ISSN 1560-8034, 1605-6582 (On-line), SPQEO, 2019. V. 22, N 4. P. 444-451.

Effect of electron-beam treatment of sensor glass substrates for SPR devices on their metrological characteristics

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Abstract. Electron-beam treatment the glass substrates for sensitive elements of SPR devices causes almost two-fold narrowing their refractometric characteristics from 0.867 down to 0.453 deg. The angular shift was also changed, which made the measuring range wider by 0.37 deg. The sensitivity of SPR devices increased by 1.7 times from 1.425 up to 2.396 deg^{-1} as a consequence of lowering the energy expenses during propagation of surface plasmons along the boundary “metal–air”. The reason for this lowering is related to higher surface uniformity of the gold metal film, its higher density as well as lower nano-roughness of the glass surface and the thickness of heterointerface “gold–air”. In this case, the dispersion value for unevenness heights on the surface relatively to the base line was lowered from ±18 down to ±5 nm, mean-square roughness was three-fold reduced from 4.67 down to 1.64 nm, and the thickness of heterointerface gold–air was lowered from 3.26 down to 1.37 nm. It was ascertained using X-ray reflectometry that the film density increased from 17.2 up to 19.3 g/cm^3 and reached the value typical for the bulk gold. It provided the changes in the refraction index and extinction coefficient of the gold film, which was ascertained using the ellipsometric method. Thus, the performed analysis of refractometric characteristics showed that electron-beam treatment the glass substrates of sensitive elements for SPR devices is able to efficiently enhance their sensitivity and to widen the range of measured resonance SPR angles.

Keywords: surface plasmon resonance, sensitivity, electron-beam processing, ellipsometry, atomic-force microscopy, X-ray reflectometry.

https://doi.org/10.15407/spqeo22.04.444
PACS 73.20.Mr

Manuscript received 22.08.19; revised version received 26.09.19; accepted for publication 29.10.19; published online 08.11.19.

1. Introduction

One of the promising optical methods enabling to analyze various compounds and micro-objects as well as processes at the molecular level is the refractometric method based on the phenomenon of surface plasmon resonance (SPR). The weighty advantages of this method, as compared with the traditional diagnostic methods (*e.g.*, immune-enzyme analysis, immunodiffusion test, polymerase chain reaction) are the following ones: possibility to study the processes of molecular interaction in nano-dimensional layers in a real-time scale; small sample volume of the studied substance (less than 10 μL); absence of necessity to use markers or fluorescent labels for the studied substance (analyte) [1]. The devices based on SPR are mostly equipped with film nano-dimensional sensors that consist of a sensitive element and physical transducer. As a plasmon-carrying layer of the sensitive element in SPR devices with the prism method to excite surface plasmons, there used metals with high electrical conductance, mainly it is gold deposited onto a glass substrate.

Surface roughness of metal essentially influences on propagation of surface plasmons and causes (through the scattering of energy) premature decay of plasmons and lowering their phase velocity [2]. So, technology of producing the metal layer for sensitive elements plays an important role. To reduce the absolute error in the analyte refraction index and enhance the sensitivity, it is necessary to lower roughness of the metal layer surface in the sensitive element. For example, lowering the mean-square surface roughness from 2 down to 0.8 nm, when depositing the metal layer under the angle 45° to the glass substrate causes 1.5 times increase in the sensor sensitivity in the analysis of liquid substances and 2 times in analyses of gas-like substances [3]. The main factor influencing on the structure and properties as well as on nanoroughness of metal layers prepared using thermal evaporation in vacuum is effect of the substrate nanorelief. To reduce this effect on the surface of the deposited metal layers, the substrate surface is usually prepared using the traditional optical technology applied in production of optical parts [4, 5]. The main problem in this case is preparation of substrates for SPR devices with the surface that is qualitatively polished, defectless and homogeneous in its chemical composition.

Applied in optical industry are the methods of mechanical, chemical, chemical-and-mechanical polishing, which can not provide avoiding the surface defects. The latter, being the reason for surface optical nonuniformity, cause losses of electromagnetic wave energy and birefringence. The methods of chemical and chemico-mechanical polishing introduce chemical changes into chemical composition of the processed surface. Traditional technologies of optical production do not allow preparation of optical surfaces meeting the requirements of the International standard ISO 10110-1/14, namely: to provide creation of surfaces with the formed on them defectless, chemically and optically homogeneous surface layers characterized by guaranteed typical micro-inhomogeneities less than 5 nm [6].

