Optica Applicata, Vol. XLII, No. 1, 2012
DOI: 10.5277/oa120117

Direct determination of the refraction index normal dispersion for thin films of 3, 4, 9, 10-perylene tetracarboxylic dianhydride (PTCDA)

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The improved approach for analysis of the thin film optical spectra exhibiting the interference fringes is presented. It is shown that, based on the positions of adjacent extrema, the interference order numbers can be easily identified allowing for determination of a model-free normal dispersion of the refraction coefficient provided the film thickness is known from an independent measurement. The usefulness of the presented method is illustrated by the analysis of the reflection spectra obtained for thin films of 3, 4, 9, 10-perylene tetracarboxylic dianhydride (PTCDA) with various thicknesses determined with the atomic force microscopy (AFM).

Keywords: optical spectra, interference fringes, refraction coefficient, PTCDA thin films.

1. Introduction

Research on optical properties belongs to basic tools for characterising thin films of various materials. The theory of the optical transmittance (T) and reflectance (R) of thin films on transparent substrates yields complicated expressions being functions of the refraction (n) and extinction (k) coefficients (which, in turn, depend on the wavelength) as well as on the film thickness d [1], thus requiring elaborated computer procedures. A much simpler procedure has been devised for the region of weak absorption (*i.e.*, for the case $n^2 \gg k^2$) involving the construction of continuous envelopes around the extrema of the interference fringes [2, 3]. This procedure, known as the Swanepoel method (see [4, 5] and references therein), was first applied to the transmission spectrum and was subsequently extended over the reflection spectrum [6]. However, the derived expressions for T and R concern homogeneous smooth films

with uniform thickness; moreover, both quantities have to be measured with an extreme accuracy. For real thin films, all these requirements can seldom be fulfilled leading to significant errors in the calculated values of the optical constants and the film thickness (even up to 100%), as already pointed out by Swanepoel himself [7].

Apart from this, it appears that various types of inhomogeneities may compensate each other resulting in a quasi-ideal spectrum for a non-ideal thin film. Namely, the geometrical inhomogeneities, such as non-uniform film thickness and surface roughness, reduce the magnitude of the interference extrema [4, 8–12], while the optical inhomogeneity may increase the transmittance even over that of the uncoated substrate in certain spectral regions [13].

Quite recently, the Swanepoel formulae were questioned [14] which gave rise to further uncertainties concerning the determination of the optical and geometrical parameters of thin films by the envelope method.

Contrary to transmittance and reflectance, the positions of the interference extrema can be determined with high accuracy and independently of the factors influencing the magnitudes of both quantities. Therefore, we propose an improved description of interference fringes in thin films resulting in a model-free refraction index in the region of normal dispersion provided that the basic geometrical parameter, *i.e.*, the film thickness, is determined by an independent method. The usefulness of our approach is illustrated by the analysis of the reflection spectra obtained for thin films of the model organic compound, PTCDA, with various thicknesses determined by AFM.

2. Spectral dependence of the refraction index from the interference fringes

At normal incidence and in the region of weak absorption, *i.e.*, where $n^2 \gg k^2$, the order number m of a given extremum of the interference fringes for a film of thickness d results from the basic equation

$$4nd = m\lambda_{\mathrm{extr}} \tag{1}$$

where the convention of treating m's as integers [2, 6], that may be simply called the interference numbers, has been used. For $n > n_s$ (the refraction index of the substrate), an odd number m (1, 3, 5, ...) corresponds to the wavelength λ_{extr} for which the reflection spectrum has a maximum, while an even m (2, 4, 6, ...) corresponds to a minimum; the situation is reversed in the case of the transmission spectrum.

As pointed out in [15], it is better to present the optical spectra as functions of the photon energy $E = hc/\lambda$ (with h the Planck constant and c the light speed in vacuum) instead of λ since, in the region of weak dispersion of the refractive index, the positions of extrema are almost equidistant and, moreover, at higher photon energies, the interference extrema are much better resolved.

