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Interaction between nitrogen plasma and tungsten

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ABSTRACT

The set of experiments using a glow discharge of direct current in the mixture of working gases (nitrogen-hydrogen) was carried out in order to choose optimal method of tungsten nitriding. In the result of experiments it was chosen nitriding mode which leads to formation of tungsten nitrides on the surface of irradiated sample. Analysis methods SEM, EDS, X-ray diffraction were used to determine parameters such as the structure, microhardness, phase and element composition of the irradiated tungsten surface, and also the depth of nitrogen penetration into tungsten.

The mode of tungsten nitriding, which can be used for studying the effect of plasma and thermal influence on nitrided surface of tungsten (forming of tungsten "fuzz" at nitrided tungsten, sputtering of nitrided tungsten) was determined.

1. Introduction

In thermonuclear reactors (TR) the thermal flux from plasma to diverter, in the case of plasma disruption, can exceed operating the limit for candidate material of diverter – tungsten ~5 MW/m^2. For more evenly distribution of thermal flux over surfaces facing to the plasma at such tokamaks as ASDEX Upgrade and JET charging the plasma with nitrogen is used. However nitrogen has specific features during interaction with tungsten namely the formation of nitrides of tungsten in the implantation area [1,2].

In a fusion reactor helium is the result of thermonuclear reaction. In the result helium impinge on the diverter area, the proposed material being tungsten. In the course of interaction between plasma containing helium and pure tungsten nanostructures are formed (tungsten "fuzz" [3,4]), which are highly brittle and can lead to increased sputtering of tungsten as well as dirtiness of plasma by tungsten particles consequently. In this regard reducing of the tungsten fuzz formation becomes an actual problem. In order to solve this problem it is required to carry out comparative analysis of tungsten fuzz formation on the pure surface of tungsten as well as on surface of tungsten, which previously was subjected to nitriding. Determination of tungsten nitriding method is the first stage of this work. Microstructure, microhardness, phase and element composition of irradiated surface of tungsten and penetration depth of nitrogen into tungsten also should be determined.

2. Material and methods

Three experiments on tungsten nitriding in glow discharge of direct current have been conducted in plasma-beam installation.

The experiments were conducted on plasma-beam installation (PBI) the main elements of which are: electron-beam projector (EBP), chamber of plasma-beam discharge (PBD), evacuation chamber of EBP, interaction chamber, magnetic coils, the target device, gateway device and loading chamber [5].

During the experiment a sample was placed in the holder of the target device, which was galvanically isolated from the housing of vacuum chamber as Fig. 1 shows. A negative potential (cathode) was supplied to the irradiated sample, the housing of the interaction chamber was grounded (anode).

The tungsten samples of 5 mm height were cut from cylinder-shaped tungsten rod with diameter of 10 mm by the method of electroerosive cutting. Then samples were grinded and polished from one side. Fig. 2 provides the picture of tungsten sample.

After sample preparation the experiments for tungsten nitriding were carried out. According to the results of experimental studies on tungsten nitriding in plasma-beam discharge with the mixture of nitrogen 60% and argon 40%, the chosen methods of tungsten nitriding lead to erosion of irradiated surface and not to nitriding. In this regard, for increasing of flux density of ions on the irradiated

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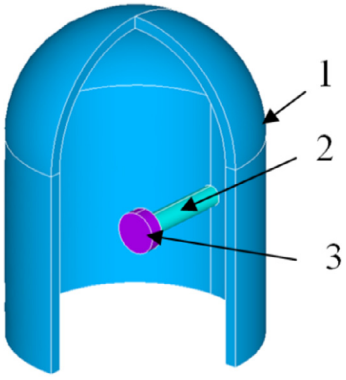
Table 1
Nitriding modes of sample.

Sample	Pressure in the chamber, Torr	Current on the sample, mA	Potential on the sample, V	Temperature of the sample, °C	Experiment time, hour	Square of irradiated surface, cm^2	Flux density of ions, $1/(cm^2 \cdot s)$	Ions fluence, $1/cm^2$
W-1	4	105	−630	900	4	10.6	$6.1 \cdot 10^{16}$	$8.8 \cdot 10^{20}$
W-2	6	100	−425	850	10	10.6	$5.8 \cdot 10^{16}$	$2.1 \cdot 10^{21}$
W-3	17	45	−456	1130	30	3.14*	$8.9 \cdot 10^{16}$	$9.6 \cdot 10^{21}$

* the area of irradiated surface of W-3 sample is less then area of W-1 and W-2 ones, due to tantalum holder, which was not used in this case, but it was used for fixing of W-1 and W-2 samples.



