

# THE OPTICAL CHARACTERISTICS OF CONTAMINATING FILMS ON Mo, SS AND Cu MIRROR SAMPLES EXPOSED IN PLASMA DEVICES

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The optical methods with the use of in-vessel first mirrors (FMs) are expected to occupy an important place in the diagnostic complex of the future fusion reactor ITER. As the surface of FMs will be subjected to physical sputtering by high-energy D and T charge-exchange atoms, this can lead to a decline in the initial FM reflectance. The second factor of FM reflectance deterioration is the redeposition of contaminants on the mirror surface. In the paper, optical indices and thickness values of the films deposited on Mo and SS mirrors in the tokamak TRIAM-1M as well as on SS and Cu mirrors in the torsatron URAGAN-3M were studied by ellipsometry at the wavelength  $\lambda=632.8$  nm. The spectral reflectance at normal incidence of light for all the samples was measured in the wavelength range  $\lambda=253-650$  nm. The optical parameters of TRIAM-1M films were determined as the film was progressively removed by deuterium plasma ions in the stand DSM-2.

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## 1. INTRODUCTION

The future fusion reactor (ITER) is to be supplied with a diagnostic complex, where the extensive use will be made of optical methods with first mirrors (FM) as the most responsible elements. The FM surface can be eroded due to physical sputtering by charge-exchange atoms (D and T) of mean energies up to several hundreds of eV, that diminishing the initial reflectance of FM. This paper is concerned with the second factor of the reflectance deterioration, namely, the occurrence of a contaminating film on the FM far from the plasma [1]. The film is a consequence of redeposition of eroded material inside the vacuum vessel or volatile impurities coming close to the FM. The optical characteristics of films on Mo, SS (stainless steel) samples of the mirrors exposed in the tokamak TRIAM-1M (Japan), and also of the films on SS, Cu samples exposed in the torsatron URAGAN-3M (Ukraine) were investigated along with the studies of the most probable places of films deposition.

## 2. PROCEDURES AND MEASUREMENTS

The basic device for experiments with mirror samples is the stand DSM-2 with low-temperature deuterium plasma created in a stationary ECR discharge [2]. The procedure of gradual removal of the film from the sample by positive ions of plasma was used. The ion energy  $\epsilon_i$  was fixed in the range 15–200 eV. The ions are accelerated to the sample by the negative potential applied to the sample holder in the stand. The ions attain the energy  $\epsilon_i \approx 15$  eV at the floating potential of the holder. The dimensions of samples were  $22 \times 22 \times 4$  mm. The ion current density to the sample was 0.3–2.0 mA/cm<sup>2</sup>, the duration of one stage of film removal varied from 3 to 20 min.

The spectral reflectance  $R_m(\lambda)$  of the samples was measured at normal incidence of light in the wavelength range  $\lambda=253-650$  nm. The film thickness  $d$  and the complex optical index  $\hat{n}_L = n - ik$  at  $\lambda=632.8$  nm were found using the ellipsometer LEF-3-M1 [1]. The optical para-

eters were measured *ex-situ* at the following moments. 1) After removal of the residual film from the sample, before its placement into the vessel (initial level). The indices  $\hat{n}_{LS}$  of all cleaned samples were determined at  $\lambda=632.8$  nm. 2) After film deposition on the sample in the device and the extraction of the sample (exposed level, or stage #0). 3) After a single-stage complete removal or after each stage of step-by-step removal of the film in DSM-2.

For the samples from TRIAM-1M the optical parameters  $R_m(\lambda)$ ,  $d$ ,  $\hat{n}_L$ ,  $\hat{n}_{LS}$  were measured at the initial and exposed levels, and also, after each stage of the step-by-step removal. For the samples from URAGAN-3M, the same parameters were measured at the initial, exposed levels and after the single-stage removal.

## 3. THE FILMS DEPOSITED IN TRIAM-1M

In the tokamak TRIAM-1M ( $R_0=0.84$  m,  $a \times b=0.12 \times 0.18$  m,  $B=6-7$  T,  $P_{RF}=10-300$  kW,  $n_e=(0.1-4.0) \times 10^{19}$  m<sup>-3</sup> [3]) the contaminating films were deposited onto one Mo sample and onto three out of four SS samples exposed in the vacuum vessel, in one experimental campaign.

