

# SPUTTERING OF TUNGSTEN EXPOSED TO HIGH-FLUX AND HIGH-FLUENCE HYDROGEN ION BEAM

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Tungsten samples were irradiated by hydrogen ion beam with high heat ( $0.1 \dots 1.05 \text{ MW}\cdot\text{m}^{-2}$ ) and particle flux  $[(0.13 \dots 1.3) \times 10^{22} \text{ m}^{-2}\cdot\text{s}^{-1}]$  generated by FALCON ion source. The sputtering yield has remained nearly constant in the given flux range. Scanning electron microscope (SEM) studies of the irradiated surface have indicated that the only influence of the ion beam in the fluence range up to  $2.6 \times 10^{26} \text{ m}^{-2}$  is the increase of roughness.

PACS: 79.20 Rf

## INTRODUCTION

The development of International Thermonuclear Experimental Reactor (ITER) had become a strong scientific and technical challenge for past decades. Current ITER design implies the use of tungsten at least in the divertor region [1]. According to Federici et al. [2] tungsten will be exposed to high particle ( $10^{20} \dots 10^{23} \text{ m}^{-2}\cdot\text{s}^{-1}$ ) and heat (up to  $3 \text{ MW}\cdot\text{m}^{-2}$ ) fluxes. The total number of discharges will exceed 104, while the duration of each discharge will vary between 300 s and 1000 s. The resulting total fluence of particles impinging on the tungsten surface is expected to be in the range of  $10^{27} \dots 10^{30} \text{ m}^{-2}$ . Present lifetime predictions for the first wall are based on pure physical sputtering. However, there is no experimental evidence that this assumption is valid for ITER like conditions. Therefore, experiments in laboratory environment are needed to investigate erosion mechanisms for ITER-like fluxes and fluences.

Presently, there are two ways for experimental simulation of plasma-surface interactions in laboratory environment. Ion beam device with a magnetic mass-separation provides high-energy ion beam, however, its particle flux is limited by value of  $10^{19} \dots 10^{20} \text{ m}^{-2}\cdot\text{s}^{-1}$  [3 - 5]. HiFIT ion beam device is capable to provide higher particle flux up to  $3.6 \times 10^{21} \text{ m}^{-2}\cdot\text{s}^{-1}$  and heat flux up to  $0.65 \text{ MW}\cdot\text{m}^{-2}$ , when mass-separation is excluded [6]. In contrast, plasma devices could generate low energy particle fluxes  $\approx 10^{22} \text{ m}^{-2}\cdot\text{s}^{-1}$  and heat fluxes in the range of  $0.1 \dots 1 \text{ MW}\cdot\text{m}^{-2}$  [7 - 9]. Therefore, the parameter range of particle fluxes  $> 10^{22} \text{ m}^{-2}\cdot\text{s}^{-1}$  and heat fluxes  $> 1 \text{ MW}\cdot\text{m}^{-2}$  is currently not available for most existing plasma and ion sources used in material research. However, high heat and particle flux range may contain new effects related to ion-surface interactions, which could be extremely important justifying material selection.

To fulfil the gap between the parameters provided by laboratory tools and ITER relevant conditions, the FALCON ion source has recently been developed [10-11]. It is based on design of closed drift thrusters (also known as Hall thrusters), which are typically used as space propulsions [12]. Intrinsic characteristics of this type of ion sources are simplicity (which makes them affordable) and extremely high ion currents, both are tempting for use in material research. It is capable to generate ion beam, which delivers high heat and particle flux to the sample surface. The fluxes are comparable or exceed ones provided by plasma devices.

Present work describes first results of irradiation of tungsten samples with ion beam of FALCON ion source at high heat and particle fluxes.

## 1. METHODS

The tungsten samples were exposed to an ion beam generated by the FALCON ion source. The peak of ion energy distribution function is located around 2 keV, while the ion energies are scattered from 0.6 up to 5.4 keV as shown in Fig. 1,a. The ion beam current was varied between 1.5 and 15 mA; the ion beam profile in the target plane has Gaussian-like shape as shown in Fig. 1,b by evaluating the an erosion profile. Calculating the flux and fluence, the effective diameter of the ion beam spot has been assumed  $\approx 3 \text{ mm}$ . Therefore, corresponding particle flux was  $(0.13 \dots 1.3) \times 10^{22} \text{ m}^{-2}\cdot\text{s}^{-1}$  and the heat flux was in the range of  $0.1 \dots 1.05 \text{ MW}\cdot\text{m}^{-2}$ . The total ion beam power was up to 30 W, which leads to heating of the tungsten sample up to a temperature of  $780^\circ\text{C}$ .

