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Evaluation of Hydrogen Retention Behavior for Damaged Tungsten Exposed to Hydrogen Plasma at QUEST with High Temperature Wall

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Abstract

The undamaged W (tungsten) and 0.3 dpa (displacement per atom) damaged W samples by 6 MeV Fe^{2+} (Fe ion) irradiation were installed in the first wall of QUEST (Q-shu University Experiment with Steady-State-Spherical Tokamak) device and exposed to H (hydrogen) plasma in 2018A/W (Autumn / Winter) or 2019S/S (Spring / Summer) campaign to evaluate the impact of damages and impurities on hydrogen isotope retention. The surface morphology and chemical states of constituent atoms were observed by TEM (Transmission Electron Microscope) and XPS (X-ray photoelectron spectroscopy). It was found that thick Al deposits was found for the samples in 2019S/S campaign, which would come from the insulating plate during the CHI (Coaxial Helicity Injection) discharge. The additional 1 keV D_2^+ was implanted into both of these samples and D (deuterium) retention enhancement was evaluated by TDS (Thermal Desorption Spectroscopy). The downward ion toroidal drift changed the impurity deposition and damage profiles. In 2018A/W and 2019S/S, the D retentions for undamaged W samples had position independent, indicating that plasma would be well-controlled in this configuration. In case of Fe^{2+} damaged samples, irradiation damages clearly changed the D retention characteristics.

Keyword: Hydrogen isotope retention, Tungsten, Deposition layer, Plasma exposure, QUEST

1. Introduction

W (tungsten) is a candidate PFM (plasma facing materials) for future fusion reactors [1,2] due to its higher melting point, lower sputtering yield and lower hydrogen solubility. In actual fusion conditions, 14 MeV neutrons will be produced by D (Deuterium) - T (tritium) fusion reaction and will be irradiated into W at temperature above 623 K. In addition, irradiation damages will be introduced into W accompanied with impurity deposition, which will change the hydrogen isotope retention property.

In our previous studies [3,4], hydrogen isotope retention behaviors in PFM were studied for LHD (Large Helical Device) at NIFS (National Institute for Fusion Science), and for QUEST (Q-shu University Experiment with Steady-State-Spherical Tokamak) at Kyushu University [5]. It has been shown that hydrogen isotope retention were affected by carbon deposition and the location in the plasma facing wall.

In addition, it was found that not only formation of deposition layer [6] but also the damage distribution controls the hydrogen isotope retention. To evaluate the contribution of damages on hydrogen isotope retention, Fe^{2+} damaged W samples was installed into QUEST wall and exposed to H (hydrogen) plasma in 2018A/W (Autumn / Winter) and 2019S/S (Spring / Summer) campaign. QUEST has several unique features, in especially only H plasma experiment (no helium discharge was done), all metal plasma facing walls and higher wall temperature of 473 K [7-10]. Therefore, the evaluation of hydrogen isotope behavior for plasma facing wall in QUEST will be quite useful to understand the dynamics of fuel retention in future fusion reactor.

