are considered to be the most suitable material for plasma-facing components in future fusion reactors such as DEMO. In these nuclear devices tritium retention in neutron damaged tungsten will become a more significant issue. Namely, the predicted neutron damage for DEMO produced by neutrons from the fusion D-T reaction is more than an order of magnitude higher than in ITER, 2-6 dpa/fpy [1]. In order to study the influence of material displacement damage on fuel retention, high energy ions are used [2]. It was shown that fuel retention both in neutron damaged [3] and in W ion damaged tungsten (so-called self-damaged W) is strongly increased as compared to undamaged tungsten [eg. 4]. Until now all retention studies were performed by sequential high energy ion damaging and subsequent plasma/gas/atom loading of the material by hydrogen isotopes. However, in a real fusion reactor both implantation of energetic hydrogen ions and neutrals as well as damage creation by the neutron irradiation will take place at the same time. The consequences of synergistic effects for retention are unknown. It is well known that in some metals impurities such as hydrogen, change the behaviour of defect creation and recovery, e.g. influence vacancy migration during recovery stage [5]. Moreover, also theory predicts that radiation damage will be increased in tungsten in the presence of hydrogen atoms [6,7].

In this paper we present the first experimental results on simultaneous defect creation by high energy self-ion implantation and D atom beam loading in tungsten. In order to sort out the observed effects comparison to the sequential damaging/post annealing/exposure experiments will be made. Namely, three sequential experiments were performed with different damaging/exposure procedures, that help to understand the processes: i) W ion damaging at room temperature + samples post annealing at different temperatures for one hour + D atom exposure at 500 K to determine the trap concentration; ii) damaging at elevated temperatures + D atom exposure at 600 K to determine the trap concentration and iii) damaging at room temperature + D atom exposure at elevated temperatures. We describe in detail the simultaneous W ion damaging and D exposure and damaging at elevated temperatures whereas details of i) and iii) were described in more detail in [8] and [9, 10], respectively

2. Experiment

We have used polycrystalline 99.997 wt. % hot-rolled tungsten samples (PW) manufactured by Plansee, 12x15 mm² in size and 0.8 mm thick. Grains are oriented parallel to the surface normal. Samples were chemo-mechanically polished to a mirror like finish at Max-Planck-Institut für

Plasmaphysik (IPP), Garching, Germany. After polishing, samples were heated for 2 min in vacuum at 2000 K for re-crystallization. This procedure enlarges the grain size to 10-50 μm .

The samples were mounted in the INSIBA chamber at 2 MV Tandem accelerator at Jozef Stefan Institute. The set up shown in Fig. 1 enables in situ Nuclear Reaction Analysis (NRA) measurements before, during or after the D atom loading, details in [11], and irradiation of the samples by high energy ion beam such as W^{6+} ion beam with energy of up to 10.8 MeV. An electrostatic quadrupole lens is positioned in the beam line (not shown in Fig. 1), 4.5 m before the sample, in order to gather and focus the ion beam on the sample. The ion beam is shaped by two orifices that can be changed for each irradiation/analysis. They are positioned before the ion beam mesh charge collector [12], mounted at the entrance to the experimental vacuum chamber. The ion beam mesh charge collector was specially redesigned for this experiment, enabling us to retract it out of the ion beam or placing it in the beam for current measurement.

The sample is mounted by two Ta clamps on a holder with a temperature controlled heater, capable of heating the samples up to 1200K. Hydrogen atom beam source (HABS) for sample exposure to D atoms is mounted on the vacuum chamber at an angle of 51° with respect to the sample surface normal. It enables us to expose samples to D neutral atom beam with $5.4 \times 10^{18} \text{ D/m}^2\text{s}$ atom flux density at the ion beam position determined by erosion crater on an a-C:H film by ellipsometry [13,14]. The atom beam is created by thermal dissociation in a hot tungsten capillary that is heated to $2170 \text{ K} \pm 20 \text{ K}$ for all cases. By assuming that the kinetic energy is determined by the capillary temperature the mean energy of neutral atoms ($E=3/2 \text{ kT}$) in the beam is 0.28 eV. The deuterium gas pressure in the gas supply side was controlled by an all-metal leak valve and measured by an absolute capacitance manometer, Baratron by MKS. The average driving pressure during the atom exposures of tungsten sample was $260 \pm 10 \text{ mbar}$ for D_2 .

