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On some important care to take when making spectrophotometric measurements on semiconductor thin films

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Abstract

Typical thin film semiconductor transmission (or reflection) spectra in the visible and near-infrared regions are composed of a transparency region with interference spectral fringes and an absorption region. Often, the position and contrast of the fringes are used to compute the film thickness and optical constants. In this work, we show experimentally that the transmission is strongly altered by the size of the spot if the film is not homogeneous in thickness, and also by the apparatus resolution if the optical thickness of the film is big enough. The experimental evidence is brought out using 1–2 μm thick amorphous silicon films, the transmission being carried out in a double-beam spectrophotometer from 0.5 to 3.2 μm. The results are analyzed using a theoretical mathematical model of the transmission of thickness inhomogeneous thin films previously developed by the authors, and briefly recalled in the present paper. The experiments confirm the model predictions particularly the alteration of fringe contrast and quasi-conservation of incoherent transmission.

Keywords: Optical properties; Semiconductors; Sputtering; Surface defects

1. Introduction

Spectrophotometric measurements: transmission (T) and/ or reflection (R) are powerful tools to determine the optical constants and thickness of semiconductor thin films. A simple and accurate method consists of exploiting correctly the interference spectral fringes appearing in the transparency region of the spectra. [1–7].

Generally, the structure used for this propose is a film deposited on a thick transparent substrate. Analytical formulas of T and R for this structure suppose often parallel interfaces and a smooth film surface. Unfortunately, this is never the case of real deposited films because they always present inhomogeneities along the illuminated region, which can be of different forms (continuous thickness variation, roughness, index fluctuations, etc.) depending on the studied material and the deposition technique. Another fact which is always omitted is the non-zero bandwidth coming onto the sample; this must be taken into consideration, especially when the bandwidth varies with wavelength, as is the case of some kinds of apparatus [7]. These two forms of departure from ideal conditions have the effect of altering substantially the experimental spectra and consequently the parameters computed when using non-corrected expressions for T and/ or R lead to estimable errors [1,4,7,8].

Few papers are devoted entirely to the treatment and correction of experimental spectra from the above problems [1,4,7]. Moreover, illustrations of the alteration of spectra due to thickness inhomogeneities or slit width have always been made on calculated spectra using empirical equations for the optical constants [4,7].

In a previous paper [1] the authors developed a theoretical treatment of the transmission in the case of inhomogeneous thin films and non-zero bandwidth; they also proposed a method to determine the thickness, optical constants and an estimation of the value of thickness or index inhomogeneities. The aim of this work is to confirm some aspects and predictions of this model: we study particularly the thickness variation over the illuminated area and bandwidth effects on the spectral behavior of the transmission spectrum. In this work our demonstrations and conclusions are brought out from the comparison between experimental spectra generated by varying experimentally the degree of thickness variation over the illuminated area of the sample (see Section 3 below) or by varying the bandwidth of the incoming radiation. The total study allows us to propose some precautions to take into account when making spectrophotometric measurements, and also to set limits (on the optical thickness of the films) for the importance of the bandwidth effect.

The theoretical analysis is briefly recalled in what follows, further details can be found in Ref. [1].

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2. Theoretical aspect

2.1. Film free of thickness inhomogeneities

When a monochromatic beam is incident normally on a film of complex index $\tilde{n}=n-jk$ and thickness t deposited on a non-absorbing substrate, if we assume smooth and parallel interfaces and multiple reflections to be coherent in the film and incoherent in the substrate, the transmission inverse is given by [9–11]:

$$\frac{1}{T}=\frac{[e^{\alpha t}+R_{10}R_2e^{-\alpha t}][1-R_2R_3A^2]}{(1-R_{10})(1-R_2)(1-R_3)A}+\frac{2\sqrt{R_{10}R_2}[1-R_2R_3A^2]}{(1-R_{10})(1-R_2)(1-R_3)A}\cos(\delta_1+\delta_2-\varphi) \quad (1)$$

