

Determination of optical constants of thin film from reflectance spectra

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We present a method of determining optical constants n and k of a thin film using only the reflectance $R(\omega)$ curve (normal incidence reflectance spectroscopy). The method is based on the simultaneous use of Fresnel laws and dispersion relations between n and k of the film, via an iterative process. To illustrate the method, optical constants in the VUV of a film grown on InP were determined. A second example with a SnO_2 film shows how the method can reduce the effect of experimental errors when two sets of spectroscopic data are available.

I. Introduction

The optical properties of a perfectly homogeneous slab of material can be basically described by its complex refraction index $\tilde{N} = n + ik$ and thickness d . Deposited onto a substrate (index \tilde{N}_s , thickness d_s), the film forms a system whose global optical properties (reflectance, transmittance, phase shifts) are well described by the generalized Fresnel laws, including coherent interferences between the different faces of the system.¹⁻³

The inverse problem, i. e., the determination of n and k of the film from optical measurements, is more difficult and numerous solutions, photometric or polarimetric, have been proposed.

The simplest photometric method is to deduce the \tilde{N} value from normal incidence reflectance R and transmittance T of the film, providing the thickness d is known. Numerous papers relate to this method, from manual graphic solutions⁴ to methods using extensive computer aid.⁵⁻¹¹ The main problem arising in this method is that, for certain values of R and T , small errors in the experimental data give large deviations of the resulting n values⁶⁻⁸; this point being correlated to multiple solutions of the Fresnel equations.

To avoid these problems, several authors have proposed additional measurements for which methods are described in Ref. 2. In the VUV range, oblique incidence reflectance and, eventually, transmittance measurements are probably the best way to determine n , k ,

and d .^{12,13} Normal incidence measurements with different film geometry¹⁴⁻¹⁶ are also proposed.

Totally different methods concern polarimetric measurements where phases of the polarized beams are determined. Ellipsometry¹⁷ and other related methods will not be discussed here because of the lack of simple polarizers in the VUV range, which is our main domain of interest. However, the thickness and the low energy optical properties of our samples are usually determined by classic ellipsometry.

Another way to obtain additional data is to use the causality laws, also known as dispersion relations or Kramers-Kronig relations. These expressions link the two parts of any analytical complex quantity such as the $n(\omega)$ and $k(\omega)$ indices or the reflectance $R(\omega)$ and the corresponding phase shift $\theta_R(\omega)$. However, these Kramers-Kronig relations, simple for bulk material, are not all valid for a thin film and several alternatives have been proposed, as follows:

The first method is to apply the dispersion relation giving the phase shift in transmission from the $T(\omega)$ curve:

$$\theta_T(\omega) = -\frac{\omega}{\pi} \int_0^{\infty} \frac{\ln T(\omega') d\omega'}{\omega'^2 - \omega^2},$$

which has the same form as the phase shift in reflection for bulk material due to the analytical properties of the Fresnel equation of transmittance. It is clear that this method can be applied only if the substrate is transparent in the studied energy range,¹⁸⁻²¹ which is not the case for semiconductors in VUV.

The second method consists in applying the dispersion relation to the reflectance $R(\omega)$ to obtain the phase shift $\theta_R(\omega)$. This relation for film on substrates²² does not have the usual form and presents new unknown quantities discussed in Sec. II.B.

The third possibility to avoid the difficulties inherent in the two previous methods is to express the optical constants of the film as a sum of functions

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satisfying the causality condition (Lorentzian or Drude expressions). The parameters and the number of these functions are adjusted until a good fit is obtained between the calculated and the experimental data.²³⁻²⁵

The method has also been applied on ellipsometric data^{26,27} and appears to be quite suitable when the optical index $\tilde{N}(\omega)$ presents few structures. However, multiple solutions can also be obtained here because the three methods presented above are based on the same mathematical relations.

The main idea of the method presented here is to combine the Fresnel laws and the dispersion relations of $\tilde{N}(\omega)$ because these relations are simple and independent of any optical configuration. We will show that our method can be applied easily to the analysis of reflectance data as well as other spectroscopic data.

