

## Microscopic and macroscopic damage in metals exposed to LHD divertor plasmas

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### Abstract

In order to study erosion/damage processes of the tungsten divertor target, material probe experiments were carried out by exposing tungsten specimens to short pulse helium or hydrogen divertor plasma in LHD (10–20 eV, about  $1 \times 10^{22}$  ions/m<sup>2</sup>s). A narrow ‘footprint’, a trace of local-melting, of divertor plasma was formed on the material probe head along the divertor-leg for both hydrogen and helium discharges. In the specimens located rather far from the footprint, where less loading of particles is expected, remarkable blistering was confirmed for both helium and hydrogen irradiation cases. The erosion of the surface was estimated to be 6.3 nm/1 s (one discharge), and 4.1 nm/24 s (19 discharges) for helium and hydrogen, respectively. One should note that erosion due to blistering occurs even for the very low energy hydrogen plasma exposure. It is considered that the abrupt change of specimen temperature due to very high flux particle load may enhance the blistering.

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### 1. Introduction

The large helical device (LHD) is the largest helion-type plasma machine with superconducting magnetic coils. It has the advantage of current-less and disruption-free steady state plasma operation, and an

intrinsic divertor configuration [1]. These features are also the great advantage for material exposure experiments.

The divertor armor tiles in large-sized plasma confinement devices are exposed to heat and plasma particles with very high flux. Studies on the modification of the divertor target's surface are indispensable for elucidation and control of impurity generation, transport and hydrogen isotope behavior and inventories in the vacuum vessel.

High-Z materials such as tungsten are potential candidates for the divertor armor tiles due to their excellent

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thermal properties, low solubility for hydrogen and low sputtering yield. In fact, tungsten has been selected as a plasma facing material in the divertor region of ITER [2]. There are long lasting attempts of plasma irradiation, e.g., in ASDEX Upgrade [3]. However, only few attempts have been made to study tungsten under plasma irradiation in other large-sized plasma confinement devices. In the present work, therefore, we have performed material exposure experiments with helium divertor plasmas and also with hydrogen divertor plasmas in the LHD, using the retractable material probe system attached to the LHD. Details of the microscopic and macroscopic irradiation effects were examined by using several kinds of analytical techniques complementarily.

## 2. Experimental procedures

To examine the surface modification caused by the divertor plasma of helium or hydrogen, plasma exposure experiments were carried out in the LHD. Pre-thinned vacuum-annealed tungsten disks of 3 mm diameter and tungsten plates of 0.1 mm thickness were mounted on the probe head of the retractable material probe system and placed at the divertor-leg position of LHD through the 4.5 low port (4.5L) as shown in Fig. 1. Labels (1), (2) and (3) in the figure are the connection length ( $L_c$ ) profile along the Z-axis, the schematic view of the experimental set up in the 4.5L-port and the probe head for these experiments, respectively.  $L_c$  was calculated by the field line tracing method. A peak section of  $L_c$  ( $Z = -1.32$  m) corresponds to the magnetic lines of the divertor-leg. The material probe head made of SUS316L

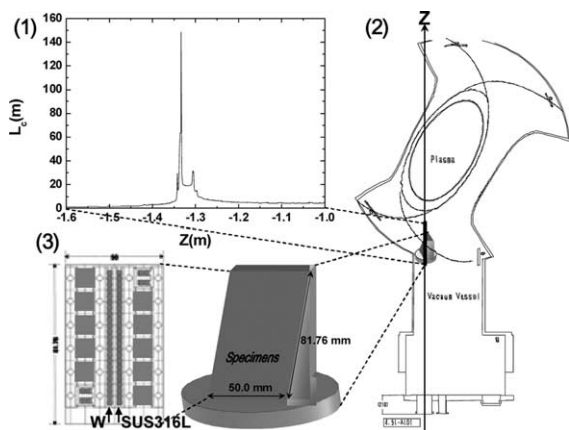


Fig. 1. Configuration of the retractable material probe system: (1) the connection length ( $L_c$ ) of magnetic lines profile as a function of location on Z-axis; (2) schematic view of the experimental set up in the 4.5L-port; and (3) the probe head for the divertor-leg.

Table 1

Typical plasma parameters of helium and hydrogen plasma discharges

	Total discharge time	Electron density ( $n_e$ )	Ion temperature ( $T_i$ )
Helium plasma	1 s (one discharge)	$3.1 \times 10^{19} \text{ m}^{-3}$	1.5 keV
Hydrogen plasma	26 s (19 discharges)	$2.3 \times 10^{19}$ – $4.0 \times 10^{19} \text{ m}^{-3}$	2.1–3.4 keV

was designed such that the specimens and the magnetic field lines of the divertor-leg cross perpendicularly. Though the temperature of the probe head was measured by thermocouples at the positions just beneath the specimens, the temperature of the specimens was not measured directly.

