

Determination of the thickness and optical constants of thin films from transmission spectra

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Abstract

A method has been developed to determine the thickness and the wavelength-dependent refractive indices and absorption coefficients of thin optical films by fitting a five parameter function to measured transmission spectra. Initial values for the fitting parameters are obtained from the transmission values at the spectral maxima and minima of the interference patterns created by the films. This is followed by a systematic variation of those parameters which can cause a failure of the calculation by reaching local minima of the residual sum of squares. The final fitting is performed with a Marquardt algorithm. A Monte Carlo simulation has indicated that the accuracy of this method is at least one order of magnitude better than that of the methods utilising only the interference extrema.

Keywords: Amorphous materials; Optical properties; Optical spectroscopy; Silicon

1. Introduction

Optically thin films are finding increasing usage in a wide variety of electronic and opto-electronic devices. A proper understanding of the behaviour of these devices frequently requires a detailed knowledge of their optical-spectroscopic properties, information which can be difficult to derive because of the interference effects which are inherently present when the film thickness is comparable with the wavelength of radiation employed to investigate the optical-spectroscopic characteristics. These effects arise because of interference between beams multiply-reflected from the air-film and film-substrate interfaces, effects which may be accentuated by the large refractive indices of the materials employed so that the observed transmission maxima and minima frequently exhibit widely varying magnitudes. In the coherent pump-probe spectroscopic techniques which are of such value in characterising the temporal behaviour of optical excitations to the picosecond-femtosecond time scales [1,2] such interference phenomena have proved to be particularly troublesome. Derivation of the true temporal response of these systems to very fast perturbations of the excited state population created by the pump laser requires a means of avoiding the spurious behaviour arising from these interference processes or/and a detailed knowledge of the optical constants for the system [3–5]. These interference phenomena are most commonly observed in the UV-Vis transmission

spectra of thin film samples and, although they can on occasion be troublesome if the desire is simply to record the absorption spectrum, they are not necessarily a drawback since they can be exploited for a simultaneous determination of the thickness of the film, d , its wavelength dependent refractive index, $n(\lambda)$, and its absorption coefficient, $\alpha(\lambda)$. Most frequently the so-called fringe pattern method is used for this purpose, which calculates d , $n(\lambda)$ and $\alpha(\lambda)$ from the transmission and wavelength values at the interference maxima and minima [6–10].

In the present paper we describe a method for the determination of the optical constants and the thicknesses of thin optical films from their UV-Vis spectra using all the measured transmission values, rather than just the transmission extrema, to obtain “best-fit” values for d , $n(\lambda)$ and $\alpha(\lambda)$. A similar target was set in the paper of Klein et al. [11], in which the thickness and $n(\lambda)$ were estimated in the first step from the low absorption part of the spectrum, the form of $\alpha(\lambda)$, the final form of $n(\lambda)$ and the final value of d were computed in a second step, and the Powell method [12] was used for optimisation. We follow a completely different route. In the first step of our method we use the positions of the interference maxima and minima for the estimation of the initial values of the thickness and of the optical constants. In the second step a systematic fitting procedure is used to make sure that the global minimum of the residual sum of squares

is approached. The third step is the final refinement of the parameters with a standard computational method to reach the best fit.

2. Experimental methods

As sample films for validating the methods developed we employed thin (200–4000 nm thickness) amorphous silicon films at various stages of crystallisation, films which have also been employed for study by infrared and Raman spectroscopy, time-resolved laser spectroscopy, transient electron microscopy, transient electron diffraction and photoconductivity [13,14]. These a:SiH films were prepared from silane in a capacitatively-coupled plasma-enhanced chemical vapour deposition (PECVD) reactor [15] in Professor J. Marshall's research group at the Department of Materials Engineering, Swansea. Corning 7059 glass plates were used as substrates. PECVD deposition conditions were: flow rate, 25 sccm SiH₄; pressure, 70 Pa; power density, 0.08 W cm⁻²; substrate temperature, 150 °C; the deposition time was varied between 10 and 40 min to control the film thickness. Furnace crystallised silicon films were made by annealing a:SiH samples at 600 °C in a 100 Pa nitrogen atmosphere for 12 h. Laser crystallised samples were obtained from a:SiH films by irradiation with the 193 nm line of a LPX-105 Lambda Physik laser. The grain size and microstructure of the films were observed by transient electron microscopy, the UV-Vis transmission spectra being recorded on a UVIKON 930 spectrometer.