The extremum position E_m for a particular interference number m is therefore given by

$$E_m = \frac{hc}{4n_m d} m \tag{2}$$

where n_m is the refraction index corresponding to the photon energy E_m. For the subsequent extremum located at a higher photon energy E_{m+1}, we have

$$E_{m+1} = \frac{hc}{4n_{m+1} d} (m+1) \tag{3}$$

Combining Eqs. (2) and (3) results in

$$m = \frac{E_m n_m}{E_{m+1} n_{m+1} - E_m n_m} \tag{4}$$

The positions of the interference extrema can be determined quite easily from experiment, and for two consecutive ones (*i.e.*, maximum and minimum or minimum and maximum) E_m and E_{m+1}, the quantity m_{exp} can be defined as

$$m_{\text{exp}} = \frac{E_m}{E_{m+1} - E_m} \tag{5}$$

which generally is not an integer. The difference between m_{exp} and m is equal to

$$\delta = \frac{E_m E_{m+1}(n_{m+1} - n_m)}{(E_{m+1} - E_m)(E_{m+1} n_{m+1} - E_m n_m)} \tag{6}$$

and is positive in the region of normal dispersion of the refractive index considered here (*i.e.*, in the region where the refraction index increases with the photon energy: $n_{m+1} > n_m$). It is also clear that with decreasing dispersion, δ approaches zero. These features of δ allow us to determine the actual interference number m as being not only an integer closest to m_{exp} but also being smaller than m_{exp} since $m = m_{\text{exp}} - \delta$.

For thicker samples with many interference fringes, the adjacent extreme positions are close to each other and the denominator of Eq. (5) may be quite small leading to an uncertainty in determining m; in such a case we can take into account a further extremum at energy E_{m+l} and then

$$m_{\text{exp}} = \frac{l E_m}{E_{m+l} - E_m} \tag{7}$$

For example, the transmission spectrum for a 106.4 μm thick film of polyethylene terephthalate (PET) in the wavelength range 740–760 nm [16] reveals the presence of 25 interference extrema ($l = 24$) between the first minimum with the interference number as high as $m = 923 \pm 2$ at 1.6318 eV and the last minimum with $m = 947 \pm 2$ at 1.6742 eV.

Knowing m for a chosen extremum at low photon energy (in the low-dispersion region), we can easily number the consecutive extrema at higher energies simply as $m + 1$, $m + 2$, *etc*. (see Fig. 3).

Having determined the interference numbers, we can make a plot of E_m vs. m (see Eq. (2)) and its deviation from linearity will be a measure of the refractive index dispersion.

Equation (2) yields the photon energy dependence of the product $n_m d$, demonstrating in fact the relative dispersion of the refractive index, as being multiplied by the constant thickness d

$$n_m d = \frac{hc}{4}\frac{m}{E_m} \tag{8}$$

Finally, a model-free absolute dispersion of the refractive index can be determined provided the film thickness is known from an independent measurement such as profilometry, scanning electron microscopy (SEM) or AFM as in this work

$$n_m = \frac{hc}{4d}\frac{m}{E_m} = \frac{309.96}{d(\mathrm{nm})}\frac{m}{E_m(\mathrm{eV})} \tag{9}$$

Frequently, d is claimed to be calculated from Eq. (8) with a very small uncertainty, even of the order of 1%, using, however, the value of n calculated from formulae for the ideal thin film which is seldom the case. For example, assuming a uniform thickness for an amorphous As-S film, $d = 806$ nm is found, while applying very complicated mathematical procedure that takes into account a small average surface roughness $A_r \cong 20$ nm, a much higher value is obtained, namely $d = 878$ nm which favorably compares with $d = 864$ nm obtained directly with a profilometer [11].

3. Experiment

In order to illustrate the usefulness of our approach, we have chosen the model organic material, *i.e.*, 3, 4, 9, 10-perylene tetracarboxylic dianhydride, abbreviated as PTCDA. Thin films of PTCDA have been deposited onto glass substrates by thermal vacuum evaporation of bulk material (from Aldrich). Five films studied have been examined with an Explorer ToMetrix AFM working in the contact mode. The films are polycrystalline, as checked by X-ray diffraction study, and possess smooth surfaces as evidenced by a small root-mean square (rms) surface roughness presented in column 3 of the Table.

