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UDC 539

CHARACTERIZATION OF COBALT PHTHALOCYANINE THIN FILM ON SILICON SUBSTRATE USING SPECTROSCOPIC ELLIPSOMETRY

The cobalt phthalocyanine film (CoPc) was prepared by an ultra-high vacuum system onto a silicon substrate. Structural features and optical properties of the organic semiconductor CoPc has been determined with the use of spectroscopic ellipsometry over the wavelength interval 300–1000 nm. By restricting it to 900–1000 nm the film thickness is determined, and, by the point-by-point fit, the behavior of the dielectric function is established in the entire spectral region. Thus, the optical properties are determined from spectral ellipsometric data using mathematical models based on Gaussian oscillators, which have led to an excellent fit to the experimental data with a relatively low mean square error. Cobalt phthalocyanine was treated as a uniaxial material.

Keywords: spectroscopic ellipsometry, cobalt phthalocyanine, optical constants, Gaussian oscillators, uniaxial material.

1. Introduction

Recently, the organic semiconductor cobalt phthalocyanine (CoPc) has been studied as a promising material for organic light-emitting devices (OLED) [1]. Kao *et al.* demonstrated an improvement in the turn-on voltage and luminance in OLEDs using the CoPc layer as a hole injection layer (HIL) in the architecture of many devices [2]. In particular, phthalocyanin has attracted a lot of attention due to its high thermal and chemical stability [3], being of interest for applications in organic optoelectronic devices such as organic solar cells, in organic field effect transistors, etc. [4]. CoPc is a p-type semiconductor in which molecules have a planar structure; these molecules are usually have anisotropic optical constants which are, in many cases, uniaxial with the optical axis oriented along the normal to the film surface [5]. The anisotropy of organic crystals is usually related to different oscillator strengths in different directions due to the particular molecular ordering of transition dipoles [6]. (Fig. 1) shows the direction of the optical axis in a uniaxial film. In this case, we take the z direction to be along the optical axis. The optical axis is expected to be perpendicular to the film. The x and y directions in the plane of the film. In anisotropic materials, the propagation speed of light varies according to the oscillating direction of the electric field. When the direction of the polarized electric field is parallel to the x, y, or z axes, it will propagate, respectively, at the speeds of $s_x = \frac{c}{n_x}$, $s_y = \frac{c}{n_y}$, or $s_z = \frac{c}{n_z}$. In a uniaxial material, if the oscillating electric field is perpendicular to the optical axis, then the speed of light diffusion remains constant, and it follows that $n_x = n_y$. While its propagation speed varies according to the direction of oscillations of the electric field, if it is parallel to the optical axis [7, 8]. In the present work, we intend to find the refractive indices $n_x = n_y$ and n_z for a cobalt phthalocyanine thin film, together with the corresponding absorption coefficients by spectroscopic ellipsometry, providing, in addition, the abundant structural information which is crucial for the development of diverse nanoscale devices.

2. Experimental

The CoPc thin film was prepared by the organic molecular beam deposition (OMBD) using an ultra-high vacuum system. The film was deposited on silicon (100). Before the film deposition, the silicon substrate was carefully cleaned using acetone, isopropyl alcohol solution, and distilled water, sequentially. After the cleaning of the sample, the pre-cleaned substrate was treated with Ar. The material was sublimated from Al_2O_3 crucible at a pressure of 10^{-8} mbar during the deposition. The rate of deposition was controlled at 0.5° A/s using a quartz crystal microbalance [9]. On the other hand, spectroscopic ellipsometry (SE) was performed using a variable-angle spectroscopic ellipsometer (VASE) in order to determine the ellipsometric parameters Ψ and Δ. The measurements were performed in air at room temperature over the wavelength interval 300–1000 nm in steps of 5 nm at incidence angles of 65°, 70°, and 75°. Finally, the spectroscopic ellipsometry data were analyzed using the WVASE software to determine the structural and optical properties of the film [10].

