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Determination of the optical properties of thin-films

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ABSTRACT. The simultaneous determination of the refractive index, absorption coefficient and thickness of thin-films in an unambiguous manner has been the subject of many research teams. Reported results show a strong dependence on the method [1]. In this work, we give an account of the methods we have developed for optical characterization of thin-films, based on external [2] and internal reflectometry [3], including the excitation and detection of surface electromagnetic waves (surface plasmons) in the case of metals and two characteristic isoreflectance angles in the case of weak absorbing and transparent films. In all cases, the samples are prepared with dielectric stepped overcoating protecting the sample degradation when exposed to ambient; these treatments give unambiguous information on the optical properties and thickness of the sample. Some examples on dielectric, weak absorbing and metallic film are given.

RESUMEN. La determinación simultánea del índice de refracción, el coeficiente de extinción y el espesor de una película delgada ha sido tema de trabajo de muchos grupos de investigación. Los resultados hasta ahora reportados han mostrado una fuerte dependencia del método utilizado y con frecuencia existe más de una solución con significado físico [1] En este trabajo hacemos un repaso de los métodos desarrollados por nuestro grupo, incluyendo excitación y detección de ondas superficiales de plasma (plasmones de superficie) en el caso de metales y dos ángulos característicos de isorreflectancia en el caso de materiales poco absorbentes y transparentes. En todos los casos, la película estudiada está recubierta por una sobrecapa escalonada de material transparente, que en el caso de metales actúa también como protección contra la degradación producida por el ambiente; el tratamiento resultante da información unívoca sobre las propiedades ópticas y el espesor de la muestra. Se presentan algunos resultados en los tres tipos de materiales: películas delgadas de metales y dieléctricos débilmente absorbentes y transparentes.

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1. INTRODUCTION

The idea of overcoating a sample with an echelled thin-film to determine optical properties was published for the first time in 1967 [1], Vincent-Geisse *et al.* have applied this technique to study five carbonated monocrystals in the IR region, at 1400 cm^{-1}.

Thereafter, Denton and Tomlin in Australia [2], reported in 1972 the description of a similar technique, without reference to the work of Vincent-Geisse; further work from the group headed by Tomlin with the participation of people from Pakistan and India continue the use of dielectric overcoatings for the optical characterization in samples of ZnS and CdS in 1975 [3], and amorphous and crystalline Ge in 1976 [4]. However, this technique presented some spectral points where data was not available, due to the lack of a unique solution. Recently, del Pozo and Díaz, in Madrid [5] pointed out this situation by studying the dependence of experimental uncertainties in the determination of the optical properties of thin films, they show the missing information in these spectral points.

Our group first reported [6] a simulated study on data taken from Rh thin films [7], where groups from different countries using multiple techniques have been characterized samples prepared under the same experimental conditions, we showed that reflectance measurements through an stepped overcoating of ZnS whose thicknesses have been conveniently chosen, allow the determination of the optical properties of opaque films of this material even in the case of great experimental uncertainties of the overcoating thickness. In another work [8], we combine this overcoating method with scanning reflectometry and internal reflection through a prism, and determine the complex index of refraction of metallic thin films; this result is valid within a given spectral range, thus allowing the determination of the dispersion relation and the thickness of the sample, with precision comparable to that of most dedicated techniques. In this work we also presented, for the first time, the reflectance behaviour with p-polarized light for angles below the critical θ_C and show the existence of a characteristic angle where reflectivity is independent of the overcoating thickness, the phase also shows no shift at this particular angle that corresponds to the well known Abelès or pseudo-Brewster angle [9], called θ_B where the Fresnel reflection coefficient is zero at the outermost interface dielectric-air, and does not contribute to the overall reflectance of the system.

In our more recent work [10] we have also detected a second characteristic isoreflectance angle, called θ_A, resulting from equal contributions to the optical phase introduced by given thicknesses of the stepped dielectric thin film. This two angles maintain the same angular position, even in the case where the steps are an overcoating to a given absorbent sample. The detection of these two experimental parameters (θ_A and θ_B) who are related to both the refractive index and thickness of the steps, allows the simultaneous determination of the optical properties of absorbent thin film samples with assistance of some other techniques such as the attenuated total reflection or any of the typical inversion routines used when spectral reflectance are to be converted into indices of refraction and thickness.