However, there is a promising method for obtaining the qualitatively polished surface – it is the electron beam treatment of surfaces, which provides ecological cleanness, simplicity in realization, possibility to control the respective technological process [7, 8]. Investigation of goods after e-beam treatment shows that the mean-square surface roughness is close to R_q = 3...5 nm, standard deviation of optically controlled surface is close to N = 1…2, residual thermotension does not exceed 0.2…0.4 MPa. These measures entirely correspond to the requirements of SSU ISO 10110-1/14 [6]. It is established in the work [9] that after e-beam treatment the mean-square surface roughness for the glasses К8, К108 is reduced from 4…9 down to 1.5…2.2 nm. After processing the elements from optical glass, there takes place the noticable increase in the value of spectral transmission coefficient in the infra-red range as a result of decreasing the amount and sizes of unwanted defects on the surface and in subsurface layers due to their melting. As a consequence, there appear some improvement of such important properties of optical elements as cleanness of their surface and the value of nanoroughness. Besides, homogenization of surface layers in the elements of optical glass occurs [8]. Therefore, the method of e-beam processing the surface of glass substrates used in SPR devices is very promising, if taking into account the reduced nanoroughness of the glass surface and improvement of refractometric characteristics inherent to devices based on the SPR phenomenon.

Since the nanoroughness of substrates plays an important role in formation of the metal film in SPR devices, in which surface plasmons are excited, it seems reasonable to study the influence of e-beam treatment on optical characteristics of the metal film, its density and surface micro-roughness. In this work, the authors performed complex investigations aimed at the effect of e-beam treatment the surface of glass substrates for SPR devices on their refractometric characteristics. In doing so, the authors involve the following experimental methods: atomic-force microscopy (AFM), ellipsometry and X-ray reflectometry (XRR), The AFM method is intended for measuring and analyzing micro- and sub-micrometer surface relief with a high resolution of objects in the micro- and nanometer scales [10]. The following widely used method for investigation of the optical parts surface is ellipsometry that is based on measuring the characteristics of polarized light reflected from the surface [11]. The XRR method is based on the total internal reflection of X-rays from the sample surface. The high sensitivity to slight changes in the electron density is one of the advantages of this method [12].

2. Materials and methods

We investigated four sensitive elements for SPR devices with different specific powers of e-beam treatment or without it. Two of them were irradiated with the power 27.84 W/mm^2 (sample 1) and 36.62 W/mm^2 (sample 2), the rest samples 3 and 4 were not irradiated. The substrates of sensitive elements were made of optical glass Ф1 with the refraction index n_D = 1.5141 ± 0.0001. These substrates were thin glass plates with the overall sizes 1×20×20 mm with a possible deviation ± 0.1 mm. The surfaces of plates were polished mechanically, which provided their mean-square surface roughness R_q = 3.18 ± 2 nm. After mechanical and e-beam treatments, one of the substrate sides was covered with the gold layer of the thickness 48 ± 4 nm by using thermal evaporation in vacuum with the installation ВУП-5М. During deposition, the thickness of the gold layer was controlled using the quartz microbalance device КИТ-1. Before deposition of the metal gold film, the glass substrates were cleaned in the ultrasound bath containing the mixture of solutions of hydrogen peroxide and ammonium hydrate in the relationship 1:1. After cleaning the surface, the samples were washed out with distilled water and dried in air flow.

E-beam treatment of glass samples was performed using the equipment based on the multi-purpose installation УВН-74П3. Mounted in the vacuum chamber of it were: a silica infrared oven providing preliminary heating and final cooling, Pierce electron gun with a special optics to form a strip electron flow (accelerating voltage 1…12 kV, cathode heater current 10…30 A), mechanism for transferring optical elements. With the aim to avoid thermotensions in surface layers caused by temperature drops that occur under action of e-beam treatment in glass elements, the samples were heated up to the temperatures 700…800 K [8] before and after treatment.