In the case of the experiment *simultaneous W ion irradiation and D atom exposure and damaging at elevated temperatures* samples were irradiated by W^{6+} ions with an energy of 10.8 MeV. The beam size was defined by two 4 mm circular slits. Before the start of the irradiation the beam was checked for homogeneity by observing the glow (by ionoluminescence) on a quartz glass inserted into the irradiating beam. The quartz can be placed instead of the first collimating slit and a blue glow is observed from the side by a mirror when it is irradiated by the beam. The current was first set to the proper value by the ion mesh charge collector that transmits 77.4% of the whole ion current. The ion mesh charge collector was then retracted in order not to have a meshprint of the mesh on the sample. The stability of the current was checked every 20-30 minutes. The ion current was stable within 5-10 %

yielding an inaccuracy for the total ion fluence of about 5% for all irradiated samples. The W ion irradiation current on the sample was set to 1.2 nA and irradiation time was 4 hours yielding a W ion fluence of $(1.4 \pm 0.07) \times 10^{18} \text{ W/m}^2$ on the irradiated 4 mm diameter spot. The displacement damage profile as calculated by SRIM is shown in Fig. 2. With a fluence of $1.4 \times 10^{18} \text{ W/m}^2$ we create damage dose of 0.47 dpa_{KP} (Kinchin-Pease calculation, 90 eV displacement damage energy, evaluating the “vacancy.txt” output) at the peak maximum yielding 3.3×10^{-5} dpa/s displacement rate.

The NRA analysis can only be performed about an hour after the W ion irradiation since one has to switch ion sources for the tandem accelerator. In the case of the W ion beam a sputter source is used and in the case of ^3He beam a duoplasmatron source is used. NRA analysis is performed by detecting protons from $d(^3\text{He},p)\alpha$ nuclear reaction using a 1500 μm thick partially depleted Passivated Planar Silicon (PIPS) detector. The NRA detector is mounted at 135° with respect to the incoming beam with a solid angle of 26.7 msr. A 24 μm Al absorber was placed in front of it, to stop the backscattered ^3He ions. The second PIPS detector with 300 μm thick depletion region is placed at 165° with respect to the incoming beam for detecting the backscattered projectile particles (RBS detector). ^3He beam was collimated by 2 mm circular aperture. The NRA analyzing diameter is smaller than the W irradiation diameter (4 mm) in order to be assured we analyse the damaged area only. In order to determine the D depth profile [15] in the studied samples up to 7 μm , six different ^3He ion beam energies were used: 730 keV, 1040 keV, 1550 keV, 2576 keV, 3394 keV and 4317 keV. We used the same collected charge for all six energies, corresponding to 14.9 μC ion dose He^+ (9.3×10^{13} He ions) for the four lower energies and 7.45 μC He^{2+} (4.65×10^{13} He ions) for the two highest energies. All samples were also analysed by thermal desorption spectroscopy (TDS) after the NRA analysis that gives us information about the binding energies of deuterium in the traps. The TDS spectra will be presented elsewhere.

The samples simultaneously damaged and exposed to D atoms at 450 K, 600 K and 800 K were after the final D atom exposure analysed also at IPP, Garching. Linear scans across the sample centre in the vertical and horizontal direction were performed at 2.5 MeV ^3He energy in order to get the information about the damaging area. There was a difference in the proton signal of a factor ~ 20 comparing the undamaged region to damaged spot. The measured diameter with increased proton signal due to W irradiation was in good agreement with the collimating slit diameter. Moreover the NRA measurement with nine ^3He energies was performed in the centre of the irradiation spot and the obtained depth profiles were in very good agreement with the ones measured after the loading at JSI.