T a b l e. Characteristics of the PTCDA thin films investigated.

Sample	d [nm]	σ_{rms} [nm]	s [eV]	n_0^{exp}	n_0^{fit}
1	216 ± 15	11	0.857	1.67	1.65
2	365 ± 10	7	0.511	1.66	1.66
3	725 ± 40	10	0.250	1.71	1.68
4	1260 ± 50	29	0.142	1.73	1.70
5	2050 ± 70	38	0.087	1.74	1.73

d and σ_{rms} denote the film thickness and rms (root-mean square) roughness, respectively, both determined by AFM; s is the slope of the dashed straight lines from Fig. 4; n_0^{exp} and n_0^{fit} are the zero-energy limits of the refraction coefficient as determined experimentally (from the slope s and thickness d) and from fitting Eq. (12) to the experimental data (Fig. 5), respectively.

As for the film thickness (column 2 in the Table), the films were scratched with a scalpel down to the glass substrate and the thicknesses were calculated by measuring the vertical distance between the substrate and the film surface with an AFM tip (for very thin organic films which are usually soft, an AFM tip may be used also for scratching [17]). An example of the AFM profile after scratching is shown in Fig. 1.

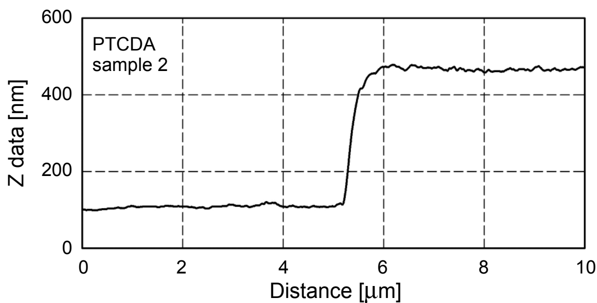

Fig. 1. AFM profile of PTCDA layer on glass (sample 2) after scratching. Height of the step yields the sample thickness $d = 365 \pm 10$ nm.

The optical spectra of the PTCDA films under investigation have been gathered with a JASCO V-570 double-beam spectrophotometer operating in the range from 190 to 2500 nm. For reflection measurements, a special two-beam JASCO unit has been used with an Al mirror in the reference beam as a reflectance standard. The angle of incident light is approximately 5°, which is very close to the normal incidence.

4. Results and discussion

The optical reflection spectra of the PTCDA films investigated (see the Table) are shown in Fig. 2 demonstrating clear interference fringes, the number of which depends on the film thickness.

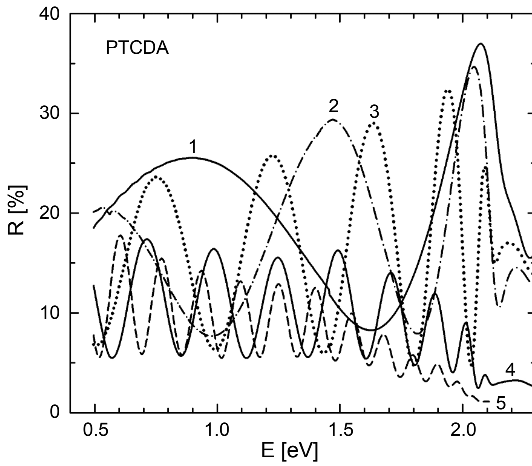

Fig. 2. Reflectance of the PTCDA films with various thicknesses.