II. Mathematical Framework

A. Basic Optical Formula

For the sake of simplicity, we will consider here the reflection and transmission properties of a single film onto a substrate. The specular reflected and transmitted intensity in the case of normal incidence are given by:

$$R = |\tilde{\rho}|^2, \tilde{\rho} = \frac{\tilde{r}_1 + \tilde{r}_2 \tilde{e}^2}{1 + \tilde{r}_1 \tilde{r}_2 \tilde{e}^2} \tag{1}$$

$$T = n_s |\tilde{\tau}|^2, \tilde{\tau} = \frac{\tilde{t}_1 \tilde{t}_2 \tilde{e}}{1 + \tilde{r}_1 \tilde{r}_2 \tilde{e}^2} \tag{2}$$

where

$$\tilde{r}_1 = \frac{1 - \tilde{N}}{1 + \tilde{N}} \quad \tilde{t}_1 = \frac{2}{1 + \tilde{N}}$$

$$\tilde{r}_2 = \frac{\tilde{N} - \tilde{N}_s}{\tilde{N} + \tilde{N}_s} \quad \tilde{t}_2 = \frac{2\tilde{N}}{\tilde{N} + \tilde{N}_s}$$

$$\tilde{e} = \exp\left(i \frac{2\pi}{\lambda} \tilde{N} d\right),$$

and \tilde{r}_1, \tilde{t}_1 and \tilde{r}_2, \tilde{t}_2 are the respective Fresnel reflection and transmission coefficients for the vacuum layer and the layer-substrate boundary (Fig. 1).

For a certain wavelength λ , R and T are easily calculated by these expressions. The inverse deduction may give several \tilde{N} solutions as discussed in Sec. IV.

B. Dispersion Relation Applied to $R(\omega)$

According to the possibility of zeros in the values of $R(\omega)$ for complex values of ω , it can be demonstrated that the phase shift at the reflection may be deduced from

$$\theta_R(\omega) = -\frac{\omega}{\pi} \int_0^\infty \frac{\ln R(\omega') d\omega'}{\omega'^2 - \omega^2} + 2 \sum_j \arctan \frac{\omega - a_j}{bj}, \tag{3}$$

the summation being over the j zeros (located at $\tilde{p}_j = \pm a_j + ib_j$) of the $\rho(\tilde{p})$ function in the complex upper plane.

This expression has already been used assuming particular approximations not valid here. In Refs. 22

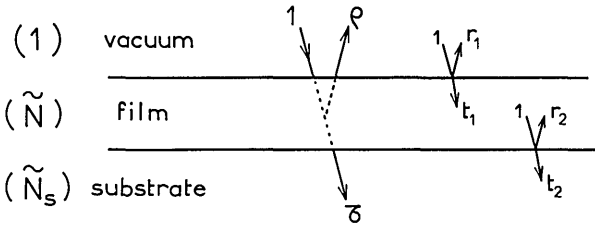


Fig. 1. Representation of the film-substrate system. Definition of the symbols used in Eqs. (1) and (2).

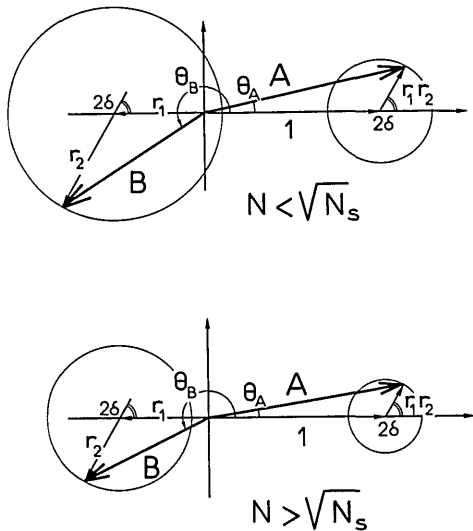


Fig. 2. Vector diagram corresponding to a transparent film of index N on a transparent substrate of index N_s in the two cases $N < \sqrt{N_s}$ (i.e., $r_1^2 < r_2^2$), and $N > \sqrt{N_s}$, with $2\delta = 4\pi/\lambda N_d = 2\omega/c N_d$.

and 28, zeros are assumed to lie on the imaginary axis, which is not generally the case for absorbing film on absorbing substrates. In Ref. 29, the arctan term is replaced by a linear expression, which applies only in some special cases.

