



## Sputtering properties of tungsten ‘fuzzy’ surfaces

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### ABSTRACT

Sputtering yields of He-induced W ‘fuzzy’ surfaces bombarded by Ar have been measured in the linear divertor plasma simulator PISCES-B. It is found that the sputtering yield of a fuzzy surface,  $Y_{\text{fuzzy}}$ , decreases with increasing fuzzy layer thickness,  $L$ , and saturates at  $\sim 10\%$  of that of a smooth surface,  $Y_{\text{smooth}}$ , at  $L > 1 \mu\text{m}$ . The reduction in the sputtering yield is suspected to be due mainly to the porous structure of fuzz, since the ratio,  $Y_{\text{fuzzy}}/Y_{\text{smooth}}$  follows  $(1 - p_{\text{fuzz}})$ , where  $p_{\text{fuzz}}$  is the fuzz porosity. Further,  $Y_{\text{fuzzy}}/Y_{\text{smooth}}$  is observed to increase with incident ion energy,  $E_i$ . This may be explained by an energy dependent change in the angular distribution of sputtered W atoms, since at lower  $E_i$  the angular distribution is observed to become more butterfly-shaped. That is, a larger fraction of sputtered W atoms can line-of-sight deposit/stick onto neighboring fuzz nanostructures for lower  $E_i$  butterfly distributions, resulting in lower ratio of  $Y_{\text{fuzzy}}/Y_{\text{smooth}}$ .

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### 1. Introduction

Sputtering and tritium retention are primary concerns for the selection of a plasma-facing material (PFM) in a fusion reactor. Tungsten (W) exhibits superior properties on these issues compared to other materials [1,2], but strong radiative loss and fuel dilution induced in the core [3,4] mean that it is crucial to reduce the sputtered W influx to the core.

Helium-induced W nano-structures, commonly called ‘fuzz’ (see Fig. 1), have been recently recognized as a potential drawback for W as a PFM, since it is fragile and may have poor thermal properties. Thus, the formation condition and the growth rate of a fuzzy layer have been extensively investigated [5,6]. It has been revealed that the surface temperature,  $T_s$ , of  $\sim 1000$ – $2000$  K is necessary for fuzz to grow, and that the thickness of a fuzzy-surface layer grows in proportion to the square root of the exposure time of a W surface in He plasma.

On the other hand, sputtering properties of W surfaces with a fuzzy layer are poorly understood. To shed more light on this issue, we have measured the incident ion energy dependence of the sputtering yield of W fuzzy surfaces bombarded by Ar. It is found that the sputtering yield of a fuzzy layer is lower than that of a smooth surface by a factor of up to  $\sim 10$ , depending on the fuzzy layer thickness and the incident ion energy. In addition, the angular distribution of sputtered W atoms from both smooth and fuzzy sur-

faces is investigated, showing no clear difference in the angular distribution between both surfaces.

### 2. Angular distribution of sputtered W atoms

In an rf plasma device [7], the angular distribution of sputtered W atoms from a smooth surface bombarded by Ar plasma was directly measured with a quadrupole mass spectrometer (QMS: Hiden EQP mass/energy spectrometer). Plasma parameters in this experiment are as follows: electron density  $n_e \sim 3 \times 10^{17} \text{ m}^{-3}$ , electron temperature  $T_e \sim 3 \text{ eV}$ , ion flux  $\Gamma_i \sim 3 \times 10^{20} \text{ m}^{-2} \text{ s}^{-1}$ , neutral pressure  $\sim 3$ – $4$  mTorr, and rf power  $\sim 1$  kW. The incident ion energy,  $E_i$ , is controlled by negatively biasing the W target. By rotating the target with respect to the QMS aperture, the angular distribution of sputtered W atoms was collected. In this plasma device, no magnetic field is applied, so that the incident angle of ions is normal to the surface due to the applied bias. The details of the data analysis for the QMS system are described in Ref. [7]. As seen in Fig. 2, the angular distribution from a smooth surface is butterfly-shaped in this  $E_i$  range up to 235 eV, and tends to become more butterfly-shaped at  $E_i < 100$  eV. This type of distribution has been also observed in other experiments, and predicted by theoretical modeling and simulations [8].