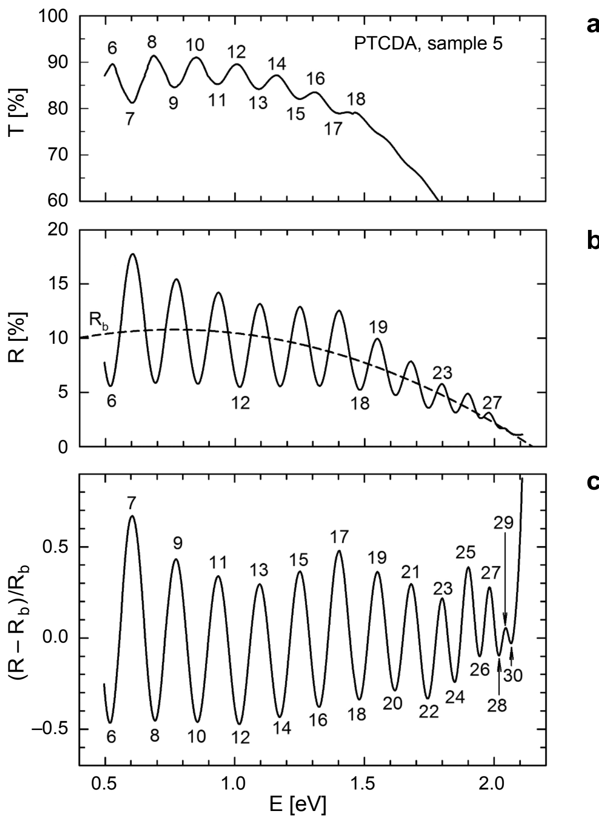

Fig. 3. Transmittance (**a**), reflectance (**b**) and relative reflectance change (**c**) for PTCDA sample 5. Integers indicate the interference numbers of extrema and R_b (dashed line) represents the non-oscillatory background reflectance fitted with a second order polynomial.

We have also measured the transmission spectra *T* and such a spectrum for the thickest sample 5, treated as an example, is shown in Fig. 3**a**, in order to compare it with the reflection spectrum *R* presented once again, in greater detail, in Fig. 3**b**. A clear onset of absorption results in vanishing of the interference fringes in *T* above 1.5 eV. However, the fringes are still observed in *R* up to 2 eV.

The effect of absorption on the optical spectra is such that Eq. (1) is not exactly valid at the interference extrema, as it is in the transparent region, but at the tangent points, *i.e.*, at the points where the envelopes, usually computer-drawn, are tangential to the actual spectrum [4, 5, 8, 18]. In order to eliminate the shift between the tangent- and extreme points, we propose a subtraction of the non-oscillatory background from the total optical spectrum by fitting it with, *e.g.*, a polynomial, as shown in Fig. 3**b** for the reflectance. The resulting relative reflectance change, shown in Fig. 3**c**, allows the interference extrema to emerge up to 2.08 eV. Applying Eq. (4), we have determined the interference numbers (see Fig. 3), clearly showing that the relative reflectance change enables one to observe much more interference fringes (up to $m = 30$) than in the case of the transmission spectrum (only to $m = 18$).

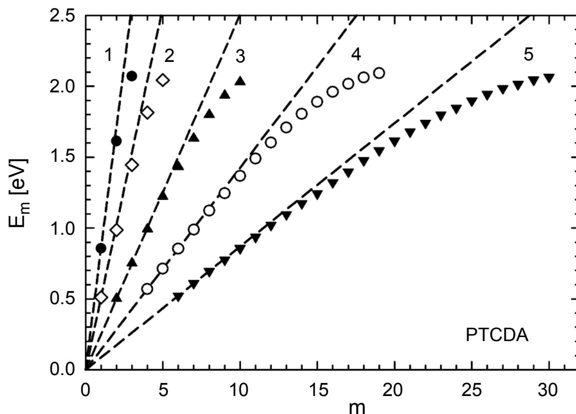

Fig. 4. Energies of the reflectance extrema vs. the interference numbers for the PTCDA films investigated. Dashed lines demonstrate a linear character of this dependence at low energy.

The positions of extrema versus the interference numbers for all the samples studied are presented in Fig. 4, indicating a clear deviation from linearity above 1.5 eV. On the other hand, the low-energy linear part of this dependence, extrapolated to $E = 0$, yields $m = 0$, evidencing the correct interference numbering and inequality $n > n_s$.