$$\frac{1}{T}=I_F(R_{10},R_2,R_3,\alpha,t)+g(R_{10},R_2,R_3)\cos(\chi) \quad (2)$$

$$\chi=\delta_1+\delta_2-\varphi=\delta_1+\delta_2-\frac{4\pi nt}{\lambda} \quad (3)$$

where R_{10}, R_2 and R_3 are the normal reflectances at the air–film, film–substrate and substrate–air interfaces respectively, δ_1 and δ_2 are the phases of the reflection Fresnel coefficient at the air–film and film– substrate interfaces respectively, $\alpha=4\pi k/\lambda$ is the absorption coefficient and A is an attenuation term taking into account eventual absorption in the substrate (in our case $A\cong 1$).

The first term in the second member of Eq. (1) represents the interference-free transmission inverse (inverse of the "incoherent" transmission). It is a curve passing through the inflection points of the interference fringes. Experimentally it is obtained from the envelope curves of the transmission pattern $T_{min}(\lambda)$ and $T_{max}(\lambda)$ supposed to be continuous functions of λ [1,3,4,6]:

$$I_F=\frac{1}{2}\left(\frac{1}{T_{max}}+\frac{1}{T_{min}}\right) \quad (4)$$

It can also be obtained by filtering the transmission inverse from interference fringes using Fourier analysis [1]; this is the reason why we call it the filtered transmission inverse. The second term of Eq. (2) represents the oscillating part of the transmission inverse.

Our preference to work on the transmission inverse instead of the transmission itself is argued by the simplicity of the derived analytical treatment and obtained formula. For instance, we do not have to consider three absorbing spectral regions to solve our equations [3,4,7].

2.2. Film including a thickness variation

If the film presents a thickness variation of a very low slope over the illuminated area and if σ is the maximum thickness departure from the average thickness, we have demonstrated (see Ref. [1] for calculus details) that two correction terms appear in Eq. (1), and the new expression for the transmission inverse is given by:

$$\frac{1}{T}=I_F(R_{10},R_2,R_3,\alpha,t)\left[\frac{sh\alpha\sigma}{\alpha\sigma}\right]+g(R_{10},R_2,R_3)\left[\frac{\sin\Delta\varphi}{\Delta\varphi}\right]\cos(\chi) \quad (5)$$

where $\mp\Delta\varphi=\mp 4\pi n\sigma/\lambda$ is the phase fluctuation due to the thickness fluctuation $\mp\sigma$.

The factor $(\sin\Delta\varphi)/(\Delta\varphi)$ in this equation is responsible of the shrinking of the interference fringes (a quick calculus leads to a decrease of some percents in the fringe contrast). Nevertheless, in the region of moderate or small absorption, the modification introduced by the term containing the hyperbolic sine is negligible: for σ as high as 60 nm the absorption coefficient needs to be higher than 5×10^4 cm^{-1} to give a correction smaller than 0.995. Eq. (5) can be written as:

$$\frac{1}{T}=I_F(R_{10},R_2,R_3,\alpha,t)+g(R_{10},R_2,R_3)\left[\frac{\sin\Delta\varphi}{\Delta\varphi}\right]\cos(\chi) \quad (6)$$

2.3. The bandwidth effect

In practice the spectrophotometer has a finite bandwidth set by the entrance and output slits of the instrument which means that a band of wavelengths is incident on the sample such that: $(\lambda-\Delta\lambda)<\lambda<(\lambda+\Delta\lambda)$.