The uniqueness of the solution is assured by the existence of this two fixed points that all the possible solutions must fulfill. We have applied this technique to Cu samples overcoated with ZnS, and once the clean metallic surface has been characterized, the study of an exposed Cu surface to the air is immediate, so the optical properties of a natural

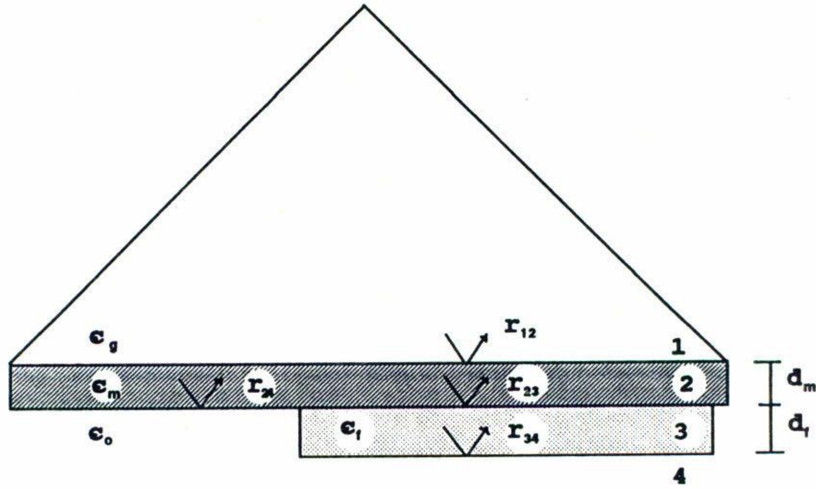

FIGURE 1. Experimental arrangement of the multilayer system under internal reflection.

overlayer may also be determined with the same method, the corresponding results are shown in Sect. 4.

2. THEORY

We describe here the development of our method, in fact, a combination of three common techniques: internal reflectometry, angular scanning and attenuated total reflection, to capitalize their sensibility and versatility. With our arrangement it is possible to characterize the optical properties of dielectric, weakly absorbent and metallic thin films. The key point is the detection of the two characteristic angles θ_A and θ_B, that characterize completely a dielectric material while introduces two fixed points in the case of absorbing thin films overcoated with the same stepped dielectric film. The solution is always found in an univocally manner.

a) Internal reflectometry

In general, we use the experimental set up in a configuration as shown in Fig. 1. The incident beam enters the sample through a prism, where a critical angle appears θ_C, and total reflectance is found for $\theta > \theta_C$. Sensibility of total internal reflection (TIR) has been shown to be better than external reflection [11]. Measurements with light in both polarization states give enough information to determine the complex index of refraction of a thick metallic layer, this has been successfully used in the case of Fe films [12].

b) Angular scanning

On analyzing transparent thin films with a fixed wavelength by changing the angle of incidence [13] it is possible to detect the two characteristic angles θ_A and θ_B, where

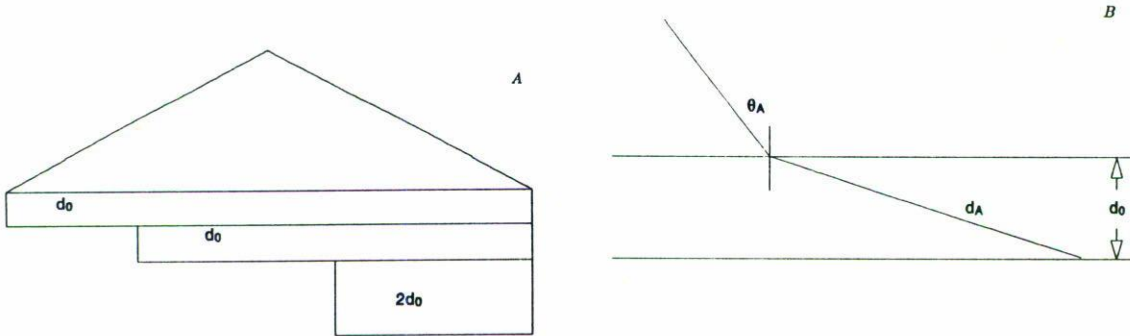

FIGURE 2. a) Stepped transparent thin-film with sequence 1, 2, $4d_0$. b) Notation for the optical thickness and optical path of a light beam entering a d_0 thick transparent layer at θ_A.

reflectance or transmittance values are independent of the thickness of the layer. The first one is detected for both polarization states of the incoming light, and appears when the sample presents a given sequence of thicknesses, thus the optical phase contributions are $\beta = 2\pi(n \cdot d)/\lambda$, while the second is valid only for p–polarization (TM or magnetic field perpendicular to the plane of incidence) and represents no phase contribution at all [14].