In the hydrogen plasma experiment, the specimens were exposed to successive 19 discharges (26 s in total), while only to one helium plasma discharge (1 s), because stronger irradiation effects were expected for the latter. Typical core plasma parameters of the helium and hydrogen plasma discharges are listed in Table 1.  $T_e$  and  $T_i$  in the divertor region and the ion flux to the specimens were expected to be  $T_e \sim T_i = 10$ – $20$  eV and  $1 \times 10^{22}$  H(He)/ $\text{m}^2 \text{ s}$ , respectively, for both the hydrogen and helium plasma. The incident ion energy distribution to the specimens must be shifted-Maxwellian (sheath-potential +  $T_i$ ). Therefore it is expected that the effective incident energy of hydrogen and helium ions to the specimens is the order of 100–200 eV.

After the exposure, surface morphology, microscopic damage inside the materials and chemical components were examined by means of scanning electron microscopy (SEM), atomic force microscopy (AFM), transmission electron microscopy (TEM) and energy dispersive spectroscopy (EDS), respectively.

## 3. Results and discussion

### 3.1. Footprints on the probe head

In the LHD, many lines of ‘footprints’, traces of surface modification such as local melting, were observed on the divertor tiles used in the third campaign [1]. ‘Footprints’ refers to the marks caused by irradiation which remain on the surface along with the strike points of the divertor plasma. The ‘footprints’, were also observed in the present probe experiments for both hydrogen and helium discharges as a trace of melting on the probe head. It was also confirmed that evaporated elements from the probe head (Fe, Cr and Ni) re-deposited on the surface of the specimens very close to the footprint. In spite of the melting, the temperature measured

by the thermocouple inserted just beneath the footprint did not change much. This result can be understood because the heat load is highly localized along the divertor-leg and its duration time is very short.

### 3.2. Damage by the helium divertor plasma

Changes of surface morphology and internal damage of tungsten specimens exposed to a single divertor plasma discharge for 1 s are summarized in Fig. 2. The broad line drawn from the upper right to the lower left (noted FP) in the schematic view of the specimen position indicates the footprint. The position of each micrograph is indicated by an arrow: the distance from the footprint is also noted for positions (a)–(e).

Morphology of the damage depends on the distance from the footprint, namely it depends on the fluxes of helium ions and heat. In the case of specimens (a), (b) and (e) (see Fig. 2), located rather far from the footprint (about 7–18 mm), dense small dimples (exfoliation of blisters) were formed on the incident surface. A high resolution micrograph of AFM in Fig. 3(a) shows that about 20% of the surface is covered with dimples of about 150–270 nm in diameter and 9–25 nm in depth. Bulges of similar size are also observed. These results indicate that the fine blisters were formed by pulse exposure to the helium plasma. The covers of the blisters remain for the bulges but are exfoliated for the dimples. The surface erosion estimated from the exfoliated blisters is about 6.3 nm per discharge. The diameter and the depth of a dimple can be estimated by AFM observation.

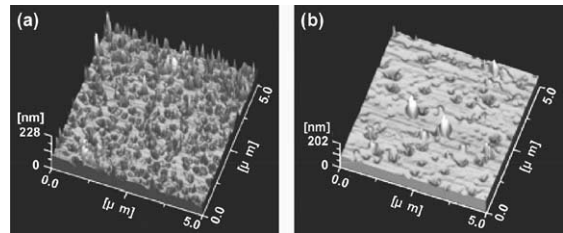


Fig. 3. Surface morphology of tungsten as observed by AFM: (a) after exposure to a helium divertor plasma, and (b) after exposure to a hydrogen divertor plasma.

TEM micrographs of positions (a), (b) and (e) in Fig. 2 show internal damage of the pre-thinned tungsten specimens. Not only dislocation loops but also bubbles are formed very densely. One should note that many of the bubbles are larger than 10 nm and some of them have an oddly shaped image like an ellipse or even a gourd. This indicates that the bubbles have grown by coalescence with the abrupt temperature increase and the non-equilibrium shape is ‘frozen’ by rapid cool-down after the termination of the discharge. According to recent results, the coalescence of helium bubbles in tungsten actively occurs above 800 °C by their thermal migration [4].