3. Results

We will first describe the experiment *damaging at elevated temperatures*. In that case the samples were first irradiated by W^{6+} ions, 10.8 MeV ion energy, at three different temperatures 600 K, 800 K and 1000 K with the same irradiation time of 4 h. Each sample was right after the damaging exposed to D atoms at 600 K sample temperature for 24 h in the same chamber. Deuterium atom fluence of 4.7×10^{23} D/m² was high enough to saturate the created displacement defects in the material by D atoms. This was also checked by in situ NRA measurement after 18 h and 19.5 h of D atom exposure at 600 K for the 600 K and 800 K damaging case, respectively. The obtained profiles were the same within the error bars as the final measured depth profiles after the end of D atom exposure. The obtained deuterium depth profiles for sequential damaging at elevated temperatures and exposure at 600 K are shown in Fig. 2 together with the damage profile as obtained by SRIM. One can observe that the increased deuterium concentration agrees with the range of the damage profile. The obtained deuterium concentration is homogeneous throughout the damaged zone not following the damage profile. This is due to the fact that deuterium retention saturates above damage dose of 0.25 dpa [16] and also TEM analysis showed that dislocation damage is homogeneous in the damaged zone [8]. The maximum deuterium concentration at the damage peak maximum decreases with increasing damaging temperature from 0.18 at. % to 0.1 at. % for 600 K and 1000 K, respectively yielding a 60 % decrease. Moreover we also performed sequential damaging at room temperature and sample loading by D atoms at 600 K for 24 h and 42.5 h. The final depth profile is also shown in Fig. 2, marked by 300 K. The maximum concentration at the peak damage is higher as compared to sequential damaging at high temperatures and exposure to D atoms at 600 K. In that case we get (0.32 ± 0.2) at. % what is 1.8 times higher D concentration than for damaging at 600 K and post exposure at 600 K.

To make one step further towards more realistic situation we have performed first study of simultaneous tungsten irradiation by 10.8 MeV W^{6+} ions and D atom loading, atom flux of 5.4×10^{18} D/m²s, at five different temperatures of 450 K, 600 K, 800 K, 900 K and 1000 K. The damaging and loading took place for 4 hours, linearly increasing the damage dose and yielding the terminal 0.47 dpa_{KP} damage dose at peak maximum, with displacement rate of 3×10^{-5} dpa/s (SRIM calculation, Kinchin-Pease formalism and displacement energy of 90 eV). The D depth profiles were measured by NRA after the damaging and loading and they are shown in Fig. 3. For the 450 K case the atoms hardly penetrated into depth whereas in the case of higher temperatures 800 K - 1000 K the atoms did diffuse through the damaging area in 4 h indicating faster diffusion. The shallow penetration depth at 450 K is in agreement with the previous studies with sequential procedures of damaging at room temperature

and D atom loading at lower temperatures ≤ 600 K where higher fluence was needed to saturate traps [17]. The D concentration in the damaging area decreases with increasing the sample temperature, except in the case of 900 K and 1000 K that yielded, within the error bars, the same D concentration. The D concentration for the different damaging/exposure temperatures is determined by the balance of D flux into the bulk and D flux out of the bulk due to trapping/de-trapping of D atoms from an active trapping site at high temperatures. Namely, the thermal desorption spectra exhibit in the case of sequential damaging at room temperature and defect population by D atom exposure at 500 K [8] a broad desorption peak starting at 600 K with maximum at 875 K. Therefore there is an active trap site at these high temperatures whose occupancy is determined by the local equilibrium of atoms in solute states, driven by the flux into the bulk, and atoms de-trapping from the trap site.

