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REGULAR ARTICLE

Ellipsometric Characterization of SiO_2/Au/Cr Nanostructures for Stable SPR Sensors

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In this research, the optical characteristics of a multilayer nanostructure SiO_2(5 nm)/Au(50 nm)/Cr(5 nm)/glass, designed for stable surface plasmon resonance (SPR) sensors, were investigated. Multi-angle spectroscopic ellipsometry was employed at angles of 50°, 60°, and 70° to precisely analyze the spectral dependences of the ellipsometric parameters Ψ and Δ over a wide wavelength range from 300 to 1500 nm. While the metal layers (Cr and Au) were deposited by vacuum thermal evaporation, the silicon dioxide protective layer was formed using the sol-gel method followed by thermal annealing at 400 °C.

The study established that the deposition of the 5 nm SiO_2 layer causes a characteristic modification of the $\Psi(\lambda)$ and $\Delta(\lambda)$ spectra, reflecting the dielectric contribution of the coating without degrading the metallic properties of the gold film. Numerical simulations based on the verified optical model showed that the introduction of the protective layer results in a minor SPR resonance shift of approximately 0.5° at the operating wavelength of 633 nm. Importantly, the high-quality factor and sensitivity of the resonance are preserved. The results confirm that the sol-gel derived SiO_2 nanolayer effectively stabilizes the sensor surface against environmental degradation while maintaining its optimal functional performance, making it a promising candidate for durable SPR-based sensing applications.

Keywords: Spectroscopic ellipsometry, Surface plasmon resonance, Optical constants, Sol-gel method, Nanostructures, Gold, Silicon dioxide.

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1. INTRODUCTION

The modern development of nanotechnology and biophotonics has led to a rapid increase in demand for highly sensitive analytical devices capable of operating in real time. Among them, sensors based on surface plasmon resonance (SPR) occupy a special place, as they enable label-free detection of biological and chemical objects [1]. Due to their ability to register extremely small changes in the local refractive index near a metal surface, SPR sensors are widely used in medical diagnostics, environmental monitoring, and pharmacology [2].

The classical configuration of an SPR sensor (Kretschmann scheme) involves the use of a thin noble metal film, most commonly gold (Au), with a thickness of about 50 nm [3]. Gold was chosen due to of its high chemical stability and ability to excite a clearly defined plasmon peak in the visible and near-infrared regions of the spectrum. However, to improve the adhesion of gold to the glass substrate, an intermediate chromium (Cr) or titanium layer with a thickness of 3–5 nm is typically required [4].

Despite the chemical inertness of gold, operation in aggressive chemical environments or prolonged contact with biological samples can lead to degradation of the metal surface and a shift of the resonance characteristics [5]. One of the most promising approaches for stabilizing the sensor is the deposition of nanoscale dielectric protective layers, particularly silicon dioxide (SiO_2). This material combines high mechanical strength, chemical inertness, and optical transparency, which minimizes its influence on the resonance quality factor [6].

The effectiveness of such protection critically depends on the parameters of the nanofilm. The thickness of the SiO_2 layer should not exceed the plasmon decay length (evanescent field penetration depth), which for gold is approximately 100–200 nm in air and significantly smaller in liquid media. In this work, an ultrathin SiO_2 layer (5 nm) deposited by the sol–gel method followed by thermal annealing is considered. The use of tetraethoxysilane (TEOS) as a precursor allows the formation of uniform amorphous films that slow down diffusion processes and dislocation motion in the gold layer [7].

However, the deposition of even nanometer-thick dielectric layers significantly changes the excitation conditions of surface plasmon polaritons. This imposes strict requirements on the accuracy of thickness control and on the determination of the actual ellipsometric amplitude parameters Ψ and phase parameter Δ of the obtained films, which may significantly differ from tabulated values for bulk materials due to nanoscale

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effects and structural porosity. Traditional photometric methods are often insufficiently sensitive for the analysis of such multilayer systems.

Spectroscopic ellipsometry is one of the most precise tools for the non-destructive investigation of nanostructures [8]. The method is based on the analysis of the change in the polarization state of light of the amplitude Ψ and phase Δ components upon reflection from a multilayer structure. This enables a comprehensive characterization of the optical properties and an estimation of the layer thicknesses, provided that appropriate dispersion models are applied to account for parameter correlation.