3. Results and Discussion

The use of the spectroscopic ellipsometry to study the sample is due to it being one of the most accurate optical techniques to characterize the film thickness fora single layers or many layers from a few angstroms or tenths of a nanometer to several micrometers, in addition, to determine the optical constants (refractive index n, extinction coefficient k) of the sample and other structural properties such as the surface roughness and uniformity. All this information is accessed by measuring two angles expressed as Ψ and Δ:

$$\widetilde{\rho} = \frac{\widetilde{R_p}}{\widetilde{R_s}} = \tan(\Psi)\exp(i\Delta), \tag{1}$$

where Ψ is the ratio of the field amplitudes of the two polarized light components, Δ is the phase difference between the $p-$ and s-polarized light [11]. In Fig. 2 and Fig. 3, the experimental data Ψ and Δ are shown for the cobalt phthalocyanine film.

These data are indirectly related to the optical and structural properties of the sample. Modeling the measured data is the appropriate procedure to determine these properties [12] and can be performed with the use of the WVASE32 software package from J.A. Woollam Co., Inc., while building an accurate physical model that optimally describes the physical system. This physical model consisting of any number of layers on a substrate, each layer which refers to specific material, and each of them carries the information about the thickness and dielectric constants. Thereafter, the model is modified using the algorithm in order to minimize the difference between the measured data for the sample and generated data from the physical model. The difference is measured with mean square error (MSE), using the following equation:

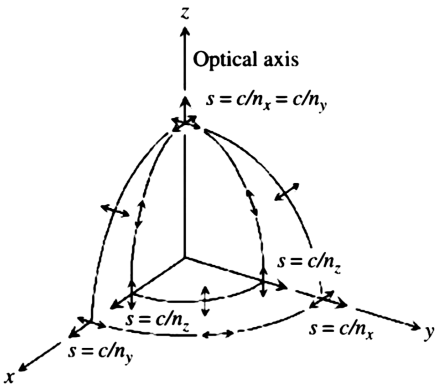


Fig. 1. Propagation of light in a uniaxial material

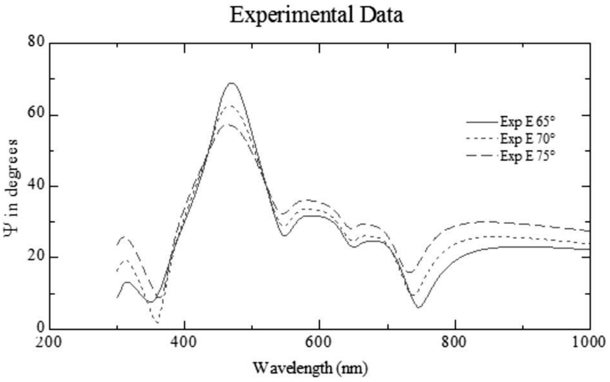


Fig. 2. Ellipsometric Ψ data for CoPc on silicon

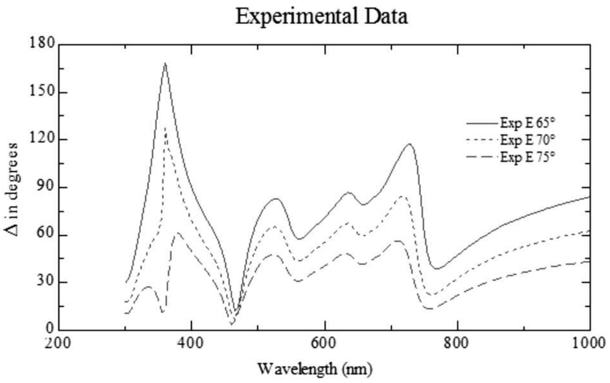
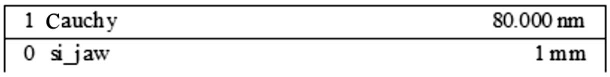


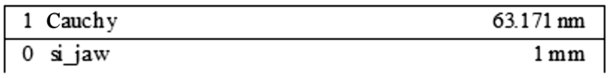
Fig. 3. Ellipsometric Δ data for CoPc on silicon

$$\mathrm{MSE} = \left(\frac{1}{2N-M}\sum_{i=1}^{N}\left[\left(\frac{\Psi_i^{\mathrm{mod}}-\Psi_i^{\mathrm{exp}}}{\sigma_{\Psi,i}^{\mathrm{exp}}}\right)^2+\left(\frac{\Delta_i^{\mathrm{mod}}-\Delta_i^{\mathrm{exp}}}{\sigma_{\Delta,i}^{\mathrm{exp}}}\right)^2\right]\right)^{1/2}, \tag{2}$$



