

# Impacts of Carbon Impurity in Plasmas on Tungsten First Wall

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**Abstract.** In order to study the effect of carbon impurity in plasma on tungsten first wall, hydrogen-carbon mixed ion beam was irradiated to tungsten. It was found that very small amount of carbon (~0.3% or more) significantly enhanced blister formation. According to XPS analysis, all of tungsten atoms in a top surface were combined with carbon atoms to form tungsten carbide (WC) layer (~6 nm thick) in the cases that significant blisters were observed. This layer could enhance diffusion of hydrogen atoms to the bulk, leading to blister formation. In addition, carbon impurity increased hydrogen retention, but the increment of the retention tended to decrease with fluence.

## 1. Introduction

In terms of fusion technologies relevant to fusion power reactors, selection of plasma facing materials is one of the key issues for the blanket and divertor design. Tungsten is quite the strongest candidate for an armor material of blankets and divertors because of low sputtering yield and a high melting point. However, we do not have absolute confidence in feasibility of tungsten in steady-state fusion reactors, because material degradation and erosion behavior under steady-state burning plasma conditions have not been understood.

For plasma material interaction (PMI) in actual fusion devices, studies on material mixing effects become increasingly important. In ITER, plasma facing materials of beryllium, carbon, and tungsten are planned to be used. Therefore, mixing of these materials will take place and significantly complicate the interaction. For example, very small amount of carbon ion in plasmas enhances sputtering erosion of tungsten, while in the case that carbon concentration exceeds some critical value (a few % depending on impinging energy), carbon deposition takes place and protects the surface from sputtering erosion. Material mixing could also affect surface morphology change (blistering, flaking, and so on), which would be related to erosion enhancement and production of dust particles. In addition, impurity effects on hydrogen retention property could not be negligible. So far in terms of material mixing effects, comprehensive database and models for tungsten are far from sufficient.

In this study, material mixing effects of carbon and tungsten on PMI have been studied with a steady-state and high-flux ion beam irradiation test device (HiFIT)[1]. We investigated the effects of carbon impurities on blister formation and hydrogen retention of tungsten by hydrogen and carbon mixed-ion beam irradiation.

## 2. Experimental

A cross sectional view of HiFIT is shown in Fig.1. This device can produce high-flux beams by using a high accel-decel ion extraction system and geometrical focusing. Hydrogen irradiation fluxes were  $3.6 \times 10^{21}$  H/m<sup>2</sup>s for an acceleration voltage  $V_{acc}$  of 3 kV and about  $4 \times 10^{20}$  H/m<sup>2</sup>s for  $V_{acc}$  from 0.15 kV to 0.5 kV. Dominant ion species is H<sub>3</sub><sup>+</sup> in these beams. These fluxes are much higher than those of conventional ion beam devices. According to the edge plasma study for ITER[2], particle flux (fast neutrals and plasma ions) to plasma facing surfaces of blankets are up to  $\sim 10^{21}$  m<sup>-2</sup> s<sup>-1</sup> with energies up to a few hundred eV. Ion flux and energy of HiFIT coincide well with these conditions.

Carbon impurity concentration in the beams can be controlled from 0.06 % to roughly 10 % by putting carbon plates in the ion source chamber and/or puffing methane gas. Oxygen impurity of about 0.05 % was always present. The other impurity concentration is less than a detection limit (about 0.01 %). Carbon species in the beam appeared as hydrocarbon molecular ions such as  $\text{CH}_x^+$  and  $\text{C}_2\text{H}_x^+$ . Carbon impurity concentration in ion beams was evaluated by using measured effective cross-sections of hydrocarbon ions in a beam transport region (including dissociation and neutralization through collision with ambient gas molecules).