3. Theory

The model for the calculations is an absorbing film deposited on a transparent substrate, on which a light beam is incident perpendicularly (see Fig. 1.). The media are char-

acterized optically by their refractive indices, n_i , and absorption coefficients, α_i , which are contracted in the complex refractive indices $N_i = n_i - ik_i$, where $k_i = \alpha_i \lambda / 4\pi$ (λ is the wavelength in vacuum). Both the film and the substrate are of uniform thickness.

The interference of light in this system is a rather complicated phenomenon, since there are multiple reflections on the inner side of the air–film interface, on both sides of the film–substrate interface and on the inner side of the substrate–air interface.

It is convenient to consider initially interference in the film only before considering the transmission/reflection characteristics of the film–substrate double layer. Since the thickness of the film is comparable with the wavelength of the light, the transmission of the film, T_{13} , and its reflectivity on its air-side boundary, R_{13} , and substrate-side boundary, R_{31} (Fig. 1(A)), can be obtained by summing the electric fields of the multiply reflected beams in the coherent limit (Fig. 1(B)), yielding [16–18]

$$T_{13} = \frac{n_3}{n_1} \left| \frac{t_{12}t_{23}\exp(-i\delta/2)}{1 - r_{21}r_{23}\exp(-i\delta)} \right|^2 \quad (1)$$

$$R_{13} = \left| \frac{r_{12} + t_{12}t_{21}r_{23}\exp(-i\delta)}{1 - r_{21}r_{23}\exp(-i\delta)} \right|^2 \quad (2)$$

and

$$R_{31} = \left| \frac{r_{32} + t_{32}t_{23}r_{21}\exp(-i\delta)}{1 - r_{21}r_{23}\exp(-i\delta)} \right|^2 \quad (3)$$

In Eqs. (1), (2) and (3) t_{ij} and r_{ij} are the Fresnel coefficients for transmission from medium i to j and for reflection in medium i by medium j , respectively,

$$t_{ij} = \frac{2N_j}{N_i + N_j} \quad (4)$$

$$r_{ij} = \frac{N_i - N_j}{N_i + N_j} \quad (5)$$

and δ is the phase angle

$$\delta = 4\pi(d/\lambda)N_2 \quad (6)$$

In contrast to the film, the thickness of the substrate is larger by orders of magnitude than the wavelength of the light, i.e. we are at the incoherent limit. If we consider a substrate covered with a film of infinite thickness, the substrate transmission, T_{24} , and the reflectivities of the film-side and air-side surfaces, R_{24} , and R_{42} can be obtained by summing the intensities of the multiply reflected beams. Here we need only the expression for T_{24} (Fig. 1(B)):

$$T_{24} = \frac{T_{23}T_{34}\exp(-\alpha_3 d)}{1 - R_{34}R_{32}\exp(-2\alpha_3 d)} \quad (7)$$

where T_{ij} and R_{ij} with $j = i \pm 1$ denotes the transmission and reflectivities of the boundaries,

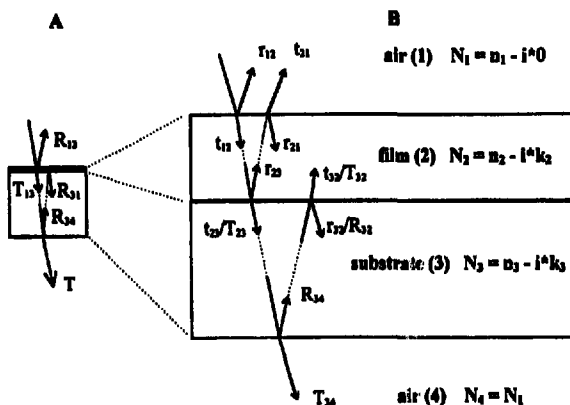


Fig. 1. Transmission of a light beam through an absorbing film-transparent substrate system. For clarity of viewing the incident beams are tilted away from the normal to the surface. (A) the transmission/reflection characteristics T/R are shown; (B) an expanded view of the film is displayed, the transmitted and reflected beam amplitudes being represented by lower case letters t and r .