2. Experimental

Experimental procedure was summarized in Fig. 1. Polycrystalline W samples (10 mm⌀, 0.5 mm^t, 99.99% purity from A.L.M.T. Corp. Ltd) were prepared from a W rod under stress-relieved conditions. These samples were preheated at 1173 K for 30 minutes under the vacuum less than 10^{-6} Pa to remove impurities and damages. Thereafter, the irradiation damage was introduced by 6 MeV Fe^{2+} irradiation with the damage level of 0.3 dpa using a 3 MV tandem accelerator in TIARA (Takasaki Ion Accelerators for Advanced Radiation Application) at QST (National Institutes for Quantum and Radiological Science and Technology). According to the SRIM calculation, the implantation depth of Fe^{2+} was estimated to be ~ 1.1 µm [11]. The Fe^{2+} irradiated W samples and undamaged W samples were introduced at 3 different positions (Top, Equator and Bottom walls) in QUEST as shown in Fig. 2. In addition, small undamaged W TEM disks (3 mm⌀, <0.2 mm^t) were also installed in the same positions to evaluate the surface morphonogy change by QUEST plasma exposure. The TEM disks were prepared using the same Polycrystalline W rods mentioned above by cutting with the thickness less than 0.3 mm and punching out the sample with the diameter of 3 mm. Thereafter, these samples were polished electrically using NaOH solution to achieve TEM disks. Therefore, all the TEM disks were undamaged condition. These samples were exposed to H plasma during 2018A/W or 2019S/S campaign. The unique future of 2018A/W campaign was long term discharge about 96 minites with downward ion toroidal drift and current start experiment such as 8.2 GHz high power klystron system [7] and CHI (Coaxial Helicity Injection) discharge (Fig. 3). [8] The total discharge number for 2018A/W campaign was 1396 times. Compared to 2018A/W campaign, long term discharges with the maximum duration of 32 minutes was performed in 2019 S/S campaign with downward ion toroidal drift. Thereafter, TF direction was reversed. Current start experiment was performed with upward ion toroidal drift. The total discharge number was 574 times. Especially in CHI discharge, the H plasma was generated from the electrode on the Bottom wall and spread throughout the vessel. These discharges would introduce significant damages to the Bottom wall. It is expected that the constituent atoms (Al, O) of the insulating plate on Bottom wall were released during the CHI discharge. After H plasma exposure at QUEST, the chemical states and the depth profiles of atomic concentrations were evaluated by XPS (X-ray photoelectron spectroscopy, ESCA1600, ULVAC-PHI Inc. Mg-Kα 400 W,1253 eV) using the combination of 3 keV Ar^+ sputtering technique (sputtering rate of 1.42 nm/ min (based on Si substrate) using Ar^+ flux of 2.0×10^{16} Ar^+ m^{-2} s^{-1} with irradiation angle of 30 degree to the surface normal) at Shizuoka University. In addition, TEM (transmission electron microscopy) observation was also performed for undamaged W at Kyushu University using JEM 2000EX, JEOL Ltd. To evaluate the enhancement of D retention after exposure of QUEST H plasma, additional 1 keV deuterium ion (D_2^+) implantation was performed with the ion flux of 1.0×10^{18} D^+ m^{-2} s^{-1} up to the ion fluence of 1.0×10^{22} D^+ m^{-2} at room temperature. The H and D desorption behaviors were evaluated by TDS (thermal desorption spectroscopy) from room temperature to 1173 K with a heating rate of 0.5 K s^{-1}. The sample temperature was measured by the K-type thermocouble attached at the center of sample holder. The sensitivities of H and D were caliburated by the standard calibration leak before the TDS experiment.

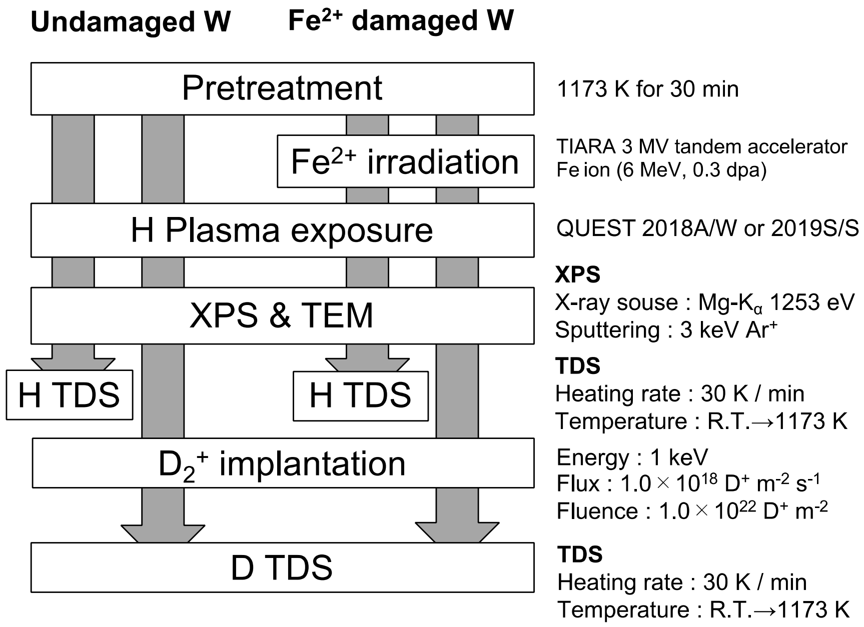

Fig. 1 Experimental procedure in this study. Several samples with same pretreatment were installed in QUEST H plasma exposure. The different samples were used for XPS, H TDS and D TDS experiment.

