

## Thermal cycling and high power density hydrogen ion beam irradiation of tungsten layers on tungsten substrate

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**Abstract.** Tungsten layers with iron impurity were deposited on tungsten substrates modeling re-deposited layers in a fusion device. The samples were tested by thermocycling and hydrogen ion beam tests. Thermocycling revealed globule formation on the surface. The size of the globules depended on iron impurity content in the coating deposited. Pore formation was observed which in some cases lead to exfoliation of the coatings. Hydrogen ion irradiation lead to formation of blisters on the coating and finally its exfoliation.

### 1. Introduction

Tungsten is considered as a plasma-facing material of the ITER divertor. The behavior of tungsten during thermal cycling and under plasma radiation was researched in a number of articles [1-3]. Tungsten atoms sputtered by plasma radiation form layers of tungsten on divertor tiles less affected by irradiation [4, 5]. This work is dedicated to researching the processes in the deposited layers during thermal cycling and high power density hydrogen ion beam irradiation.

### 2. Experimental

Tungsten layer deposition and ion flux irradiation was conducted on the CODMATT (COating Deposition and MATerial Testing) stand [6]. The layers were formed on polished tungsten plates with the dimensions of  $15 \times 7.5 \times 1 \text{ mm}^3$  using atoms sputtered from the tungsten target by plasma ions. The layers of  $2 \text{ }\mu\text{m}$  thickness were formed with the deposition rate of  $2 \text{ }\mu\text{m/hour}$ .

It should be noticed that while researching the behavior of tungsten re-deposited in the ITER conditions, beryllium and, possibly, stainless steel impurities sputtered from different parts of the installation will be present in the layers. As a safety precaution, beryllium cannot be used in our laboratory, which is why stainless steel (Fe, Cr, Ni) atoms were directed towards the tungsten surface to be used as an impurity.

Thermal cycling of samples was conducted in the Multifunctional Research Complex of Mass-spectrometry Analysis (MIKMA) [7]. The samples were heated by heat radiation from the tungsten coil located at the opposite side of the sample and heated by applied current in a vacuum of at least  $10^{-6}$  Torr. The upper limit of the temperature was  $1200 \text{ }^\circ\text{C}$ . The temperature gradient on the sample did not exceed  $\pm 10^\circ\text{C}$ . The samples were heated at the rate of  $5^\circ\text{C/sec}$  and cooled for 5 minutes down to  $300 \text{ }^\circ\text{C}$ . A number of samples with a small (0.2% at.) and large (2% at) concentration of iron impurity in the deposited layer has been prepared for testing.

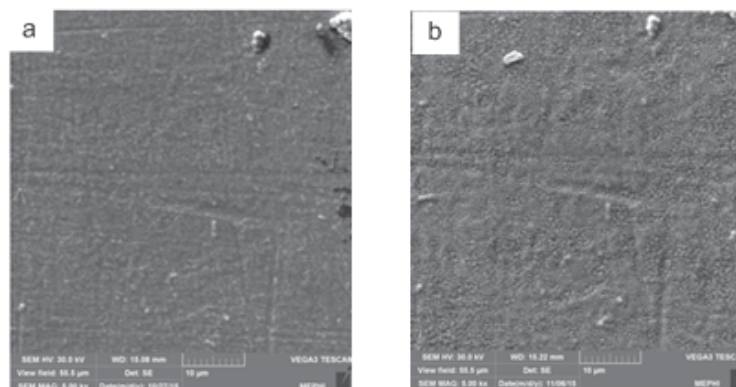


The deposited layers' structure and texture analysis before and after the tests was conducted on the Vega Tescan 3 scanning electron microscope (SEM), the cross-section was prepared using the FEI Quanta 200 3D system.

### 3. Results

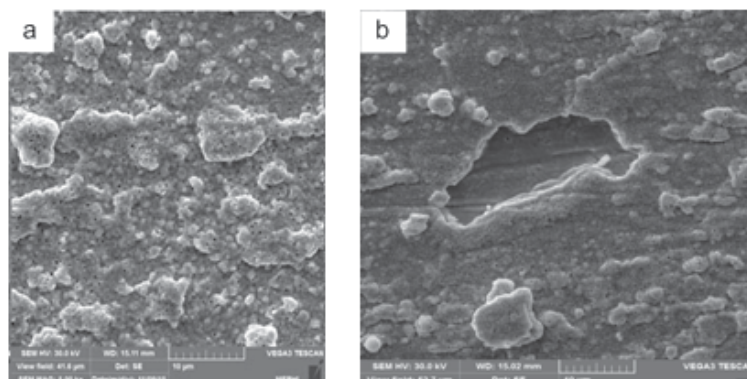
#### 3.1. Thermal cycling of the deposited layers

After 260 cycles signs of restructuring have begun appearing on the surface of low impurity level deposited film. Distinct globules 200-500 nm in diameter have appeared (figure1). No signs of cracking or exfoliation were found.



**Figure 1.** Deposited tungsten with small impurity concentration. a) directly after deposition, b) after 260 heating cycles.