$$T_{ij} = \frac{n_j}{n_i} |t_{ij}|^2 \quad (8)$$

$$R_{ij} = |r_{ij}|^2 \quad (9)$$

The transmission of the film–substrate double layer system of Fig. 1 can finally be derived by replacing the front surface transmission and reflectivity of the substrate in Eq. (7), T_{23} and R_{32} in Fig. 1(B), by T_{13} and R_{31} obtained for the film in the coherent limit (Fig. 1(A)) and taking into account that the substrate is transparent, i.e. $\alpha_3 = 0$. These substitutions lead to [18]

$$T = \frac{T_{34}T_{13}}{1 - R_{31}R_{34}} \quad (10)$$

The most widely applied version of the “fringe pattern” method has been developed by Swanepoel [8]. The determination of the thickness and the optical constants is based upon an approximate formula for the transmission of the film–substrate system,

$$T = \frac{Ax}{B - Cx \cos \varphi + Dx^2} \quad (11)$$

where

$$A = 16n_1^2n_2^2n_3 \quad (12a)$$

$$B = (n_1 + n_2)^2 [n_1(n_2^2 + n_3^2) + n_2(n_1^2 + n_3^2)] \quad (12b)$$

$$C = 2n_1(n_2^2 - n_1^2)(n_2^2 - n_3^2) \quad (12c)$$

$$D = (n_2 - n_1)^2 [n_1(n_2^2 + n_3^2) + n_2(n_1^2 + n_3^2)] \quad (12d)$$

$$\varphi = 4\pi n_2 d / \lambda \quad (12e)$$

$$x = \exp(-\alpha_2 d) \quad (12f)$$

Eq. (11) can be derived by substituting Eqs. (1), (3), (8) and (9) into Eq. (10) and simplifying the expression obtained by neglecting terms containing k_2 in comparison with terms containing only n_1 , n_2 and n_3 (since $k_2 \ll n_1, n_2, n_3$, almost throughout the spectrum). The expressions (12a)–(12d) differ from those cited in Ref. [8], because we retain n_1 rather than replacing it by unity.

The spectrum contains transmission extrema at wavelengths which fulfil the condition

$$2n_2 d = m\lambda \quad (13)$$

where m is an integer for maxima and a half-integer for minima. The maxima can be connected by a continuous envelope with transmission maxima, T_M , and minima T_m . If we describe the spectrum with the approximate expression (11), the T_M function is obtained with the substitution $\cos \varphi = 1$, the T_m function $\cos \varphi = -1$.

Swanepoel's method calculates the refractive index at the positions of the transmission maxima and minima from the expression

$$n_2 = [E + (E^2 - n_1^2 n_3^2)^{1/2}]^{1/2} \quad (14)$$

where E contains the values of T_M and T_m at the same wavelengths (one of them has to be obtained by interpolation),

$$E = 2n_1 n_3 \frac{T_M - T_m}{T_M T_m} + \frac{n_1^2 + n_3^2}{2} \quad (15)$$

Values for the thickness are obtained from the expression

$$d = \frac{\lambda' \lambda''}{2(\lambda' n_2'' - \lambda'' n_2')} \quad (16)$$

where the upper indices denote two adjacent transmission maxima or minima. To improve the accuracy of the calculations the average of the results for d obtained directly from Eq. (16) is substituted in Eq. (13) and the values of m for the individual extrema are determined. They are rounded and using these integer and half-integer numbers for m , d is calculated again from Eq. (13). Taking the average of the new d values, n_2 is also calculated from Eq. (13). Finally, the absorption coefficient is computed from the expression

$$x = \frac{F - [F^2 - (n_2^2 - n_1^2)^2 [n_1(n_2^2 + n_3^2) + n_2(n_1^2 + n_3^2)]^2]^{1/2}}{(n_2 - n_1)^2 [n_1(n_2^2 + n_3^2) + n_2(n_1^2 + n_3^2)]} \quad (17)$$

where for the transmission maxima

$$F = \frac{8n_1^2 n_2^2 n_3}{T_M} + n_1(n_2^2 - n_1^2)(n_2^2 - n_3^2) \quad (18)$$

and for the minima

$$F = \frac{8n_1^2 n_2^2 n_3}{T_m} - n_1(n_2^2 - n_1^2)(n_2^2 - n_3^2) \quad (19)$$

The data calculated from expressions (14), (16) and (17) were employed as starting parameters in the fuller analysis of the extensive data contained within the observed spectra. A description of the procedures involved in fitting the observed spectra with Eq. (10) is given in Section 4.