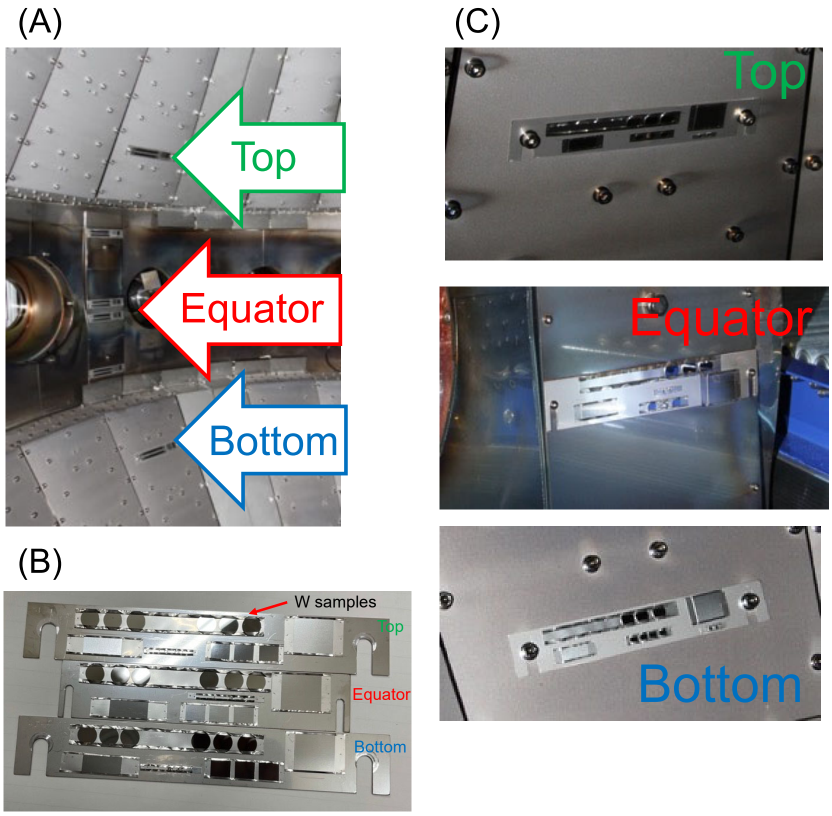

Fig. 2 Photos of (A) sample mounting on QUEST first wall, (B) the sample holders and (C) photomacropraphs of sample mountings on QUEST.

