MODIFICATION OF OPTICAL PROPERTIES OF TUNGSTEN EXPOSED TO LOW-ENERGY, HIGH FLUX DEUTERIUM PLASMA IONS

V.Kh. Alimov*[1]*, A.I. Belyaeva*[2]*, A.A. Galuza*[2]*, K. Isobe*[1]*, V.G. Konovalov*[3]*,
***A.A. Savchenko*[2]*, K.A. Slatin*[2]*, S.I. Solodovchenko*[3]*, V.S. Voitsenya*[3]*, T. Yamanishi*[1]**

[1]Tritium Technology Group, Japan Atomic Energy Agency, Tokai, Ibaraki, Japan;
[2]National Technical University "Kharkov Polytechnical Institute", Kharkov, Ukraine;
[3]Institute of Plasma Physics, NSC "Kharkov Institute of Physics and Technology", Kharkov, Ukraine
E-mail: voitseny@kipt.kharkov.ua; belyaeva@kharkov.com

Anomalous change of optical properties of recrystallized W caused by exposure to D plasma ions at sample temperature of ~535 K was studied by ellipsometry and reflectometry. There is a qualitative difference between the samples reflectivity values measured directly and calculated using ellipsometric data. A physical model of the phenomenon is suggested. It is shown that on the W surface exposed at ~535 K two processes take place 1) blistering and 2) modification of electron structure in the upper-most layer.

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1. INTRODUCTION

Due to its favorable physical properties, like low erosion yield and high melting temperature, tungsten (W) is foreseen as one of plasma-facing materials in fusion reactors, such as ITER [1] and DEMO [2]. Moreover, W is considered as a candidate for in-vessel mirror materials for optical diagnostic systems in ITER [3]. As a plasma-facing component, W mirrors will be subject to intense fluxes of low-energy (energy is below the displacement threshold) D and T particles including helium ash. This implantation process leads to concerns about a change of the optical properties after long-term hydrogen-helium plasma exposure. The effect of irradiation with low-energy H and He ions on the optical reflectivity of W mirrors was investigated [4]; and the degree of the optical reflectivity degradation was demonstrated to depend on type of ions, irradiation temperature, ion energy, ion flux, as well as on the properties of material itself.

Blistering is one of phenomena influencing the optical properties of W mirrors. There is evidence of blistering occurred on tungsten surface exposed to hydrogen plasmas with ion energies much below the displacement threshold [5, 6].

Significant temperature dependence of surface topography was found for re-crystallized W exposed to low-energy (38 eV/D), high-flux (10^{22} D/m^{2}s) D plasma ions up to ion fluence 10^{26} D/m^2 [6]. After plasma exposure at temperatures, T_{exp}, in the range from 320 to 400 K, only sparse blisters with diameters of 0.5…2 μm are formed on the W surface. As the exposure temperature increases, the blisters become much denser. Two kinds of blisters appear after exposure at T_{exp} = 520…570 K: large low-dome blisters with sizes of 10…30 μm and small cone-shaped blisters with diameters of less than a few μm. No blisters appear at T > 700 K.

This work focuses on study of the temperature dependence of the optical characteristics of recrystallized W mirror exposed to the low-energy, high flux D plasma ions.

2. EXPERIMENTAL

Plates of polycrystalline tungsten (A.L.M.T. Corp., Japan), 10×10×2 mm^3 in size, fully recrystallized in hydrogen (protium) atmosphere at 2073 K after cutting and polishing, with a purity of 99.99 wt%, were used as W mirror samples. The linear plasma generator (JAEA, Tokai, Japan) used for delivering plasma beam comparable to the edge plasma in ITER divertor is described elsewhere [7].

The samples numbers exposed at corresponding temperature are shown in the Table for convenience.

Temperature of the samples while exposure

Sample #	W42	W29	W30	W43	W35	W20
T_{exp}, K	320	405	483	535	600	695

The optical measurements included: (i) direct measuring a specular reflectance *R* at normal incidence of the light [8], and (2) measuring the optical constants *n* and *k* by means of ellipsometry [9]. In the second case, measurements of ellipsometry parameters (Ψ and Δ) at different incidence angle at fixed wavelength (633 nm) were used [10]. The apparent reflectance at normal incidence, R, as calculated from the ellipsometric data is a good measure of the real normal-incidence reflectance. Surface pictures and the surface relief parameters were obtained with the use of an interferometric microscope [11].

3. RESULTS

3.1. MICROSCOPY AND INTERFEROMETRY DATA

Optical microscopy of the samples irradiated at different temperatures clearly shows distinctive feature of the surface of the specimen W43 (535 K) (Fig. 1). An interference image of clean (without blisters) parts of sample surface consists of straight-line interference strips.