The aim of this work is to establish the physical regularities governing the formation of the ellipsometric spectra $\Psi(\lambda)$ and $\Delta(\lambda)$ of multilayer plasmonic nanostructures SiO_2/Au/Cr/glass and to determine their spectral characteristics using spectroscopic ellipsometry. To achieve this goal, several tasks were addressed. First, the spectral dependences of the amplitude parameters $\Psi(\lambda)$ and phase $\Delta(\lambda)$ were determined in a wide spectral range (300-1500 nm) using experimental measurements and analysis of a complex metal-dielectric system. Second, the influence of the SiO_2 nanolayer was evaluated by performing a comparative analysis of the ellipsometric parameters $\Psi(\lambda)$ and $\Delta(\lambda)$ of the structure before and after the deposition of the protective coating formed by the sol-gel method. Finally, the suitability of the sol-gel method for the formation of optically homogeneous protective nanofilms that do not introduce significant parasitic losses into the operation of the SPR sensor was confirmed by analyzing the measured $\Psi(\lambda)$ and $\Delta(\lambda)$ spectra.

2. MATERIALS AND METHODS

2.1 Preparation of the Multilayer Structure

The object of the study was a multilayer nanostructure (Fig. 1) formed on optical glass substrates of TK-14 grade with dimensions of 20×20 mm. The sensor fabrication process included several sequential stages of deposition and chemical coating formation.

To create an adhesion layer, a chromium (Cr) film with a thickness of 5 nm was deposited onto the cleaned glass substrate by vacuum thermal evaporation, ensuring reliable adhesion of gold to the glass. A plasmon-active gold layer (Au) with a thickness of 50 nm was subsequently deposited on top of the chromium layer, which is optimal for the excitation of surface plasmon resonance in the visible spectral region. The protective silicon dioxide (SiO_2) film with a thickness of 5 nm was formed using the sol-gel method.

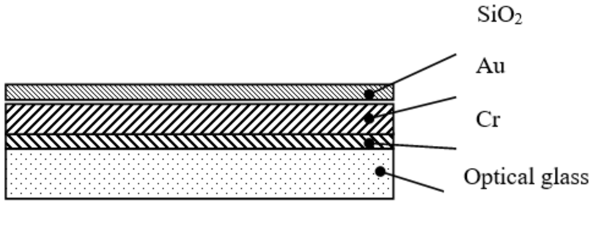

Fig. 1 – Schematic representation of the multilayer structure of the SPR sensor

2.2 Sol-Gel Synthesis of the Protective Coating

Tetraethoxysilane (TEOS) produced by Evonik Industries (Germany) was used as the precursor for the formation of the protective layer. The deposition process included the following steps:

1. Deposition of the working solution onto the gold surface by spin coating at a rotation speed of 3000 rpm.
2. Preliminary drying of the formed films at 150 °C for 30 minutes.
3. Final annealing in a SNOL 12/1200 LSC01 muffle furnace at 400 °C for 1 hour to form a dense amorphous SiO_2 structure.
4. Gradual cooling of the samples together with the furnace for 24 hours to prevent the formation of microcracks caused by thermal stresses.

2.3 Ellipsometric Measurement Procedure

The optical parameters of the thin-film structures were researched using spectroscopic ellipsometry (SE) with an automated M-2000 ellipsometer (J.A. Woollam Co.) in the wavelength range of 300-1500 nm. The measurements were performed at multiple angles of incidence (50°, 60°, 70°). This set of angles was chosen to encompass the Brewster angle of the glass substrate (approximately 56-57°), which ensures maximum sensitivity of the ellipsometric parameters to the properties of the nanolayers. The use of multi-angle spectroscopic ellipsometry (MASE) allows for a significant reduction in the correlation between the thickness and dispersion parameters during the modeling process, thereby increasing the reliability of the obtained results. The measurements were based on the rotating-compensator ellipsometry principle.

The relationship between the experimental data and the optical properties is described by the fundamental ellipsometric equation of the complex reflectance ratio:

$$\rho = \frac{r_p}{r_s} = \tan(\Psi) e^{i\Delta}, \quad (1)$$

where r_p and r_s are the complex reflection coefficients for the parallel and perpendicular components of the electric field vector, respectively; Ψ, Δ – amplitude ratio angle and phase shift.

The obtained experimental spectra $\Psi(\lambda)$ and $\Delta(\lambda)$ were modeled using Complete EASE software by constructing a multilayer optical model. For each layer, the thickness d and the dispersion parameters were determined to achieve the best fit for the measured amplitude parameter Ψ and phase parameter Δ.

The following approaches were used to describe the dispersion of the materials:

1. Cauchy model – for approximating the optical constants of the transparent SiO_2 layer and the glass substrate.
2. Drude-Lorentz model (GenOsc) – to accurately describe the dielectric functions of the metallic Au and Cr layers, the General Oscillator (GenOsc) layer was utilized. The model included a Drude oscillator to account for the free-carrier response and several Lorentz oscillators to model interband transitions. This approach ensures that the ellipsometric parameters are physically

consistent and satisfy the Kramers-Kronig relations.

3. Surface roughness model – the Bruggeman effective medium approximation (EMA) was applied, describing the top-most layer as a mixture of the base material and voids (50 vol. %).

4. Interface layers – thin transition layers between the substrate and the metal film, as well as between metal layers, were included to improve the fit quality.