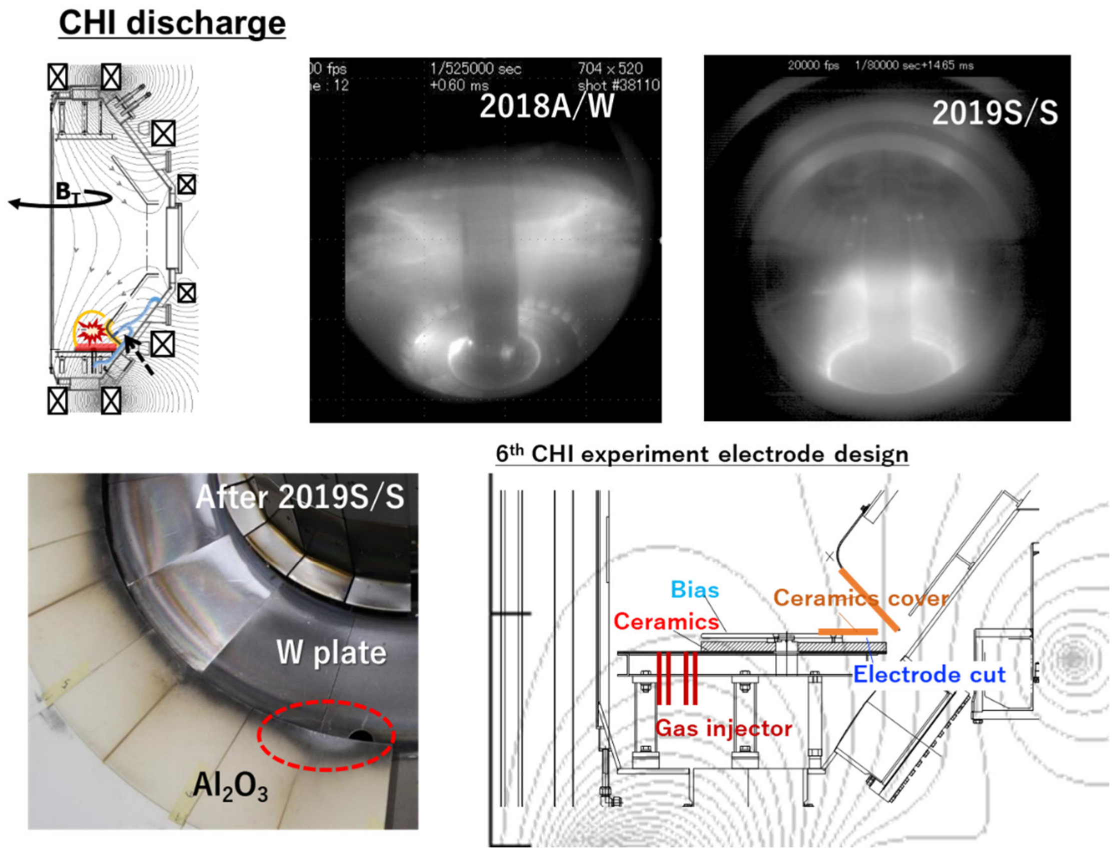

Fig. 3 Schematic diagram of CHI discharge device and photos during CHI discharge.

3. Results and discussion

3.1. Atomic concentration in the depth direction evaluated by XPS

Fig. 4 shows the XPS depth profiles of constituent atoms for the undamaged W samples placed on Top, Equator and Bottom walls after QUEST H plasma exposure. For Equator in 2019 S/S, the deposition layer was not completely removed even after 400 second of Ar^+ sputtering. It was found that carbon was the major deposit for all the samples, but the source of carbon was not confirmed at this time. The typical constituent atoms beneath the deposit were Fe and Cr, coming from stainless steel. In 2019S/S, not only stainless steel but also Al with Al_2O_3 state, was deposited as shown in Fig. 5. This may be coming from inslating plate for CHI discharge as the discharge trace was observed as shown in Fig. 3. In addition, the erosion/deposition profile was changed, namely deposition dominated area was located at Bottom wall. These facts indicated that the opposite profile was observed due to the reversed direction of toroidal magnetic field.