1	Cauchy	80.000 nm
0	si_jaw	1 mm

Fig. 4. Model for the analysis of ellipsometric data from CoPc on silicon



1	Cauchy	63.171 nm
0	si_jaw	1 mm

Fig. 5. Model for CoPc on silicon, after the determination of the thickness, the Cauchy parameters A, B and C as fit parameters and after the normal fit

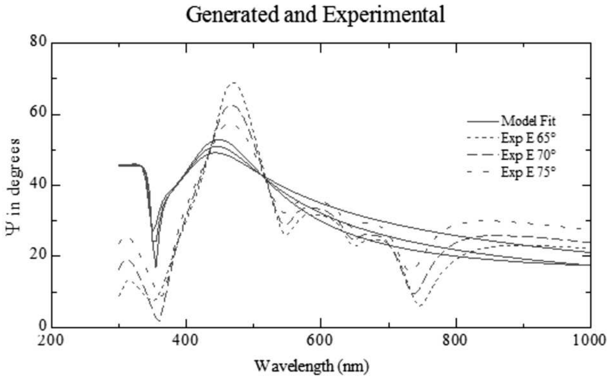


Fig. 6. Fit to the ellipsometric Ψ data for a CoPc film on silicon, using a Cauchy model for the optical constants of the CoPc layer

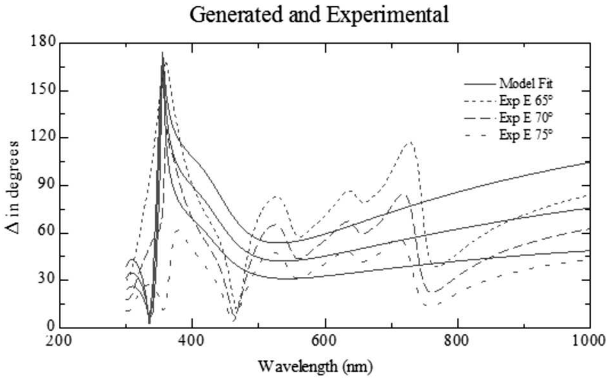


Fig. 7. Fit to the ellipsometric Δ data for a CoPc film on silicon using the Cauchy model for the optical constants

Ψ_i and Δ_i represent the measured Ψ and Δ angles at the ith wavelength, σ is the standard deviations of the experimental data point, upper indices mod and exp denote the generated and experimental data, respectively, while N and M represent the number of measured values and fitted parameters, respectively.

A built physical model represents the structure of the sample with silicon as the substrate with a thickness of 1 mm, and a Cauchy layer on top of the substrate which was used as an initially represented for the CoPc thin film. In the software package WVASE, the implemented Cauchy layer is described by the equation (3) [13]

$$n = A + \frac{B}{\lambda^2} + \frac{C}{\lambda^4}. \tag{3}$$

This equation describes the refractive index of a transparent material. It is evident that the refractive index of the medium decreases with an increase in the wavelength of light, which means the normal dispersion. The parameters A, B, and C are called Cauchy parameters [14]. We chose 80 nm as an initial estimate for the Cauchy layer thickness and defined the thickness and the Cauchy parameters as a fit parameter. The physical model is represented in (Fig. 4).

After generating the data and the normal fit, we got a thickness of 63.171 ± 0.97 nm, as shown in Fig. 5, the Cauchy parameters $A = 2.146 \pm 0.0922$, $B = 0.032304 \pm 0.0473\ \text{nm}^2$, and $C = -0.022686 \pm \pm 0.00531\ \text{nm}^4$. This yields an MSE of 463.1 and a very bad fit for the experimental data, as shown in Figs. 6 and 7.

From these figures, it can be seen that the behavior of the experimental data and the generated data are similar qualitatively at wavelengths longer than 800 nm. So, we have located the measured wavelengths in the region, where the film is transparent ($k = 0$), or, in other words, there is no absorption, or the imaginary part of the complex dielectric function in the region equals zero ($\varepsilon_2 = 0$), in order to fit as much of the measured data as possible.