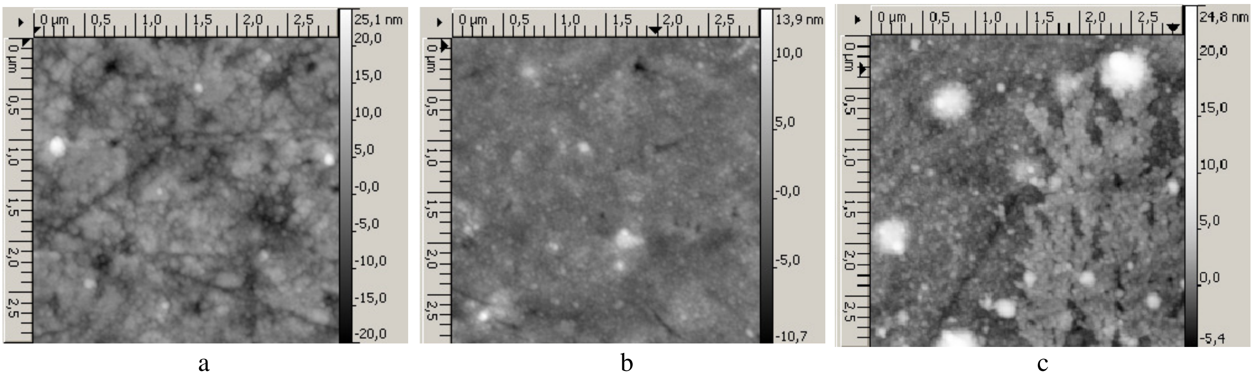

Fig. 1. AFM images of sample surfaces: without e-beam treatment – sample 3 (a); treated with different specific powers – 27.84 W/mm^2, sample 1 (b) and 36.62 W/mm^2, sample 2 (c).

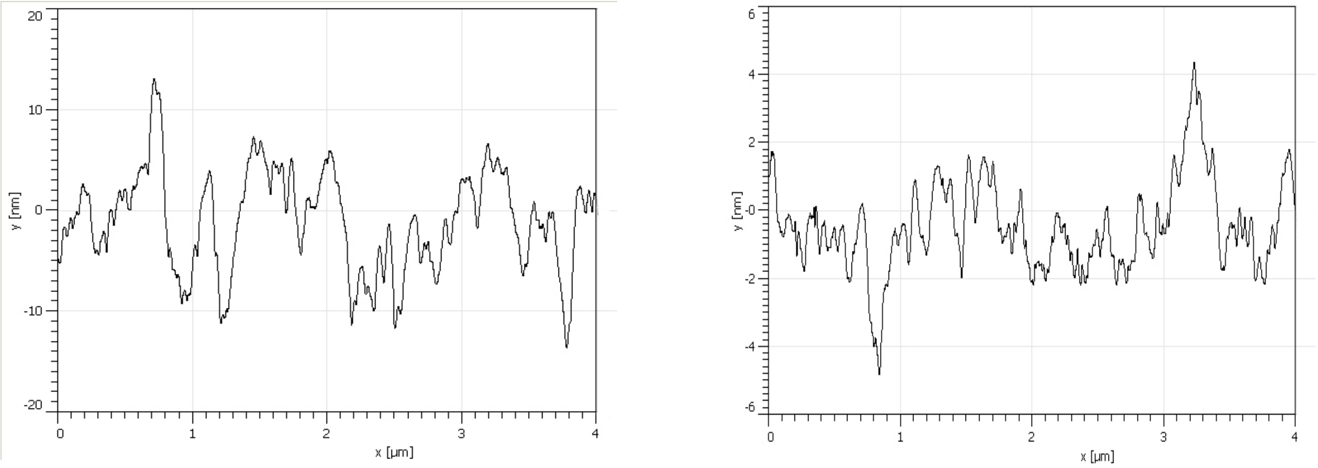

Fig. 2. Height profiles of studied surfaces: without e-beam treatment – sample 3 (a), with treatment by the specific power 36.62 W/mm^2 – sample 2 (b).