Taking into account the slit width value $\Delta\lambda$ in the analytical treatment, another correcting term appears in Eq. (6) and the transmission inverse becomes:

$$\frac{1}{T}=I_F(R_{10},R_2,R_3,\alpha,t)+g(R_{10},R_2,R_3)\left[\frac{\sin\Delta\varphi}{\Delta\varphi}\right]\left[\frac{\sin\left(\frac{4\pi nt\Delta\lambda}{\lambda^2}\right)}{\frac{4\pi nt\Delta\lambda}{\lambda^2}}\right]\cos(\chi) \quad (7)$$

and can be written as:

$$\frac{1}{T}=I_F(R_{10},R_2,R_3,\alpha,t)+\frac{C}{2}\cos(\chi) \quad (8)$$

where

$$C=2g(R_{10},R_2,R_3)\left[\frac{\sin\Delta\varphi}{\Delta\varphi}\right]\left[\frac{\sin\left(\frac{4\pi nt\Delta\lambda}{\lambda^2}\right)}{\frac{4\pi nt\Delta\lambda}{\lambda^2}}\right] \quad (9)$$

is the fringe contrast for a given wavelength.

On the basis of Eq. (6), it can be said that both the thickness variation and slit width have the effect of shrinking the fringe contrast without altering significantly the filtered transmission inverse (related to the incoherent transmission).

3. Experimental details

An a-Si:H film presenting an important thickness variation is used in this study; it was grown by radio-frequency reactive sputtering, under a gas mixture of 80% argon and 20% hydrogen on a glass substrate. Details of the experimental procedure are described elsewhere [12]. This film has a surface area of 4×2 cm^2 and a thickness varying continuously from 1.4 μm (edge of the film) to 1.6 μm (center). Transmission spectra were carried out between 500 and 2 800 nm in a SHIMADZU 3101PC double-beam spectrophotometer. Film refraction index, thickness and degree of thickness variation (σ) were determined using a fast converging algorithm and a fully interactive graphic program running on an MS-DOS compatible computer [1]. The total treatment of a spectrum including the determination of the absorption coefficient takes about 15 mn. The accuracy of the method for n and t is always around or smaller than 1%; and it has been successfully applied to a variety of semiconductor thin films (a-Si:H, CdTe, CdS, ZnS, V_2O_5, polymers, etc.) which are transparent in the visible and/or near IR parts of the electromagnetic spectrum.

A thickness profile of the used film was made in order to have an idea about its degree of thickness variation. First, the sample was fixed in a mounting allowing its accurate horizontal movement: the illuminated position was known to better than 0.5 mm (the light spot being 1 mm large and between 5 and 14 mm high depending on the used masks). Then, starting from the edge and all along the film surface we took several transmission spectra over the longitudinal direction with 16/9 mm gap distance. This allowed us to find out the thickness at each position and a surface thickness profile was drawn. This is illustrated in Fig. 1, where we have reported the computed thicknesses with their relative error bars (r.m.s. of the difference between the mean thickness and the values found for different fringes [1]). It can be seen that the thickness variation is less than 1% in a circular area having

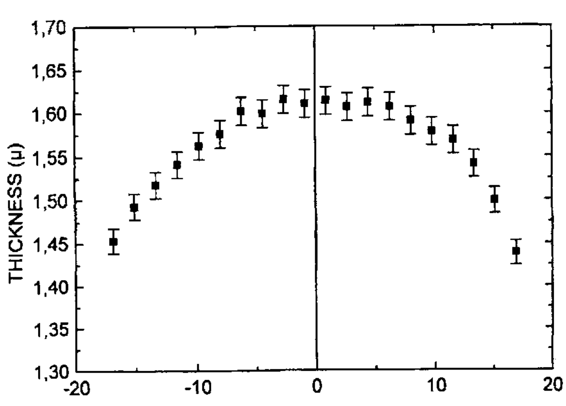

Fig. 1. Thickness profile for the sputtered a-Si:H film used in this study.