We started by restricting the wavelengths to 800–1000 nm and performed a normal fit. This gave an MSE of 41.39, thickness equal to 82.645 nm, and a rather poor fit. Restricting the interval to 850–1000 nm and performing a normal fit gave an MSE of 21.41, thickness equal to 83.683 nm, and a better fit. Restricting the interval to 900–1000 nm and performing a normal fit again, we got an MSE of 9.872. This reduction in the spectral interval leads to a pretty good fit to the experimental data, as shown in Figs. 8 and 9. So, we stopped at this point.

In this method, we determined the thickness of the film, where its refractive index in the transparent spectral regions is almost identical, from the last

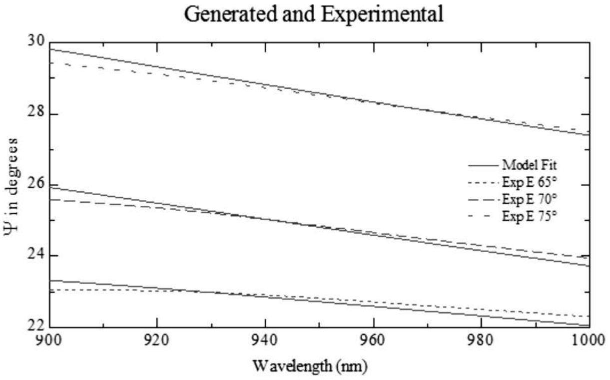


Fig. 8. Generated and experimental Ψ data for a CoPc film on silicon, fit with a Cauchy model over the restricted spectral interval 900–1000 nm

fit to be 84.407 nm, as shown in Fig. 10. Thus we have additional information available to determine the optical properties of the thin film over the entire spectral interval [15].

After that, we returned the selected spectral interval to 300–1000 nm. Then we opened the Cauchy layer dialog box and turn off the thickness and the Cauchy parameters A, B, and C, as a fit parameters, because their values were determined from the previous procedure, and defined the optical constants n and k, as the fit parameters. Then we performed a point-by-point fit starting from the long-wavelength end. The results of the fit of Ψ and Δ are shown in Figs. 11 and 12. This procedure will allow the determination of the behavior of the optical constants (n and k) or the dielectric constants (ε_1 and ε_2) of the thin film in the entire spectral region.

The simultaneous point-by-point fits of tan(Ψ) and cos(Δ) was the procedure following A.B. Djurišić *et al.* to determine the optical function data of cobalt phthalocyanine (CoPc), nickel phthalocyanine (NiPc), and iron phthalocyanine (FePc). In addition, we note that Z.T. Liu *et al.* determined the optical functions by simultaneous point-by-point fits of five metal phthalocyanine thin films (cobalt phthalocyanine (CoPc), copper phthalocyanine (CuPc), iron phthalocyanine (FePc), nickel phthalocyanine (NiPc), and zinc phthalocyanine (ZnPc)) by spectroscopic ellipsometry [4, 16].

Since CoPc is an absorbent material, the Cauchy model is not sufficient to describe the data. Therefore, the CoPc film is described by using mathemat-

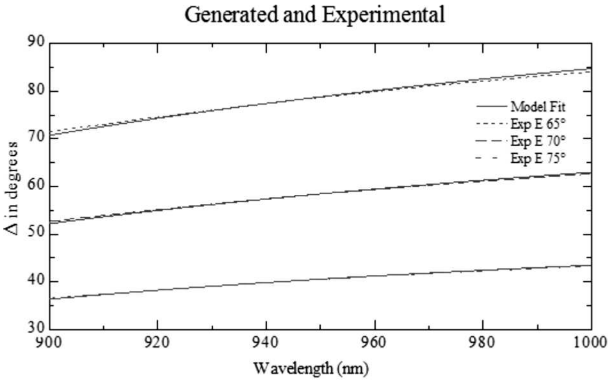
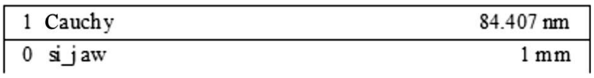


Fig. 9. Generated and experimental Δ data for a CoPc film on silicon, fit with a Cauchy model over the restricted spectral interval 900–1000 nm



1	Cauchy	84.407 nm
0	si_jaw	1 mm

Fig. 10. Model for the determination of the CoPc film thickness over the restricted spectral interval 900–1000 nm