1 – housing of interaction chamber of PBI;
2 – glass quartz tube-holder of irradiated sample;
3 – irradiated sample

Fig. 1. Placement of the sample in the interaction chamber during irradiation.

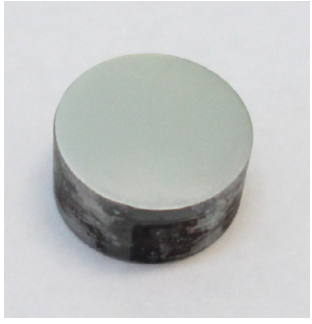


Fig. 2. Outer view of tungsten sample.

surface by increasing of the pressure in the interaction chamber and nitriding of tungsten surface, the method of surface nitriding in glow discharge of direct current was applied, which is widely used for obtaining thin films and for cleaning of materials' surfaces [6].

As plasma-supporting gas the mixture of nitrogen (70%) and hydrogen (30%) was used. Hydrogen was added for minimizing tungsten oxide formation on the irradiated surface.

Before experiments, the chamber cavities of the installation were pumped down to the pressure of 10^{-7} Torr and then working mixture of gases and negative potential were supplied to the sample.

Nitriding modes of tungsten samples are shown in Table 1.

The density of ion current during tungsten nitriding was 9 – 14 mA/cm^2.

The ion energy in the glow discharge was determined by the cathode drop and made 630 eV, 425 eV and 456 eV for the samples W-1, W-2 and W-3 respectively.

For comparison the density of ion current during titanium nitriding in glow discharge amounted to 1.5 – 4 mA/cm^2, potential on nitride sample – 600 V, pressure of the environment ~20 Torr [7].

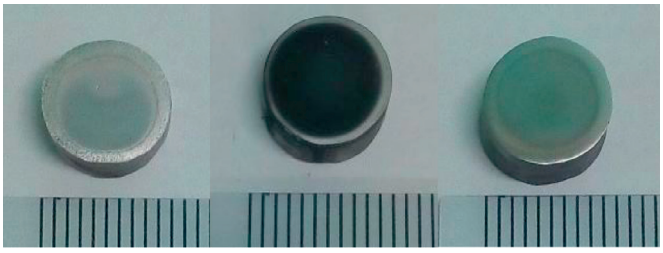


Fig. 3. Outer view of the samples after nitiriding.

Fig. 3 shows outer view of samples after nitriding.

The sample W-2 changed its color to black at the site of irradiation. This change is currently undergoing additional research.

3. Research results and discussion

X-ray diffraction phase qualitative and quantitative analysis in the automatic mode of a "HighScore" program was carried out. To identify phase composition of the sample via the "HighScore" program for processing and searching the Crystallography Open Database (COD) [8] was used.

Fig. 4 provides the range of diffractogram pictures, horizontally from 5° to 153°, vertically from 0 to 8000 impulses.

Fig. 5 provides the range of diffractogram pictures horizontally from 10° to 95°, vertically from 0 to 250 impulses.

At the diffractograms of W-1 and W-2 samples the lines of basic phase of tungsten and additional phases were revealed. Some of the peaks correspond to the lines of tungsten nitride in high-intensity area, but some lines of medium intensity are absent for the rest angular range.

For strict phase identification, the presence of all peaks is required. While the presence of tungsten oxide phase was identified, the pattern overlapping of one of the tungsten oxide phase on the diffractograms of W-1 and W-2 samples shows close agreement on lines with intensity up to 50%, but the agreement of two lines with intensity more than 50% in the angle range between 22°- 23° is absent. On the diffractogram of W-3 sample the absence of the lines corresponding to tungsten oxide as well as the presence of lines of tungsten nitride phase are indicated.

Thus, by the results of peak identification additional phases are revealed on the surface of W-1 and W-2 samples together with the basic phase of tungsten. The peaks of additional phases sufficiently match to the phases of tungsten nitride and tungsten oxide.

Table 2 provides a comparative analysis of accepted patterns with the results of semi-quantitative estimation of the mass fraction of the phase content [9].

A code of patterns is presented in the "Reference pattern code on COD" column according to the database of reference pattern of COD. The "Chemical formula and the title of compound" column contains a structural formula of reference pattern of the phase.

The column "Weight ratio" shows estimation data of semiquantitative estimation.

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