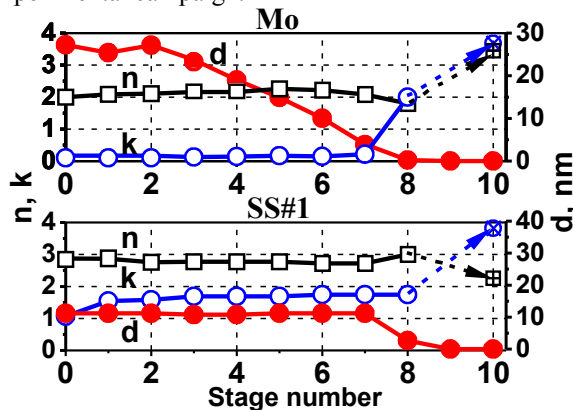


Fig. 1. Film removal from Mo and SS#1 samples. The arrowed dashed lines are drawn to the indices of the clean mirror

The film removal from the Mo sample as well as the reflectance  $R_m(\lambda)$  recovery were not attained at  $\varepsilon_i \approx 15$  eV. They occurred at  $\varepsilon_i = 60$  eV, starting with stage #3, as is shown in Fig. 1. The saturation of  $R_m(\lambda)$  began from stage #8. The closest approach to the initial  $R_m(\lambda)$  and to the index  $\hat{n}_{1,S}$  of the clean sample was achieved at stage #10. At stages # 0 to 7 the average index of the film on the Mo sample was  $\hat{n}_{L,av} = 2.15 - i0.14$ , which was close to the index  $\hat{n}_1 = 2.02 - i0.08$  of a-C:H film [4]. By analogy with the Mo sample, the cleanings of other samples were performed (Tables 1 and 2). The sample SS#2 had traces of a film, which were easily removed. The films on samples SS#1 and SS#3 could be removed completely only at  $\varepsilon_i = 200$  eV, and on SS#4 – at  $\varepsilon_i = 100$  eV, because the films were more resistant, than on the Mo sample, for which  $\varepsilon_i = 60$  eV was sufficient for film removal. The films on samples SS#1, #3 and #4 had atypical higher extinction coefficients  $k$  than those of a-C or a-C:H films [4].

Table 1. Stage numbers (#), at which the reflectance  $R_m(t)$ : S – starts to recover, H – saturates almost up to the initial value, I – becomes the closest to initial. The energy  $\varepsilon_i$  (eV) is given in parentheses

Sample	S	H	I
Mo	#2 (60)	#8 (60)	#10 (60)
SS#1	#7 (100)	#9 (200)	# 10–12 (200)
SS#2	#1 (~15)	#1 (~15)	#2 (~15)
SS#3	#5 (100)	#7 (200)	#8 (200)
SS#4	#1 (~15)	# 9–12 (100)	#13 (100)

Table 2. The film parameters:  $d$  – maximal thickness,  $\hat{n}_{L,av}$  – averaged index,  $t$  – time of complete removal. The averaging stages are shown in parentheses

Sample	$d$ , nm	$\hat{n}_{L,av}$	$t$ , min
Mo	27	2.15–i0.14 (# 0–7)	60
SS#1	11	2.80–i1.60 (# 0–8)	60
SS#2	traces	almost clean SS	6
SS#3	5	2.65–i2.50 (# 0–6)	29
SS#4	16	2.22–i1.90 (# 0–8)	68

#### 4. THE FILMS DEPOSITED IN URAGAN-3M

At pulse operation in the torsatron URAGAN-3M with hydrogen plasma ( $l=3$ ,  $m=9$ ,  $R_0=1$  m,  $\bar{a} \approx 0.12$  m,  $B=0.7$  T), the line-averaged electron density  $\bar{n}_e$  attains  $\sim 10^{19}$  m $^{-3}$ , the electron temperature in the plasma core  $T_e(0)$  does not exceed 300–400 eV [5]. The SS and Cu samples were regularly exposed in operational and cleaning regimes, during annual experimental campaigns (2002–2007). The samples were installed on the helical coils and sidewall flanges of the diagnostic ports. The top view of the vessel for all campaigns is shown in Fig. 2.