The model parameters (thickness d and oscillator parameters) were optimized using the Levenberg-Marquardt algorithm. The optimization was performed by minimizing the Mean Square Error (MSE) between the experimental and calculated $\Psi(\lambda)$ and $\Delta(\lambda)$ spectra.

3. RESULTS AND DISCUSSION

As a result of the mathematical processing of the experimental ellipsometric spectra using the Complete EASE software, the optical response of the investigated nanostructures was analyzed. Fig. 2 presents the obtained experimental and modeled spectral dependences of the amplitude parameter Ψ and the phase parameter Δ for samples with and without the protective SiO_2. The measurements and subsequent modeling were performed at three angles of incidence 50°, 60°, and 70° to encompass the region near the Brewster angle of the glass substrate (56°-57°). This multi-angle approach ensures maximum sensitivity of the Ψ and Δ parameters to the properties of the nanolayers and significantly reduces the correlation between the thickness and dispersion characteristics during the construction of the optical model.

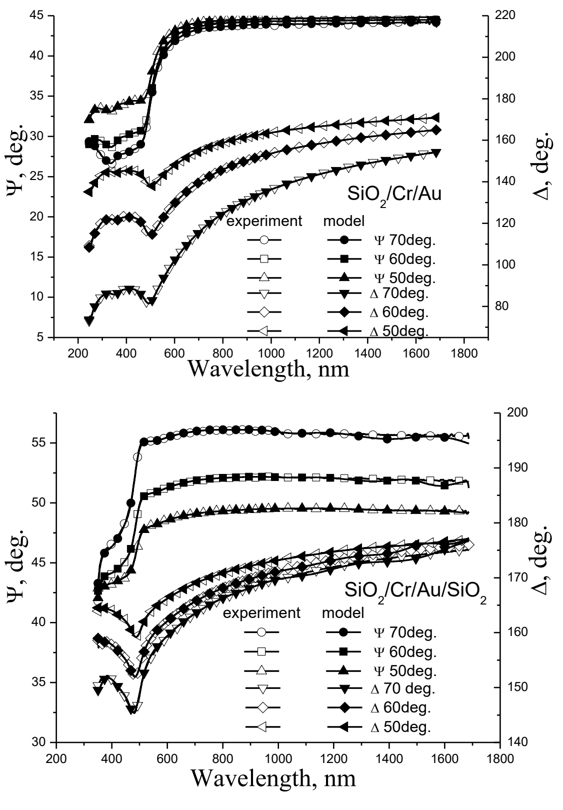

Fig. 2 – Experimental and modeled spectral dependences of the amplitude parameter Ψ and the phase parameter Δ for samples with and without the protective SiO_2

The analysis of the obtained $\Psi(\lambda)$ and $\Delta(\lambda)$ curves made it possible to establish the following physical regularities. The deposition of a 5 nm SiO_2 layer formed by the sol-gel method leads to a measurable modification of the ellipsometric response of the entire structure, which is especially noticeable in the infrared spectral region above 800 nm. This is attributed to the additional dielectric contribution of the silicon dioxide film to the overall optical response of the system. The characteristics of the measured $\Psi(\lambda)$ and $\Delta(\lambda)$ spectra indicate the high optical quality of the amorphous SiO_2 layer, which does not introduce significant parasitic absorption losses.

The spectral behavior of the measured $\Psi(\lambda)$ and $\Delta(\lambda)$ parameters in the long-wavelength region is consistent with the classical response of metallic nanofilms. A characteristic feature of the obtained spectra is the preservation of the specific plasmonic response after thermal annealing at 400 °C [9], which confirms that the gold layer maintains its metallic nature and structural integrity. The stability of the ellipsometric signal across the 300-1500 nm range indicates that the interposition of the 5 nm SiO_2 layer and the subsequent heat treatment do not lead to significant oxidation or degradation of the Au/Cr interface. This is a crucial factor for the efficient excitation of surface plasmon resonance in the final sensor device.

To further validate the obtained results, the experimental ellipsometric parameters $\Psi(\lambda)$ and $\Delta(\lambda)$ were converted into the refractive index n and extinction coefficient k for each layer of the sample using the Complete EASE software. A quantitative comparison of these calculated values with classical reference data at the operating wavelength $\lambda = 633$ nm is presented in Table 1. The observed slight discrepancies between our results and the tabulated values for bulk gold [10] specifically slight increases in the refractive index are attributed to size-dependent effects. In nanofilms, charge carrier scattering at grain boundaries and surface roughness significantly influences the final optical constants derived from the $\Psi(\lambda)$ and $\Delta(\lambda)$ spectra. At the same time, the parameters obtained for the SiO_2 layer are in good agreement with reference data, confirming the high quality and density of the protective coating synthesized by the sol-gel method.