The surfaces of samples were studied using AFM, ellipsometry and X-ray reflectometry. The refractometric characteristics of SPR (*i.e.*, dependences of the reflected by the sample light intensity on the angle of incidence onto it) inherent to the studied samples in ambient air were determined using the respective device. These AFM and SPR investigations were carried out under normal conditions ($P = 10^5$ Pa, $T = 293$ K).

To study the nanoroughness of the sample surface by using AFM, we used the device NanoScope IIIa Dimension 3000$^{\text{TM}}$ (Veeco Inc.). The samples were mounted on the sample stage, and to scan we used the silicon probes “Ultrasharp CSC38” (Mikromash, Germany) with the nominal nib radius 10 nm. To study the samples, we also used the method of X-ray reflectometry with the diffracttometer Philips X’Pert PRO–MRD (CuK$_\alpha$ radiation, $\lambda = 0.15406$ nm). The tube anode voltage was 45 kV, current – 40 mA). The samples were scanned in the symmetric (mirror) geometry, when the angle of incidence is always equal to the angle of reflection. The step of scanning was 0.0008°, data acquisition time in one point – 1.5 s.

The ellipsometric method (SE-2000 spectroscopic multiangle ellipsometer, Semilab) provided measurements of the phase shift Δ between the components of electric field for the polarized reflected light in the cases of parallel and normal orientations of these components relatively to the plane of incidence, as well as the azimuth ψ of the restored linear polarization. These ellipsometric parameters obtained in experiments enable to calculate optical constants of the studied samples, namely: refraction index n, absorption index k as well as optical conductivity σ [13].

The refractometric characteristics of the studied samples were determined using the SPR device “Plasmon-6” designed in V. Lashkaryov Institute of Semiconductor Physics, NAS of Ukraine. This device is based on the prism method for excitation of plasmons in the Kretschmann geometry [14]. To excite surface plasmons, there used is a semiconductor laser with the wavelength 650 nm. The refractometer was used in the regime of angular periodical scanning with the simultaneous registration of the reflection curve. By their turn, the samples were mounted onto the operation face of glass (K8, n_D = 1.514) semipentaprism inside “Plasmon-6” with using the immerse liquid (polyphenyl ether Н-ПФЭ possessing the refraction index $n_D = 1.63025 \pm 0.0007$), and their refractometric characteristics were measured. As a reference substance, we used ambient air.

Table 1. Results of AFM and XRR measurements.

Structure	Irradiation power, W/mm^2	AFM method			XRR method			
		R_q, nm	R_a, nm	R_z, nm	R_q Au–air, nm	R_q SiO–Au, nm	Density of metal film, g/cm^3	Thickness of metal film, nm
Sample 1	27.84	2.32	0.66	3.94	2.0	1.2	19.3	43.00
Sample 2	36.62	1.64	0.53	3.42	1.3	0.8	19.2	43.46
Sample 3	0	4.67	1.21	7.41	2.6	0.5	17.4	-
Sample 4	0	4.32	1.36	7.79	2.6	0.5	17.2	-

Table 2. Calculated optical constants for the studied samples.

Structure	n	k	ε_r	ε_i	$\lvert\varepsilon\rvert$	δ_d, nm
Sample 1	0.4207	3.4141	–11.4791	2.8726	11.8330	334.7
Sample 2	0.3521	3.4484	–11.7672	2.4284	12.0152	339.3
Sample 3	0.6022	3.2141	–9.9679	3.8713	10.9085	309.7
Sample 4	0.3699	3.2820	–10.6349	2.4277	10.6934	321.0

Table 3. Results of measurements and calculations.

Structure	θ_{spr}, deg	θ_c, deg	S, deg^{-1}	$W_{0.2}$, deg	S_{slope}, RIU^{-1}	S_θ, deg·RIU^{-1}
Sample 1	43.462	41.268	2.396	0.453	2.511	1.048
Sample 2	43.676	41.346	1.948	0.648	2.034	1.044
Sample 3	43.804	41.360	1.578	0.622	1.703	1.079
Sample 4	43.829	41.346	1.425	0.867	1.516	1.064

Table 4. Calculated optical constants of the gold layer and heterointerfaces.