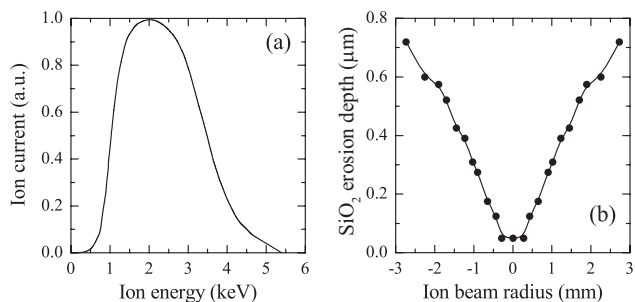


Fig. 1. The distribution function of the ion energy in the beam for accelerating voltage of 5.4 keV (a). The ion beam current density profile of the ion beam was obtained by sputtering of the  $\text{SiO}_2$  layer with hydrogen ions (b)

All measurements on erosion yields were performed estimating sample temperature through the calculation to avoid the disturbance of the weight-loss measurements. The sample temperature has been calculated by Stephan-Boltzmann law and compared to one measured by chromel-alumel thermocouple. The results of the comparison are shown in Fig. 2,a. The overestimation of the calculations over actually measured values occurs because some heat is lost through the thermal conductivity instead of emissivity.

The measurement of the erosion yield has been performed by using ex-situ weight-loss measurements before and after the exposure. The absolute accuracy of the weight measurements was  $\pm 5 \mu\text{g}$ , while typical weigh-change of the sample before and after the exposure was above 1 mg.

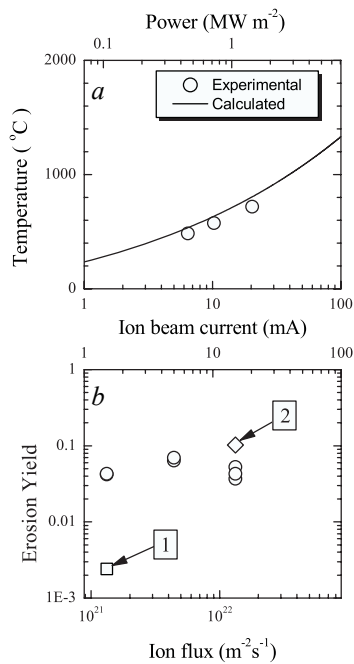


Fig. 2. The dependence of the W sample temperature on the ion beam current measured with a thermo couple (a). The dependence of measured sputtering yield as a function of the ion flux (b). Data point (1) is the value for sputtering yield, which has been taken for 2 keV ions incident on W surface from [13]. Data point (2) shows the erosion yield for sample with top polished surface layer

The charge collection measurements are assumed being a good representative of total number of incident particles. Details of ion beam characterization could be found in [11]. Therefore, weight-loss measurements of erosion in given conditions introduce negligible errors.

After the exposure, the samples were examined a scanning electron microscope (SEM) (ESEM XL30, FEI) and a second SEM with focused ion beam (FIB) (HELIOS Nanolab 600/FEI), both installed in Garching IPP. Each single sample was exposed to certain fluence and then examined in SEM, which had allowed studying fluence dependent surface morphology of tungsten samples. The focused ion beam was used to produce cross-sections of the surface layer and the resulting cross-section was imaged by SEM under an angle of 38°.

## 2. RESULTS AND DISCUSSION

Flux dependent erosion has been investigated by weight-loss measurement using only one single sample, which has been irradiated and weighted sequentially. The sample has been pre-irradiated up to a fluence of  $2 \times 10^{26} \text{ m}^{-2}$  with a ion flux of  $1.3 \times 10^{22} \text{ m}^{-2} \cdot \text{s}^{-1}$ . The pre-irradiation has been performed in order to etch away the damaged layer still remaining after polishing. This layer is suspected to be weaker than other underlying layers resulting in an elevated erosion yield. Fig. 2,b shows the measured erosion yield of the sample with the damaged top layer. This is somewhat higher than other values, obtained after the pre-irradiation. Similar elevation of erosion yield was observed in other experiments. Pre-irradiation of the surface turns its mirror-like surface into matte one due to increased roughness of the surface (see below).