The thickest opaque film having  $\hat{n}_1 = 1.88 - i0.8$ ,  $d \geq 200$  nm was deposited in 2002 on the SS sample placed horizontally on the coil, at a distance of  $\sim 0.1$  m, ccw from RF antenna №1 (Fig. 2). In 2004, only samples SS#1, SS#2, Cu#2, Cu#4 showed a decrease in the reflectance  $R_m(\lambda)$ , but the other 8 samples kept it up (Fig. 2). Sample Cu#4 was placed on the coil in section “B4”, at a distance of  $\sim 0.09$  m, ccw from RF antenna №2, at a distance of  $L_{SX} \sim 0.1$  m from the plasma separatrix. The sample became coated with a strongly absorbing film having  $\hat{n}_1 = 2.14 - i0.38$ ,  $d \approx 24$  nm. As a result, after the exposure the reflectance dropped significantly, e.g., the decrement

amounts to  $\Delta R_1 = -13.6\%$  at  $\lambda = 300$  nm and  $\Delta R_2 = -34.5\%$  at  $\lambda = 632.8$  nm, as is shown in Fig. 3. The values of deposited film thickness were maximal near the both antennas.

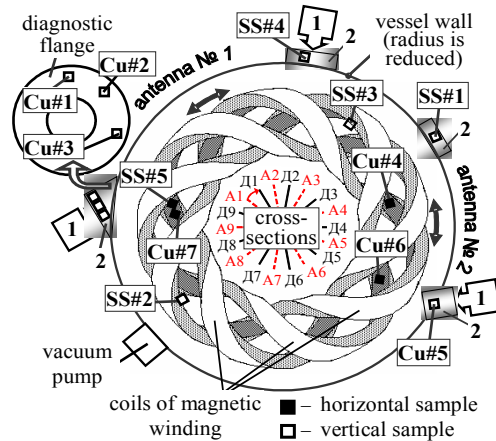


Fig. 2. Positions of samples SS # 1–5 and Cu # 1–7 in URAGAN-3M in 2004 (top view); 1 – monochromator, 2 – diagnostic port with a window

The sample SS#1 was fixed vertically on the flange in section “E3”, at a distance of  $L_{SX} \approx 1.5$  m. It was coated with a thin, weakly absorbing film, having  $\hat{n}_1 = 2.22 - i0.08$ ,  $d \approx 11$  nm. The decrements of reflectance for this sample were  $\Delta R_1 = -19.5\%$ ,  $\Delta R_2 = -5.9\%$  (Fig. 3). For samples SS#2 and Cu#2 the decrement values  $\Delta R_1 = -3.8\%$ ,  $\Delta R_2 = -4.4\%$  and  $\Delta R_1 = -4.7\%$ ,  $\Delta R_2 = -7.9\%$ , correspondingly, were probably the result of an apparent layer, which had appeared after bombardment with high-energy charge-exchange atoms from plasma. The former sample was fixed vertically on the coil in section “A8”, with  $L_{SX} \sim 0.1$  m, and the latter one was fixed vertically on the flange in section “B9”, with  $L_{SX} \approx 1.5$  m.

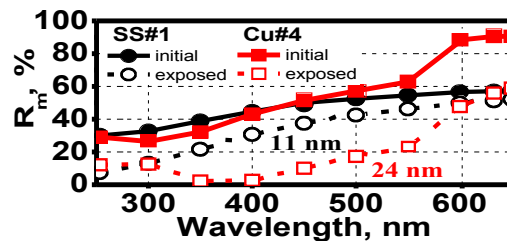


Fig. 3. The reflectance  $R_m(\lambda)$  initially and after the exposure of samples SS#1 and Cu#4 (2004). The film thickness is given near the curve

In 2005, only two out of five Cu samples and none of three SS samples were coated with films. The reflectance of sample Cu#6 (in position of Cu#4, 2004) changed moderately, by  $\Delta R_1 = -6.7\%$  and  $\Delta R_2 = -3.4\%$ , because the deposited film was absorbing,  $\hat{n}_1 = 1.88 - i0.33$ , and thin,  $d = 6$  nm. The reflectance at  $\lambda = 632.8$  nm of sample Cu#4 (in position of SS#2, 2004) changed strongly in ultra-

violet, by  $\Delta R_1 = -15.1\%$ , and moderately in visible, by  $\Delta R_2 = -1.4\%$ , due to deposition of a transparent thin film,  $n_1 = 2.61 - i0.00$ ,  $d = 5$  nm. In 2006, very thin films were deposited. The growth rate of the films in 2002–2007 went down from year to year.