In order to determine how many traps/defects were actually created in the material during the simultaneous damaging and D loading each sample was after the NRA analysis additionally exposed to D atoms at 600 K for 19 h corresponding to a fluence of 3.7×10^{23} D/m². After stopping the exposure we again performed the NRA analysis and the depth profiles are shown in Fig. 4. Ideally this additional loading should be performed at lower temperature e.g. 450 K not to anneal the created damage but this would mean much longer exposure time > 5 days. Beside this, since we have a lot of measurements already performed at 600 K on self-damaged W material in the INSIBA set-up [11] we have decided for exposure at this temperature. The additional exposure was enough for deuterium to penetrate throughout the whole damaged zone and saturate the created defects. As expected the highest concentration was obtained for the 450 K case, decreasing for higher temperatures up to 900 K. In the case of simultaneous damaging and exposure at 900 K and 1000 K the maximum concentration is the same. This could already indicate that the atoms that diffused into the layer at 1000 K during the irradiation prevented the defect recovery during irradiation/damaging. The maximum concentration at the peak damage for 600 K case is 0.24 at. % what is between the sequential damaging at 300 K and exposure at 600 K yielding 0.32 at. % and damaging at 600 K and exposure at 600 K yielding 0.18 at % both at damage peak maximum, see Fig 2.

4. Discussion and conclusions

In order to discuss whether there are any synergistic effects we will compare the simultaneous experimental results to the sequential experiments. A comparison between maximum concentrations at peak damage maximum for different damaging and exposure conditions is shown in Fig 5. In our opinion this is the relevant value that needs to be compared since it can be extrapolated to larger depths in the case of damaging by neutrons or damaging by different W ion energies where damage range is

different. The results obtained for damaging at high temperatures, indicated as “Dam. at high temp.”, simultaneous damaging and D atom exposure, “Simult. dam. & D loading” are shown there. The data are shown at the damaging/annealing temperature, whereas the traps were populated at 600 K for both cases. Additionally, we have performed a study where W samples were damaged at room temperature (RT) by 20 MeV tungsten ions at IPP, Garching, damage dose $0.25 \text{ dpa}_{\text{KP}}$, and annealed for one hour at temperatures from 600 K to 1200 K and then finally the defects were populated by D atoms, atom density flux $2.6 \times 10^{19} \text{ D/m}^2\text{s}$, at 500 K with fluence of $1.3 \times 10^{25} \text{ D/m}^2$ (144 h). Such high fluence was needed to saturate the damaged layer. Details about this experiment and results can be found in [8]. The results for this so-called “post annealing” are shown at the annealing temperatures in Fig. 5 (indicated as “Dam. + post annealing + D @ 500 K”). Since the exposure for the simultaneous damaging and loading and damaging at elevated temperatures were performed at 600 K we need to extrapolate the post annealing data for 600 K exposure. At 600 K there is already small defect annealing and atom de-trapping that decreases the maximum concentration for about 10 % as compared to exposure at 500 K. In the case of room temperature damaging and exposure at 600 K we have obtained a maximum D concentration of 0.32 at %, the depth profile is also shown in Fig 2. In order to compare with the other two data sets we use this data point as the reference through which we have put the straight line parallel to the damage post annealing and D exposure at 500 K, and indicated it as “post annealing”.

There is a difference between sequential damaging at elevated temperatures and exposure and simultaneous damaging and exposure. The simultaneous experiment yields higher maximum concentration for about a factor of 1.3 as compared to damaging at those temperatures and then populating the defects at 600 K. In the post annealing study we obtained an almost linear decrease of maximum concentration with the annealing temperature up to 1000 K and a more drastic decrease for 1200 K. One can observe that the post annealing at high temperatures yields the highest D retention. It is about a factor of 2 higher as compared to damaging at elevated temperatures. This difference between the two damaging procedures could be due to the larger diffusion of interstitials and vacancies at elevated temperatures that are mobile during the damaging at elevated temperature and can annihilate [18]. Whereas on the other hand when annealing is performed after the damaging at RT, the interstitials possibly spread away from the damaged area and get lost at the surface or at grain boundaries and are not available for annihilation. More over the vacancies start to create larger vacancy clusters and dislocation loops when only annealing is performed. This was also confirmed by the transmission electron microscopy study in [8,19]. The obtained maximum D concentrations for simultaneous exposure and damaging at different sample temperatures are just in between the two sequential procedures, damaging at high temperatures yielding the lowest retention and post annealing the highest