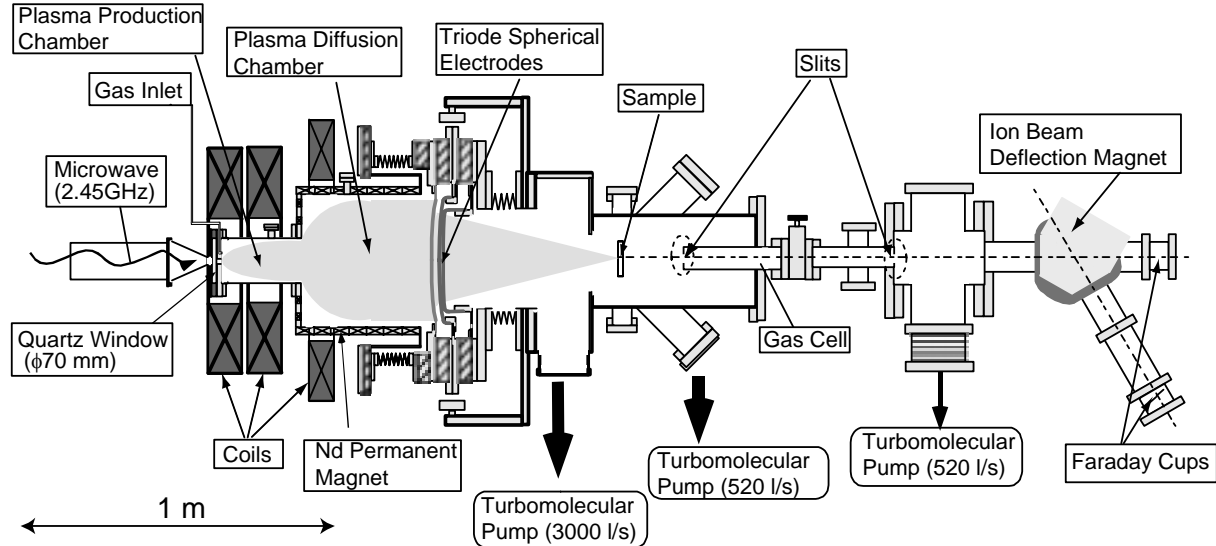


Fig.1 Cross sectional view of high-flux ion irradiation device (HiFIT)

### 3. Effects of carbon impurity on blister formation

The most important result in this paper is the fact that very few carbon impurities significantly affect blister formation, which was not reported before by other research groups. Figure 2 shows SEM photograph of irradiated surface of tungsten. In these experiments, sintered tungsten plates (99.95 % purity) with mirror polishing surfaces were used (Nilaco. Co.). Irradiation was made with 1 keV  $\text{H}_3^+$  beam (mainly 333 eV H) at 653 K. It was clearly observed that quite a lot of blisters with its size up to about 1 mm were formed for the carbon concentration of 0.35% case (b) and 0.95% case (c). In these cases, many small blisters lay on large blisters. The number of blisters was larger for the case of 0.95% (c) than the case of 0.35% (b). On the other hand, no significant blisters were formed for the low impurity case (0.11% (a)). As carbon concentration was raised to 2.35%, carbon accumulated on tungsten samples and no blisters were found.

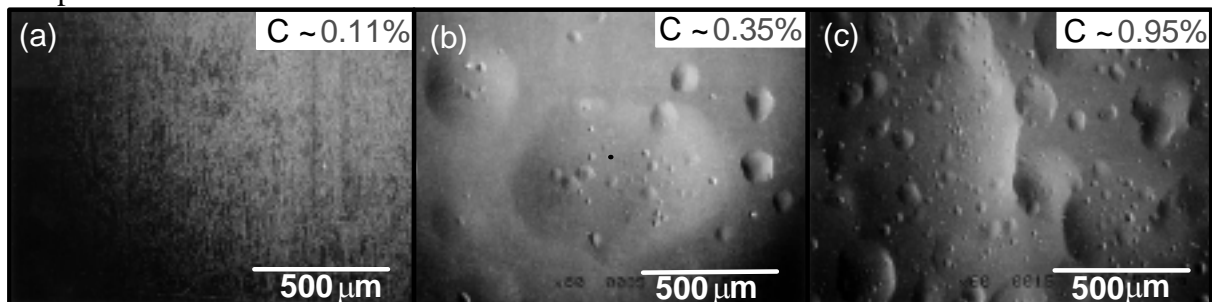


Fig. 2 Surface morphology of irradiated tungsten at 653K with carbon concentration as a parameter. Irradiation beam energy and fluence were 1 keV  $\text{H}_3^+$  and  $(6-8) \times 10^{24} \text{H/m}^2$ .