For a fuzzy surface, the QMS system is difficult to use, since this technique is time-consuming and the surface changes during a measurement at each angle. Instead, the vertical profile of W I (429.4 nm) line emission intensity from sputtered W atoms was measured in front of a W target exposed to He/Ar mixture plasma in the PISCES-B linear divertor plasma simulator [9], where the axis of the plasma column is horizontal. The plasma radius of  $\sim 25$  mm is larger than that (11 mm) of the W target. Thus, the vertical

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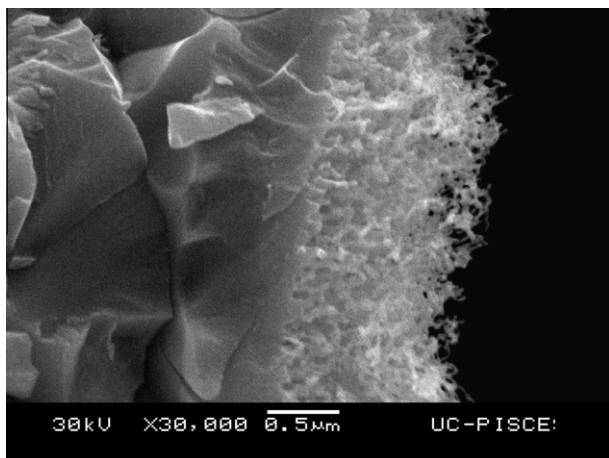


Fig. 1. Cross-sectional SEM image of a W fuzzy surface.

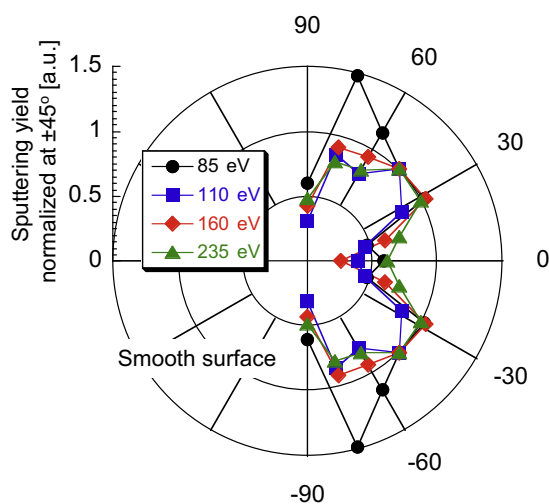


Fig. 2. Polar plot of the angular distributions of sputtered W atoms from a smooth surface bombarded by Ar plasma at various  $E_i$  of  $\sim 85$ –235 eV, measured with a quadrupole mass spectrometer in an rf plasma device. The sputtering yield is normalized at  $\pm 45^\circ$ .

profile reflects the angular distribution of sputtered W atoms. Line-integrated vertical profiles of the normalized W I line emission intensity from a smooth surface are shown in Fig. 3a. It is clearly seen that the profile becomes broader at lower  $E_i$ , which is consistent with the distributions measured with the QMS system in the rf device. Note that the vertical profile of the Ar II (434.8 nm) line intensity did not change with  $E_i$ , indicating that the background  $n_e$  and  $T_e$  profiles did not change. Vertical intensity profiles between smooth and fuzzy surfaces are compared for  $E_i \sim 110$  eV in Fig. 3b. There is no observed clear difference, in addition, no difference is observed for all attempted  $E_i$  down to  $\sim 70$  eV. This indicates that the angular distribution of sputtered W atoms from a fuzzy surface is very similar to that from a smooth surface, and shows the similar  $E_i$  dependence.

