Growth of nanostructured highly porous surface on refractory metals under plasma treatment in the PLM device

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Abstract. Refractory metals are irradiated with powerful plasma in the PLM plasma device which is a linear magnetic trap with a 8-pole multicusps magnetic confinement. A feature of this device is the many hours stationary confinement, which is an advantage for testing materials of the divertor and wall of a thermonuclear fusion reactor and for the development of novel plasma technologies for metal processing. Helium plasma parameters were measured in the PLM by using the reciprocated Langmuir probe. Tungsten, titanium and molybdenum test plates were tested in stationary helium discharges in the PLM during 200 minutes. The thermal load on the surface of the test plates was more than 1 MW / m². The temperature of the plates reached 1000 °C. Scanning electron microscopy analysis revealed a stochastic nanostructured surface with dimensions of structural elements less than 100 nm. Such materials are of interest for using in nuclear, chemical and biomedical technologies.

1. Introduction

Material tests of tungsten and refractory metals in modern fusion devices have revealed the significant change of surface structure under the powerful plasma loads [1]. The process of plasma-surface interaction (PSI) in magnetic fusion devices involves several mechanisms of surface erosion including melting and resolidification of surface layers, melted material motion over surface, sputtering, evaporation, redeposition of the eroded material on the surface, recrystallization, reformation of surface layers from tens of nanometers to hundreds of microns [1-3]. The uniqueness of the PSI under high heat load in fusion devices is that many elementary processes can affect simultaneously. As a result, the surface morphology evolution is influenced by not a single of the above listed elementary processes, but by a cumulative integral effect of many processes. This leads to
synergistic effects considered by the theory (see, e.g. [3,4]) taking into account surface growth instability driven by stochastic motion of agglomerated particles and clusters. In results, a structure of such surface obeys inhomogeneous hierarchical granularity, statistical self-similarity and scale invariance of the surface structure with unusual shape; e.g., materials with cauliflower-like [1,3,4] and fuzz-like surface recently found in fusion devices [1-7]. In plasma-surface interaction, physical and chemical sputtering, thermal annealing due to plasma heat flux, material erosion and redeposition, melting, cracking should be considered in the problem depending on their intensity and coupling. The specific property of material solidification and clustering under plasma influence in fusion devices is due to a material’s (ions, clusters, melt on the surface etc.) motion under the influence of stochastic electromagnetic field formed by the near-wall turbulent plasma [8-14]. For such purposes, the PLM device was constructed [15,16]. In this article we describe nanostructure surface grown on refractory metals irradiated with plasma in the PLM plasma device.

2. Irradiation of samples in the PLM plasma device

Tungsten, molybdenum and titanium was exposed to helium plasma in the PLM linear divertor-plasma simulator at NRU “MPEI”. The PLM device is a linear plasma trap with a multi-cusp configuration of a magnetic field and a stationary plasma discharge that provides the powerful plasma-thermal load up to 5 MW/m² on test materials, Fig.1. The test samples were irradiated with helium plasma of many hours discharge. Plasma parameters measured by reciprocating Langmuir probe and spectroscopic diagnostics were as follows: plasma density was up to \(3 \times 10^{18} \text{ m}^{-3}\), the electron temperature was up to 10 eV with a fraction of hot electrons of temperature up to 50 eV, the ion plasma flow onto the test sample was up to \(3 \times 10^{21} \text{ m}^{-2} \text{ s}^{-1}\), discharge current reached the value of more than 15 Amps. Magnetic field was of 0.01 Tesla on the trap axis and up to 0.1 Tesla in the cusps. During the plasma exposure, sample temperature was monitored. The duration of the plasma exposure was 200 minutes. The plasma conditions were as following: electron temperature was of ~4 eV, plasma density was of \(~3 \times 10^{18} \text{ m}^{-3}\). The target samples have no active cooling in these experiments, plasma heat load on test target was of 0.5 - 1 MW/ m². The incident ion energy of 30-80 eV was achieved by applying a negative bias to the sample manipulator. The sample temperature in the range 900 - 1200 K was maintained.

Figure 1. (a) View of the PLM plasma device. (b) Fast reciprocated Langmuir probe diagnostics. (c) View of helium plasma discharge with material target exposed.
3. Nanostructured highly porous surface on refractory metals

The surface morphology of tungsten sample was examined using a scanning electron microscope before and after He plasma exposure. The surface of samples has changed after exposure. The metallographic sections of tungsten sample after irradiation with the high heat plasma in the PLM device have revealed nanostructured “fuzz”-type structure grown on the surface with fibers of 20-50 nanometers, Fig. 2. Scanning electron microscopy (SEM) analysis revealed the nanostructured fuzz layer of the depth of approximately 1.6 µm on the tungsten. The density of fuzz layer was observed to depend on the plasma load, Fig. 2a,b and Fig. 2c,d. The obtained results on tungsten nanostructure should be taken into account when analyzing the conditions of erosion and modification of tungsten divertor plates in the ITER.

![Figure 2](image1.png)

**Figure 2.** SEM micrographs of the nanostructured tungsten surface of “fuzz” type, irradiated with the PLM plasma. Dense (a,b) and rare (c,d) fuzz-type structure.

A nanostructured highly porous surface is formed on titanium surface under plasma load in the PLM device during 200 minutes with loads of more than 1 MW / m², Fig. 3. The layers of corrugated
surface with deep holes and stochastic agglomerated hills are observed on the titanium surface which is of highly porous.

Figure 3. SEM micrographs of the nanostructured titanium surface irradiated with the PLM plasma.

A nanostructured highly porous surface is formed on molibdenum surface under plasma load in the PLM device during 200 minutes with loads of more than 1 MW / m², Fig. 4. The sample was irradiated with plasma under condition with alternative voltage from -100 V to +30 V on the sample. Scanning electron microscopy analysis revealed a stochastic nanostructured surface with dimensions of structural elements less than 50 nm, Fig. 4b. The surface structure is roughen and similar to the structure of stochastically agglomerated elongated elements observed on tungsten surface under the same plasma load.
4. Conclusion

Refractory metals are irradiated with powerful plasma in the PLM plasma device which is a linear magnetic trap with 8-pole multicusp magnetic plasma confinement. The feature of this device is the many hours stationary confinement, which is an advantage for testing materials of the divertor and first wall of a thermonuclear fusion reactor and for the development of novel plasma technologies for metal processing. Helium plasma parameters were measured in the PLM by using the fast reciprocated Langmuir probe and spectroscopic diagnostics. The measured temperature of the hot fraction of electrons were up to 50 eV, the cold fraction of electrons was up to 10 eV. The electron density reached $3 \times 10^{18} \text{ m}^{-3}$. Tungsten, titanium and molybdenum test plates were irradiated with stationary helium discharges in the PLM during 200 minutes. The thermal load on the surface of the test plates was more than 1 MW / m$^2$. The temperature of the plates reached 1000 °C. Scanning electron microscopy analysis revealed a stochastic nanostructured surface with dimensions of structural elements less than 100 nm. The growth of a nanostructured surface with a “fuzz” type structure and high porosity is observed. Such novel materials are of interest for nuclear, chemical, hypersonic technologies, as well for biotechnologies and biomedical applications.

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