## 5. CONCLUSIONS

One Mo sample and three out of four SS samples exposed in the tokamak TRIAM-1M showed a decrease in the spectral reflectance because of deposition of absorbing contaminating films. The thickness and indices of the films were determined at  $\lambda = 632.8$  nm through step-by-step cleanings. The films on three SS samples had high coefficients  $k$ , higher than  $k$  of a-C film [4]. Probably, the absorbing metal atoms implanted into the film were dominant in the contaminating flow to the SS mirrors, because the in-vessel structures are metallic (SS and Mo) [3]. The film on the Mo sample was weakly absorbing, most likely due to remoteness from metal sources.

The SS and Cu mirror samples were exposed in the torsatron URAGAN-3M every year, during experimental campaigns of 2002–2007. The thickness and optical indices of the films on the samples, for which the spectral reflectance has decreased, were measured at  $\lambda = 632.8$  nm. All the films deposited on samples in campaigns had unequal thickness values. The thickest absorptive or even opaque films were deposited on the samples near the RF

antennas. The majority of indices of the films were close to those of a-C:H films [4]. The cleanest places were inside the diagnostic ports of optical spectroscopy. The film growth rate decreased every year due to the improvement of vacuum regime.

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## ОПТИЧЕСКИЕ ХАРАКТЕРИСТИКИ ЗАГРЯЗНЯЮЩИХ ПЛЕНОК НА ОБРАЗЦАХ Мо, SS и Cu ЗЕРКАЛ, ЭКСПОНИРОВАННЫХ В УСТАНОВКАХ С ПЛАЗМОЙ

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Оптические методы с использованием внутрикамерных первых зеркал (ПЗ) будут занимать важное место в диагностическом комплексе будущего реактора синтеза ИТЕР. Так как поверхность ПЗ будет подвергаться физическому распылению высокоэнергетическим D и T атомами перезарядки, это может привести к снижению исходного коэффициента отражения (КО). Вторым фактором ухудшения КО будет переосаждение загрязнителей на поверхность зеркала. Для пленок, осажденных на Mo и SS зеркала в токамаке TRIAM-1M, а также на SS и Cu зеркала в торсатроне УРАГАН-3М методом эллипсометрии изучались толщины и константы на длине волны  $\lambda = 632.8$  нм. Спектральный КО всех образцов при нормальном падении света был измерен в диапазоне  $\lambda = 253-650$  нм. Оптические параметры пленок из TRIAM-1M были определены на стадиях постепенного удаления пленки ионами дейтериевой плазмы в стенде ДСМ-2.

## ОПТИЧНІ ХАРАКТЕРИСТИКИ ЗАБРУДНЮЮЧИХ ПЛІВОК НА ЗРАЗКАХ Мо, SS і Cu ДЗЕРКАЛ, ЕКСПОНОВАНИХ В УСТАНОВКАХ З ПЛАЗМОЮ

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Оптичні методи з використанням внутрікамерних перших дзеркал (ПЗ) будуть займати важливе місце в діагностичному комплексі майбутнього реактора синтезу ИТЕР. Тому що поверхня ПЗ буде піддаватися фізичному розпиленню високоенергетичними атомами перезарядження D і T, це може привести до зниження початкового коефіцієнта відбиття (КВ). Другим фактором погіршення КВ буде переосадження забруднювачів на поверхню дзеркала. Для плівок, осаджених на Mo і SS дзеркала в токамаку TRIAM-1M, а також на SS і Cu дзеркала в торсатроні УРАГАН-3М, методом еліпсометрії вивчалися товщини і константи на довжині хвилі  $\lambda = 632.8$  нм. Спектральний КВ усіх зразків при нормальному падінні світла був вимірний у діапазоні  $\lambda = 253-650$  нм. Оптичні параметри плівок із TRIAM-1M були визначені на стадіях поступового видалення плівки іонами дейтерієвої плазми в стенді ДСМ-2.