The values of \tilde{p}_j are the solutions of $\tilde{r}_1(\tilde{p}) + \tilde{r}_2(\tilde{p})\tilde{e}^2(\tilde{p}) = 0$ and can only be deduced if $\tilde{N}(\tilde{p})$ and $\tilde{N}_s(\tilde{p})$ are known, which is not the case. We will show below that, in general, neither the existence nor the position of zeros in $\tilde{R}(\tilde{p})$ can be deduced from experimental data, greatly reducing the use of the aforementioned dispersion relation.

Let us suppose for simplicity, that the film and the substrate are transparent (indices N and N_s real). The phase shift θ_R can then be determined graphically by the sketches in Fig. 2, where \tilde{B} and \tilde{A} are the numerator and denominator of the expression of $\tilde{\rho}$. The phase shift θ_R is given by the difference of the arguments of \tilde{B} and \tilde{A} :

$$\theta_R = \theta_B - \theta_A.$$

It can be seen that θ_B can make one or several complete turns around the origin, in the case of $N < \sqrt{N_s}$, when ω and δ varies. When ω varies from zero to infinity, the phase shift θ_R can then vary by several 2π units, depending on the relative magnitude of N and N_s in the different spectral ranges.

This deduction is similar for absorbing media and only a precise knowledge of $\tilde{N}(\omega)$ and $\tilde{N}_s(\omega)$ allows us to predict the behavior of $\theta_R(\omega)$. This result can explain the role of the two terms in Eq. (3): as the integral term takes values usually within a limited range ($0-\pi$), the arctan term is related to the possibility of \tilde{A} turning around the origin.

In conclusion, the number of zeros \tilde{p}_j of the relation (1), is related to the number of interference fringes in the whole spectral range of $R(\omega)$, counting only those with $|\tilde{r}_1|^2 < |\tilde{r}_2|^2$. This number cannot be easily known because interference structures can be confused with optical constant structures on the reflectance curve. Equation (3) giving θ_R can, then, only be used in some special cases as for very thin films when absolutely no interference minimum occurs in the studied energy range.

C. Dispersion Relation for \tilde{N}

The dispersion relations for the index of the film are the same as for bulk materials:

$$\begin{cases} n = 1 + \frac{2}{\pi} \int_0^\infty \frac{\omega' k(\omega') d\omega'}{\omega'^2 - \omega^2} \\ k = -\frac{2\omega}{\pi} \int_0^\infty \frac{n(\omega') d\omega'}{\omega'^2 - \omega^2} \end{cases} \quad (4)$$

There is no way of inserting these relations into the Fresnel laws in order to eliminate n or k . We will show that an iterative process works as well.

Let us first discuss how dispersion relations can be related to the Fresnel laws giving R . For each ω value there is an infinity of couples (n, k) giving the corresponding measured value of reflectance R . These couples lie in the complex \tilde{N} plane on one or several continuous closed lines (Fig. 3). If one draws successive complex \tilde{N} planes for different ω values, the preceding lines in the (n, k, ω) space, form a surface having the shape of several tubes which can mix or diverge for certain ω values (Fig. 4).

If we introduce the dispersion relations, these solutions reduce to one line drawn on the aforementioned surface. According to the following equivalent form of Eqs. (4):

$$\begin{cases} n = 1 + \frac{1}{\pi\omega} \int_0^\infty \frac{d[\omega' k(\omega')]}{d\omega'} \ln \left| \frac{\omega' + \omega}{\omega' - \omega} \right| d\omega' \\ k = -\frac{1}{\pi} \int_0^\infty \frac{d[n(\omega')]}{d\omega'} \ln \left| \frac{\omega' + \omega}{\omega' - \omega} \right| d\omega', \end{cases}$$

it can be seen that the maxima of n correspond to the maxima of the slope of $[\omega k(\omega)]$ and the maxima of k to the maxima of the slope of $[-n(\omega)]$. Consequently, the line solution of the dispersion equations circles the tube solution of the Fresnel laws, helicoidally in an anticlockwise direction, each loop corresponding to a multiple of 2π change in the phase shift θ_R .