The formation of helium bubbles in tungsten by helium ion irradiation has been studied extensively. It is remarkable that helium bubbles are formed even if the incident energy of helium is much less than the threshold energy for the displacement damage (about 0.25 keV for tungsten), because the injected helium atoms can

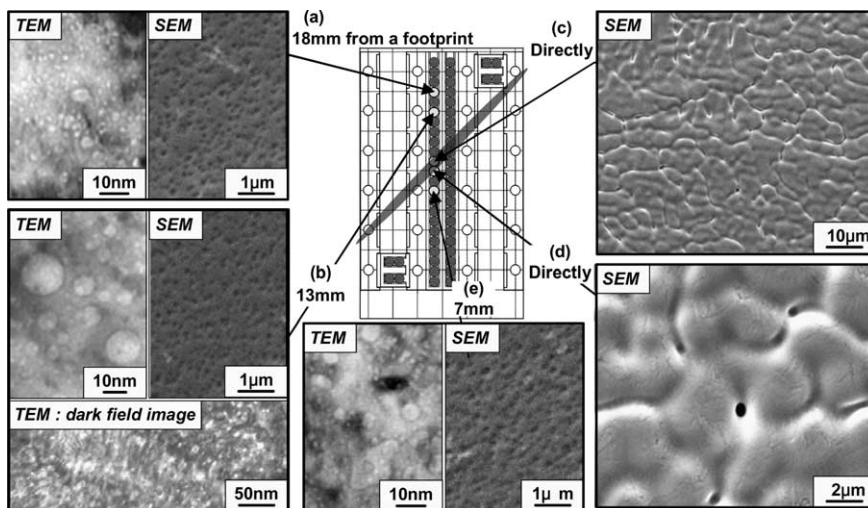


Fig. 2. Changes of surface morphology and internal damage for tungsten specimens exposed to a helium divertor plasma discharge for 1 s. The broad line drawn from the upper right to the lower left in the schematic view of the specimen position indicates the footprint. Position of each micrograph is indicated by an arrow, and the distance from the footprint is noted for each positions under the labels (a)–(e).

aggregate without preexisting vacancies [5]. It is suggested that the present case (order of 100–200 eV, about  $1 \times 10^{22} \text{He}^+/\text{m}^2$ ) belongs to this category. Moreover the bubbles are formed in a very wide temperature range, at least from room temperature up to 2500 °C [5,6]. It is known that blistering in tungsten occurs at rather low temperatures, less than about 700 °C for steady state irradiation at fixed temperature. Though the histories of the specimen temperature and helium flux during the discharge are not well known, features of the helium irradiation effects mentioned above indicate that the blistering occurs during temperature increase, when the helium gas pressure in the bubbles exceed the critical value. The increase of the inner gas pressure and thermal expansion which locally occurs at the surface due to the abrupt surface temperature increase may enhance blistering.

The surfaces of the specimens closer to the footprint (positions (c) or (d) in Fig. 2), where higher fluxes of heat and helium are expected, show different types of modification, namely, no blisters but a wavy surface consisting of grooves and hills. The size of the hills is a few  $\mu\text{m}$ . Small deep holes of about 500 nm in diameter are also formed at the grooves. Though it is difficult to discuss the formation mechanism of these structures, recent results of helium irradiation at 1000 °C [4] strongly suggest that large helium bubbles beneath the incident surface form the hills as ‘balloons’ of helium gas. Judging from the size of the hills, the maximum temperature for the present case must be much higher than 1000 °C. Most of the deep holes are formed at the nodes of the grooves. We can understand this phenomenon by assuming them as pin-holes connecting the bubbles

and the surface. Once the pin-hole is formed, the ‘balloon’ is shrunk and the area of the balloon will sink in and become the node of grooves.

### 3.3. Damage by hydrogen divertor plasma

The changes in surface morphology and internal structure of tungsten after exposure to a hydrogen divertor plasma for 26 s (19 discharges) are summarized in Fig. 4. The tungsten specimen located at the footprint position (c), shows a unique morphology; in addition to the roughening of the surface; the surface along the grain boundaries rose as a long bank. Details of the surface morphology of the bank indicate that melting and solidification were repeated locally near the grain boundaries. Cracking along the banks was also observed. It is known that melting starts at areas with lattice imperfections such as grain boundaries and highly deformed areas. It can be expected that once melting occurred at the surface area along the grain boundaries, the area rose in order to compensate for the local thermal expansion at the surface, and the shape was frozen by the rapid cooling down of the surface after the termination of the discharge. Large residual stresses at the banks may induce the clacking along the banks. Formation of the banks indicates that the surface temperature was close to the melting point during the pulse discharge.