retention. Therefore we can say that we have observed a synergistic effect but it is not very dramatic. Namely there is a competition between defect annihilation at high temperatures and defect stabilization due to the presence of D atoms. From surface to bulk rate equations in a steady state [20] one can calculate the solute concentration of D atoms at 600 K, which is for our flux density of 5.4×10^{18} D/m²s, $\sim 10^{-10}$ at. fraction. Meaning that this concentration of solute atoms does influence on the defect formation/stabilization.

The maximum concentration obtained for simultaneous damaging and D atom exposure for individual temperatures after 4 hours (“Simult. dam & D loading”) is compared to the room temperature damaging and exposure at high temperatures (“Dam. at RT + Exposure”) in Fig 6. The details about samples damaged at room temperature and exposed at high temperatures can be found in [9,10] and details about thermodesorption spectra in [21]. In short W samples were damaged at RT by 20 MeV tungsten ions at IPP, Garching, damage dose 0.45 dpa_{KP}, and exposed to D atoms at different temperatures from 500 K to 900 K, atom density flux 3.5×10^{19} D/m²s. For samples exposed at temperatures ≥ 700 K a fluence of 3.8×10^{23} was sufficient for atoms to penetrate through the whole damage zone whereas in the case of 500 K and 600 K higher fluences were needed to saturate the whole damaged zone. When one exposes the damaged sample at high temperatures two effects take place at the same time. Namely, defects are being annealed and D atoms are being de-trapped from trapped to solute site. The steady state concentration is a balance between flux of atoms into the bulk and flux of atoms out of the bulk due to de-rapping. We have learned from the post annealing study that the defects are annealed at elevated temperatures what is shown by a decrease in D concentration by 70 % when going from 500 K to 1200 K, also shown in Fig 6 and indicated as “Dam. at RT + Post annealing”. On the other hand the maximum D concentration in the case of exposure at elevated temperatures decreases for almost two orders of magnitude comparing D concentration at 500 K and at 900 K. Therefore the thermal D de-trapping is the dominant process at high temperatures. However, even though the concentration is low at high temperatures the diffusion of deuterium is much faster and this could have drastic influence on the total retention in a neutron damaged material where defects extend much deeper (cm) as compared to ion damaged samples. The concentrations obtained for simultaneous damaging and exposure are below those of the post exposure for 450 K to 800 K. However at 900 K it is reversed. This effect could be dramatic for a fusion scenario of a hot divertor but additional experiments are needed to clarify the significance of this single data point.

In conclusion the first study of simultaneous damaging by tungsten ions and D atom exposure was performed. Synergistic effects were observed where higher maximum concentrations were found

in the case of simultaneous damaging and exposure as compared to corresponding damaging at elevated temperatures and post D exposure. Therefore part of the defects that would annihilate at high temperatures do not due to the presence of solute deuterium atoms in the bulk, that stabilize the defects as was predicted by theory [6,7]. We have also observed the effect of exposure temperature on the retention and depth range. At low temperatures the penetration depth is smaller for a given time i.e. fluence but the concentration is higher. On the other hand the maximum concentration is smaller at high temperatures but the penetration depth is larger due to faster diffusion.