As the shape of the solution surface may be complicated, we cannot prove definitively that the solutions $n(\omega)$ and $k(\omega)$ satisfying all the equations are unique. This fact is mathematically equivalent to the problem of the number of zeros of $\tilde{\rho}(\tilde{p})$, which cannot be pre-

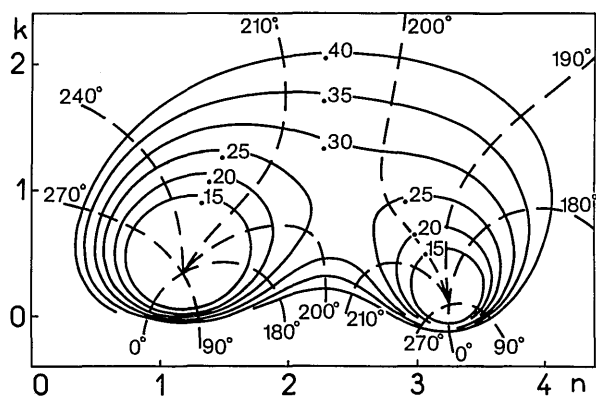


Fig. 3. Plot of the contours of constant normal incidence reflectance $R(n, k)$ (solid lines) and constant phase shift θ_R (dashed lines) of a thin film (index $\tilde{N} = n + ik$, thickness $d = 220$ Å) on a substrate ($n_s = 0.742$, $k_s = 1.015$) at $h\nu = 9.5$ eV).

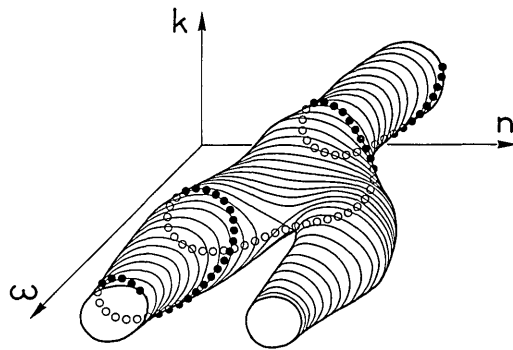


Fig. 4. Example of surface representing the normal incidence reflectance solutions $R(n, k, \omega) = R_{\text{exp}}(\omega)$. The dotted line represents the solution obtained when the dispersion relations $k \leftrightarrow n$ are introduced.

dicted unambiguously. In fact, as Eqs. (4) are better defined than Eq. (3), every system we have tested at present has given only one solution with Eqs. (4) when $R(\omega)$ is known in a sufficiently large spectral range.

The problem of unicity is very different when only a part of the spectrum of $R(\omega)$ is measured. To perform the integrals of Eq. (3) or Eqs. (4), we must arbitrarily introduce extrapolations of R , n , or k . These extrapolations induce different values of the calculated constants inside the domain where $R(\omega)$ is measured and may introduce or suppress zeros of $\tilde{\rho}(\tilde{p})$. Numerous tests have been performed on experimental data, trying different extrapolations and/or truncating the experimental range. It appears that the smallest measured range corresponds to the less defined curve, including several sets of different solutions. On the contrary, as the measured range increases, the number of observed solutions reduces accordingly.

To resume, when the interval of the measured values of $R(\omega)$ increases from zero (one experimental point) to the whole $(0, \infty)$ range, the number of $\tilde{N}(\omega)$ solutions of both Fresnel and dispersion equations reduces from infinity to one solution, but in an unforeseeable manner. This rather qualitative result is nevertheless useful in practical analysis.