4. Results and discussion

As mentioned in the previous section, our fitting procedure starts with an initial estimation of the thickness and the $\alpha(\lambda)$ and $n(\lambda)$ functions of the silicon films by using Swanepoel's version of the fringe pattern method [8]. Profiles of the transmission maxima and of the transmission minima were calculated by parabolic interpolation through the measured transmission maxima, T_M , and the transmission minima values, T_m , with the exception of very thin films ($d < 500$ nm), for which an exponential function with three constants, $C_1 \exp[(C_2 - \lambda)/C_3]$, was found to represent the envelope curves much better. The fringe pattern method has been found to give satisfactory results for the ~ 1000 nm thick films: in the case of much thinner films the obtained d values seemed reliable, but because of the low number of transmission extrema in the spectra the forms of the $\alpha(\lambda)$ and $n(\lambda)$ func-

tions are uncertain. The method proved unsatisfactory for films much thicker than 1000 nm since the fringes in the spectra fall very close together, the d values calculated from Eq. (16) showed large scatter and the orders of interference obtained from Eq. (13) became ambiguous.

We chose the same type of functions to describe the wavelength dependence of the optical constants as those in Ref. [8]:

$$n(\lambda) = a_1/\lambda^2 + a_2 \quad (\lambda \text{ in nm}) \quad (20)$$

and

$$\log_{10}\alpha(\lambda) = a_3/\lambda^2 + a_4 \quad (\alpha \text{ in nm}^{-1}, \lambda \text{ in nm}) \quad (21)$$

This means that the transmission function (10) contains five parameters, a_1 , a_2 , a_3 , a_4 and d . This function can be fitted to the measured spectrum by minimising the sum of the squares of the deviations of the calculated transmission from the measured one,

$$S^2 = \sum_i [T_m(\lambda_i) - T(\lambda_i, a_1, a_2, a_3, a_4, d)]^2 \quad (22)$$

where $T_m(\lambda_i)$ is the measured transmission at wavelength λ_i and $T(\lambda_i, a_1, a_2, a_3, a_4, d)$ is the transmission at wavelength λ_i calculated from Eq. (10), after substituting Eqs. (1)–(9) in this equation and using the $n(\lambda)$ and $\alpha(\lambda)$ functions given by Eqs. (20) and (21).

Much care has to be taken in the fitting of a non-linear function to find the global minimum of the sum of the squares when local minima may exist in the parameter space. The existence of local minima was checked by computer simulation, values of the transmission were calculated by Eqs. (1)–(10), (20) and (21) in 1 nm steps in the 500–900 nm wavelength range attributing the following values for the individual parameters of the selected sample film:

$$\begin{aligned} a_1 &= 3 \times 10^5 & a_2 &= 2.6 \\ a_3 &= 1.5 \times 10^6 & a_4 &= -8.0 & d &= 1000 \text{ nm} \end{aligned} \quad (23)$$

The resulting simulated spectrum was used with each $T_m(\lambda_i)$ value for all the λ_i values considered to determine/plot the sum of the squares S^2 defined by Eq. (22) as a function of the a_1 parameter. a_1 was varied systematically between 0 and 10^6 , the values of the other four parameters being kept unchanged. The obtained $S(a_1)^2 - a_1$ curve characterises the sensitivity of the sum of the squares to a_1 . Analogous curves were computed for the other four parameters, varying the values of the individual parameters in the following ranges: $0 < a_2 < 5.0$; $0 < a_3 < 3 \cdot 10^6$, $-20 < a_4 < 0$ and $0 < d < 5000$. The sum of the squares functions are displayed in Fig. 2. As can be seen, $S(a_1)^2$ does have local minima, although only few and they are relatively shallow, $S(a_2)^2$ and $S(d)^2$ display more local minima, $S(a_3)^2$ and $S(a_4)^2$ do not have local minima.

For the same film further calculations were performed for $S(a_1)^2$, $S(a_2)^2$, $S(a_3)^2$ and $S(a_4)^2$, with $d = 300$, 500 and 1000 nm. It was found that the number of minima of $S(a_1)^2$ and $S(a_2)^2$ increases with the thickness, $S(a_3)^2$ and $S(a_4)^2$ always have a single minimum.