This firstly indicates that all samples maintained a smooth surface, and secondly shows the lack of sputtering which would lead to formation of a step structure [12].

The cone-shaped blisters in overwhelming majority are of 1…5 μm in diameter (Fig. 1). The plane blisters are significantly larger in size (7…12 μm).

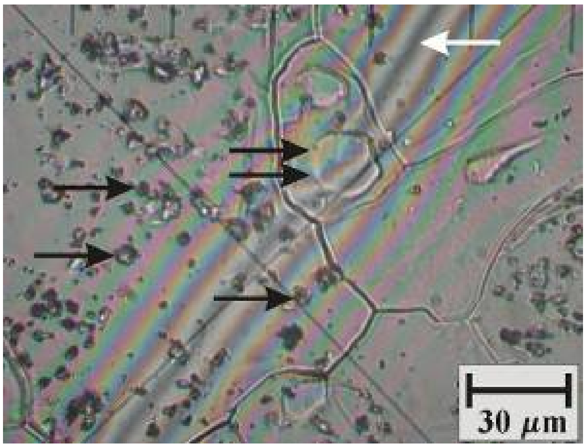

Fig. 1. W43 interference picture (single arrows - small blisters, double arrow - large ones). White arrow indicates interference minimum stripe

3.2. ELLIPSOMETRIC AND REFLECTOMETRIC DATA

The ellipsometric parameters Ψ and Δ were measured for all exposed samples and the unexposed one – to know the initial state of the mirror samples. To analyze the samples surface various models were used and the bare surface model turned out to be optimal. Fig. 2 shows dependences of the samples optical constants *n* and *k* calculated within this model on exposure temperature.

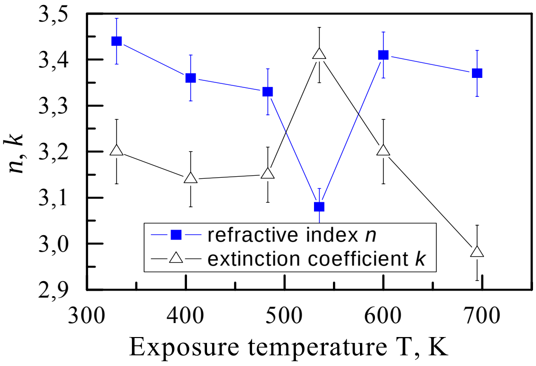

Fig. 2. Optical constants n and k of recrystallized W mirror as a function of the exposure temperature

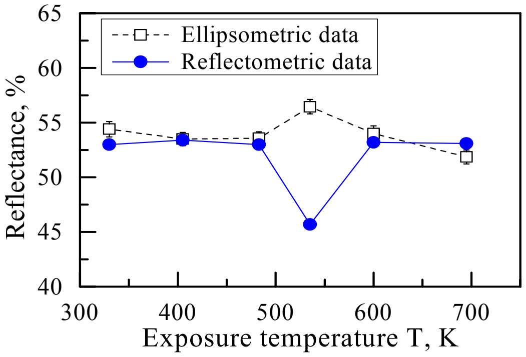

Fig. 3. Normal incidence reflectance of recrystallized W as a function of the exposure temperature (measured directly and calculated from ellipsometric data)

One can see the optical constants "fall-out" for the W exposed at T_{exp} = 535 K: the refractive index *n* decreases and the extinction coefficient *k* increases. Using these indices, the reflection at normal incidence, R, was calculated. In Fig. 3 these calculated R values are compared with the values directly measured at the same wavelength for normal incidence of light. Importantly to pay attention on quantitative difference between R for W43 specimen directly measured (fall down of R) and found using ellipsometric data (rise of R).

4. DISCUSSION

Fig. 1 demonstrates that blistering is grain-dependent, i.e., some grains are almost free from blisters but others are almost fully covered with blisters. This is probably connected with the fact that on the W surface the grains with orientation (111) are most subjected to blistering in comparison with grains of other orientations [5]. The experimental conditions [5] were very similar to ours. Thus, there is a probability that optical indices of the smooth areas of this particular specimen can be in some degree different from those characteristics for the W surface with blisters.

Now we have to analyze the principal difference between ellipsometrically estimated and directly measured values of reflectance (see Fig. 3). It is worthy to emphasize that ellipsometry and reflectometry are based on different physical effects. Reflectometry of the specular reflection measures the full energy specularly reflected from the surface. Correspondingly, even small defects of the surface result in increase of scattering and corresponding decrease of specular reflection. Ellipsometry is based on the study of changing the polarization state of only specular component of the probing radiation, and therefore brings information about specularly reflecting parts of the surface.