### 3. Sputtering yield

To produce a W fuzzy layer, a W target was first exposed to He plasma at  $T_s \sim 1150$  K and  $E_i \sim 90$  eV for  $\sim 800$  s in PISCES-B. The thickness of the fuzzy layer,  $L$ , is expected from the exposure parameters to be  $\sim 1$   $\mu\text{m}$ , according to Ref. [5]. The fuzzy W surface

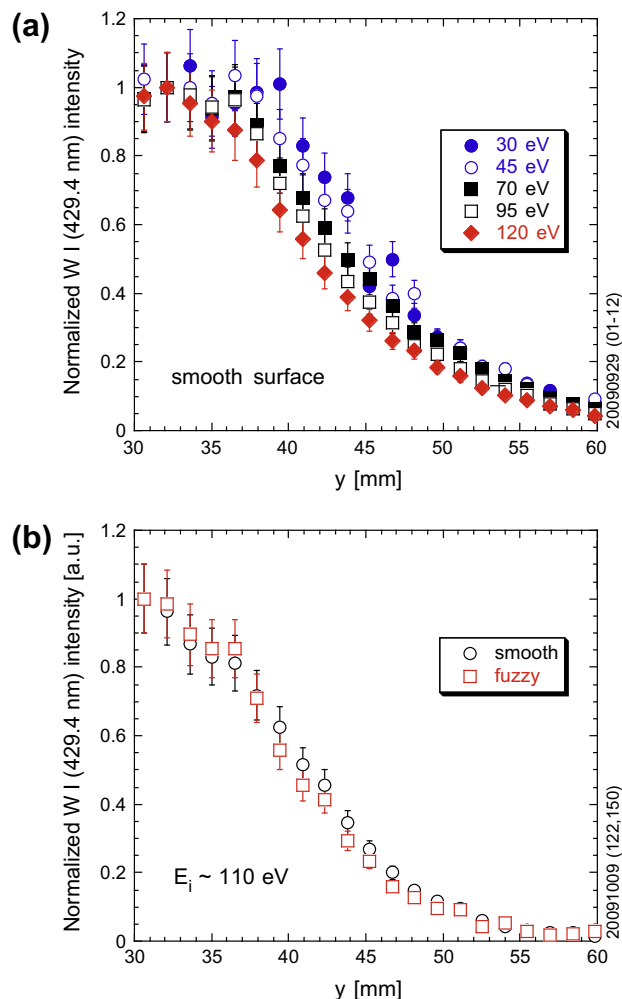
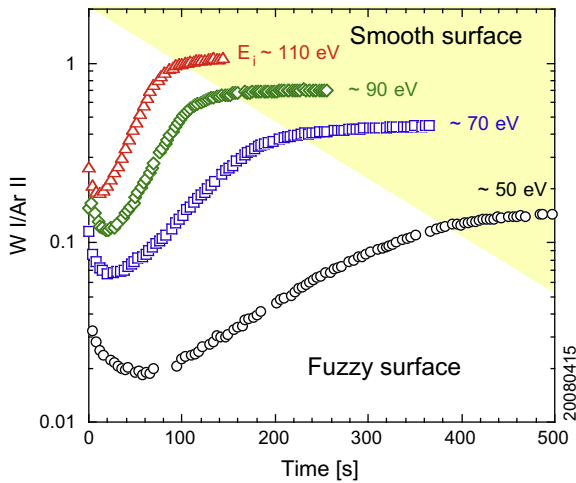


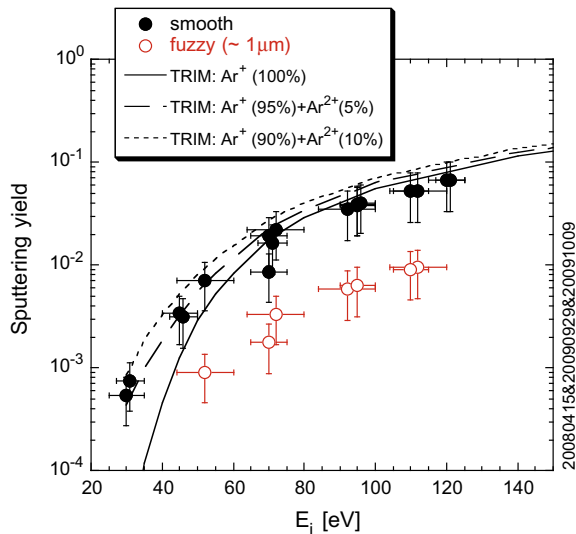
Fig. 3. (a) Vertical profiles of normalized W I (429.4 nm) emission intensity from W atoms sputtered from a smooth surface by Ar bombardment at various  $E_i$  from 30 to 120 eV. (b) Comparison of vertical W I line emission intensity profiles between smooth and fuzzy surfaces at  $E_i \sim 110$  eV. The center of the plasma column is at a vertical position  $y \sim 30$  mm, and the measurements were done at an axial position  $z \sim 4$  mm from the sample surface.