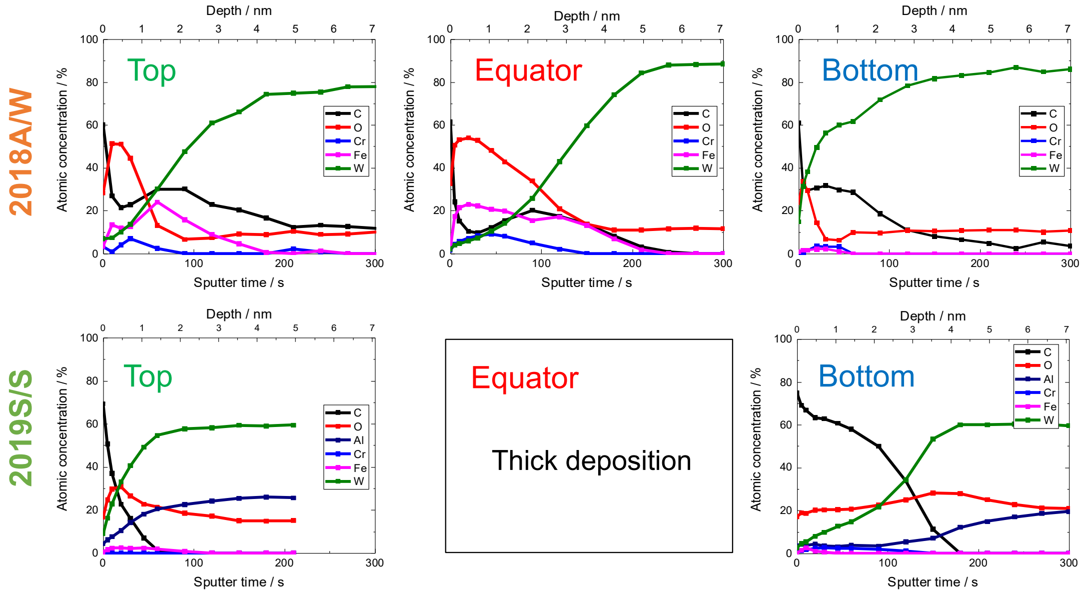

Fig. 4 Depth profiles of constituent atoms for Top, Equator and Bottom wall samples exposed to QUEST plasma.

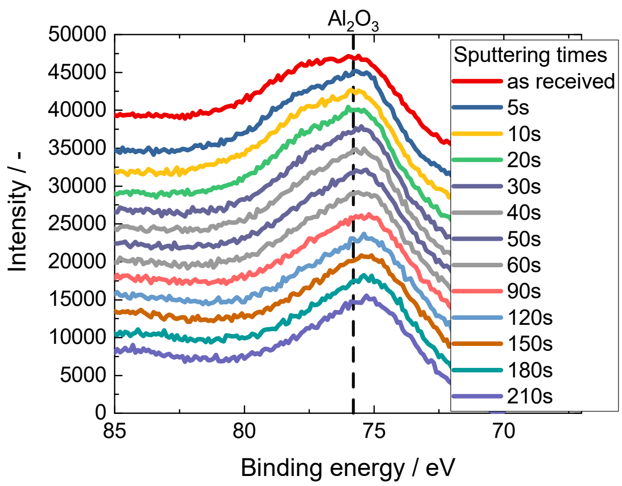

Fig. 5 Chemical states of Al for bottom sample in 2019S/S as a function of sputtering times

3.2. Evaluation of morphology change after QUEST H plasma exposure

TEM images of the undamaged W samples exposed to QUEST H plasma were summarized in Fig. 6. To observe the change of surface morphology by annealing, TEM disk was heated in vacuum at 873 and 1073 K and the TEM images were shown in almost the same position. For 2018A/W sample in Fig. 6 (b), dislocation loops were formed in the near-surface layer for Bottom wall sample after annealing at 873 K. However, almost all the dislocation loop was recovered at 1073 K as shown in Fig. 6 (c). In 2019S/S, thick deposits were formed at the egde of TEM disk as shown in Fig. 6(d). Figs. 6(e) and (f) show the result of

BF (Bright Field) and DF (Dark Filed) mode for Bottom wall sample. A lot of white spots were observed at DF image, indicating the formation of crystalline structure, namely W-C structure at 873 K. It was thought that W-C was formed by C irradiation and heat load during the plasma operation. Therefore, dense plasma would introduce the dislocation loops (Fig. 6 (b) and interaction of impurity carbon with W to form W-C structure (Fig. 7) on Bottom wall, which would be caused by higher heat load during CHI discharge as can be found in the picture of Fig. 3.