Let's consider a well characterized prism (known n_p), we evaporate a three-steps transparent thin film whose thicknesses keep the sequence 1, 2 and $4d_0$ (Fig. 2a), the system is illuminated with monochromatic light of wavelength λ and angle scanned to values below the critical angle, $\theta < \theta_C$, around the center of the prism taken as the rotational axis. By using the notation of the optical path $n \cdot d$, corresponding to a quarter wave (QW) inside the film at the angle θ_A (Fig. 2b) is easy to show the relation

$$n \cdot d_A = n \cdot d_0 \sqrt{n^2 - S_A^2},$$

that may take values multiples of $\lambda/4$, here $S_A = n_1 \sin\theta_A$ is the Snell invariant and d_0 is the thin film physical thickness at normal incidence.

The variations of p-polarized reflectance in terms of the effective optical thickness in terms of the angle of incidence, and in particular for $\theta = 0$, $\theta = \theta_A$ and $\theta = \theta_B$, are shown in [10].

It is worth noting that for the angle of incidence θ_A, where the optical thickness is $n \cdot d_A = m\lambda/6$, the corresponding reflectance R_A is the same for each step, and if we would take into account the behaviour of a thickness equal to $3d_0$, the value of R_A is the actual value of the bare prism internal reflectance. This is valid for both states of light polarization, in both reflection and transmission.

Then, the condition to find θ_A is $R(d) = R(md)$, with $m \neq 3n$, $m, n \in N$ corresponding to the optical thickness $n \cdot d$ of each step equal to $\lambda/2$ for this particular angle of incidence,

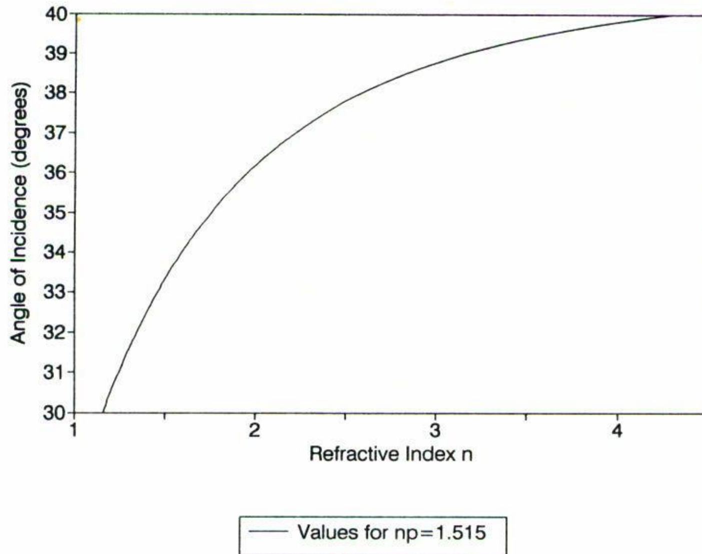

FIGURE 3. Determination of n through the detection of θ_B in a dielectric film.

reflectance in both cases is given by

$$R(d) = |\rho_1|^2; \qquad \rho_1 = \frac{r_{1,2} + r_{2,3}e^{-2i\beta}}{1 + r_{1,2}r_{2,3}e^{-2i\beta}}; \qquad \beta = \frac{2\pi}{\lambda} nd\cos(\theta_A).$$

And the condition $R(d) = R(md)$ implying $\exp(-2i\beta) = \exp[-2i(\pi+\beta)]$, means $\beta = m\pi$ and consequently the physical thickness is

$$d_A = \frac{m\lambda}{2} \frac{1}{\sqrt{n^2 - S_A^2}}. \tag{1}$$

From the same configuration we can observe also that the reflectance R_B at the angle of incidence θ_B is a constant for every thickness value of the dielectric layer. The condition to detect this characteristic angle is that the Fresnel internal reflection is to be zero at the exit interface, $r_{23} = 0$, then the steps act as *absentee* layers for the monitoring wavelength and do not contribute to the overall reflectance of the system that remains a constant.

The condition $r_{23} = 0$ implies that $n^2/(n^2 - S^2) = 1/(1 - S^2)^{1/2}$, that means,

$$n = \frac{S_B^2}{\sqrt{1 - S_B^2}}. \tag{2}$$

Figure 3 illustrates clearly the relation between θ_B with the refractive index n. Another useful expression is obtained by combining Eqs. (1) and (2):

$$d_A = \frac{m\lambda}{2} \frac{1}{\sqrt{(1 - S_B^2)^{-1} + (1 + S_A^2)}}. \tag{3}$$

It is possible to determine both the refractive index and the thickness of the steps once we can measure the characteristic angles θ_A and θ_B.