was later exposed to He/Ar mixture plasma at a certain  $E_i$ , and the time evolution of the W I (429.4 nm) line emission intensity of sputtered W atoms was monitored in front of the target. This procedure was repeated to obtain the  $E_i$  dependence. The reason for the gas mixture is to delay the sputtering process by reducing the  $\text{Ar}^+$  ion concentration for better time resolution. Note that sputtering of W by He is negligible compared to that by Ar in this  $E_i$  range up to  $\sim 110$  eV [10]. Fig. 4 shows the time evolution of the ratio of line emission intensities of W I at 429.4 nm to Ar II at 434.8 nm during He/Ar mixture plasma exposures at various  $E_i$ . The normalization by the Ar II intensity is to compensate for changes in plasma parameters and in the transmission of the spectroscopic window during the exposure. It is found that, after a slight decrease, the line intensity ratio increases with time, and reaches a saturation level, showing a transition from a fuzzy surface to smooth. The transition was also confirmed from metal like appearance after sputtering rather than black fuzz color.

A mass loss measurement was performed to obtain an absolute sputtering yield at  $E_i \sim 110$  eV for a smooth W surface, which was exposed for 2100 s to a pure Ar plasma [11]. The sputtering yield of  $\sim 0.05 \pm 0.002$  was obtained, which agrees well with the TRIM calculation [10] and measurements from ion beams [12]. The W I/Ar II



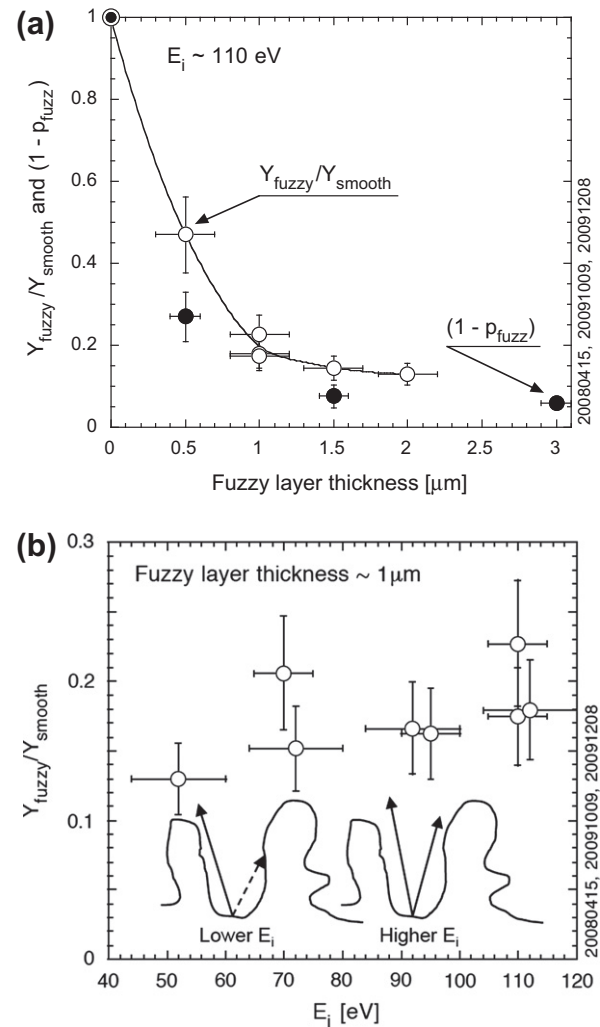
**Fig. 4.** Time evolution of the emission intensity ratio of W I (429.4 nm) to Ar II (434.8 nm) during He/Ar mixture plasma exposures to a W surface at  $E_i \sim 50$ –110 eV, showing a transition from a fuzzy surface to smooth. The fuzzy layer thickness was  $\sim 1\ \mu\text{m}$  at  $t = 0\ \text{s}$ .