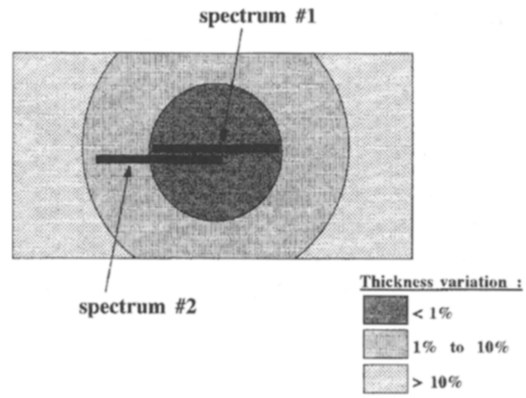

Fig. 2. Surface topological model for the used film showing the different thickness variation regions. Arrows indicate the positions of the light spot on the film for the spectra of Fig. 3.

no more than 14 mm diameter (because of the symmetry of the deposition process around the target axis). The thickness drops rapidly out of this region and the decrease is 10% at 17 mm from the target axis. Attempts were made to obtain the thickness profile using a Talystep, but it was impossible because the profile thus obtained included the substrate sag with a mechanical deformation in the order of the film thickness. We think that this is an advantage for our method because it uses a local analysis of the film in which the substrate thickness inhomogeneities can be ignored in a great extent. Analog profiles have been reported for other sputtered materials: V_2O_5 [13] and M_nO_4 [14].

Taking into account the circular symmetry of the deposition technique, we propose in Fig. 2 a surface topological model for the film in which we consider three regions delimited by concentric circles.

- Region 1: the variation of thickness is less than 1% (quasi-flat region).
- Region 2: the thickness decrease varies between 1 and 10% of the central value.
- Region 3: the thickness is more than 10% smaller than the central value.

4. Results

4.1. The thickness variation effect

In Fig. 3, solid lines represent two transmission spectra: the first taken over the quasi-flat region (spectrum #1), and the second at a position presenting an important thickness variation (spectrum #2). The size (standard, 14×1 mm^2) and position of the light spot on the film surface are indicated in Fig. 2. Dashed lines represent the envelope curves calculated by a polynomial fit and non-linear interpolation between extrema: they are drawn so as to determine the fringe contrast, which is not strongly dependent on the film thickness and is used to calculate the refraction index [1–4,7]. Dashed pointed lines are the incoherent (or filtered) transmissions ($1/I_F$) for the two spectra.

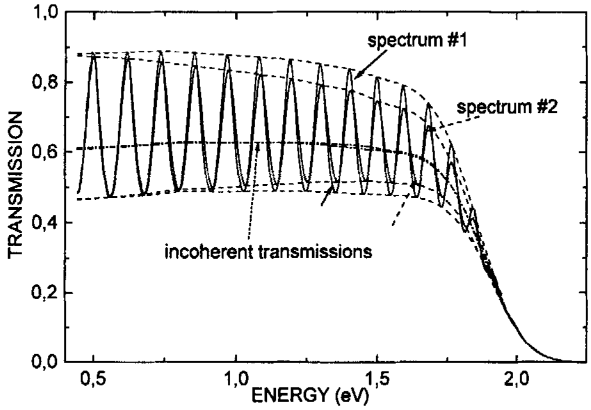

Fig. 3. Transmission spectra taken at different thickness variation regions (see Fig. 2). The envelope and incoherent transmission curves are also illustrated.

Table 1
Data computed for the spectra of Fig. 3. The last column gives the thickness variation estimated from Fig. 1 for comparison

	$(t \pm \Delta t)$ (nm)	n_∞	σ_{cal} (nm)	σ_{est} (nm)
Spectrum #1	1614 ± 9	3.084	26.5	15
Spectrum #2	1578 ± 8	3.120	47	54

Table 2
Results obtained when neglecting the thickness variation effect

	t(nm)	n_∞
Spectrum #1	1629	3.049
Spectrum #2	1633	3.027

The first remark is that the fringe contrast is smaller for spectrum #2; this is because the degree of thickness variation in this region is important: it can reach 80 or 100 nm between both sides of the spot (see Fig. 2). Nevertheless, the medium transmission seems not to be affected significantly by this fact. It is indeed practically the same for both spectra. The slight shift of the fringes between the spectra is due simply to a slightly different optical thickness (see Table 1 where we have summarized the results obtained from the two spectra).