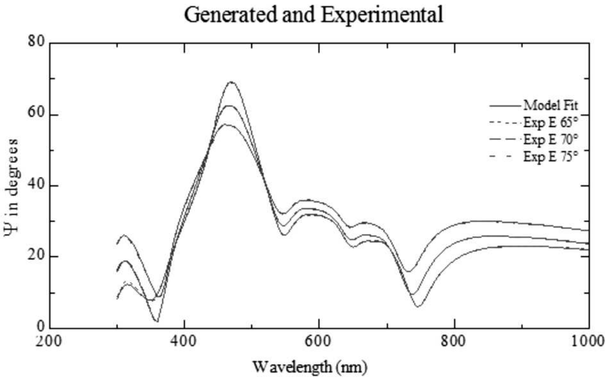


Fig. 11. Generated and experimental Ψ data for CoPc on silicon from a point-by-point fit with the film thickness fixed

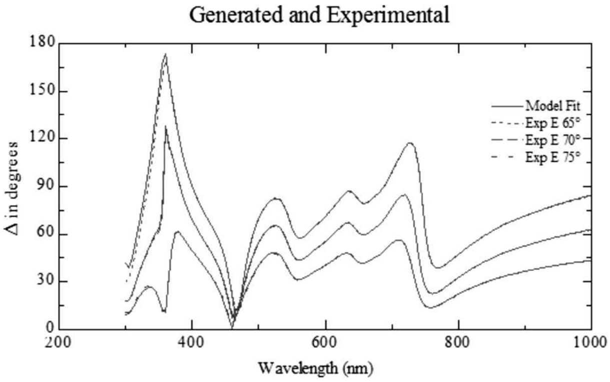


Fig. 12. Generated and experimental Δ data for CoPc on silicon from a point-by-point fit with the film thickness fixed

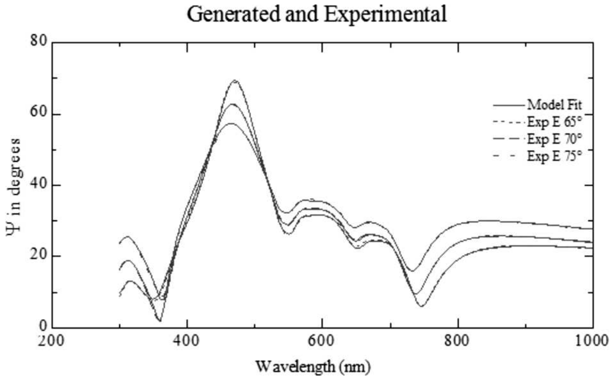


Fig. 13. Generated and experimental ellipsometric Ψ data for a CoPc film on silicon using the GenOsc layer

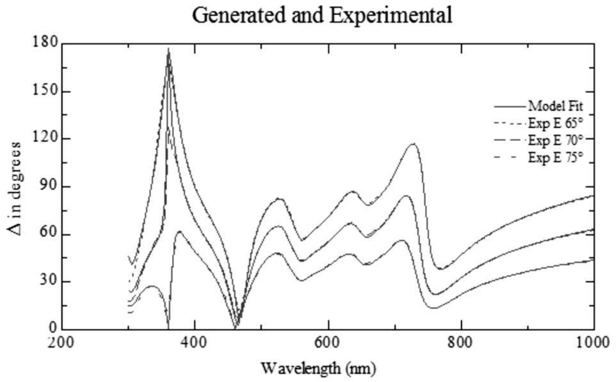
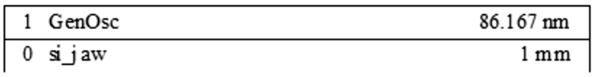


Fig. 14. Generated and experimental ellipsometric Δ data for a CoPc film on silicon using the anisotropic GenOsc layer



1	GenOsc	86.167 nm
0	si_jaw	1 mm

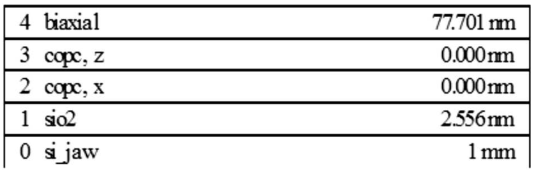
Fig. 15. Model for a CoPc film on silicon using the GenOsc layer