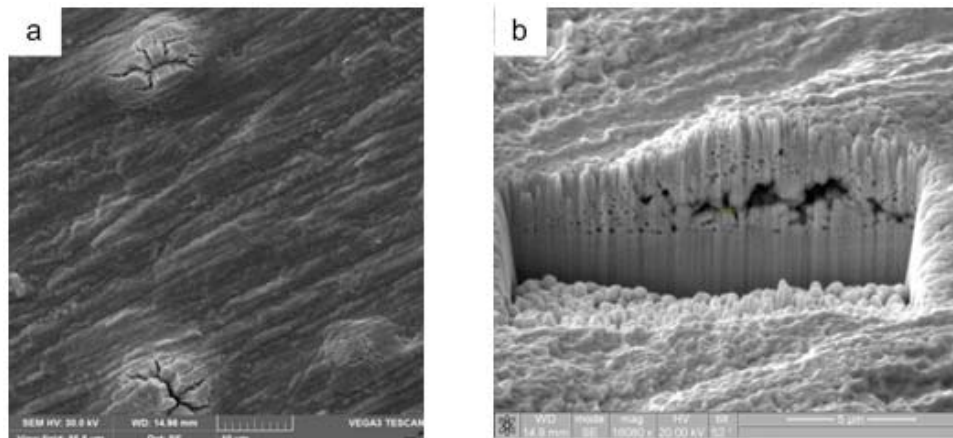
The same experiments were carried on the tungsten layer with a large iron concentration (2% at.). Thermal cycling has lead to the formation of larger globules (1-3  $\mu\text{m}$  in diameter, figure 2), which may be connected with the crystallization processes in the deposited layer. After 250 cycles the formation of pores on the surface is clearly visible. After 350 cycles the pore formation on the surface continues, with signs of exfoliation of the deposited layer in some areas, which may hint towards the formation of a large number of pores in the bulk of the deposited tungsten.



**Figure 2.** The deposited layer of tungsten with 2% at iron impurity content a) pores on the surface after 250 cycles, b) exfoliation after 350 heating cycles.

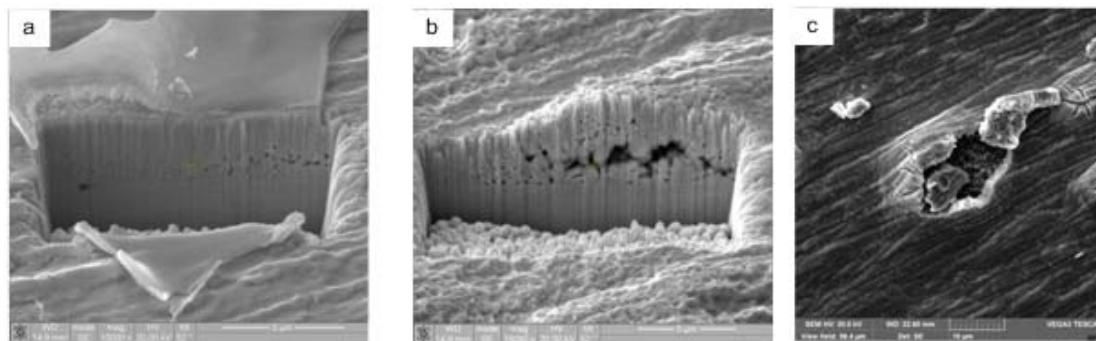
### 3.2. Hydrogen ion irradiation of the deposited layers

During the sample irradiation of tungsten film with 2% at iron impurity concentration with 5 Mw/m<sup>2</sup> power density H<sub>2</sub><sup>+</sup> ions (ion energy is 10 KeV, cyclic irradiation mode with 20 pulses of 5 seconds each), blister formation was observed on the surface of the layer, with the temperature of the surface not exceeding 1200°C, with the sample cooled down to 600°C between the pulses. Fig. 3a shows the image of the film after the irradiation. Blister formation was not observed on the film with low impurity level irradiated under the same conditions. Formation of a large number of microscopic gas bubbles near the film-substrate boundary can be seen on figure 3b.



**Figure 3.** Deposited layers of tungsten after hydrogen ion beam irradiation. a) distinct blisters, b) blister cross-section.

In the figure 4 one could observe the stages of blister formation under hydrogen ion irradiation.



**Figure 4.** The stages of blister formation: a) gas bubbles formation, b) growth and association of adjacent bubbles, c) destruction of blister.

On the first stage the gas bubbles near the separation line between deposited layer and the substrate are formed (figure 4a). Then the bubbles growth and association lead to the occurrence of stresses in deposited layer and as a consequence blister formation (figure 4b). Further hydrogen accumulation leads to the blister cover destruction.

Hydrogen accumulation in these bubbles could lead to formation of tensions in the deposited layer parallel to the surface and the subsequent blister formation. Interesting thing to note is that open pores were formed when the sample was heated to the same temperature without irradiation which could

assist with gas captured during the deposition escaping the sample and, as a result, the blisters and tensions did not form.

#### 4. Conclusions

Thermal cycling and hydrogen ion beam irradiation of samples of tungsten film on tungsten with different concentrations of iron impurity in the deposited layer (0,2% at. and 2% at.) were conducted. During the thermal cycling, globules were formed on the surface of the films, with larger globules appearing on the sample with higher impurity concentration, with the diameter of 1-3  $\mu\text{m}$ . Pore formation was also observed on the samples with higher impurity concentration, which lead to the exfoliation of the deposited layer. This phenomenon was not observed on the sample with 0,2% at. iron impurity concentration. During the hydrogen ion beam irradiation of the samples the pore development on the samples with high impurity concentration lead to exfoliation of the deposited layer and formation of blisters.

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