**Fig. 5.** Sputtering yields of smooth (closed circles) and fuzzy (open circles) W surfaces bombarded by Ar for the fuzzy layer thickness of  $\sim 1\ \mu\text{m}$ . The curves show theoretical yields for a smooth surface from the TRIM code [10]: solid line for 100%  $Ar^+$ , long-dashed line for 95%  $Ar^+$  and 5%  $Ar^{2+}$ , and short-dashed line for 90%  $Ar^+$  and 10%  $Ar^{2+}$ .

line intensity ratio taken at  $E_i \sim 110\ \text{eV}$  and from a smooth surface in Fig. 4 is scaled to the yield of 0.05, and the scaling factor is applied to other  $E_i$  as well as fuzzy surfaces. In Fig. 5, the sputtering yields of both smooth,  $Y_{\text{smooth}}$ , and fuzzy,  $Y_{\text{fuzzy}}$ , W surfaces are plotted as a function of  $E_i$ .  $Y_{\text{fuzzy}}$  values are taken to be the minima of the curves in Fig. 4. The  $E_i$  dependence of  $Y_{\text{smooth}}$  is in good agreement with TRIM, except at lower  $E_i$ . This deviation may be due to the existence of a small amount of  $Ar^{2+}$  in the plasma, as demonstrated with the dashed curves in Fig. 5, for which 5% and 10%  $Ar^{2+}$  concentrations are assumed.

As a cause of the reduction in the sputtering yield due to fuzz, its porous structure is considered here. Fig. 6a shows the dependence of  $Y_{\text{fuzzy}}/Y_{\text{smooth}}$  on the fuzzy layer thickness,  $L$ , at  $E_i \sim 110\ \text{eV}$  (open circles). The layer thickness was again estimated from  $T_s$  and the plasma exposure time [5]. It is found that  $Y_{\text{fuzzy}}/Y_{\text{smooth}}$  rapidly decreases with increasing  $L$ , and almost saturates to  $\sim 10\%$  at



**Fig. 6.** (a) Fuzzy layer thickness,  $L$ , dependence of  $Y_{\text{fuzzy}}/Y_{\text{smooth}}$  (open circles) taken at  $E_i \sim 110\ \text{eV}$  and the change in fuzz porosity,  $(1 - p_{\text{fuzzy}})$  (closed circles). The curve is drawn only for a guide for the eye. As a definition, the curve crosses one at  $L = 0\ \mu\text{m}$ . (b) Incident ion energy dependence of  $Y_{\text{fuzzy}}/Y_{\text{smooth}}$  for  $L \sim 1\ \mu\text{m}$ . In the inset, it is schematically depicted how the line-of-sight deposition (dashed arrow) of sputtered W atoms onto fuzz increases with a decrease in  $E_i$ , because of the change in the angular distribution.

$L > 1\ \mu\text{m}$ . Note that the curve is drawn only to guide the eye, but it crosses unity at  $L = 0\ \mu\text{m}$  by definition. This observed trend is consistent with the change in  $(1 - p_{\text{fuzzy}})$ , where  $p_{\text{fuzzy}}$  is the fuzz porosity (closed circles), as shown in Fig. 6a. The porosity of a fuzzy layer was calculated from the geometrical volume,  $V_{\text{fuzzy}}$  [13], obtained from cross sectional observations with a scanning electron microscope (SEM), and the mass change,  $\Delta m_{\text{fuzzy}}$ , measured by removing the layer:  $p_{\text{fuzzy}} = 1 - [(\Delta m_{\text{fuzzy}}/V_{\text{fuzzy}})/\rho_{\text{bulk}}]$ , where  $\rho_{\text{bulk}}$  is the mass density of bulk W. At  $L \sim 1.5$  and  $3.0\ \mu\text{m}$ ,  $p_{\text{fuzzy}}$  was approximately 0.90–0.95, i.e.  $(1 - p_{\text{fuzzy}}) \sim 0.05$ –0.10, compared to that of bulk W, while  $p_{\text{fuzzy}}$  at  $L \sim 0.5\ \mu\text{m}$  is only  $\sim 0.73$  [ $(1 - p_{\text{fuzzy}}) \sim 0.27$ ]. Furthermore, high-resolution SEM images also confirm that the porosity increases as fuzz forms with increasing  $E_i$  [14]. At the beginning of the fuzz formation, the structure is micron-sized and less porous, and then it becomes smaller and more porous as fuzz continues to develop. One could make a reasonable assumption that the time dependence of fuzz growth exhibits a similar change in scale during the formation process, but no data is yet available to confirm this assumption.