Energy dependence was shown in Fig. 3. When the energy was reduced to 300 eV  $H_3^+$  (mainly 100eV H), similar large blisters were still observed for the carbon concentration of 0.7%, though the number of blisters decreased. There was no significant difference between the cases of 1 keV  $H_3^+$  and 3 keV  $H_3^+$ .

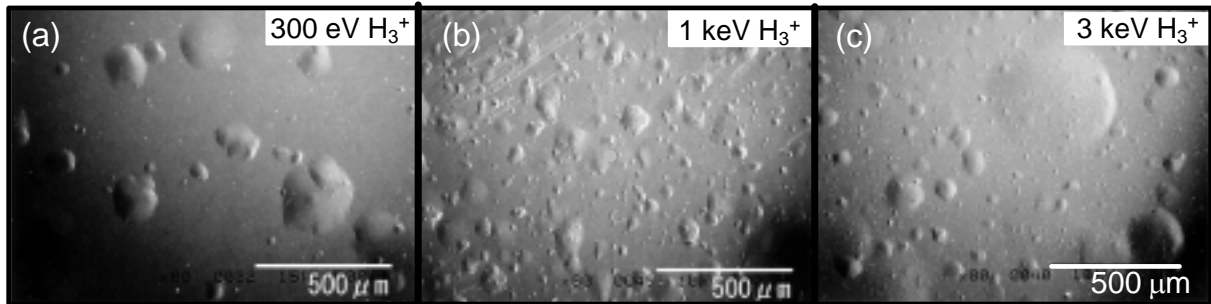


Fig. 3 Surface morphology of irradiated tungsten at 653K with beam energy as a parameter. Carbon concentration and irradiation fluence were 0.7-0.8% and  $3 \times 10^{24} H/m^2$ .

Temperature dependence was also studied. At the temperature of 653 K, large number of blisters (about 400 blisters/ $mm^2$ ) was formed in the case of 1keV  $H_3^+$  and 0.8% C. As the temperature was changed higher or lower than 653 K, the size of blisters was reduced and the number of blisters decreased. At 388 K and 873 K, no significant blisters appeared.

Irradiation fluence dependence of blister formation was shown in Fig. 4. In the case of  $3.1 \times 10^{23} m^{-2}$  (b), small blisters with the sizes of less than  $10 \mu m$  started to appear. The number of blisters increased to 300-400 blisters/ $mm^2$  at the fluence of an order of  $10^{24} m^{-2}$ . As the fluence increased more, the number of blisters does not change much and blister size became larger (up to about 1 mm), see Fig. 4 (d).