Structure	Gold layer			Heterointerface SiO–Au			Heterointerface Au–air		
	n	k	d, nm	n	k	d, nm	n	k	d, nm
Sample 1	0.1931	3.6502	44.59	0.1274	4.5388	1.33	0.8105	3.7375	1.85
Sample 2	0.1556	3.6424	46.53	0.1579	4.9983	0.82	0.7887	4.2605	1.37
Sample 3	0.1827	3.6040	47.02	0.1483	4.2516	0.41	0.8125	3.6415	3.05
Sample 4	0.1941	3.6130	43.69	0.1457	4.0522	0.49	0.8324	3.2296	3.26

3. Results and discussion

Using the AFM method, we studied the main characteristics of surfaces of these samples by measuring surface topography (Fig. 1). This figure does not contain the image of topography inherent to the sample 4, because its topology is identical to that of the sample 3 (Fig. 1a). Besides, with account of scales shown in the figure it is seen that the samples after e-beam treatment possess the lower dispersion of relief heights. Being based on the program product Gwyddion 2.36, we performed the statistical analysis of these images and determined: mean-arithmetic deviation of the profile R_a and heights of nanoroughness R_z relatively to the base line of 4.24 μm.in length (Fig. 2), as well as the mean-square micro-roughness R_q for the images of the area 9 μm^2 (Table 1).

The performed investigations of surfaces of optical elements after e-beam treatment show that the surface becomes more homogeneous (Fig. 2b) by the values of dispersion between heights of roughness relatively to the base line, as compared with that before treatment (Fig. 2a).

Our analysis of topology showed that, after e-beam treatment of the glass substrate surface, the nanoroughness of the deposited on it metal film is reduced.

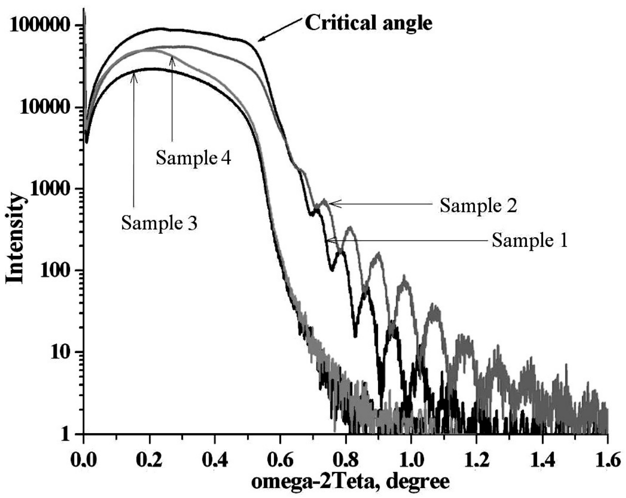

Fig. 3. X-ray reflectivity profiles.