Table 1. **Values of parameters of Gaussian oscillator models for an CoPc film on silicon**

ε_1 Offset $= 2.2348 \pm 0.182$		E_n (eV)	B_r (eV)
Type osc.	Amp (eV^2)		
Gauss. 1	1.3036 ± 4.69	4.2787 ± 1.85	1.1679 ± 1.24
Gauss. 2	1.1869 ± 7.19	4.5358 ± 0.838	0.60006 ± 1.91
Gauss. 3	1.9559 ± 0.58	3.6692 ± 0.041	0.50567 ± 0.047
Gauss. 4	0.642 ± 0.0189	2.2585 ± 0.0009	0.08348 ± 0.0027
Gauss. 5	0.25465 ± 0.016	2.5642 ± 0.0927	1.344 ± 0.428
Gauss. 6	0.60048 ± 0.016	2.1908 ± 0.00515	0.26506 ± 0.0089
Gauss. 7	0.60526 ± 0.046	1.7523 ± 0.00948	0.52685 ± 0.0381
Gauss. 8	2.8336 ± 0.0787	1.7278 ± 0.0005	0.075829 ± 0.0013
Gauss. 9	2.5621 ± 0.0665	1.7654 ± 0.00141	0.17701 ± 0.003

ical models based on Gaussian oscillators. Because the Cauchy model was initially used only to obtain the thickness of the film and the optical constants over the restricted spectral interval 900–1000 nm. In terms of the procedure, through the WVASE software, we converted the Cauchy layer into a GenOsc layer. This layer allows the user to choose from a large variety of oscillator models. Using the menu, we added nine Gaussian oscillators to model the dielectric function of CoPc. We started the modeling by manually adjusting the amplitudes, the center energy, and the broadenings of the oscillators. Subsequently, we performed the generation of data and the normal fit and got the thickness 86.167 ± 0.182 nm as shown in Fig. 15 and the GenOsc parameters, as shown in Table 1. This yields an MSE of 13.9 and a pretty good fit for the experimental data, as shown in Figs. 13 and 14.

CoPc is a p-type semiconductor. Usually, these materials are classified as anisotropic and exhibit various optical properties depending on the polarization direction of the light beam. The WVASE software allows assuming such an option and the film description by using different in-plane and out-of-plane components. In terms of the procedure, the GenOsc layer was saved twice as the (CoPc, x) and the (CoPc, z), where (CoPc, x) is the in-plane index and (CoPc, z) is the out-plane index. Let us replace the GenOsc layer with the (CoPc, x) layer. It now represents the complex indices of the anisotropic film N_x or N_y along the x or y axes. Then we added the (CoPc, z) layer above. It represents the complex index N_z along the z-axis. Both (CoPc, x) and (CoPc, z) are coupled into the Biaxial.mat layer at the Mat#2 position [17]. This procedure is similar to that done by O.D. Jordan *et al.* In it, two different Cauchy relations were used to simulate the in-plane refractive index of the film (x, y-direction) and, respectively, the out-of-plane refractive index of the film (z-direction), to determine the anisotropic dielectric function for several copper phthalocyanine (CuPc) layers by spectroscopic ellipsometry [18]. Likewise, J. Sindu Louis *et al.* used two different Cauchy relationships to simulate the dielectric functions inside and outside the plane of the film for zinc phthalocyanine (ZnPc) layers by spectroscopic ellipsometry [19]. In order to obtain an improvement of the model, we have made an assumption that the silicon substrate is covered by SiO_2 on the top, whereas the WVASE software allows



4	biaxial	77.701 nm
3	copc, z	0.000 nm
2	copc, x	0.000 nm
1	sio2	2.556 nm
0	si_jaw	1 mm

Fig. 16. Best fit model for a CoPc film on silicon using the anisotropic GenOsc layer

Table 2. **Values of the parameters of Gaussian oscillator models for an (CoPc, *x*) layer**