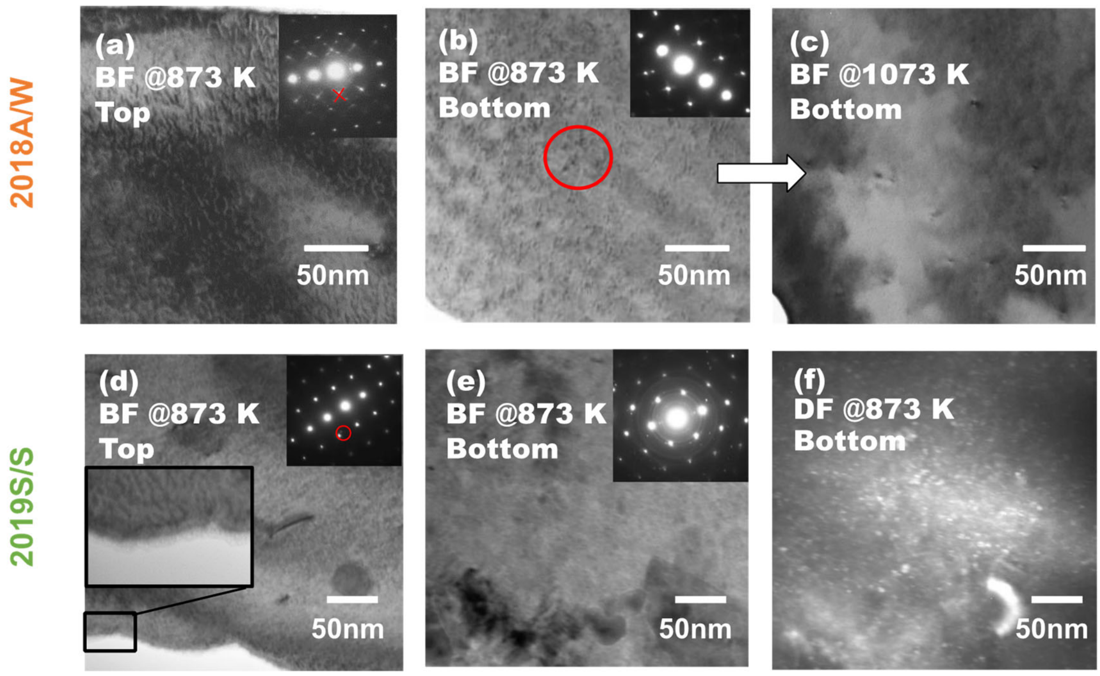

Fig. 6 TEM images focused on dislocation loops for Top and Bottom samples after QUEST H plasma exposure in 2018A/W or 2019S/S. Figs. 6 (a), (b) and (c) show the surface condition in 2018A/W, and Figs. 6 (d), (e) and (f) show that in 2019S/S. These TEM images were observed at almost the same position to observed the change of damage diostribution.

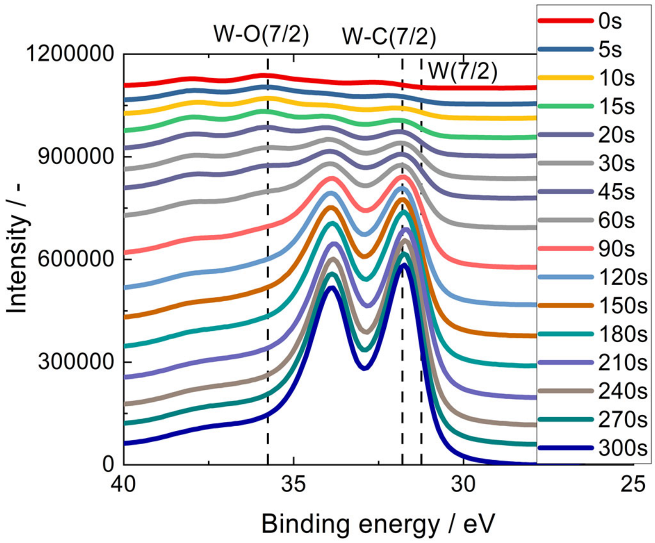

Fig. 7 W 4f XPS spectra for Bottom sample in 2019S/S as a function of Ar^+ sputtering time. Tungsten oxide was formed on the top surface, but the tungsten carbide was the major chemical state beneath the surface.

3.3. The hydrogen isotope retention behavior for undamaged samples

The H retention was evaluated by TDS using undamaged W samples exposed to QUEST H plasma only. For the evaluation of D retention enhancement, additional 1 KeV D_2^+ implantation was performed at Shizuoka University for undamaged W exposed to QUEST H plasma to reveal the impact of QUEST plasma exposure on D retention.