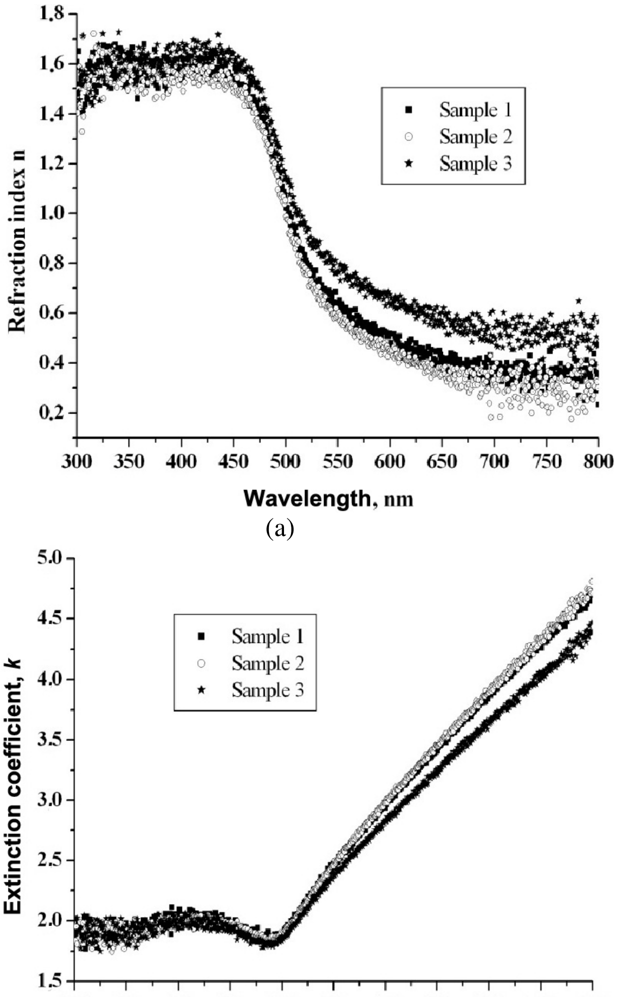

Fig. 4. Calculated spectral dependences of the refraction index *n* (a) and the extinction coefficient *k* (b) for the samples: without any e-beam treatment – sample 3, treated with different specific powers – 27.84 W/mm^2 – sample 1; 36.62 W/mm^2 – sample 2.

So, the mean-square roughness R_q is two-fold reduced from 4.67 down to 2.32 nm for the irradiation power 27.84 W/mm^2 and three-fold reduced from 4.67 down to 1.64 nm for the irradiation power 36.62 W/mm^2.

Availability of interferential oscillations on the characteristics of X-ray reflections from the samples studied using the XRR method enabled to estimate the thickness of metal films, and different values of critical angles are indicative of their different density (Table 1). For instance, the density of gold films is increased from 17.2 up to 19.3 g/cm^3 after e-beam treatment as a consequence of the higher velocity of grain creation than the velocity of their growth, which provides more fine-dispersed structure and higher compactness between them. In this case, the density value reached that of bulk gold – 19.32 g/cm^3 [15].

Absence of oscillations in reflectograms inherent to the samples 3 and 4, where the substrate surface remains without any e-beam treatment, can be caused by three factors, namely: i) large thickness of the films (oscillations are considerably superposed); ii) considerable inhomogeneity of these films by thickness; iii) strong heterogeneity of properties over the film plane.

Estimation of the decay velocity for the reflected X-rays enabled to determine the mean-square roughness that was two-fold reduced from 2.6 down to 1.3 nm, which agrees with the experimental results obtained using the AFM method (Table 1, columns 3 to 5).

Using the measured ellipsometric characteristics of the samples, we calculated the optical constants of the heterointerface "glass–gold" in accord with the method [16]. The spectral dependences of the refraction index *n* and extinction coefficient *k* within the range of wavelengths 300…800 nm are shown in Fig. 4. The figure does not contain the plots of spectral dependences for the refraction index *n* and extinction coefficient *k* inherent to the sample 4, since they are similar to those of the sample 3 and show superposition on them (Fig. 1a).

To determine the complex relative dielectric function for the heterostructure "glass–gold–air", we calculated *n* and *k* at the operation wavelength 650 nm. These values are summarized in Table 2. Also, the table contains the values for the effective depth of penetration inherent to surface plasmons δ_d into dielectric medium. The values were calculated using the formula [17].

As seen from Table 2, e-beam treatment leads to the decrease in decay of the surface plasmon field. It causes the increase in the absolute value of the real part of dielectric permittivity of metal film and expands the depth δ_d of surface plasmon field penetration into the studied medium (air, in our case), which enhances the sensitivity of the SPR device.

The measured refractometric characteristics (Fig. 5) are the dependences of the normalized intensity for the reflected light (reflection coefficient) on the angle of light incidence θ onto the boundary of media "glass–air" with the thin gold film between them.

The measured SPR characteristics show that e-beam treatment increases glass absorption, which can be concluded from the increase of the reflection coefficient value in the minimum of refractometric characteristic.