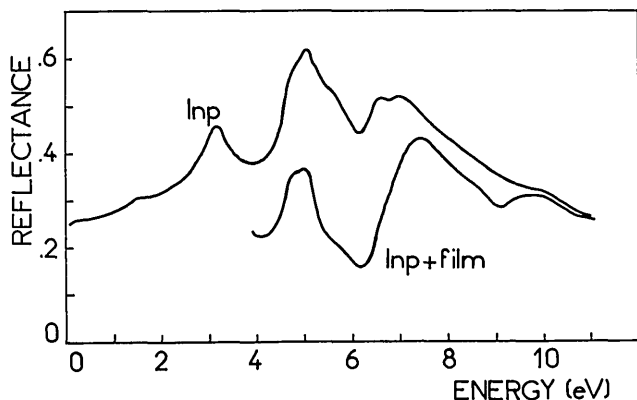


Fig. 5. Normal incidence reflectance of InP and InP + InPO₄ film (thickness $d = 220$ Å).

III. Determination of the Optical Constants From $R(\omega)$ Data

As an example, we have chosen reflectance data in the VUV range for a native dielectric layer of InPO₄ grown electrochemically on InP.^{30,31} This compound is transparent up to 5.5 eV as deduced from ellipsometric data, the optical gap of this dielectric appearing at higher energy where the substrate InP is totally opaque. Figure 5 represents normal incidence reflectance of InP and InP + film, 220 Å thick as deduced from ellipsometry.

In the measurement range (4–12 eV), according to the low value of the thickness, we expect to see the onset of the absorption of the film superposed with few interference structures.

To combine dispersion relations with the Fresnel laws, we adopt a symmetrical iterative method, similar to the one we have used in Ref. 32.

We start with an arbitrary $\tilde{N}_0(\omega)$ function (constant or Lorentzian) as close as possible to the expected result.

For each ω value, we introduce a correction $\delta\tilde{N}(\omega)$ which adjusts the calculated reflectance to the experimental one.

We adopt, in fact, the linear approximation along the gradient of R :

$$\delta n = (R_{\text{exp}} - R_{\text{calc}}) \frac{\frac{\partial R}{\partial n}}{\left(\frac{\partial R}{\partial n}\right)^2 + \left(\frac{\partial R}{\partial k}\right)^2}$$

$$\delta k = (R_{\text{exp}} - R_{\text{calc}}) \frac{\frac{\partial R}{\partial k}}{\left(\frac{\partial R}{\partial n}\right)^2 + \left(\frac{\partial R}{\partial k}\right)^2}$$

which leads to the smallest modulus $|\delta\tilde{N}|$.

Neither index $\tilde{N}_0(\omega)$ nor index $\tilde{N}_1(\omega) = \tilde{N}_0(\omega) + \delta\tilde{N}(\omega)$ are coherent, relative to the dispersion relations. We then compute $\tilde{N}_2(\omega)$ using Eqs. (4):

$$\begin{cases} n_1 \rightarrow k_2 \\ k_1 \rightarrow n_2 \end{cases}$$

As (n_1, k_2) and (n_2, k_1) are coherent, the value

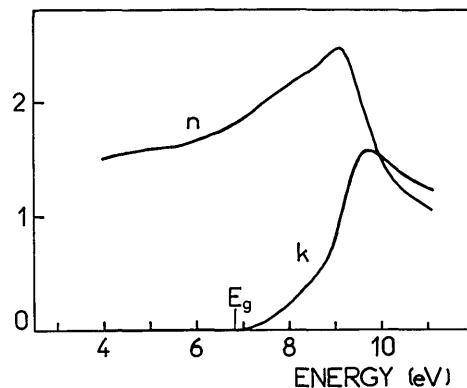


Fig. 6. Real part n and imaginary part k of the refractive index \tilde{N} of the InPO₄ film as deduced from our iterative calculation.

$$\tilde{N}_3 = \frac{1}{2}[(n_1 + n_2) + i(k_1 + k_2)]$$

is a complex constant coherent and symmetrical in the treatment of n and k .