ε_1 Offset $= 1.6457 \pm 1.83$		E_n (eV)	B_r (eV)
Type osc.	Amp (eV^2)		
Gauss. 1	2.6914 ± 0.261	1.7304 ± 0.00115	0.080691 ± 0.00202
Gauss. 2	0.16971 ± 0.174	2.2095 ± 0.0189	0.064814 ± 0.0256
Gauss. 3	2.5842 ± 0.983	1.8114 ± 0.0143	0.2219 ± 0.0248
Gauss. 4	0.99246 ± 0.286	2.1365 ± 0.0505	0.26844 ± 0.0533
Gauss. 5	0.47272 ± 0.9	1.7936 ± 0.255	0.38439 ± 0.136
Gauss. 6	1.8775 ± 0.691	4.0826 ± 0.378	1.2115 ± 0.466
Gauss. 7	4.0389 ± 24.2	6.6276 ± 7.87	1.6595 ± 13.6
Gauss. 8	0.14524 ± 0.0503	2.5909 ± 0.0633	0.68064 ± 0.211
Gauss. 9	1.5417 ± 0.422	3.6597 ± 0.0225	0.39656 ± 0.0755

Table 3. **Values of the parameters of Gaussian oscillator models for an (CoPc, *z*) layer**

ε_1 Offset $= 1.8822 \pm 4.75$		E_n (eV)	B_r (eV)
Type osc.	Amp (eV^2)		
Gauss. 1	5.528 ± 2.95	1.8124 ± 0.0147	0.079052 ± 0.0105
Gauss. 2	1.734 ± 0.425	2.152 ± 0.0165	0.10142 ± 0.0263
Gauss. 3	1.292 ± 0.627	1.8702 ± 0.0376	0.10487 ± 0.0457
Gauss. 4	1.3163 ± 0.51	2.1313 ± 0.0508	0.25513 ± 0.0663
Gauss. 5	0.816 ± 0.482	1.6253 ± 0.242	0.56021 ± 0.25
Gauss. 6	3.299 ± 2.34	3.7339 ± 0.205	0.53961 ± 0.44
Gauss. 7	3.503 ± 16.2	5.8581 ± 14.7	1.8023 ± 19.2
Gauss. 8	0.582 ± 3.42	1.2126 ± 10.4	3.0338 ± 11.7
Gauss. 9	2.874 ± 1.44	3.3405 ± 0.0579	0.2489 ± 0.0638

adding a layer called "SiO_2.mat". This layer exists in the WVASE library. Defining the Biaxial layer thickness, the SiO_2 thickness, and the parameters Amp, E_n, B_r, and ε_1 Offset for both (CoPc, x) and (CoPc, z) as a fit parameter and performing a normal fit, we have determined the thickness of 77.701 ± 2.68 nm of the CoPc film, and the SiO_2 layer thickness to be 2.556 ± 2.46 nm, as shown in (Fig. 16). This yields an MSE of 7.01 and a pretty excellent fit to the experimental data, as shown in Figs. 17 and 18. Tables 2 and 3 list the parameters for both (CoPc, x) and (CoPc, z) given by the Fit window in the WVASE software.

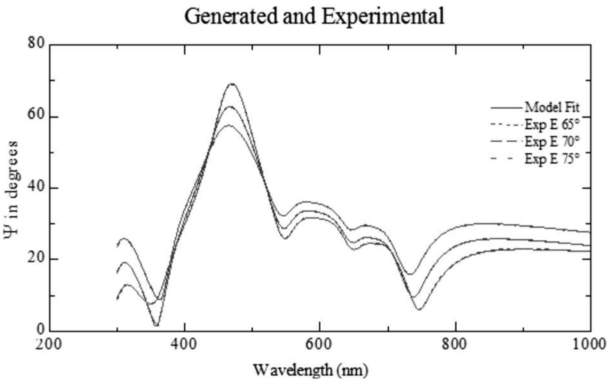


Fig. 17. Best fit generated and experimental ellipsometric Ψ data for a CoPc film on silicon using the anisotropic GenOsc layer

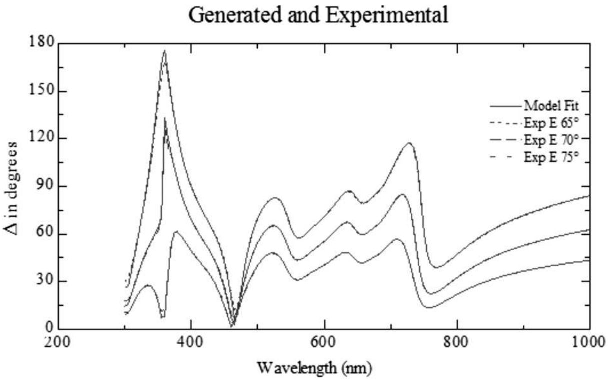


Fig. 18. Best fit generated and experimental ellipsometric Δ data for a CoPc film on silicon using the anisotropic GenOsc layer