t is the thickness and Δt is the r.m.s. of the calculated values of t on each interference fringe; n_∞ is the value of the refraction index extrapolated to infrared frequencies, $\pm\sigma_{cal}$ is the calculated variation of thickness along the illuminated area and $\pm\sigma_{est}$ is its homologue estimated from Fig. 1. We note for spectrum #2 that the agreement for both values of σ is good (σ_{cal} is 14% lower than σ_{est}), while for spectrum #1 σ_{cal} is practically twice σ_{est}; this will be a subject of discussion in later in Section 5. On the other hand the refraction index does not present a significant change: the variation (around 1%) remains inside the margins of accuracy of the method.

4.2. *Effect of neglecting the inhomogeneities*

The calculation method in this case does not take the inhomogeneities into account and can be summarized as follows. The first step is the determination of the refraction index $n(\lambda_i)$ on each fringe using the experimental contrast; λ_i being the wavelength of the ith fringe (extrema positions). Then we obtain a value t_i of the thickness on each fringe using the optical thickness [11,15]:

$$D(\lambda_i) = [nt](\lambda_i) = (p+i-1)\frac{\lambda_i}{4} \tag{10}$$

where p is the order of the fringe situated at higher wavelength ($i=1$ in Eq. (10)). p can be easily known [1] and the spectral optical thickness too. The knowledge of n at a given wavelength gives then the thickness and the spectral refraction index. As the contrasts tends to be the same for both spectra at low energy, it may be thought to use the contrast of the first fringe to do this without taking into account the thickness inhomogeneity of the film: the results thus obtained are shown in Table 2.

As the experimental contrast (without correction) has been used directly in the calculation, it gives lower values of the refraction index than those listed in Table 1. This leads to higher thickness values because the optical thickness D, determined from the fringe positions, has a fixed value for a given wavelength.

For spectrum #1 (taken over the region 1) the difference between results in Table 1 and 2 is not important (0.96% for t and 1.1% for n_∞). This is not the case for spectrum #2 taken over the region 2 of higher thickness inhomogeneities where this difference is more appreciable (3.5% for t and 3% for n_∞).

When more than one fringe is used in the calculations more serious errors may be introduced. This is indeed obvious from Fig. 4, where we have reported for spectrum #1 the thicknesses calculated at each one of the first 12 fringes: circles represent the thicknesses obtained when we consider the effect of inhomogeneities and the straight line is their mean value, squares are the thicknesses obtained using the above described procedure. In this last case, the value of t from the first fringe (low energy) is the nearest one to the straight line

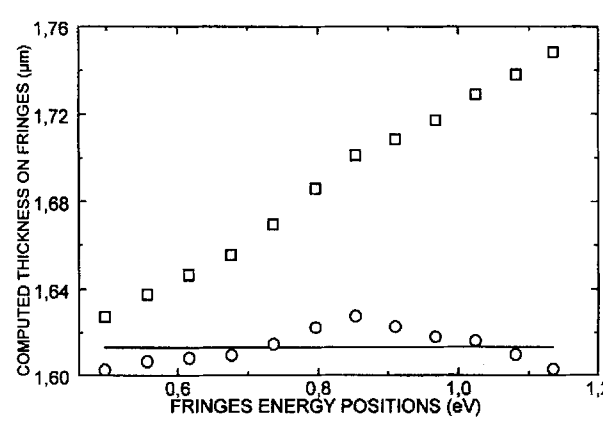

Fig. 4. Computed thickness at the first 12 fringes positions obtained with: $\sigma=0$ (□) and $\sigma\neq0$ (○); (———) mean value in the last case.

