SIMULATING STUDY OF PLASMACHEMICAL EROSION OF A-C:H FILMS IN A ECR DISCHARGE PLASMA

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The dynamics of interference figure of the reflection spectrum for stainless steel and copper mirrors with specially deposited carbon-containing film (a-C:H) were studied when the film was gradually eroded under impact of a deuterium plasma produced in conditions of electron cyclotron resonance (ECR). The refraction coefficient of the film was estimated and the rate of the film removal was obtained.
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1.INTRODUCTION

For decreasing the influx of impurities into the plasma confinement volume, the wall conditioning procedures are in practice at every fusion device, with the use of different kind stationary or pulse discharges in hydrogen: glow, Ohmic, radio frequency (RF) in conditions of electron cyclotron resonance (ECR). The control for the state of wall surfaces is realized by means of special collectors, the surface of which is time to time analyzed with some standard methods. For providing surface analyses, the collectors are taken out from the vacuum vessel.

Recently it was suggested to control the quality of cleaning the walls of vacuum chamber of the stellarator type fusion device Uragan-2M by means of mirrors installed into the vacuum chamber. The idea of this suggestion is as follows. Before mirror installation, the carbon-containing film (i.e., a-C:H film) is deposited on its surface, and the *in situ* measurements of reflectance (e.g., at the He-Ne laser wavelength) during the conditioning procedure can give possibility to make conclusion about the cleaning efficiency. Or such measurements can be provided *ex situ* with taking samples out through a lock chamber. For evaluation of the prospect of this method, the modeling experiments with the use of ECR discharge in deuterium were provided.

2. EXPERIMENTAL DETAILS

The main procedures, when providing simulation experiments, were as follows:
i) deposition on metallic mirror of a carbon-containing film thick enough for obtaining an interference figure in reflection spectrum (λ=0.3…0.6 µm); ii) step-by-step sputtering of deposited film in plasma of ECR discharge; iii) *ex situ* measurements of reflectance; iv) monitoring of dynamics of interference figure in the course of gradual film erosion, with an aim to obtain an estimation of the film erosion rate.

Films were deposited in the device described in [1]. The stainless steel (SS) samples (carbide-forming material) and Cu samples (metal that does not form stable carbides [2]) were coated with a-C:H film in a non-self-maintained discharge in a propane-butane mixture at pressure 3×10^{-3} Torr. The mirror specimens (22×22 mm^2) were immersed into a flow of ions ejected from the hollow anode. On a check test piece the step of deposited material was formed which was used for measuring the deposited film thickness. The analysis showed that the film is amorphous; its thickness was measured by means of interference microscope (M I I-4). The error in the range of thickness of 0.3…0.6 µm is within 10%. Besides, the samples were weighted (with accuracy ± 50 µg) before and after film deposition for additional control of the film sputtering rate.

An erosion of the deposited film down to its full disappearance was provided in the DSM-2 stand [3], which is a simple double-mirror magnetic trap with a SS vacuum chamber pumped by a turbomolecular pump up to ~$2\text{x}10^{-6}$ Torr. Working gas deuterium was continuously flowing through the chamber at pressure $(2…4)\times10^{-4}$ Torr. ECR plasma was produced by UHF power ≈200 W at frequency 2.37 GHz. The electron temperature did not exceed 5 eV, and the plasma density in the place of mirror location was ~5×10^{9} cm^{-3}. No any voltage was applied to the mirror holder thus the ion energy was <15 eV. The gas composition was monitored before and during discharge. After every exposure in the stand, the sample was drawn out for weighing and measuring the reflectance.

For measurements of reflectance the scheme suggested by S.Tolansky [4] was used; it includes two stabilized light sources for UV and Visible ranges, the monochromator MDR-2 and a couple of gage units with photomultipliers. The reflectance of mirror samples, R(λ), at normal incidence was measured in the range 220…650 nm with the step 10 nm along the wavelength spectrum. The issued repeatability of measurements along a uniform surface was 1…1.5 %. The size of the irradiated spot Ø=2 mm (S=3 mm^2). Because of possible non-uniformity of the deposited film, very important is to fix the spot, what was assured by a special pattern. The processing of interferograms was provided with the use of a well-known relation $2d\cdot(n^2-\sin^2\varphi)^{0.5} - 0.5\lambda = m\lambda$, where d is a geometrical depth, n - refractive index, φ - angle of incidence, m = 0, 1, 2. During step-by-step sputtering, the number of interference periods for a fixed wavelength was tracked down to the full disappearance of the interference figure.