Acknowledgments

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Figures and figure captions

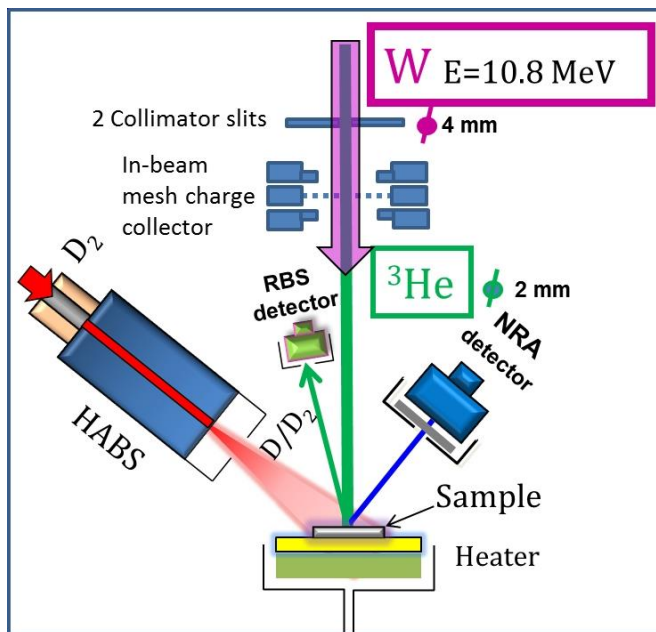


Figure 1 : Schematic figure of experimental set-up for simultaneous W ion irradiation and D atom exposure, together with the detector for Nuclear Reaction Analysis (NRA detector) and Rutherford Backscattering Spectroscopy (RBS detector).

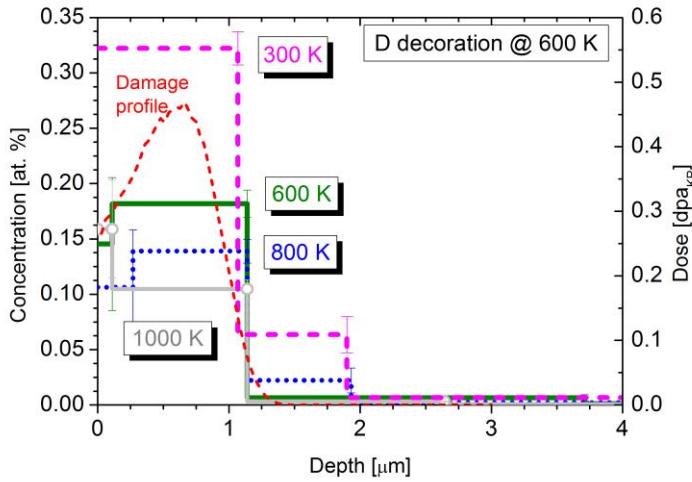


Figure 2 : Deuterium concentration depth profiles obtained after D atom exposure for 24 h at 600 K, atom fluence 4×10^{23} D/m², of samples damaged by 10.8 MeV W⁶⁺ ions at high temperatures indicated in the figure. For comparison the depth profile obtained for sample damaged at room temperature (indicated by 300 K) and exposed to D atoms at 600 K is also shown. The damage depth profile as obtained by SRIM is also shown in the figure by red dotted line.

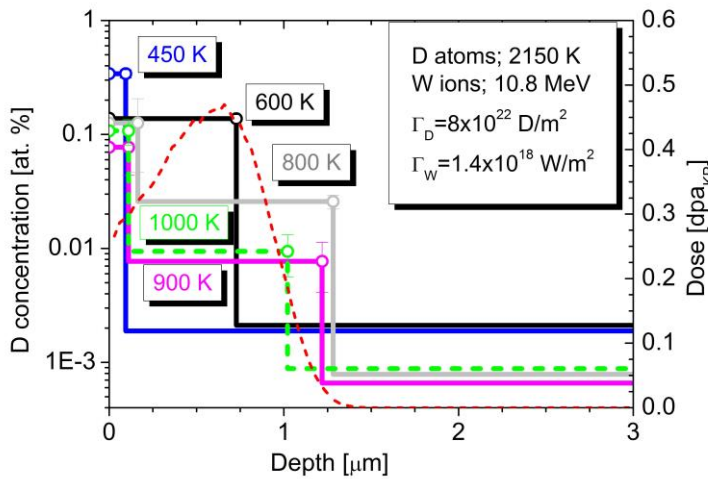


Figure 3: Deuterium concentration depth profiles in logarithmic y-scale obtained after 4h of simultaneous W ion irradiation and D atom exposure at the indicated temperatures. The terminal damage depth profile as obtained by SRIM is also shown in the figure by red dotted line.