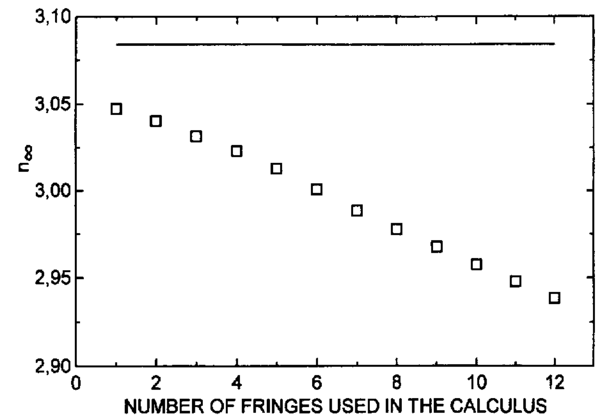

Fig. 5. n_∞ found when the thickness is calculated using an increasing number of fringes. The straight line represents the value found by the calculus program.

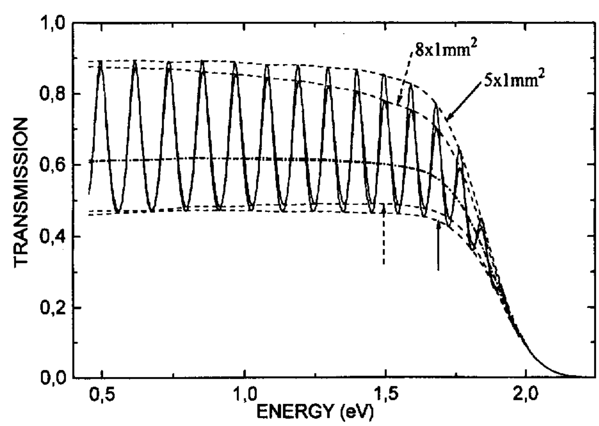

Fig. 6. Transmission spectra taken at the same position and varying only the spot size. The corresponding envelope and incoherent transmission curves are also shown.

and consequently is the more accurate one, but still not accurate enough. If other fringes are used, the mean thickness increases causing a further decrease in the refraction index. This is clearly illustrated in Fig. 5 which represents the values of n calculated when using an increasing number of fringes. The straight line represents the final value found by the computation program when taking into account the thickness inhomogeneities (from Table 1).

It is also notable that these errors become much serious when the analyzed spectrum contains few fringes. This is the case for example of low optical thickness films ($nt \leq 800$ nm), as the spectral width between successive fringes becomes greater and the effect of inhomogeneities (and bandwidth also, see Section 4.4) is more contrasted at higher energies (see Fig. 3 or Fig. 6 for example).

4.3. *Effect of lowering the size of the spot*

In Fig. 6 we present two spectra. The first with lower contrast was taken with a standard light spot size (14×1 mm^2), and the second by introducing a mask at the monochromator output without changing the spot position between measurements. The mask reduces the light spot area on the film to

Table 3
Data computed when using two diffrent spot sixes, for the same explored position on the film

Size of the spot (mm^2)	($t \pm \Delta t$) (nm)	n_∞	σ_{cal} (nm)
14×1	1594 ± 8	3.114	37
5×1	1604 ± 12	3.095	24

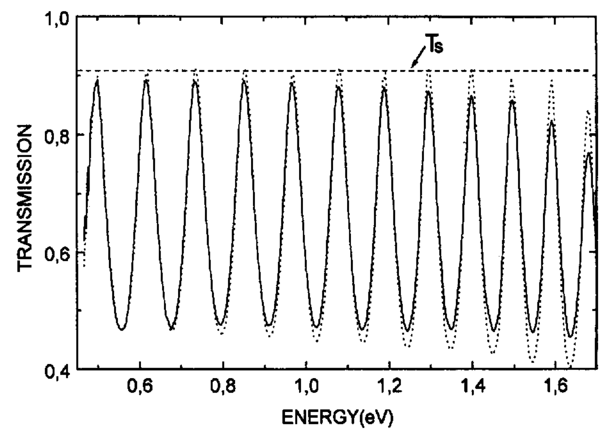

Fig. 7. Matching between the ideal and experimental spectra when using a small spot size. T_s is the substrate transmission.

