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Influence of mixed material layer formation on hydrogen isotope and He retentions in W exposed to 2014 LHD experiment campaign

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ABSTRACT

Influence of mixed material layer formation on hydrogen isotope retention in W exposed to 2014 Large Helical Device (LHD) experiment campaign was evaluated by Thermal Desorption Spectroscopy (TDS), X-ray Photoelectron Spectroscopy (XPS), Scanning Electron Microscope (SEM) and Transmission Electron Microscope (TEM). It was found that a lot of hydrogen isotopes were trapped by the carbon-dominated mixed-material layer deposited on the plasma facing materials. Most of He was also trapped in the carbon-dominated mixed-material layer and the corresponding desorption temperature was limited to be about 600 K, 900 K and 1200 K, respectively. However, the hydrogen retention behavior for erosion dominated area was clearly different from those for deposition dominated area and typical Plasma Wall Interaction (PWI) area, where He bubbles were introduced near the sample surface, leading to the introduction of various types of trapping sites in W.

1. Introduction

Tungsten (W), which possesses higher energy threshold for sputtering, low ability to hydride formation and low hydrogen solubility level, has been selected as the most likely candidate of plasma facing materials (PFMs) for future fusion reactors [1–3]. Evaluation of fuel retention in W is one of important issues to sustain the safety fusion operation with sufficient supply of deuterium/tritium. However, our recent studies [4–7] showed that the surface of PFMs after plasma exposure was quite different from that before experiment, leading to the hydrogen isotope behavior should be also changed and enhanced larger than that for undamaged W. Even if the full metallic PFMs are used in fusion device, the accumulation of impurity layer on the surface of PFMs cannot be ignored, which was demonstrated in the spherical tokamak, Q-shu University Experiment with Steady-State Spherical Tokamak (QUEST)[8].

The recent studies [4–7] related with hydrogen isotope retention enhancement for W exposed to Large Helical Device (LHD) plasma campaign showed that the surface nature was clearly changed in most places except for erosion dominated area, where the metal surface still existed after plasma exposure, however huge amount of irradiation damages were introduced. These facts indicate that the combination of damage introduction and impurity deposition would control total fuel retention in fusion device, which is the important results that have been studied in this work.

In this study, W samples were installed in 2014 LHD plasma discharge campaign and exposed to total of 6441 H and He shots, and the surface change and hydrogen retention enhancement were evaluated and compared with those in previous plasma campaigns. In addition, High Temperature Thermal Desorption Spectroscopy (HT-TDS) was applied to heat the samples above 1800 K to evaluate He desorption behavior in 2014 LHD plasma exposed W [9]. The correlation between hydrogen isotope retention enhancement and He retention in 2014 LHD plasma exposed W will be discussed.

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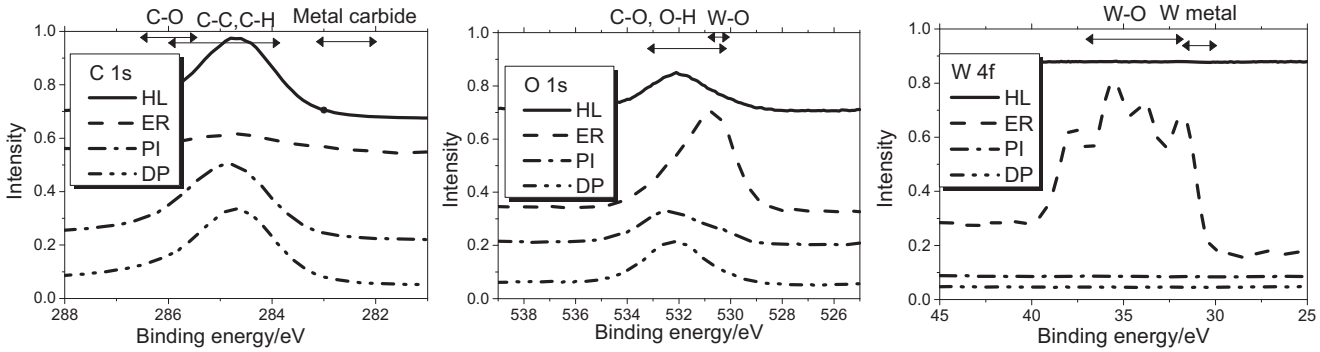

Fig. 1. XPS spectra for C-1s, O-1s and W-4f for 2014 LHD plasma exposed W.