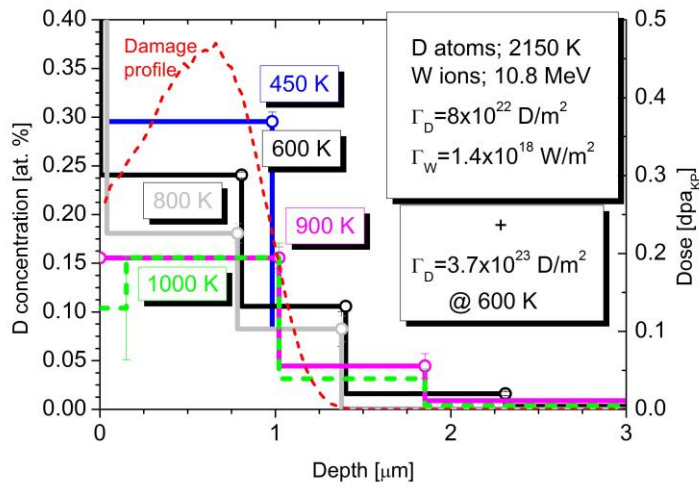


Figure 4: The deuterium concentration depth profiles obtained for samples simultaneously damaged and exposed to D atoms and additionally exposed to D atoms for 19 h at 600 K for defect population determination.

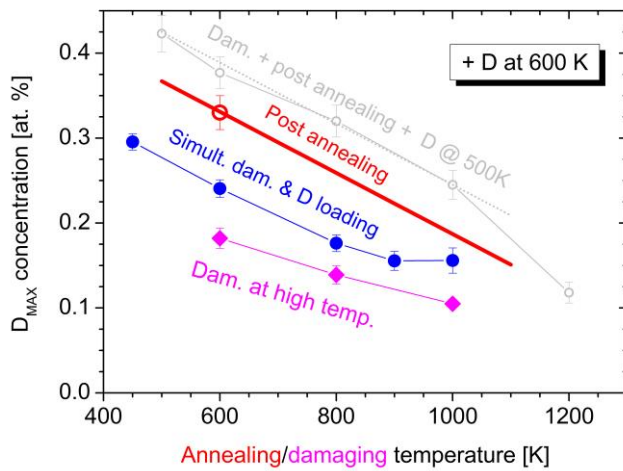


Figure 5: Maximum D concentration obtained at the maximum peak damage profile versus damaging/annealing temperatures for different damaging and loading experiments. Details about individual procedures are given in the text.

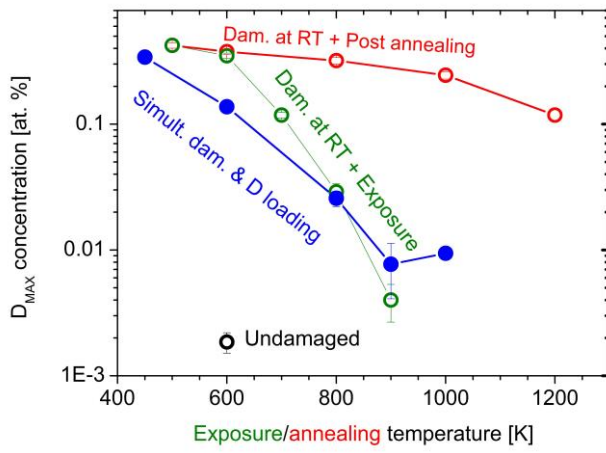


Figure 6: Maximum deuterium concentrations obtained at the maximum peak damage profile versus exposure/annealing temperature. The data are shown for three different damaging/exposure experiments. Details about individual damaging/exposure conditions are given in the text.