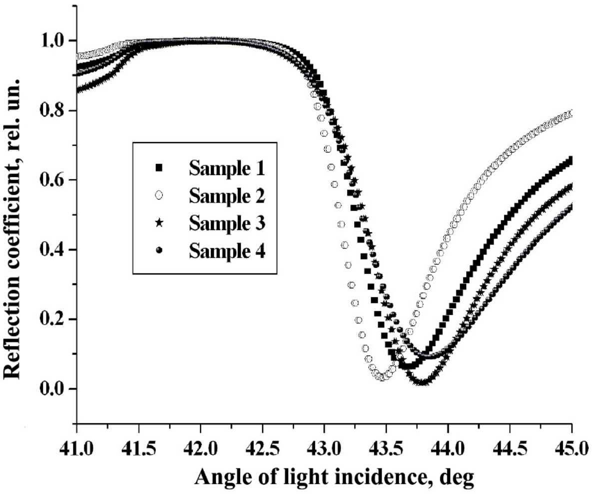

Fig. 5. Measured refractometric characteristics of the samples: with different specific powers of e-beam treatment of glass substrates 27.84 W/mm^2 – sample 1, 36.62 W/mm^2 – sample 2, and without this treatment – samples 3 and 4.

Besides, one can observe the shift of SPR minima to the side of lower angles, which is indicative of lowering the refraction index in the heterointerface "glass–gold" and "gold–air".

The measured refractometric characteristics were used to determine the following parameters: critical angle θ_C, angle corresponding to the reflection minimum θ_{spr}, half-width of the refractometric characteristic $W_{0.2}$ and the sensitivity S at the point of refractometric curve where the slope reaches the highest value on the left wing S_{slope}, as well as the sensitivity by the shift of the resonance curve minimum S_θ [17], which was calculated with account of the obtained optical constants for gold (see Table 3).

The half-width value defines the approximation error that is used for determination of the SPR minimum in the course of measuring the kinetics with the SPR device "Plasmon-6". As seen from Table 3, the half-width $W_{0.2}$ is practically two-fold decreased after e-beam treatment from 0.867 down to 0.453 deg. In this case, the sensitivity S_{slope} grows due to the 1.7 times increase in the slopes of resonance curve from 1.425 up to 2.396 deg^{-1}, while the sensitivity by the shift of the resonance curve minimum S_θ has no essential changes. As a consequence of resonance curve shift to the side of lower angles, the range of measurements for this device is additionally widened by 0.37 deg.

In addition, the obtained refractometric characteristics enabled to calculate the optical constants and thickness of gold layers as well as heterointerfaces glass–gold and gold–air (Table 4) by using the soft package WinSpell3.0.

The calculated optical constants for the gold film are in accord with the existing table data [18, 19], and the thickness values agree with those obtained using the data of X-ray reflectometry. The thickness of heterointerfaces glass–gold and gold–air is in good accordance with the values of mean-square roughness R_q obtained using the X-ray method (see Table 1, the columns 6 and 7), which confirms the reliability of results obtained in calculations.

Thus, the analysis of measured refractometric characteristics has shown that application of electron-beam treatment of glass substrates for sensitive elements of SPR devices enhances their sensitivity and widens the range for measuring the resonance angles of SPR due to reducing the nanoroughness both of the glass surface and gold film.

4. Conclusions

Experimentally proved in this work is the efficiency of electron-beam treatment the surfaces of glass substrates for sensitive elements of SPR devices. This treatment results in practically two-fold narrowing their refracttometric characteristics from 0.867 down to 0.453 deg. and leads to the angular shift of their minimum by 0.37 deg. In this case, the sensitivity of the SPR device is 1.7 times increased from 1.425 deg^{-1} up to 2.396 deg^{-1} due to lowering the energy losses on propagation of surface plasmons along the boundary metal–air. The reason for this decrease in losses is higher homogeneity of the gold film surface, its higher compactness and lower nanoroughness as well as reduced thickness of the intermediate layer gold–air. After e-beam treatment of glass substrates, the value of surface irregularity height dispersion relatively to the base line has been reduced from ±18 nm down to ±5 nm, mean-square roughness of the surface has been three-fold reduced from 4.67 down to 1.64 nm, and the thickness of the gold–air heterointerface – from 3.26 down to 1.37 nm. The density of gold film has increased from 17.2 up to 19.3 g/cm^3, *i.e.* reached the value inherent to bulk gold. It has an effect on the refraction index and extinction coefficient of the gold film. Thus, the analysis of the obtained refracto-metric characteristics has shown the efficiency of applying electron-beam treatment of glass substrates to sensitive elements of SPR devices for enhancing their sensitivity and widening the range of measurements of SPR resonance angles.

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