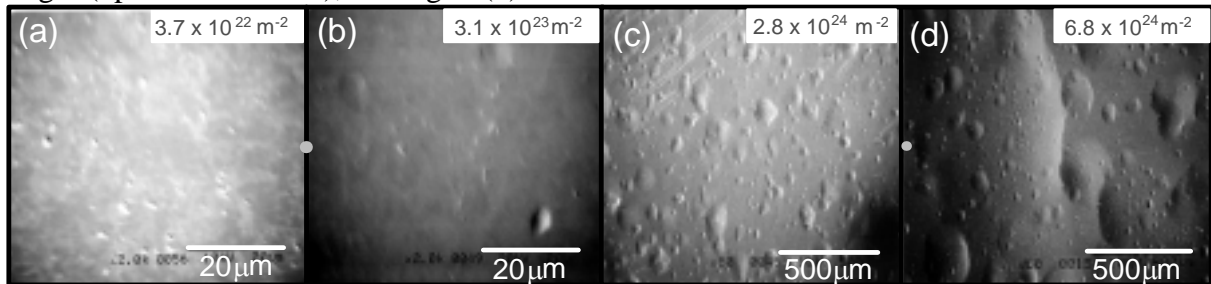


Fig. 4 Surface morphology of irradiated tungsten at 653K with beam fluence as a parameter. Carbon concentration and beam energy were 0.7-0.8% and  $1keVH_3^+$ . Note that magnification was different between [(a), (b) low fluence] and [(c), (d) high fluence].

Enhancement mechanisms of blister formation by carbon impurity could be related to the formation of mixed material layer of carbon and tungsten in a top surface. Impurity concentration in the tungsten samples were measured by XPS, see Fig. 5 (0.85% C) and Fig. 6 (0.11% C). For the high carbon concentration case (Fig. 5), all of tungsten atoms near the surface (up to the depth of  $\sim 6$  nm [etching time of  $\sim 200$  sec]) combined with carbon atoms to form tungsten carbide (WC) as the irradiation fluence exceeded about more than  $3 \times 10^{23} m^{-2}$ . It is noted that WC layer thickness roughly corresponds to carbon ion range ( $R_p \sim 4$  nm). After this surface mixing layer was formed, blisters appeared, see Fig. 4. On the other hand, for the low carbon concentration case (Fig. 6), some of tungsten atoms near the surface did not combine with carbon atoms. In this case, no blisters appeared. Since solubility of hydrogen in WC is low, this WC top surface layer can control hydrogen diffusion back to the surface. Then significant part of implanted hydrogen atoms can diffuse to the bulk to form voids, which eventually grow up to be blisters.

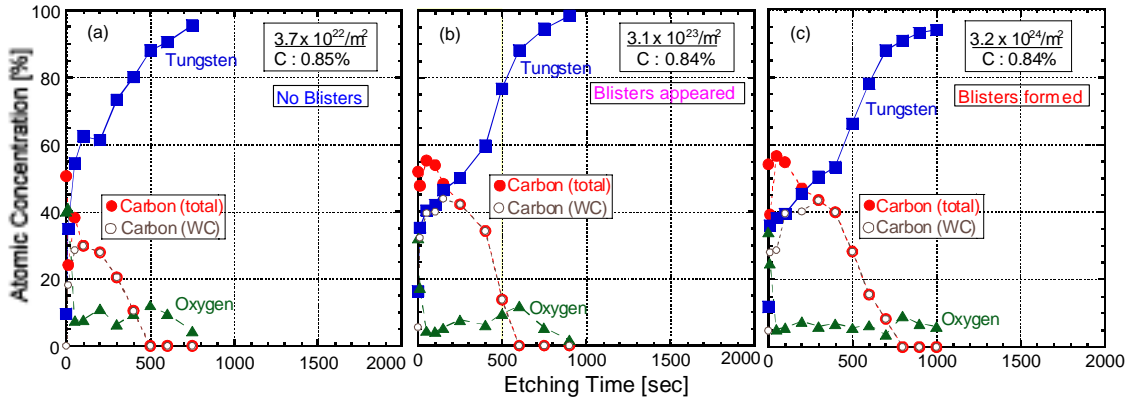


Fig.5 Depth distribution of W, C, and O in tungsten samples with  $1 \text{ keV H}_3^+$  irradiation at 653K for  $\sim 0.84\%$  C case. Etching time of 1,000 sec corresponds to about 30 nm for pure W.