3. RESULTS

To work-out the method, the SS and Cu mirror samples were deposited with a-C:H films of approximately equal thickness. In Fig. 1, a and Fig. 1, c are shown the initial reflectance values and the ones just after deposition of the film of ~0.5 μm in thickness with typical interference figures. The film density was estimated as 2.06 g/cm^3. After plasma exposing during 185 min, the interference figures disappeared (Fig. 1, b and 1, d) and R(λ) became to be similar to the initial ones but at much lower level (open squares). With that, only ~10% of the initial film thickness remained on specimens according to weight loss measurements. Taking into account the accuracy of weight loss measurements, one can say that practically the whole film was removed. The mean removal rate was estimated to be 2.9 nm/min. However, the optical data indicate that a quite strongly absorbing film does still exist. For the full removal of the rest film the ion energy has to be increased. In the case of SS specimen the 50-minute exposure to ions with accelerating voltage -20 eV results in practically full restoration of reflectance (Fig. 1, b, triangles) but for the Cu specimen the rate of reflectance restoration was much slower, as Fig. 1, d demonstrates (triangles). Note, that Cu mirror specimens were chosen for this experiment because copper does not form stable carbides.

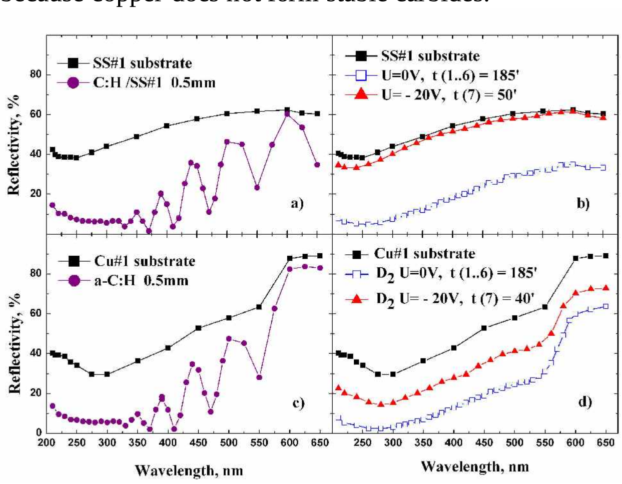

Fig. 1. a), c) - initial stage of R(λ) for SS and Cu samples; b), d) – pre-final stage (disappearance of the interference figure with total time equals 185 min (6 time-steps), U-biasing of mirror) and the final stage, i.e., return to the initial R(λ) stage of the substrate

After sample experiments the optimal characteristics of a-C:H films and the time for step-by-step film erosion were chosen. On the specimen SS #3 the film thickness was ~0.35 μm. The film density was as above, 2.06 g/cm^3.

On the assumption of the full visible disappearance of the interference figure and taking into account periodicity for every chosen wavelength 360, 440 and 540 nm the refraction coefficients of the deposited films were estimated: n ≈ 1.3 (accordingly: 1.3 -1.34 -1.29).

Similar processing of the interferograms for samples Cu #2 and SS #2 with initial film thickness 0.25 μm gave for wavelengths 340 , 420 and 550 nm, correspondingly, n = 1.43-1.45-1.43 and n = 1.47-1.47-1.47**,** and the rate of erosion 3.1-3.5 nm/min. These results were obtained by processing all interference figures for every specimen curves similar to those shown in Fig. 2.

According to [5], such n values indicate that the hydrogen content in the film is 40-60 %, i.e., the film is so called 'soft a-C:H film'.

At the same time, if additional exposure time for R(λ) restoration to the initial value (SS #3, Cu #2, SS #2) is taken into account, then applying similar procedure of data processing, one can obtain for the same wavelengths n ≈ 1.63-1.68. Such a rise of n can be connected with transformation (at the latest phase of sputtering) of the film into a 'hard a-C:H film' with lower hydrogen content, which has lower rate of erosion.