After pre-irradiation, the tungsten sample was used to measure flux-dependent erosion yield. The sample was weighted before and after each exposure; few measurements were performed at the same flux to evaluate scattering. The ion beam current, and corresponding flux, was stable during the exposure within the deviation of <2%. The experiments were performed in the flux range between  $1.3 \times 10^{21}$  and  $1.3 \times 10^{22} \text{ m}^{-2} \cdot \text{s}^{-1}$ . The results of experiments are shown in Fig. 2,b. One could observe nearly constant erosion yield in the investigated flux range taking into account the scattering. The erosion at lower fluxes should be explained by physical sputtering. There are two measurements were performed at flux of  $1.3 \times 10^{21} \text{ m}^{-2} \cdot \text{s}^{-1}$ , however, the data points are too close to each other. The elevation of erosion yield over values typical for interaction of hydrogen ions with tungsten surface could be most probably explained by presence of light impurities in the ion beam, like carbon or oxygen. Since the erosion yield remains independent of ion flux, the erosion at higher fluxes could also be explained by impurities. The scatter of data may be the result of variation of the impurity content, which was not controlled. Therefore, in the given flux range, there has not been observed a flux dependent erosion yield. Erosion effects related to high-flux, if present, should be equal or lower than observed scattering of the data on erosion yield.

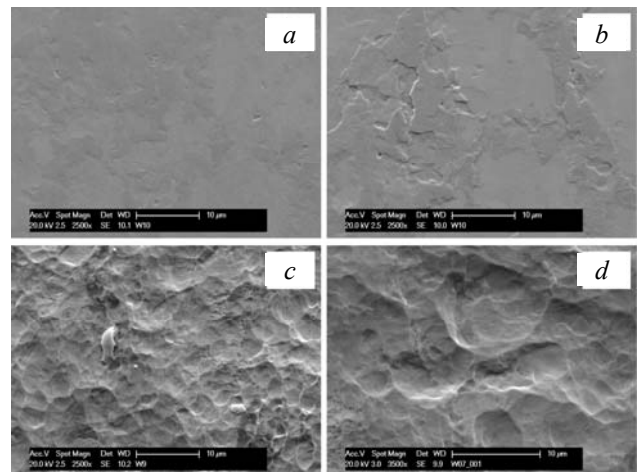


Fig. 3. SEM images of the tungsten surface exposed to hydrogen ion beam. Initial surface conditions (a); ion fluence of  $2.4 \times 10^{24} \text{ m}^{-2}$  (b); ion fluence of  $2.3 \times 10^{25} \text{ m}^{-2}$  (c); ion fluence of  $2.6 \times 10^{26} \text{ m}^{-2}$  (d)

To investigate morphology evolution with fluence  $2.6 \times 10^{21} \text{ m}^{-2}$  of the tungsten sample, several samples were exposed to the different fluence. During the exposition, the ion flux was varied in the range of  $(4.5 \dots 9) \times 10^{21} \text{ m}^{-2} \cdot \text{s}^{-1}$ . The respective temperature of the samples was 400...600°C. After the exposition the sample surface has been examined in SEM, the images are shown in Fig. 3 for three fluences. One can see that the initially polished surface becomes rougher as fluence increases, however, no blisters were found over the surface at any fluence. After the ion fluence of  $2.4 \times 10^{24} \text{ m}^{-2}$  the surface remains still smooth, however, the grains of lamina reach the surface and become well visible. Increasing the fluence up to  $2.3 \times 10^{25} \text{ m}^{-2}$  the roughness level increases and the roughness covers the surface completely. Small parts of the lamina (see central part of Fig. 3,c) may rarely be found. Increasing the ion flu-

ence further up to  $2.6 \times 10^{26} \text{ m}^{-2}$  the surface roughness still increases. The cross-section of the surface in Fig. 4 shows no internal cavities or cracking along the grain boundaries.