The slope of the linear part (column 4 in the Table) gives the product $n_0 d$ (at least its upper limit for the thinnest sample), with n_0 being the zero-energy limit of the refraction coefficient. Knowing the film thicknesses *d* from AFM, we have calculated the values of n_0 (column 5 in the Table).

Finally, the experimentally determined photon energy dependences of the refractive index $n(E)$ for the samples studied at the extreme points are presented in Fig. 5.

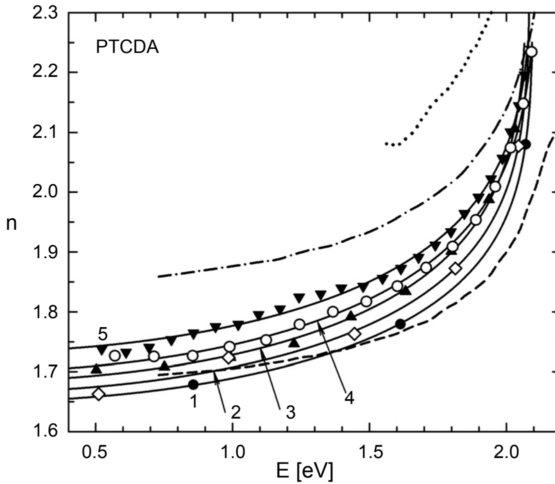

Fig. 5. Dispersion relation of the refractive index of PTCDA thin films. Various symbols represent our experimental data and solid curves – fitting with Eq. (12). For comparison, the ellipsometric results from Refs. [20], [21] and [22] are also shown as dotted, dash-dotted and dashed curves, respectively.

The relative uncertainty $\Delta n/n$ is, at least, equal to $\Delta d/d = 3\%–7\%$ depending on the sample (see the Table), which means that differences between the $n(E)$-dependences for our PTCDA samples lie within the film thickness uncertainty. Moreover, the films under investigation may be more or less compact, which can also contribute to these differences, as clearly seen for a number of TiO_2 thin films [19].

For comparison, we have also inserted in Fig. 5 some ellipsometric results (requiring, however, a model to calculate n) obtained for thin films of PTCDA [20–22]. It should be mentioned that the refraction coefficient of PTCDA samples may change in a wide range, depending on their structure. The highest values are reported for PTCDA single crystals, with the maximal value of $n_0 = 2.3$ [23]. Therefore, the $n(E)$-dependences for PTCDA thin films, which may be polycrystalline and/or less compact than the bulk material, are expected to lie below the highest $n(E)$-dependence of the single crystal, as actually observed in Fig. 5, where $n = 2.3$ is just the upper limit on the scale for the refraction coefficient.

Applying the commonly used phenomenological Cauchy and Sellmeier relations, we came to the conclusion that they did not fit our model-free experimental data well. Therefore, following [24], we have made use of the physically-based Solomon approach [25]. Considering a simple two-band model, the variation in the refractive index takes the form [25]

$$n^2 - 1 = \left(n_0^2 - 1\right)\left[1 + \frac{E}{4\Delta}\ln\frac{E_M^2 - (\Delta - E)^2}{E_M^2 - (\Delta + E)^2}\right] \tag{10}$$

where Δ is the bandwidth, assumed to be the same for both bands, and E_M is the average gap, *i.e.*, the energy difference between the "centres of gravity" of the bands. Within this model, one can introduce the optical gap E_{opt} and then [25]

$$E_M = E_{\mathrm{opt}} + \Delta \tag{11}$$

Substituting Eq. (11) into Eq. (10) we get the final $n(E)$-dependence in the form

$$n^2 - 1 = \left(n_0^2 - 1\right)\left[1 + \frac{E}{4(E_M - E_{\mathrm{opt}})} \ln \frac{(2E_M - E_{\mathrm{opt}} - E)(E_{\mathrm{opt}} + E)}{(2E_M - E_{\mathrm{opt}} + E)(E_{\mathrm{opt}} - E)}\right] \tag{12}$$

clearly showing that the Solomon model is valid for $E < E_{\mathrm{opt}}$. In view of this, it is worth noticing that fitting the exact formula (12) to a 1120 nm thick As–S–Se glassy alloy film from [24], the value of $E_{\mathrm{opt}} = 2.78$ eV is found, while fitting the approximation of Eq. (10) yields $E_{\mathrm{opt}} = 2.08$ eV making the impression that the $n(E)$-dependence may be extended beyond E_{opt} [24].