The transmission curves of the thinner ($d \leq 1000$ nm) films were directly fitted with the Marquardt algorithm [19], which is said to be relatively insensitive to the local minima of the target function. In these calculations the initial values for a_1 , a_2 , a_3 , a_4 and d were those obtained with the fringe pattern method. In the case of the thicker films it has been found easier to avoid the local minima by varying first, a_1 , a_2 and d systematically in a $\pm 50\%$ range around the values obtained with the fringe pattern method and to use the Marquardt algorithm only for a final refinement.

To compare the accuracies of the fringe pattern method and our fitting procedure, computer simulations were performed with a Monte Carlo method. 100 spectra were simulated, using the values of Eq. (23) for the five parameters and adding errors to the transmissions and to the wavelengths. It was presumed that the errors can be described with a normal distribution and standard deviations of $\sigma_T = 0.005$ for transmission and $\sigma_\lambda = 0.5$ nm for the wavelength, respectively.

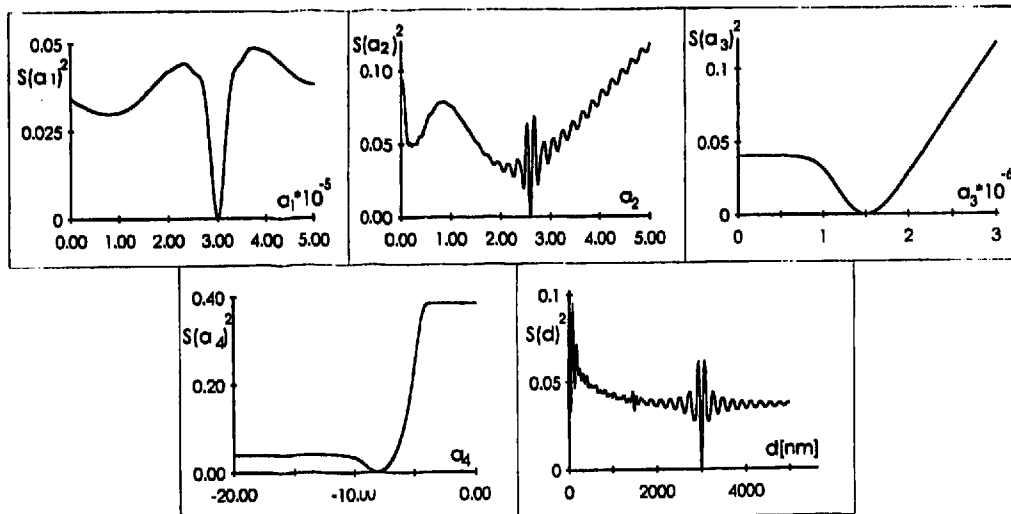


Fig. 2. The dependence of the sum of the squares in Eq. (22) on the parameters, a_1 , a_2 , a_3 , a_4 and on the film thickness.

Table 1

The average mean square errors of the parameters of the transmission function (in %) obtained in the Monte Carlo simulation

Method	$\chi(a_1)$	$\chi(a_2)$	$\chi(a_3)$	$\chi(a_4)$	$\chi(d)$
Swanepoel's	6.9	3.4	9.5	11	0.11
Fitting of full spectrum	0.044	0.021	0.14	0.084	0.018

The values of the five parameters were determined from each spectrum with the two methods and the average mean square errors, related to the original values of the parameters

$$\chi(\Theta) = \frac{[\sum_i^M (\Theta_i - \Theta^*)]^2}{M \cdot \Theta^*} \quad (24)$$

(Θ stands for a_1 , a_2 , a_3 , a_4 or d and $M=100$ is the number of the simulated spectra) were computed to characterise the magnitude of the deviations from the original values, Θ^* . The results are summarised in Table 1. As can be seen, the fitting method leads to $\chi(a_1)$, $\chi(a_2)$, $\chi(a_3)$ and $\chi(a_4)$ values two orders of magnitude smaller than those obtained by the fringe pattern method. In the case of the thickness the improvement is one order of magnitude. As was hoped, the accuracy of the calculations utilising all the spectral points are significantly better than those utilising only the interference extrema.