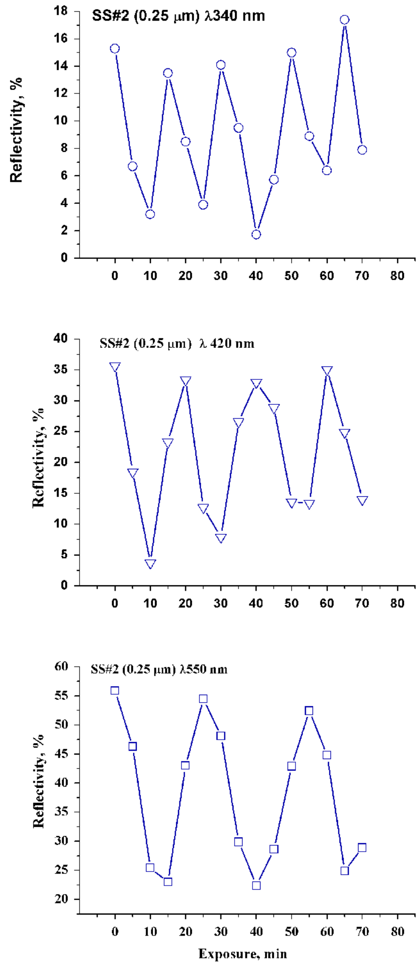

Fig. 2. Interchange of interference orders for λ = 340, 420 and 550 nm depending on the time of the film etching. (The total exposure time for the full disappearance of the interference figure is 70…80 min)

The rate of erosion of the film with initial thickness 0.35 and 0.25 μm was found ~3.3 nm/min on average. Taking into account the accuracy of weighing, the accuracy of film thickness measurement, the moment of disappearance of the interference figure, and peculiarities of the film deposition, one can consider that the rates of film removal, 2.9 (for 0.5 μm) and 3.3 nm/min for thinner films, are in a good agreement. Note, that during cleaning the mirrors in ECR plasma, the traces of water vapors (H_2O, D_2O, HDO) are present in discharge. This can intensify the effect of cleaning in comparison with pure deuterium plasma. However, similar situation will be probably met in the U-2M device during wall conditioning.

4. CONCLUSIONS

The experiments were provided with SS and Cu mirror specimens aiming a quantitative evaluation of the efficiency for the vacuum chamber walls to be cleaned from hydrocarbons by means of ECR discharge in hydrogen. The a-C:H films were deposited on SS mirrors, i.e. made from a carbide-forming material and on Cu mirrors – from material which does not form a stable carbides.

1. The dynamics of the shift of interference picture in the spectral reflectance was studied when the artificially deposited hydrocarbon film was gradually eroded from SS and Cu mirror specimens by plasmachemical treatment in deuterium plasma.
2. The rate of a-C:H film erosion was measured.
3. The refraction coefficient of the film in the range 340…550 nm was estimated.
4. It was found that after pronounced disappearance of the interference picture the rest of film can be taken off only with negative biasing of the specimen holder in the range 20…100 **V** for both SS and Cu specimens.

This fact indicates that:
(i) taking into account the behavior of R(λ) for SS and Cu samples at a completion phase of cleaning, the rest film is probably not the result of carbides formation, (ii) for effective cleaning of the vacuum chamber walls in U-2M it will be necessary to combine the low-ion-energy plasma (ECR plasma) with high-ion-energy plasma (e.g., glow or RF discharge plasma) during wall conditioning procedures.

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ИМИТАЦИОННЫЕ ИССЛЕДОВАНИЯ ПЛАЗМОХИМИЧЕСКОЙ ЭРОЗИИ А-С:Н ПЛЁНКИ В ПЛАЗМЕ ЭЦР-РАЗРЯДА

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Исследовалась динамика интерференционной картины спектра отражения стальных и медных зеркал с искусственно нанесённой углеродсодержащей плёнкой (a-C:H) по мере распыления в дейтериевой плазме ЭЦР-разряда. Произведена оценка коэффициента преломления плёнки. Получена количественная оценка скорости распыления.

ІМІТАЦІЙНІ ДОСЛІДЖЕННЯ ПЛАЗМОХІМІЧНОЇ ЕРОЗІЇ А-С:Н ПЛІВКИ В ПЛАЗМІ ЕЦР-РОЗРЯДУ

В.Г. Коновалов, М.М. Махов, І.В. Рижков, А.М. Шаповал, А.Ф. Штань, О.О. Скорик, С.І. Солодовченко, О.І. Тимошенко, В.С. Войценя

Досліджувалася динаміка інтерференційної картини спектру віддзеркалення сталевих і мідних дзеркал з штучно нанесеною вуглецьвмісною плівкою (a-C:H) по мірі розпилювання в дейтерієвій плазмі ЕЦР-розряду. Проведена оцінка коефіцієнта заломлення плівки. Отримана кількісна оцінка швидкості розпилювання.