High-Z Dust Generation on Tungsten Surfaces due to Synergetic Erosion of Deuterium/Helium Plasma Exposures

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Low-energy and high-flux plasma exposures on tungsten surfaces result in blister/hole formation. Subsequent He plasma exposure at 2,000 K on the blister-rich surface forms a hole structure on the surface, including the blister surface, and some blisters collapse due to recrystallization and bubble formation on the blister skin. Grain ejections were observed after deuterium plasma exposure at 550 K on a W surface on which a hole structure had formed due to He pre-exposure at 1,600 K.

Keywords:
plasma facing material, tungsten, blister, hole, dust, high-Z impurity, Nagoya Divertor Simulator (NAGDIS)

1. Introduction
The generation of erosion and impurities on wall material due to plasma exposure is an important problem in a fusion reactor such as the International Thermonuclear Experimental Reactor (ITER). In ITER, tungsten (W) is one of the most important candidates for the armor material of the divertor and first wall because of its good material properties such as low sputtering yield, low tritium inventory and high melting point. However, contamination in the form of W impurities into core plasma significantly increases radiation loss because W is a high-Z material. W impurities could degrade the plasma performance when the relative density of W atoms to the fuel particles reaches the order of $10^{-3}$ [1]. A serious concern is the case of a long-pulse burning plasma if W impurities get mixed in the core plasma in the form of micron-sized dust particles.

Hydrogen isotopes (fuel) and helium (ash) are the primary plasma species in a fusion reactor. Wall temperatures are assumed to fluctuate during some operation cycles because heat flux on the wall is not constant in the startup/shutdown phases, L-mode, ELMy H-mode, disruptions etc. This means that wall materials in fusion reactors are exposed to mixture plasmas of hydrogen (isotope) and helium at various temperatures. The W surface is damaged due to plasma exposure even when the incident ion energy is lower than the threshold energy of ion-induced defects [2-4]. The surface damage depends on the plasma species and on the surface temperature. Hydrogen plasma exposure results in blister formation on a W surface at temperatures of less than 900 ~ 1,000 K [2,4], while a hole structure is formed in the case of helium (He) plasma exposure at temperatures of more than ~1,500 K [3]. These surface modifications are also observed in deuterium/He mixture plasma exposure [5]. The present paper examines the possibility that the generation of micron-sized W particles is promoted on the W surface with blister or hole structure.

2. Experimental Setup
Two powder metallurgy W (PM-W) samples provided by Niraco Co. (Tokyo, Japan) were used in the present experiments. The thickness and purity of the PM-W samples were 0.2 mm and 99.95%, respectively. Figure 1 shows the surface

Fig. 1 The surface of a representative PM-W sample as received from the supplier.
of the PM-W sample as it was received. Deuterium and He plasma exposures were performed on the two samples in the linear divertor plasma simulator Nagoya Divertor Simulator NAGDIS-I [6] and NAGDIS-II [7]. In NAGDIS-I, where the deuterium plasma exposures were performed, the samples were fixed on a water-cooled sample stage with a tantalum clip, and the temperature of the samples was measured using a chromel-alumel thermocouple in contact with the back surface of the sample. In the case of He plasma exposure, the samples were mounted on the head of a W wire by the spot welding method and the exposures were performed in NAGDIS-II, where the surface temperature of the samples increased to more than 1,500 K due to the incident plasma heat flux. The surface temperatures were measured by a radiation pyrometer through the quartz window. The experimental procedures have been described in detail elsewhere [4,8].

Plasma exposure was carried out twice on each sample. Deuterium plasma exposure was carried out on sample W1 at 500 K as the first exposure, which results in blister formation on the surface. As the second exposure, W1 was subsequently exposed to He plasma at 2,000 K, in which a hole structure can form on the W surface. Sample W2, on the other hand, was exposed to He plasma at 1,600 K as the first exposure, followed by deuterium plasma exposure at 550 K as the second exposure. The surface conditions of each sample were investigated by scanning electron microscopy (SEM) after each plasma exposure. All plasma exposures were carried out with an incident ion energy of less than 100 eV, which means that neither ion-induced displacement nor sputtering on the W surface occurred. The details of the present experimental conditions are described in Table I.

### Table I. Experimental conditions.

<table>
<thead>
<tr>
<th>Sample name</th>
<th>W1</th>
<th>W2</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>First exposure</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Plasma</td>
<td>Deuterium</td>
<td>Helium</td>
</tr>
<tr>
<td>Surface temp. [K]</td>
<td>500</td>
<td>1600</td>
</tr>
<tr>
<td>Fluence ([\text{m}^{-2}])</td>
<td>(2.7 \times 10^{25})</td>
<td>(9.0 \times 10^{26})</td>
</tr>
<tr>
<td>Ion flux ([\text{m}^{-2}\text{s}^{-1}])</td>
<td>(2.5 \times 10^{17})</td>
<td>(4.8 \times 10^{22})</td>
</tr>
<tr>
<td>Exposure time [s]</td>
<td>10800</td>
<td>1800</td>
</tr>
<tr>
<td>Ion energy [eV]</td>
<td>80</td>
<td>25</td>
</tr>
</tbody>
</table>

| **Second exposure** | | |
| Plasma | Helium | Deuterium |
| Surface temp. [K] | 2000 | 550 |
| Fluence \([\text{m}^{-2}]\) | \(2.5 \times 10^{25}\) | \(2.9 \times 10^{25}\) |
| Ion flux \([\text{m}^{-2}\text{s}^{-1}]\) | \(7.0 \times 10^{12}\) | \(4.0 \times 10^{21}\) |
| Exposure time [s] | 3800 | 7200 |
| Ion energy [eV] | 20 | 80 |