The situation is possible when, with a developed relief, a significant portion of the scattered light can also get to the detector as the result of complex re-reflections (Fig. 4, a). With that, the reflecting light is depolarized and thus the full darkening (when null-method is used) does not occur [13]. In our case during ellipsometry measurement of the W43 specimen, practically total blanking of reflected light took place what authenticates the lack of depolarization and negligible contribution of light scattered by defects (Fig. 4, b).

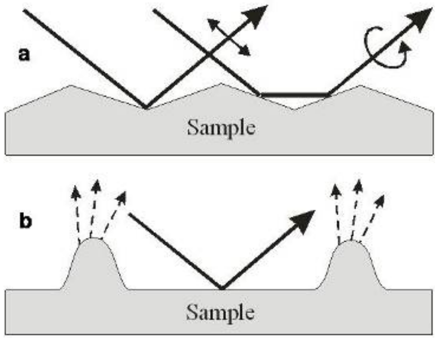

Fig. 4. Light scattering from surfaces with different structures: a) specular reflection and depolarization; b) specular reflection and scattering without depolarization of specular component

The light scattered due to blistering results in decrease of the specular reflection, thus leading to decrease of reflectance measured by reflectometry (see Fig. 3).

The increase of reflectance found by ellipsometry (see Fig. 3) indicates that the properties of the W43 specimen surface do change not only due to blistering but also because of modification of the electronic structure of those surface areas which are not affected by blistering; the ellipsometry gave the optical indices for just these parts of surface.

A small rise of reflectance (see Fig. 3) found by ellipsometry can be connected with either increase of extinction coefficient observed (see Fig. 2) or formation of a quasifilm on the W43 surface. For better interpretation of ellipsometric data, several models of W43 surface were checked. It was ascertained that the bare surface model is the optimal one. It means that the thickness of the modified near-surface layer exceeds the penetration depth of light or this layer has no sharp boundary and thus cannot act as a film. Therefore, it was concluded that an increase of R in Fig. 3 is due to significant rise of the extinction coefficient seen in Fig. 2.

5. CONCLUSIONS

The optical properties of W mirror specimens exposed to low-energy, high flux D plasma ions at various temperatures were examined. The sharp change of reflectance was found for the specimen exposed at 535 K (W43).

Summarizing results obtained, we can conclude that two processes are realized on the surface of the W43 specimen: (i) blistering and (ii) modification of the electronic structure in the upper-most layer.

So, evidently ellipsometry and reflectometry supplement each other effectively when such complicated phenomenon as blistering occurs on the surface.

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МОДИФИКАЦИЯ ОПТИЧЕСКИХ СВОЙСТВ РЕКРИСТАЛЛИЗОВАННОГО ВОЛЬФРАМА, ОБЛУЧЕННОГО НИЗКОЭНЕРГЕТИЧЕСКИМИ ИОНАМИ ДЕЙТЕРИЕВОЙ ПЛАЗМЫ

В.Х. Алимов, А.И. Беляева, А.А. Галуза, K. Isobe, В.Г. Коновалов, А.А. Савченко, К.А. Слатин, С.И. Солодовченко, В.С. Войценя, T. Yamanishi

Методами эллипсометрии и рефлектометрии обнаружено аномальное изменение оптических свойств рекристаллизованного W в результате бомбардировки ионами D при температуре ~535 К. Имеет место принципиальное отличие между значениями коэффициента отражения, измеренными рефлектометрией и рассчитанными по данным эллипсометрии. Предложена физическая модель обнаруженного эффекта. Показано, что на поверхности W, облученного при 535 К, имеют место два процесса: 1) блистеринг и 2) модификация электронной структуры поверхностного слоя.

МОДИФІКАЦІЯ ОПТИЧНИХ ВЛАСТИВОСТЕЙ ВОЛЬФРАМУ В НАСЛІДОК ОПРОМІНЕННЯ ВЕЛИКИМ ПОТОКОМ НИЗЬКОЕНЕРГЕТИЧНИХ ІОНІВ ДЕЙТЕРІЄВОЇ ПЛАЗМИ

В.Х. Алімов, А.І. Беляєва, О.А. Галуза, K. Isobe, В.Г. Коновалов, А.О. Савченко, К.О. Слатін, С.І. Солодовченко, В.С. Войценя, T. Yamanishi

Методами еліпсометрії та рефлектометрії виявлено аномальне змінення оптичних властивостей рекристалізованого W внаслідок бомбардування іонами D при температурі ~535 К. Існує принципова різниця між значеннями коефіцієнту відбиття, що отримано рефлектометрією та розраховано за даними еліпсометрії. Запропоновано фізичну модель виявленого ефекту. Показано, що на поверхні W, що опромінено при 535 К, мають місце два процеси: 1) блістерінг и 2) модифікація електронної структури поверхневого шару.