We reiterate the process, taking \tilde{N}_3 as a new approximation of the result. Several tens of iterations are needed to fit the experimental $R(\omega)$ curve properly. In Fig. 6, we report the results of the deduced $\tilde{N}(\omega)$ with the following extrapolations:

$k(\omega)$ has been taken as zero below 4 eV according to the results of ellipsometry;

$k(\omega)$ is assumed to be of the form $\omega^{-\alpha}$ above 12 eV. The factor α is determined to get the correct ellipsometric n and k values between 4 and 5 eV; and

$n(\omega)$ is constant outside the measurement range.

Observing Figs. 5 and 6, we can conclude that the optical gap occurs around 6.9 eV and is not related to any reflectance structure. Further studies not detailed here have shown that:

the 6-eV minimum is related to the first interference minimum;

the reflectance maxima at 5 eV and 7.5 eV are due to the structures in the substrate reflectance seen through the film, and that

the structure above 9 eV is the onset of the opacity of the film, the reflectance of the system reducing to $|\tilde{r}_1|^2$.

IV. Use of Dispersion Relations in the Case of Classic \tilde{N} Determination

When two independent measurements such as R and T are performed (at one wavelength), the complex indices \tilde{N} of the film can be easily deduced according to Eqs. (1) & (2). Unfortunately, experimental errors or surface alterations can affect the R and T values thus giving a (n, k) result, at times very far from the true values. We shall show below that by forcing $n(\omega)$ and $k(\omega)$ to be related by the dispersion relations, this can help to detect and reduce those experimental errors. As the structures of $n(\omega)$ and $k(\omega)$ are perfectly related by Eqs. (4), physically absurd values of n (or k) resulting from the application of Fresnel laws can be rejected as inconsistent with that of k (or n).

As an example, we have chosen R and T measurements on a 4200 Å-thick film of SnO₂ on silica between

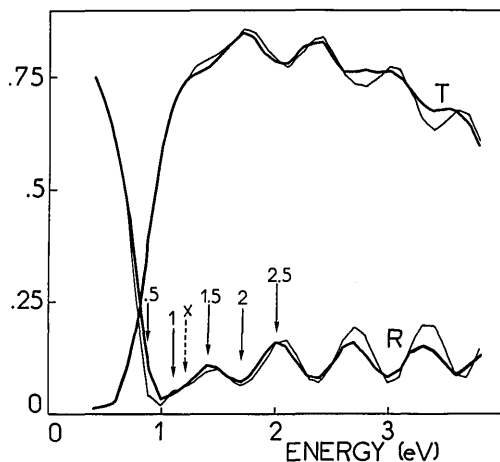


Fig. 7. Normal incidence reflectance R and transmittance T of a SnO_2 film (thickness $d = 4200 \text{ \AA}$) on silica. The heavy lines represent the experimental data; the thinner lines represent the best fit by our iterative method. Interference order $l = (2Nd/\lambda)$ are indicated, X refers to the point where $r_1 = r_2$.

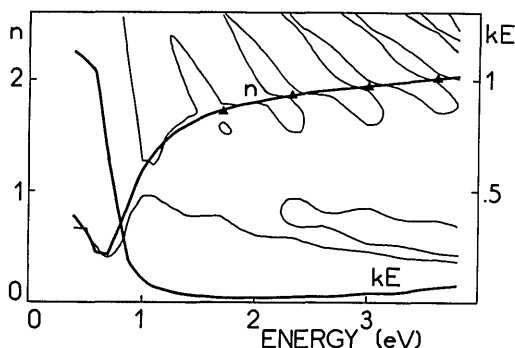


Fig. 8. Real part n and imaginary part k of the refractive index \tilde{N} of the SnO_2 film. Thin lines are deduced from R_{exp} and T_{exp} data by simple inversion of the Fresnel laws. Dots represent values of n deduced from the positions of the interference extrema of R . The heavy lines are the result of our one-way iterative treatment.