Solid lines in Fig. 5 represent the global fit of the Solomon relationship yielding $E_M = 4.33 \pm 0.15$ eV and $E_{\mathrm{opt}} = 2.10 \pm 0.02$ eV, in excellent agreement with the absorption data [26]. The assumption of weak absorption used in this work ($n^2 \gg k^2$) is very well fulfilled up to $E_{\mathrm{opt}} = 2.10$ eV, since for this energy $n \approx 2$ and $k \approx 0.1$ [20–23]. The fitting of Eq. (12) to the experimental data gives also the values of n_0 (column 6 in the Table) consistent with those found previously from the slopes in Fig. 4 (column 5 in the Table).

The interference numbering for thin films of PTCDA (see Fig. 3) is a consequence of inequality $n > n_s$, as mentioned in Section 4. For the opposite case ($n < n_s$), the interference numbering is reversed, *i.e.*, odd m's correspond to maxima in T and minima in R, while even m's – on the contrary. This can be illustrated, for example, by the reflection spectrum for a 525 nm thick thermal SiO_2 layer on Si [27]. In the wavelength range 400–800 nm, the spectrum reveals the presence of 4 interference extrema between the first maximum at 1.653 eV and the last minimum at 2.886 eV for which our approach yields the interference numbers $m = 4$ and $m = 7$, respectively. This indicates that $n < n_s$ as expected, since $n = 1.42$–1.44 [27], while $n_s \geq 3.42$ [28].

In a quite recent paper [12], it is stated that the interference numbering requires the prior knowledge whether $n > n_s$ or $n < n_s$; our approach, including extrapolation to $E = 0$ (see Fig. 4), clearly demonstrates that it is not necessary.

The possibility of verifying whether $n > n_s$ or *vice versa* may be very useful in description of various thin film structures, especially new ones. An interesting example is provided by the optical data for amorphous GdN thin films of various thicknesses deposited onto glass with $n_s = 1.51$ [29]. The quantitative analysis of the transmission spectra based on the Swanepoel method (assuming *a priori* that $n > n_s$) yields the minimal value (at 1.1 μm) $n = 1.62 > n_s$, in contradiction with the interference

numbers being odd and even m's for the transmittance maxima and minima, respectively, which means just the opposite, *i.e.*, $n < n_s$.

This example clearly shows that the interference numbering should be treated as a primary feature when analyzing the optical spectra of thin film structures.

5. Conclusions

This work presents the improved approach for analysis of the thin film optical spectra exhibiting the interference fringes. It has been shown how to determine easily the correct interference order numbers without the knowledge of the film thickness. The correct interference numbers, in turn, enable one to distinguish whether the refraction coefficient of a particular thin film is smaller or greater than the refraction coefficient of the substrate. It has also been demonstrated how to determine the exact positions of the interference extrema by subtracting the non-oscillatory background of an optical spectrum. All these procedures serve as a tool for determination of a model--free normal dispersion of the refraction coefficient $n(E)$, provided the film thickness is known from an independent measurement. It has to be stressed that the directly measured film thickness allows one to determine reliable values of the optical constants and to avoid the use of complicated and frequently uncertain mathematical procedures.

The usefulness of our approach has been illustrated by the analysis of the reflection spectra obtained for thin films of PTCDA with various thicknesses determined by AFM, and the experimentally obtained $n(E)$-dependences have been described within the physically-based Solomon model.

The values of $n(E)$, fixed below the optical gap E_{opt}, can also serve as an anchoring point when analyzing the ellipsometric data which require a model for calculation of $n(E)$ in the whole photon energy range under investigation, including that beyond E_{opt}.

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Received May 25, 2011
in revised form October 19, 2011