### 3. Experimental Results and Discussion
#### 3.1 Blister formation and blister exfoliation due to deuterium plasma exposure

Many blisters of varying sizes were observed on sample W1 after deuterium plasma exposure, as shown in Fig. 2(a). Blister exfoliations as shown in Fig. 2(b) were observed on the whole surface; the size of the exfoliated blisters was approximately 10 \(\mu\text{m}\) or less. This result indicates that blister exfoliation can occur in the case of low-energy plasma exposure as well as in high-energy ion irradiation experiments [9-11]. No exfoliation is observed in the case of larger blisters, and it remains unknown why only blisters of a particular size are exfoliated.

#### 3.2 He plasma exposure on the blister-surface at high temperatures

He plasma exposure was carried out at 2,000 K on sample W1 on which blisters had formed. Figure 3(a) and (b) show the same point of W1 after the first exposure (deuterium) and after the second exposure (He), respectively. The blisters remain with a dome-like form even after the second exposure at a high temperature. However, some other blisters have collapsed, as shown in Figs. 4(a) and (b), and a hole structure is observed on the surface (Fig. 4(b)). Figures 5(a) and (b) show a cross-section of a blister and a part of blister skin, respectively. The material recrystallized due to He exposure at the high temperature of 2,000 K. Recrystallized grains are observed beneath the blister dome and no holes are seen on the grains, as shown in Fig. 5(a). The blister skin has also recrystallized and submicron-sized He bubbles have formed on the skin. We believe that the recrystallization and He bubble formation on the skin lead to blister collapse and generate dust particles.

#### 3.3 Grain ejection on the W surface with a hole structure due to deuterium post-exposure

Figures 6(a) and (b) show the surface and a cross-section of sample W2 after He plasma exposure at 1,600 K for 30 minutes. Holes and bubbles with a diameter of a few hundred nm are observed on the surface, and grains with a size of more than 10 \(\mu\text{m}\) are seen on the cross-section. Sample W2 was then subjected to deuterium plasma exposure at 550 K for 2 hours. No particular surface modification was observed after this exposure except grain ejections. Figures 7(a) – (d) show the ejected grain particles on the W2 sample surface. We believe that an increase of pressure at the grain boundaries due to deuterium gas accumulation ejected the surface-layer grains. Hole structure due to He pre-exposure may not be an essential factor for grain ejection because a similar grain ejection was also observed on a recrystallized molybdenum surface which was irradiated by 25 keV hydrogen ions [12]. However, He atoms could accumulate in a grain boundary and this He accumulation could decrease the cohesive strength of the boundary, which could make grain ejection easier.

### 4. Conclusion

High-Z dust particles can be ejected from a W surface...
Fig. 2  Surface modification of sample W1 after deuterium plasma exposure at 500 K for 3 hours. (a) Many blisters with sizes up to ~200 μm are observed. (b) Some blisters of less than ~10 μm in size are exfoliated. No blister exfoliation was observed for large blisters, that is, those larger than ~10 μm.

Fig. 3  Comparison of blister modification on sample W1 (a) before and (b) after He plasma exposure at 2,000 K. The blisters retain their dome-like structure even after He plasma exposure at high temperatures.

Fig. 4  Surface modification of the blister-rich surface of W1 due to He plasma exposure at 2,000 K for 1 hour. (a) Some blisters have collapsed. (b) A clearly collapsed blister and porous structure with a diameter of 200–300 nm, which is usually formed due to He plasma exposure at high temperatures, are observed on the surface.

Fig. 5  Cross-section of a blister on sample W1 after He plasma exposure. (a) The substrate has recrystallized. (b) Enlarged image of the blister skin. The blister skin has also recrystallized and submicron-sized He bubbles are observed in the skin. These He bubbles become holes when they appear at the surface.
Fig. 6  Surface and cross-sectional views of sample W2 after He exposure at 1,600 K for 30 min. (a) Hole structure is observed on the surface. (b) The substrate has recrystallized and submicron-sized He bubbles have formed on each grain within the depth of ~1 μm.

Fig. 7  Grain ejection on the W2 surface after deuterium plasma exposure at 550 K subsequent to He plasma pre-exposure at 1,600 K. It is predicted that gas accumulation increases gas pressure at the grain boundaries, which then ejects the gains on the top.
due to low-energy plasma (deuterium and He) exposures. Deuterium plasma exposure at 500 K on a W surface was found to lead to blister formation; some of the blisters were then exfoliated. Subsequent He plasma exposure at 2,000 K on the blister-rich surface forms a hole structure on the surface, including the blister surface, and some blisters collapse due to recrystallization and bubble formation on the blister skin. Grain ejections were observed after deuterium plasma exposure at 550 K on a W surface on which a hole structure formed due to He pre-exposure at 1,600 K. In a fusion reactor, the presence of high-Z dust could be serious as well as that of carbon dust. Quantitative estimation of dust production is an important future work.

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References