0.5 and 3.5 eV, performed in our department and reported in Fig. 7. The SnO_2 film is expected to be transparent above 1.5 eV, producing the interference fringes so well seen on the $R(\omega)$ experimental curve. In the infrared range below 1 eV, the film becomes opaque and its index n may be low. In the 1–1.5-eV range, the interpretation of the curve is ambiguous as the structures can arise either from interference or from absorption.

The direct deduction of n and k by inversion of the Fresnel laws of Eqs. (1) and (2) gives several solutions represented in Fig. 8. The values of n can also be deduced in the transparent range from the positions λ_m of the extrema of R using the classical expression:

$$n = \frac{l\lambda_m}{2d},$$

l being integer or semi-integer. These values are represented by dots on the n curve in Fig. 8.

The determination of n as seen in Fig. 8 is not fully satisfactory: the l -order seems well defined in the

interference range, but cannot be estimated around 0.8 eV, when N may go below N_s .

To obtain a single solution with coherent variations of $n(\omega)$ and $k(\omega)$, we introduce the dispersion relations of Eqs. (4).

A problem arises here due to the large thickness (4200 \AA) of the film, the transmission of the system being very sensitive to the k index.

In this case, a symmetrical n - k treatment as discussed in the previous section will fail because the Kramers-Kronig transformation reveals itself not to be accurate enough; errors in the k_2 value will make the process diverge. We then apply a one-way iterative treatment, fitting k by the Fresnel laws and obtaining n by dispersion relation. The iteration is then done in the following way:

We start with an $\tilde{N}_0(\omega)$ function as above.

We correct this value to fit the experimental R_{exp} and T_{exp} value for each ω value. In fact, we only determine a δk correction, keeping the n_0 value constant because it is supposed to be obtained more precisely in the following step.

We apply the dispersion relation [first of Eqs. (4)] on k_1

$$k_1 = k_0 + \delta k \rightarrow n_2.$$

The new approximation for the result is now taken as $\tilde{n}_3 = n_2 + ik_1$ which is obviously coherent with dispersion relations.

Several tens of iterations are needed to obtain stable curves. But a problem not encountered in the preceding section remains because we do not fit n at any stage of the iteration. Therefore, this method does not give a correct representation of the interference fringes which are very sensitive to n absolute value. To correct this effect, we have added a smooth correction in the form $\delta n_2 = a + b\omega^2$ (approximation of a Lorentzian function). The constants a and b are estimated at each loop of the iteration to fit the $R(\omega)$ and $T(\omega)$ experimental curve as well as possible. This correction is in fact only a simple attempt to take into account the main absorption part of the spectrum (>4 eV) which cannot be easily introduced in the extrapolation of $k_1(\omega)$.

The result of the iterative calculation is reported in Fig. 8. It can be seen that the interference fringes above 1.5 eV cannot be correctly fitted, which indicates a surface or roughness effect: the reflectance is lower than expected by calculation. On the other hand, a smooth n curve is obtained between 0.5–1.5 eV. One can now explain the odd behaviour of $R(\omega)$ around 1 eV, where interference and absorption coexist.

Below 0.7 eV, the film is opaque, which is in relation to the Drude conductance of SnO_2 . At 0.82 eV, the first interference maximum and minimum of the film appear corresponding to the order $l = 1/2$ and 1. At 1.2 eV, the film and the substrate have similar indices, the film is no longer visible and a phase shift of π occurs in the reflection coefficient \tilde{r}_2 . Above this energy value, a normal interference regime begins, the first maximum at 1.4 eV corresponding to the order $l = 3/2$.

V. Conclusion

It appears in the two examples studied here that introducing both the dispersion relations (of the index of the film) and the Fresnel laws is not only possible but valuable. Two general cases can be emphasized:

(1) when the complex index $\tilde{N}(\omega)$ is to be deduced from only one measured curve (the reflectance or any other optical data curve), the dispersion relations supply the second curve; and

(2) when two or more optical measurements are made (like normal incidence R and T , Δ and φ in ellipsometry, R_0 and R_{60} in oblique incidence measurements), the dispersion relations behave as supplementary, useful in checking or correcting the primary experimental data.

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