2. Experimental

2.1. LHD plasma discharge exposure

Disk-type stress relieved tungsten (W) samples with the size of ϕ 10 mm × t 0.5 mm for TDS and X-ray Photoelectron Spectroscopy (XPS) and ϕ 3 mm × t ~ 0.1 mm for Transmission Electron Microscopy (TEM) were placed in 4 unique positions, namely the plasma wall interaction area (PI), the deposition area (DP), the higher heat load area (HL), and the erosion dominated area (ER). The detail positions can be found in our previous papers [4–6]. All the samples were mirror-polished and annealed at 1173 K for 30 min before installation. These samples were exposed to 2014 LHD experimental campaign with the total discharge of 6441 shots including 1545 shots of He and 4896 shots of H. S. Masuzaki et al. have mentioned that the closed divertors made by graphite were installed in 9 sections to enhance the plasma performance [9]. The typical wall temperature during the experimental campaign was less than 373 K.

2.2. Additional D_2^+ implantation and D retention evaluation in LHD plasma exposed W

After the LHD plasma discharge campaign, the LHD vacuum vessel was purged by air and all the samples were picked up. The samples were transferred to Shizuoka University and the 1.0 keV D_2^+ were additionally implanted into these samples with the flux of 1.0×10^{18} $D^+ m^{-2} s^{-1}$ up to the fluence of 5.0×10^{21} $D^+ m^{-2}$ by ion gun to evaluate the D trapping enhancement by LHD plasma exposure. The H and D retentions were estimated by TDS using two quadrupole mass spectrometers including high resolution mass spectrometer to distinguish D_2 and He. For He desorption observation, high temperature TDS system up the temperature of 1773 K at NIFS was used as mentioned in our previous paper [10]. TEM observation was also done to evaluate the microstructure change by LHD plasma exposure using the W samples with size of ϕ 3 mm × t ~ 0.1 mm at Institute of Applied Mechanics, Kyushu University. The chemical states of constituent atoms and their depth profiles were evaluated by X-ray Photoelectron Spectroscopy (XPS) with combination usage of Ar^+ sputtering technique at Shizuoka University.

3. Results and discussion

Fig. 1 shows the XPS spectra of C-1s, O-1s and W-4f for top surface of 2014 LHD plasma exposed W. Positive peak shift to C—O bond of C-1s for all the samples was found except for ER, where no remarkable peak was observed. This result may reflect the growth of O-1s peaks for all the samples, indicating that the existence of oxygen on the samples contribute on C-1s peak shift toward higher binding energy side. For W 4f XPS spectra, no existence of W was found except for ER sample, which was consistent with our previous campaign results [5–8]. The chemical state of W for ER sample consisted of two major chemical states, namely metal W and W-O bond states. Fig. 2 summarizes the depth profiles of consistent atoms for all the samples using XPS with Ar^+ sputtering technique. Note that the depth scale was changed depending on the sample due to the difference of thickness of carbon-dominate mixed-material layer. The depth profiles for PI and HL showed almost the same trend and the deposition layer was formed on the surface within the depth of 500 nm. For ER sample, almost no or very thin deposition layer was found. The existence of oxygen was limited near surface region, indicating that oxidation of W was formed as shown in O-1s XPS spectra in Fig. 1. In case of DP, thick deposition layer was covered on the sample. Even if long time Ar^+ sputtering was performed, it was quite difficult to reach the bulk. The surface morphology was observed as shown in Fig. 3. The trend of their surface morphology was almost consistent with the results derived in previous experimental campaign [4–7]. Most of the surface except for ER was deposition-dominated. In DP sample, granule and imbricate deposition layer was formed, which was a little different from that installed in last plasma campaign [4]. This would be caused by the derivation of high temperature plasma, leading to the formation of more thick deposition layer in this plasma discharge campaign. For PI and HL samples, the surface was quite smooth, but some precipitates were adsorbed, which were almost the same situation with the previous campaign. The black dots were distributed throughout the sample surface for ER, which would be the erosion trace and the morphology was quite different from the others. The detail microstructures for DP, ER and PI were observed by TEM in Fig. 4. The clear amorphous carbon mixed material layers were formed for both PI and DP samples. In LHD, the first wall and closed divertor were made by stainless steel and carbon, which led to the formation of carbon mixed material layers, not simple carbon deposition layer. In ER sample, both of He bubbles and H blisters were clearly observed, which was quite different microstructure from the PI and ER samples. As mentioned above, both of H and He discharges were performed throughout the experimental campaign, which would introduce them.

Fig. 5 shows He TDS spectra for ER and PI samples derived by HT-TDS device at NIFS [10]. He desorption for PI sample consisted of three major desorption stages located at 600 K, 900 K and 1200 K, respectively. No He desorption at temperature above 1300 K was found. However, for ER samples, continuous He desorption between 450 K and 650 K was found and it was extended above 1773 K. These facts indicate that the most of He for PI was retained in carbon dominated mixed material layers, although that for ER would be trapped by W and formation of He bubbles led to various trapping sites in W. These He retention was caused by 1545 shots of He discharge during LHD plasma campaign, which is consistent

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