Fig. 8 shows the H_2 and D_2 TDS spectra for undamaged W samples with QUEST H plasma exposure. It was found that major H_2 desorption was located at the temperature above 900 K for all the samples as shown in Figs. 8 (a) and (c). In 2019S/S, H retention was decreased because the number of plasma shots and their durations were reduced. The D desorption was consisted of one major desorption stage at 400 K for all the samples. Based on our previous studies [12,13], the D_2 desorption stage at around 400 K was attributed to the desorption of D trapped by dislocation loops or surface. The D_2 TDS spectrum for undamaged W without QUEST H plasma exposure was found in our previous paper [11]. The D desorption at 400 K was slightly reduced for QUEST H plasma exposed sample due to the existence of H. The D retentions for Top and Bottom walls was enhanced due to surface adsorption of D with impurities. Therefore, the impact of QUEST H plasma exposure on D retention would be small for undamaged samples.

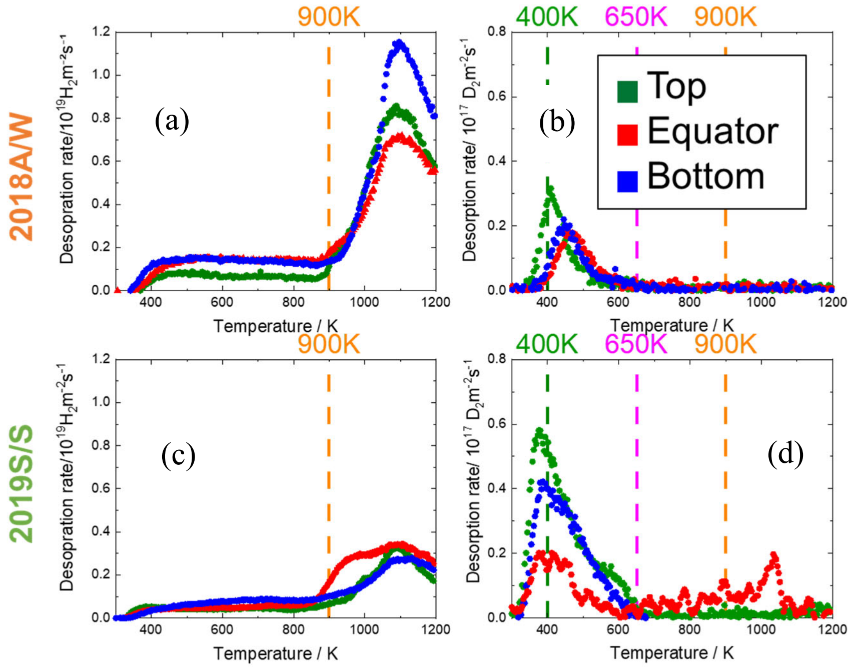

Fig. 8 (a) H_2 and (b) D_2 TDS spectra for the undamaged W samples placed at Top, Equator and Bottom walls during 2018A/W or 2019S/S QUEST plasma campaign. In case of D_2 TDS spectra, additional 1 keV D_2^+ implantation was performed at Shizuoka University before TDS.