5×1 mm^2. We have also drawn in this figure the mean and envelope curves for these two spectra. We can see that lowering the size of the light spot increases the fringe contrast because the thickness variation seen by the spot is smaller. This fact is confirmed by the calculated value of σ reported in Table 3. For the other calculated parameters shown in Table 3, we may note that the values of t and n_∞ do not present a significant change as the mean thickness in the explored position was practically conserved. Both parameters differ by less than 0.6% and overlap if we take into account the accuracy of the method. The fringe contrast alteration in Fig. 6 has then been successfully corrected for two different degrees of thickness variation using our model. The incoherent transmission, as in Fig. 3, remains practically unchanged.

In Fig. 7, we compare the spectrum taken with a smaller spot size, to the ideal spectrum which would have been obtained if the film was free of inhomogeneities. The latter is calculated using Eq. (7) setting $\sigma_{cal}=0$, $\Delta\lambda=2$ nm (experimental value) and keeping the values of the optical constants and thickness found by the program. It can be seen that the matching between the ideal and the experimental spectra is good only for the first three fringes (lower energies), and the disagreement becomes relatively important at higher fringes order (higher energies). This disagreement is even higher for the high spot size spectrum (see Fig. 6 for comparison). These are notes to keep in mind for our final discussion.

4.4. *The bandwidth effect*

Fig. 8 represents two transmission spectra taken over the quasi-flat region by changing the bandwidth of the incident

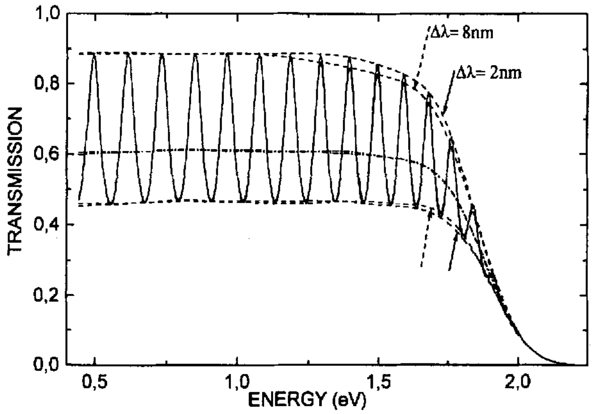

Fig. 8. Transmission spectra taken at the same position using different bandwidth values, and their respective envelope and incoherent transmission curves.

light from 2 to 8 nm, without changing neither spot position nor its size. The latter was fixed to its small value (5×1 mm^2) to minimize, as far as possible, the thickness variation effect and to ensure that the results will depend only on the bandwidth variation.

No appreciable change is noted in both spectra until 1.1 eV; it seems that the bandwidth have no influence on the spectra at lower energies, and that its unique effect is to lower the fringe contrast at higher energies where the value of $\Delta\lambda$ becomes more and more appreciable with respect to the wavelength. The incoherent transmission is conserved like in the previous cases and as predicted by Eq. (7).

Table 4 shows the results obtained when we take into account the value of the spectral bandwidth $\Delta\lambda$ in the program (value indicated), and in the case where we suppose no bandwidth ($\Delta\lambda=0$).

For the spectrum taken with lower bandwidth we note that the values found for all the calculated parameters are practically the same with or without using the bandwidth correction. It seems then that measuring with a small bandwidth such as 2 nm does not affect significantly the transmission spectrum and consequently the fringe contrast decrease is only due to the thickness inhomogeneities effect.