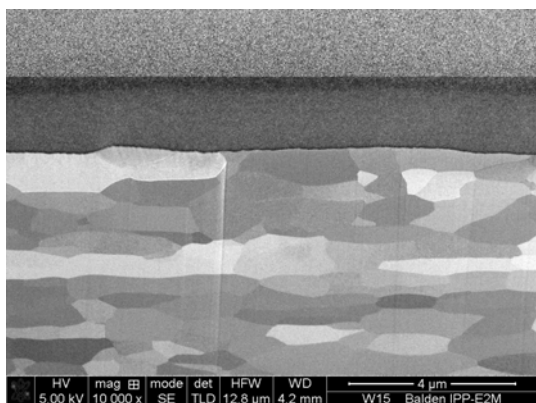


Fig. 4. The cross-section of the tungsten surface exposed to ion fluence of  $1 \times 10^{26} \text{ m}^{-2}$

The observed surface morphology could be compared to one, obtained by Shu, et al. [14] with recrystallized tungsten irradiated with low energy ions at similar fluxes and fluence in linear plasma generator. The main difference is that the surface features and blisters have been observed on the surface of the recrystallized tungsten. Shu et.al. have also observed melting and flakes at heat fluxes down to heat fluxes  $0.1 \text{ MW} \cdot \text{m}^{-2}$ . This is somewhat contradicting to the present observations, no such features have been found. Since ion beam irradiation intrinsically lacks side-effects like redeposition or arcing, it is difficult to say at present point, where the differences are coming from.

## CONCLUSIONS

The tungsten samples were irradiated with keV hydrogen ion beam generated by newly developed FALCON ion source. In our experiments the tungsten samples were irradiated with particle flux of  $(0.13 \dots 1.3) \times 10^{22} \text{ m}^{-2} \cdot \text{s}^{-1}$  and the heat flux of  $0.1 \dots 1.05 \text{ MW} \cdot \text{m}^{-2}$ . The weight-loss measurements have shown nearly constant erosion yield in the given range, which indicates low or non-existent erosion effects related to high flux irradiation of tungsten with hydrogen ions. The investigation of surface morphology shows only increase of the roughness with fluence increase. This is somewhat contradicting to results obtained in plasma devices, where flakes and melting droplets were observed on tungsten surface exposed to hydrogen plasma at much smaller heat loads. Possible differences may originate from the irradiation procedure, because ion beam bombardment allows to avoid side-effects like re-deposition or arcing.

Author acknowledges T. Schwarz-Selinger, M. Balden from Max-Planck-Institut für Plasmaphysik, EURATOM Association, Garching, Germany, for collaborating in surface diagnostics.

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Article received 20.03.2013.

## РАСПЫЛЕНИЕ ВОЛЬФРАМА ПОД ДЛИТЕЛЬНЫМ ВОЗДЕЙСТВИЕМ ПУЧКА ИОНОВ ВОДОРОДА С ВЫСОКОЙ ПЛОТНОСТЬЮ

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Образцы вольфрама облучались пучком ионов водорода с высокой плотностью частиц  $[(0.13...1.3)\times 10^{22} \text{ м}^{-2}\text{с}^{-1}]$  и мощностью  $(0.1...1.05 \text{ МВт}\cdot\text{м}^{-2})$ , который генерировался ионным источником FALCON. Коэффициент распыления оставался приблизительно постоянным в заданном диапазоне плотности ионных потоков. Изучение поверхности с помощью РЭМ показало, что воздействие пучка на поверхность приводит только лишь к увеличению шероховатости в диапазоне флюенсов  $< 2.6\times 10^{26} \text{ м}^{-2}$ .

## РОЗПИЛЮВАННЯ ВОЛЬФРАМУ ПІД ТРИВАЛИМ ВПЛИВОМ ПУЧКА ІОНІВ ВОДНЮ З ВИСОКОЮ ЩІЛЬНІСТЮ

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Зразки вольфраму опромінювалися пучком іонів водню з високою щільністю частинок  $[(0.13...1.3)\times 10^{22} \text{ м}^{-2}\text{с}^{-1}]$  і потужністю  $(0.1...1.05 \text{ МВт}\cdot\text{м}^{-2})$ , який генерувався іонним джерелом FALCON. Коefіцієнт розпилення залишався приблизно постійним в заданому діапазоні щільності іонних потоків. Вивчення поверхні з допомогою РЕМ показало, що вплив пучка на поверхню призводить лише до збільшення шорсткості в діапазоні флюенсів  $< 2.6\times 10^{26} \text{ м}^{-2}$ .