The verified optical model, incorporating the precise thicknesses and complex dielectric functions determined from the spectroscopic ellipsometry analysis, was utilized for numerical simulation of the surface plasmon resonance (SPR) reflectance curves in the Kretschmann configuration. This simulation was performed using the Transfer Matrix Method [11], which allows for a rigorous account of each constituent layer (Cr, Au, and SiO_2) within the multilayer stack rather than using simplified effective parameters. Calculations conducted at the operating wavelength of $\lambda = 633$ nm indicate that the deposition of the 5 nm SiO_2 protective layer results in a resonance angle shift of approximately 0.5°. Such a shift is relatively small and falls within a range that does not compromise the overall sensitivity of the sensor. Furthermore, the numerical analysis confirms that the high-quality factor of the plasmon resonance is fully preserved. These results demonstrate that the sol-gel derived SiO_2 nanolayer effectively enhances the chemical and mechanical stability of the sensor surface while maintaining its optimal functional performance without the requirement for significant system recalibration.

Table 1 – Comparison of experimental *n* and *k* values with classical reference data

Material	Parameter (λ = 633 nm)	Reference data [10]	Experimental data	Explanation of difference
Au	*n*	0.18	0.22 – 0.35	Size effects, grain boundary scattering
Au	*k*	3.43	3.00 – 3.30	Surface roughness, influence of Cr adhesion layer
SiO_2	*n*	1.457	1.42 – 1.45	Features of the sol-gel method

4. CONCLUSIONS

In this work, the optical properties of multilayer SiO_2/Au/Cr/glass nanostructures designed for stable surface plasmon resonance (SPR) sensors were comprehensively investigated using multi-angle spectroscopic ellipsometry. The analysis of the experimental and modeled $\Psi(\lambda)$ and $\Delta(\lambda)$ spectra in the range of 300-1500 nm, performed at angles of incidence of 50°, 60°, and 70°, ensured high accuracy in characterizing the ultrathin layers and reduced the correlation between thickness and dispersion parameters.

The study established that the formation of a 5 nm SiO_2 protective layer by the sol-gel method followed by thermal annealing at 400°C does not lead to the degradation of the underlying metallic films. The conversion of the ellipsometric parameters into optical constants confirmed that the SiO_2 layer is optically homogeneous and dense, with properties closely matching those of the bulk material. For the gold layer, a slight deviation in the optical response compared to reference data was observed, which is explained by size-dependent effects and charge carrier scattering at grain boundaries in the nanofilm.

Numerical simulations based on the verified optical model and the transfer matrix method demonstrated that the introduction of the 5 nm SiO_2 layer causes a minor SPR resonance shift of approximately 0.5° at λ = 633 nm. This shift is considered negligible for practical sensor applications and does not compromise the sensitivity or the quality factor of the resonance. Thus, the sol-gel derived silicon dioxide nanolayer is shown to be an effective protective coating that enhances the chemical and mechanical stability of SPR sensors while maintaining their optimal functional characteristics without the need for complex recalibration.

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Еліпсометрична характеристика наноструктур SiO_2/Au/Cr для стабільних ППР-сенсорів

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Досліджені оптичні характеристики багатошарової наноструктури SiO_2(5 нм)/Au(50 нм)/Cr(5 нм)/скло, розробленої для сенсорів стабільного поверхневого плазмонного резонансу (SPR). Багатокутова спектроскопічна еліпсометрія використовувалася під кутами 50°, 60° та 70° для точного аналізу спектральних залежностей еліпсометричних параметрів Ψ та Δ у широкому діапазоні довжин хвиль від 300 до 1500 нм. У той час як металеві шари (Cr та Au) були нанесені методом вакуумного термічного випаровування, захисний шар діоксиду кремнію був сформований золь-гель методом з подальшим термічним відпалом при 400 °C.

Дослідження показало, що осадження шару SiO_2 товщиною 5 нм викликає характерну модифікацію спектрів $\Psi(\lambda)$ та $\Delta(\lambda)$, що відображає діелектричний внесок покриття без погіршення металевих властивостей золотої плівки. Чисельне моделювання на основі перевіреної оптичної моделі показало, що введення захисного шару призводить до незначного зсуву резонансу ППР приблизно на 0,5° при робочій довжині хвилі 633 нм. Важливо, що висока якість та чутливість резонансу зберігаються. Результати підтверджують, що наношар SiO_2, отриманий золь-гель методом, ефективно стабілізує поверхню сенсора від впливу навколишнього середовища, зберігаючи при цьому його оптимальні функціональні характеристики, що робить його перспективним кандидатом для довговічних сенсорних застосувань на основі ППР.

Ключові слова: Спектроскопічна еліпсометрія, Поверхневий плазмонний резонанс, Оптичні константи, Золь-гель метод, Наноструктури, Золото, Діоксид кремнію.