3.4. The hydrogen isotope retention behavior for Fe^{2+} damaged samples

To evaluate the enhancement of D retention after exposure of QUEST H plasma for Fe^{2+} damaged samples, additional 1 keV D_2^+ implantation was performed at room temperature. Fig. 9 shows the TDS spectra of Fe^{2+} damaged sample exposed during 2018A/W or 2019S/S campaign. The gray dots shows the TDS results for Fe^{2+} damaged sample without QUEST H plasma exposure, but 1 keV D_2^+ implantation was performed at Shizuoka University. For 2018A/W samples, the amount of desorbed D at 800 K was reduced for all the samples (Top, Equator and Bottom) compared to those with QUEST H plasma exposure sample as the effect of irradiation damages on D retention was mitigated by the accumulation of deposition on the surface. In Bottom wall, the addition D desorption stage at 700 K was appeared due to the erosion dominanted area where the downward ion toroidal drift would introduce addition damages. In case of the samples exposed to 2019S/S QUEST H plasma, the amount of desorbed D at 800 K was also reduced. Especially, the D retention for Bottom wall was significantly reduced as the implanted D would not penetrate through the deeper region due to the acculation of dislocation loop near surface, where large amount of H was retentaion, which would work as the hydrogen diffusion barrier by the formation of the crystalline deposition layer [14] confirmed by XPS and TEM observations. The additional D desorption peak at 550 K was observed at Top wall, which would be caused by the upward of ion toroidal drift and the existence of irradiation defects introduced by QUEST plasma. The position of this desorption stage was shifted compared to that for Bottom wall sample at 2018A/W. Since there was a peak near 600 K for the undamaged sample, it was considered that D would be traped by the damages. The D desoption spectra on the Equator wall had a lot of peaks. In Equator, H plasma was difficult to hit due to far distance from the plasma, it could be thought that the enhancement of D retention would be caused by the accumulation of deposits. Therefore, the enhancement of D retention for Fe^{2+} irradiated sample would be caused by the introduction of irradiation damages, which was not observed for the undamaged sample.

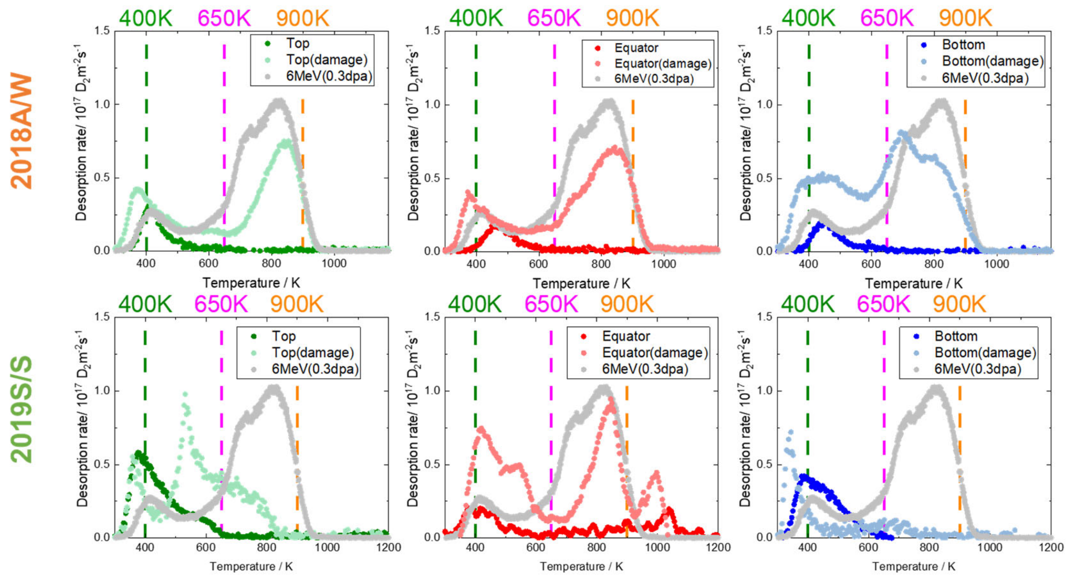

Fig. 9 D_2 TDS spectra for Fe^{2+} damaged samples placed at Top, Equator and Bottom walls after 2018A/W and 2019S/S QUEST plasma exposure. The gray spectra show D_2 TDS results for only Fe^{2+} damaged samples (no QUEST plasma exposure).

4. Conclusions

The hydrogen isotope retention behaviour for Fe^{2+} damaged samples after QUEST H plasma exposure at 2018A/W and 2019S/S campaign was studied by TDS with taking account of erosion/deposition profiles and plasma condition. It was found that the CHI discharge of 2019S/S induced the Al deposition. The downward ion toroidal drift changed the wall deposition and damage profiles. In 2018A/W and 2019S/S, the D retentions for undamaged W samples had position independent, indicating that plasma would be well-controlled in this configuration. In case of Fe^{2+} damaged samples, irradiation damages clearly changed the hydrogen isotope retention characteristics. The D retention was controlled by the sample position in QUEST.

Acknowledgments

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