Another cause of the reduction in the sputtering yield with fuzz could be the direct line-of-sight deposition of sputtered W atoms

onto neighboring fuzz before ejecting into the plasma. This effect can be seen in the  $E_i$  dependence of  $Y_{\text{fuzzy}}/Y_{\text{smooth}}$ , as shown in Fig. 6b for  $\sim 1 \mu\text{m}$  thick fuzzy layers. It is observed that  $Y_{\text{fuzzy}}/Y_{\text{smooth}}$  slowly increases with  $E_i$ . Note that the data for  $Y_{\text{fuzzy}}/Y_{\text{smooth}} > 0.2$  were possibly taken from a slightly thinner layer, since  $Y_{\text{fuzzy}}/Y_{\text{smooth}}$  rapidly changes at  $L < 1 \mu\text{m}$ . As shown in Section 2, the angular distribution of sputtered W atoms becomes more butterfly-shaped with decreasing  $E_i$ . This may lead to an enhanced deposition of sputtered W atoms onto neighboring fuzz, as schematically drawn in the inset of Fig. 6b. Thus,  $Y_{\text{fuzzy}}/Y_{\text{smooth}}$  scales weakly with  $E_i$ . It should be noted that the actual situation is more complicated than the schematic drawing. However, the effect of gyro-motion of incident ions can be neglected in the present condition, since ions are accelerated rapidly at the sheath potential drop and then move parallel to the local electric (and magnetic) field, normal to the surface, within the sheath.

#### 4. Conclusion

Sputtering properties of a He-induced fuzzy W layer have been investigated. The angular distribution of sputtered W atoms from a smooth surface by Ar plasma bombardment was directly measured with a quadrupole mass spectrometer in an rf plasma device. At lower  $E_i$ , sputtered W atoms were observed to be emitted with a more butterfly-shaped distribution. This trend is consistent with observations in PISCES-B, where vertical profiles of the W I line emission intensity from sputtered W atoms are broader at lower  $E_i$ . From comparison of vertical emission intensity profiles between smooth and fuzzy surfaces, it is found that no clear difference in the angular distribution exists.

The sputtering yield of a W fuzzy surface is measured, and is found to depend on the fuzzy layer thickness,  $L$ , and  $E_i$ . With increasing  $L$ ,  $Y_{\text{fuzzy}}/Y_{\text{smooth}}$  rapidly decreases and saturates to a level of  $\sim 10\%$  at  $L > 1 \mu\text{m}$ . It is revealed that this tendency correlates with an increase in the porosity of a fuzzy layer, compared to bulk W. The  $E_i$  dependence of  $Y_{\text{fuzzy}}/Y_{\text{smooth}}$  may be explained by the  $E_i$  dependence of the angular distribution of sputtered W atoms.

Since more W atoms are sputtered at larger angles at lower  $E_i$ , the probability of direct line-of-sight deposition onto neighboring fuzz structures increases. Thus, the ratio  $Y_{\text{fuzzy}}/Y_{\text{smooth}}$  should be expected to decrease slightly with a decrease in  $E_i$ .

At the beginning of the growth of fuzz, the layer thickness quickly changes [5]. For instance, the fuzzy layer is expected to grow to around  $0.5 \mu\text{m}$  at  $T_s \sim 1120 \text{ K}$  for only  $\sim 400 \text{ s}$ , which is comparable to an ITER DT discharge duration. Therefore, the sputtering yield of W might be expected to rapidly decrease in the ITER divertor, as shown in this paper. However, it should be noted that, if the sputtering rate of W, depending on  $E_i$  and impurity concentrations, is larger than the growth rate of fuzz, depending on  $T_s$  and He ion flux, then fuzz would not form.

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