In the case of specimens (a), (b) and (d) (see Fig. 4), located rather far from the footprint (9–14 mm), unexpected damage was observed; formation of fine bubbles and blisters was observed just as in the helium irradiation case described in 3.2. According to the hydrogen ion irradiation experiments at fixed temperature, no

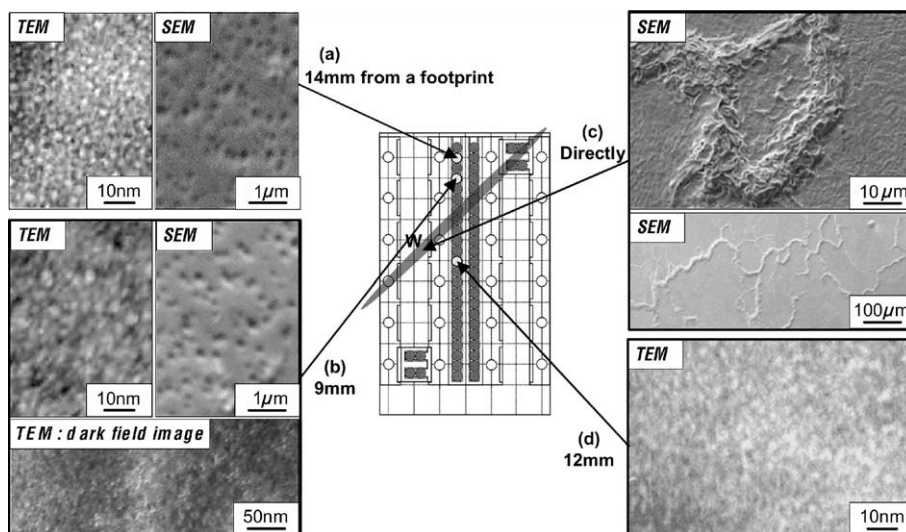


Fig. 4. Changes of surface morphology and internal structure after exposure to hydrogen divertor plasmas for 26 s (19 discharges). (Details of this figure are the same as in Fig. 2.)

defect clusters are formed if the incident energy of the ions is less than 2 keV, because the energy is insufficient for displacement damage [7]. The present experiments belong to this category. Even if the energy is higher than the threshold value, formation of dense cavities has not been observed for the irradiations up to the order of  $10^{22}$  ions/m<sup>2</sup> [8]. It is believed that the formation of blisters due to pressurized fine hydrogen bubbles at the projected range region is very difficult for hydrogen irradiation because the trapping energy of a hydrogen atom in vacancies and cavities is much lower than that of helium [9,10]. For understanding the present unexpected phenomena, more fundamental information is needed about the behavior of hydrogen under conditions of very high fluxes of hydrogen intense heat pulses.

According to the observation with the AFM in Fig. 3(b), the diameter and depth of dimples formed by the exfoliation of the blisters were estimated to be 35–475 nm and 6–34 nm, respectively. Taking into account the area density, the surface erosion was estimated to be about 4.1 nm. One should note that the expected erosion of graphite by sputtering will be about 20 nm under exposure to similar discharges (100–200 eV,  $2.6 \times 10^{23}$  H<sup>+</sup>/m<sup>2</sup>). The present results indicate that serious consideration should be given to the surface erosion of tungsten divertors due to the blistering even for hydrogen plasma discharges.

#### 4. Summary

The material probe experiments were carried out by exposing tungsten specimens to helium and hydrogen divertor plasmas in the LHD. ‘Footprints’ were observed in both hydrogen and helium discharges as traces of

melting. At the surfaces of specimens close to the footprints, complicated surface morphologies were observed; wavy surface consisting of grooves and hills (in helium discharge) and melting of grain boundaries (in hydrogen discharges) were observed. Furthermore, in the case of specimens located rather far from the footprints, not only bubbles and cavities, but also small dimples were formed by blistering. The erosion of the surface was estimated to be 6.3 nm per one discharge (1 s) in helium, and a total of 4.1 nm for 19 discharges (24 s) in hydrogen. One should note that erosion due to the blistering occurs even for very low energy hydrogen plasma exposures, which has not been expected from previous investigations. It is considered that the abrupt change of specimen temperature due to a very high particle flux may enhance the occurrence of blistering.

The present results indicate that synergistic effects due to high heat and helium/hydrogen particle loads leading to damage and erosion of metal divertor targets must be seriously considered for the design of divertors in burning plasma experiments.

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