Formation of titanium highly porous nanostructured surface under plasma irradiation in the PLM device

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Abstract. The titanium plates were irradiated with plasma in stationary helium discharges in the PLM plasma device. The duration of discharges in the PLM installation reached 200 minutes. The thermal load on the surface of the test plates is more than 1 MW / m². The heating temperature of the plates reached 700-900 °C. Scanning electron microscopy of the samples revealed a highly porous stochastic nanostructured surface with dimensions of structural elements less than 500 nm. Such novel material is of interest for nuclear, chemical, hypersonic technologies, as well for biotechnologies and biomedical applications.

1. Introduction

Tests of materials in fusion plasma devices showed a significant change of the surface micro- and nanostructures under the powerful plasma loads [1-10]. The growth of nanostructured surface with high porosity is observed. Such changes were observed after plasma irradiation of tungsten, molybdenum, titanium, tantalum, iron, and some other metals. To study such processes and the development of surface modification technologies, the PLM installation was constructed [5]. In recent years, the effects of extreme thermal plasma loads on the material have revealed universal effects of stochastic clustering of a surface with hierarchical granularity and statistical self-similarity [2-4]. Such a structure of the solid surface is formed in fusion devices after the plasma testing of materials when multiple mechanisms of erosion and redeposition of eroded material, melting and melt movement of surface layers effect simultaneously at scales from tens of nanometers up to hundreds of micrometers. 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 [2-5,10-16]. The properties of the unique scale invariance (self-similarity) of the structure of such highly porous materials with a high-porosity nanostructured surface are in demand for operation under extreme thermal and plasma-beam loads, to cover the streamlined surfaces of aircraft in order to reduce aerodynamic drag at supersonic and hypersonic speeds, in biomedical applications.
This article reports on the experimental observations of novel titanium material with stochastic surface formed under irradiation with the high-temperature plasma in the PLM plasma device.

2. Irradiation of samples with stationary plasma

The plasma irradiation of titanium samples from the T-10 tokamak were performed on the plasma linear multicusp (PLM) plasma device [5,17]. 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. The test Ti samples were irradiated with helium plasma of discharge duration up to 200 minutes. Plasma parameters measured by Langmuir probe and spectroscopic diagnostics were as follows: plasma density was up to $3 \times 10^{18}$ m⁻³, the electron temperature was up to 4 eV with a fraction of hot electrons of temperature up to 50 eV, the ion plasma flow onto the test sample was up to $3 \times 10^{21}$ m⁻² 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. Incident angle between magnetic field and target surface was of 90°. The target samples have no active cooling in these experiments, plasma heat load on test target samples was of 0.5 - 1 MW/m².

As a result, after plasma irradiation, a rough surface with stochastic surface is formed on the test sample, Fig.1. This surface exhibits rough stochastic topography with different scales of granularity, beginning from hundred nanometers (Figs. 1a and 1b), with the unique hierarchic “cauliflower-like” shape granularity, Fig. 1a, which was observed previously for carbon and tungsten materials [2-4].

Figure 1. (a) SEM micrographs of the plasma-faced surface of Ti

3. Surface structure of irradiated Ti samples

Height profiles of titanium sample after plasma irradiation in the PLM exhibits intermittent behavior, Fig. 2a. The relief differ from a trivial stochasticity (Brawnian relief). Such deviation from trivial randomness is illustrated by spectral and statistics analysis. To quantify the statistical properties of self-similarity of the stochastic structure, the probability distribution function (PDF) of the relief heights is constructed as the normalized histogram of the heights (see [2-4]). Let us remind that the Gaussian law describes a ordinary (trivial) surface roughness, for example, the Brownian surface. The PDF for sample surface exposed in the high-temperature plasma typically have “heavy” tails and is not described by the Gaussian (normal) law, Fig. 2b. The samples exposed to high-temperature plasma
are characterized by the non-Gaussian PDF of relief. Figure 2b shows the PDF of heights of the titanium sample irradiated by plasma in the PLM device (see Fig.1). This PDF significantly deviates from the Gaussian law and cannot be fitted by other trivial laws of theory of probability, e.g., the Cauchy–Lorentz law.

Figure 2. (a) Heights profile of a titanium surface after high-temperature plasma processing in the PLM. (b) PDF of the profile heights. The Gaussian (dotted line) and the Cauchy-Lorentz (solid line) distributions are shown for comparison

The Fourier spectrum of relief heights profile of experimental samples (Fig. 3) characterizes the scale distribution of heights (of structure sizes on the surface). The spectra are broadened and do not exhibit resonances, which indicates the absence of dominant periodic structures in the relief. The spectra have a decay shape usually observed in objects with scale invariance and statistical self-similarity.

Figure 3. The Fourier spectrum of the stochastic nanostructured relief of titanium sample after irradiation with plasma in the PLM.

The dependence of the Fourier spectrum on the wavenumber can be fitted by a power law (see Fig. 3) with power law exponent \( p = -2.4 \). The exponents \( p \) of a power law \( S(k) \sim k^p \) characterized samples after the irradiation by the high-temperature plasma in fusion plasma facilities are in the range from -2.4 to -2.8 and below, see [3]. Such a difference in \( p \) exponent can be described within the kinetic approach to the problem of stochastic clustering (see [3,4]), which predicts a difference in spectra for
different properties of self-similarity of agglomeration. The Hurst exponent H for the profile was estimated as H=0.6 – 1 which characterizes self-similarity. This property allows comparing the studied object with the known theoretical models of stochastic objects for which the Hurst exponent is known. The nanostructured Ti surface has a large specific area, which is of great importance for its application to the adsorption of gases and catalysis.

4. Conclusion

Tests of materials in modern plasma installations showed the possibility of a significant change in the surface micro- and nanostructures under the action of powerful plasma loads. The growth of nanostructured surface with high porosity is observed. Such changes were observed during plasma irradiation of samples from tungsten, molybdenum, titanium, tantalum, iron, and some other metals. To study such processes and the development of surface modification technologies, the PLM plasma device was constructed. The PLM is a linear magnetic trap with 8-pole multicusp magnetic plasma confinement. A feature of this device is the stationary plasma confinement of many hours of plasma confinement, which is an advantage for material test for development of plasma technologies for metal processing. The titanium test plates were irradiated with plasma in stationary helium discharges in the PLM installation. The thermal load on the surface of the test plates is more than 1 MW / m². Electron microscopy of the plates revealed a highly porous stochastic nanostructured surface with dimensions of structural elements less than 500 nm. Such novel material is of interest for nuclear, chemical, hypersonic technologies, as well for biotechnologies and biomedical applications.

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