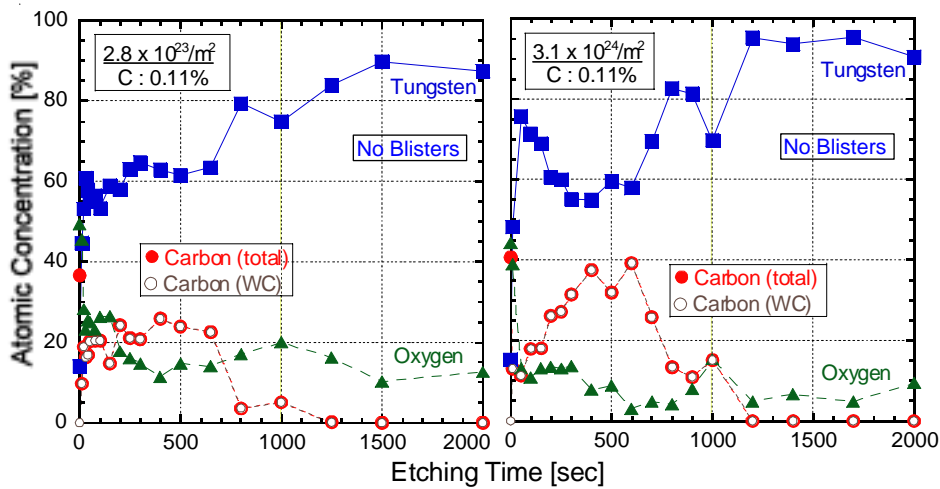


Fig.6 Depth distribution of W, C, and O in tungsten samples with  $1 \text{ keV H}_3^+$  irradiation at 653K for  $\sim 0.11\%$  C case. Etching time of 1,000 sec corresponds to about 30 nm for pure W.

According to the previous D ion beam experiments [3] by other research groups, blisters (size of about  $10 \mu\text{m}$  or more) were formed over the fluence of about  $10^{24} \text{ m}^{-2}$ . But its size was limited to about  $100 \mu\text{m}$  (fluence of up to  $10^{25} \text{ m}^{-2}$ ) and no accumulation of blisters was found. In our experiments, however, much larger blisters and accumulation of blisters were observed. Similar blister characteristic to our results was found in a plasma simulator result (PISCES)[4], which showed blister sizes more than  $100 \mu\text{m}$  and accumulation of blisters. In this experiment, plasmas contained about 0.25 % carbon impurity, which could affect blister formation in consideration of our results.

#### 4 Effects of carbon impurity on hydrogen retention

This small amount of carbon impurity in the beams also affects hydrogen retention. Thermal desorption spectra for tungsten specimens irradiated with  $1 \text{ keV H}_3^+$  beam at 380 K was measured for the carbon concentration of 0.07 % and 0.8 %. Ion fluence for these experiments was between  $10^{24}$  and  $10^{25} \text{ H/m}^2$ . At this temperature, only small number of blisters (about  $10 \mu\text{m}$ ) was formed even in the case of 0.8 % C. For thermal desorption spectra measurements, sample temperature was raised from RT to 1100 K with the ramping rate of 1K/s. When the fluence was low, thermal desorption spectra for 0.07 % C showed an emission peak around 600 K and a gradual decrease from 600 K to 1100 K, see Fig. 7(a) and (b). For the 0.8 % C in

the low fluence case, hydrogen desorption rate was higher than that of 0.07% C in most of the temperature range, especially over 800 K, where new shoulder appeared. This difference in desorption rate, however, decreased as the fluence increased. Eventually, hydrogen retention for different carbon impurity concentration became similar at the fluence of  $7.7 \times 10^{24} \text{ H/m}^2$ , see Fig. 8. The number of carbon atoms in tungsten, which can be traps for hydrogen, increased with fluence in the beginning of irradiation. But for 0.84% C the number of carbon atoms in W started to saturate in the fluence of an order of  $10^{24} \text{ H/m}^2$ . On the other hand, saturation appeared in the higher fluence for 0.07% C case, which could affect this behavior.