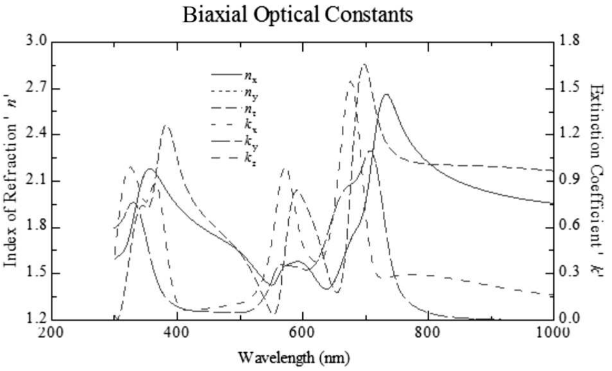


Fig. 19. CoPc film optical constants

Using the built physical model and fitting the generated and experimental data, we got parameters that accurately represent the physical structure of this sample, where a CoPc thickness of 77.701 nm, and the SiO_2 thickness is equal to 2.556 nm. We obtained the optical constants $n\lambda$ and $k\lambda$ of the CoPc film that appear in Fig. 19 for the in-plane and out-of-plane components. These results were presented at the 6th International Conference on Materials Science and Nanotechnology for Next Generation [20]. What was obtained in our research with respect to the optical constants are compatible with the results obtained by A.B. Djurišić *et al.* and Z.T. Liu *et al.* [4, 16], but with a difference that the sample we studied is anisotropic. We mention that Q. Chen *et al.* studied thin films of cobalt phthalocyanine (CoPc) in the wavelength interval 550–800 nm. The complex dielectric function and optical constants of the film were determine. We note that there is a comparatively large absorption region at 600–750 nm for the CoPc film [21], this is clearly shown in our results.

4. Conclusions

In this study, the CoPc thin film was deposited on a silicon substrate by an ultra-high vacuum system. We applied the spectroscopic ellipsometry (SE) using a variable-angle spectroscopic ellipsometer (VASE). We have shown that the uniaxial optical constants of the CoPc thin film can be obtained by using the WVASE software by representing the film in three layers on the silicon substrate. It was necessary to include the additional SiO_2 in order to obtain the reliable optical constants. Finally, the optical constants for the in-plane and out-of-plane components have been found, and the thickness of the film has been precisely determined to be 77.701 nm. The present study can be applied to many kinds of similar organic thin films. We believe that our results will sound importantly in the field of optical properties of organic thin films used as promising elements of devices in optoelectronics.

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ДОСЛІДЖЕННЯ ТОНКИХ ПЛІВОК ФТАЛОЦИАНІНІВ КОБАЛЬТУ НА КРЕМНІЄВИХ ПІДКЛАДИНКАХ МЕТОДОМ СПЕКТРОСКОПІЧНОЇ ЕЛІПСОМЕТРІЇ

Плівку фталоцианіну кобальту (CoPc) на кремнієвій підкладинці було виготовлено за допомогою системи з ультрависоким вакуумом. Особливості структури та оптичні властивості органічного напівпровідника CoPc були визначені методом спектроскопічної еліпсометрії в інтервалі довжин хвиль 300–1000 нм. При обмеженні цього інтервалу до 900–1000 нм знайдено товщину плівки. Поточковою підгонкою визначено поведінку діелектричної функції у всій спектральній області. Таким чином, оптичні властивості були отримані за даними спектроскопічної еліпсометрії із застосуванням математичних моделей на основі гаусових осциляторів. Отримано хороший опис експериментальних даних з відносно малою середньоквадратичною похибкою. При цьому фталоцианін кобальту розглядався як одноосний матеріал.

Ключові слова: спектроскопічна еліпсометрія, фталоцианін кобальту, оптичні константи, гаусові осцилятори, одноосний матеріал.