To illustrate the method we have discussed the calculation of $\alpha(\lambda)$, $n(\lambda)$ and d for a relatively thick a:SiH film. The measured and the calculated spectra are displayed in Fig. 3. When we computed the five fitting parameters with Swanepoel's method using only those T_M and T_m values which fall above 700 nm (i.e. neglecting the interval of strong absorption), we obtained $a_1 = 7.2 \cdot 10^4$, $a_2 = 3.876$, $a_3 = 7.827 \cdot 10^5$, $a_4 = -5.399$ and $d = 2664$ nm. Following the systematic variation a_1 changed to $1.07 \cdot 10^5$, a_2 remained unchanged, d became 2504 nm. The fitting of the transmission function minimising the sum of the squares with a Marquardt algorithm produced $a_1 = 1.03 \cdot 10^5$, $a_2 = 3.180$, $a_3 = 7.91 \cdot 10^5$, $a_4 = -5.430$ and $d = 2590$ nm. The mean squares function,

$$s = \sum_i^L \frac{[T_m(\lambda_i) - T(\lambda_i, a_1, a_2, a_3, a_4, d)]^2}{L-5} \quad (25)$$

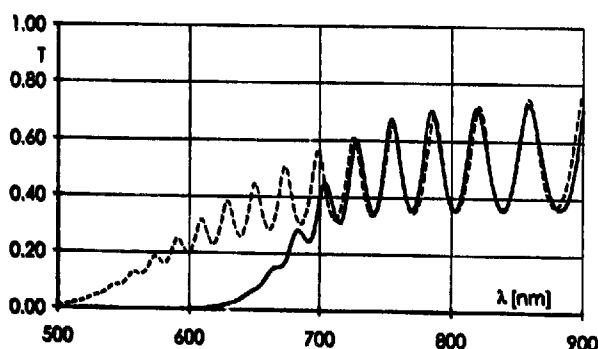


Fig. 3. Comparison of the measured (solid line) and the calculated (dashed line) spectra of an a-Si:H film.

was calculated as 1.42×10^{-3} using only the fringe pattern method, decreasing to 1.13×10^{-3} after the systematic variation and to 1.11×10^{-3} following the fitting with a Marquardt algorithm. The agreement is very good in the 700–900 nm range for which the data was fitted. Below 700 nm, however, there is a large deviation between the calculated and measured spectra. This deviation on the shorter wavelength side is due to the fact that $n(\lambda)$ and $\alpha(\lambda)$ cannot be described with the simple empirical formulae (20) and (21) as the absorption becomes significant. The poorer fit on the long wavelength side might be due to the imperfect performance of the spectrometer at the end of its operational range.

5. Conclusion

We have tested this method on a great number of amorphous and polycrystalline silicon films. It has proved efficient for the determination of the thickness and the optical constants. The empirical formula of Eq. (20) proved satisfactory to describe the $n(\lambda)$ functions of all the samples. The $\alpha(\lambda)$ function of the amorphous silicon samples could be represented satisfactorily by the logarithmic formula of Eq. (21), but the slower rising absorption edge of the polycrystalline films could be described much better by replacing $\log_{10} \alpha(\lambda)$ by $\alpha(\lambda)$ in the second order function of Eq. (21). Clearly the quality of a fit will depend on the choice of the functions employed to represent the wavelength-dependent refractive indices and absorption coefficients for the materials of which the films are composed and a better representation of $n(\lambda)$ and $\alpha(\lambda)$ in place of those represented by Eqs. (20) and (21) would undoubtedly improve the fit in the shorter wavelength regions of strong absorption (e.g. below 700 nm in Fig. 3). An empirical form of $\alpha(\lambda)$ could be derived from the wavelength-dependent contours of the fringe transmission maxima and minima, thereby providing a key to a suitable analytical function to represent the absorption of any particular sample. Without a well-defined analytic function for $\alpha(\lambda)$, however, it is doubtful whether a simple function of the form given by Eq. (21) will provide a better fit in regions of strong sample absorption.

It is believed that our method can be employed for the determination of the thickness and the optical constants of films made of any material, within the limitations of the assumed form of the $n(\lambda)$ and $\alpha(\lambda)$ functions. It is to be stressed that the evaluations of the optical constants described in this paper are based only on the measurements of the transmission spectra of the thin film samples studied. Further information is undoubtedly available, however, from the related reflectance spectra of the same sample, information which could well be employed to refine the fitting procedure and perhaps avoid the dependence on assumed forms for the $n(\lambda)$ and $\alpha(\lambda)$ functions. Investigations into these possibilities are currently under way.

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