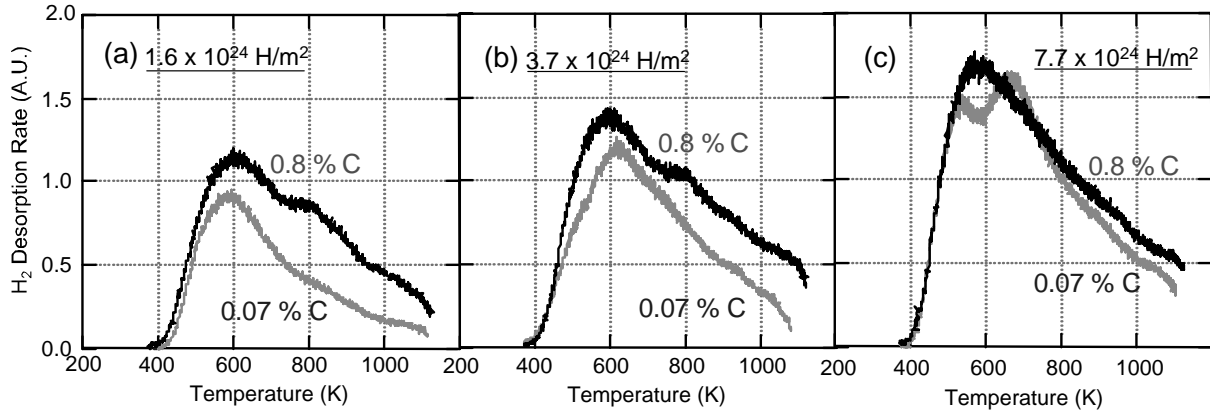


Fig.7 Thermal desorption spectra of H from tungsten samples irradiated by 1 keV  $\text{H}_3^+$  at 380K for 0.07% C and 0.8% C with fluence as a parameter.

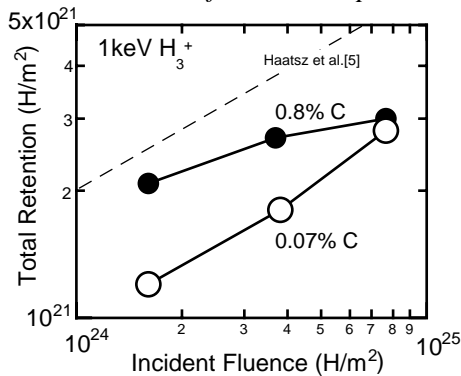


Fig.8 Total retention of hydrogen as a function of incident fluence by 1 keV  $\text{H}_3^+$  irradiation at 380 K. Dashed line shows retention data by 300 eV  $\text{D}^+$  irradiation at 500K by Haasz et al.[5].

## 5. Summary

Carbon impurity in 1 keV  $\text{H}_3^+$  ion beam significantly enhanced blister formation on tungsten as carbon concentration exceeded about 0.3%. The reason could be attributed to the formation of tungsten carbide layer in top surface of tungsten, which enhanced hydrogen diffusion to the bulk and blister formation. In addition, carbon impurity also affected hydrogen retention.

- [1] SHIMADA, T., et al., "Development of new steady-state, low-energy, and high-flux ion beam test device, Rev. Sci. Instrum. **73** (2002) 1741.
- [2] JANESCHITZ, G., ITER JCT and HTs, "Plasma-wall interaction issues in ITER", J. Nucl. Mater. **290-293** (2001) 1.
- [3] HAASZ, A., et al., "The effect of ion damage on deuterium trapping in tungsten", J. Nucl. Mater. **266-269** (1999) 520.
- [4] SZE, F., et al., "Investigation of plasma exposed W1%La<sub>2</sub>O<sub>3</sub> tungsten in a high ion flux, low ion energy, low carbon impurity plasma environment for the International Thermonuclear Experimental Reactor", J. Nucl. Mater. **264** (1999) 89.
- [5] HAASZ, A., et al., "Deuterium retention in tungsten for fusion use", J. Nucl. Mater. **258-263** (1998) 889.