For the spectrum taken with a bandwidth of 8 nm, if we set the exact value $\Delta\lambda=8$ nm for the computation, in principle we must find the same results as for the previous spectrum, but it is not so, for example, the value of σ_{cal} is 10% higher than the previous one; n and t also have a trend to increase and decrease respectively. Our opinion is that the

Table 4
Data computed for the spectra of Fig. 8 when the value of the experimental bandwidth is set for the computation ($\Delta\lambda=2$ or 8 nm) and when assuming no bandwidth ($\Delta\lambda=0$)

Spectrum	$\Delta\lambda$ (nm)	$(t\pm\Delta t)$ (nm)	n_∞	σ_{cal} (nm)
2 nm	2	1613 ± 10	3.098	21.6
	0	1611 ± 10	3.102	21.4
8 nm	8	1596 ± 13	3.128	24.1
	0	1594 ± 14	3.132	26.3

chosen bandwidth value in this case is very high and exceeds the range of validity of our formula for the bandwidth correction. From a computational point of view, the convergence criterion is the minimization of the difference between experimental and calculated contrasts [1], the program being incapable to correct the spectrum from such a high bandwidth, it attributes the residual difference to an inhomogeneity effect which rises the value of σ_{cal}. The fact that this value in the case where we set $\Delta\lambda=0$ is even higher means that our calculation program compensates uniquely in part the bandwidth effect.

5. Comments and discussion

Eq. (7) shows that either thickness variation and bandwidth effects give no change in the filtered transmission inverse: the experimental results reported in Fig. 3, 6 and 8 confirm this fact unambiguously.

In another paper we have shown that for a value of $\Delta\lambda=1$ nm, the correction given to the transmission by taking into account the bandwidth effect remains less than 0.2% if the film has an optical thickness less than 3 μm. Here, we have shown for a thin film having an optical thickness of about 5 μm that the bandwidth effect can be neglected for $\Delta\lambda$ as high as 2 nm, which is not restrictive from an experimental point of view.

The interference fringes method is largely used to determine film thickness. If thickness inhomogeneities are ignored, it is better to use a low size spot so as to minimize thickness variations over the illuminated region and to consider only the first fringe (low order) situated in the transparency region of the spectrum to compute the thickness because it is the less affected by inhomogeneities and the spectral resolution of the apparatus. This procedure gives a slightly overestimated thickness value. Nevertheless, it can be very close to the exact one ($\cong 1\%$ higher in our case) and this is better than multiple other techniques of thickness measurements; the refraction index can be determined from Eq. (10) with a similar accuracy.

From the thickness profile of Fig. 1, we have evident reasons to deal with the transmission spectra taken on the used thin film on the basis of a thickness variation model. Sputtered materials are generally homogeneous, but this does not exclude the existence of a weak index fluctuation over the illuminated area and this means that the calculated values of σ can be slightly overestimated: the program may attribute the residual contrast difference due to the index fluctuation to an eventual thickness variation effect. This fact can explain for instance the highest value of σ_{cal} for spectrum #1 in Table 1 compared with its corresponding estimated value from Fig. 1. Nevertheless, this index fluctuation can be neglected when the thickness variation is greater: for example the value of σ_{cal} for spectrum #2 in Table 1 is in better agreement with that estimated from the profile. When the samples are expected to contain a high degree of index fluctuations (presence of different phases of a component) the problem is more complicated because we have to take into account also the thickness inhomogeneities from which thin films are in general not free of. A correct approximation in this case would be to lower as possible the light spot area so as to minimize the thickness variation effect on the spectrum and to deal with the problem using an index fluctuation model [1,3].

6. Conclusion

This work demonstrates clearly from experimental spectra that transmission measurements are very sensitive to thickness inhomogeneities but also to the spectrophotometer resolution if the film optical thickness is big enough. Results obtained directly from experimental spectra without the appropriate corrections are then to be taken with special care, especially when thickness inhomogeneities are significant and when spectra present few fringes. The experimental analysis of spectra obtained on films having a non-uniform thickness confirms all the predictions of our theoretical model, namely reduction of fringe contrasts and conservation of incoherent transmission.

According to the discussion above, a correct procedure to make spectrophotometric measurements begins by lowering as far as possible the size of the light spot coming onto the film, which reduces efficiently the thickness inhomogeneity effect. Then, one must check that the bandwidth of the incident light is not too high, especially when the film optical thickness is big enough.

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