The variation of reflectance with wavelength λ for the two states of polarization s– and p–, are also shown in Ref. [10] for the case of an incidence of $\theta_A = 20°$, the isoreflectance are localized at $\lambda = 620$ nm. Here, we have neglected dispersion effects, but the observation of the angle θ_A for both states of polarization suggests the possibility of making spectrophotometric measurements instead of angle scanning for the detection of the desired parameters.

Once the transparent material is characterized, it can be used as a protecting overlayer with the same steps onto thin film samples to be studied, weakly absorbent or metallic materials. Then the determination of the optical constants of this new layer may be obtained simultaneously with those of the overcoating. In the case of metallic films it is possible to take advantage of the excitation of surface plasma waves (SPW) formed at the metal-dielectric interface, by using the well known ATR technique.

c) Surface plasma waves in metals

Excitation of SPW in the attenuated total reflection technique has been widely used for the characterization of metallic surfaces. The experimental configuration proposed by Kretschmann in 1970 [15] is the same as in Fig. 1, where the film under study is a metallic one. For angles above the critical, $\theta > \theta_C$, the evanescent waves couple with natural oscillation modes of the electrons at the interface metal-dielectric (air), giving place to the generation of surface plasma waves or surface plasmons (SP), propagating along the surface; this leaking frustrates the total reflection of the incoming light that should take place for this angular incidence, the reflected signal presents an absorption peak, as shown in Fig. 4, this effect, first reported by Otto [15] in 1968, gives the name to the attenuated total reflectance (ATR) technique.

Reflectance measured through the prism presents a minimum at the angle of incidence θ_{sp}, at this moment, coupling of light with the natural modes of the collective electron oscillation at the interface is achieved. The peak shape (angular position, absolute value and width) gives enough information about the complex refractive index and thickness of the metallic layer. This technique is very sensitive to surface modifications [16], the presence of a fraction of monolayer may be detected with great resolution by just measuring the shift of θ_{sp} and the corresponding reflectance value.

Usually, the formation of natural overlayers on metallic thin films does not contribute significantly to the optical response of the system, and is always neglected, so the interpretation of reflectance or transmittance data for the determination of the optical constants of the sample is not affected. But in this case of ATR, the presence of these natural overlayers may not be neglected and the condition of a clean surface is very important. Otherwise the results of an inversion may correspond to effective values of the complex refractive index and thickness. The preparation of metallic samples together with a protecting stepped overcoating will permit the reduction of the corrosion rate at the time that facilitates the treatment of the experimental information by fixing two points given by the two characteristic angles.

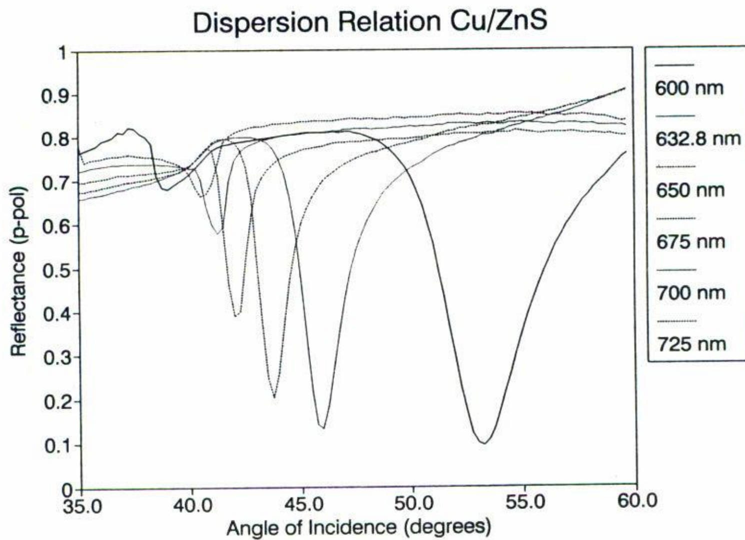

FIGURE 4. **Dispersion relation of a Cu/ZnS interface detected by ATR minima.**

d) Comparison to other techniques

There are many other methods for the optical characterization of thin films, the most widely used in different working groups throughout the world are described in [7].

One of the most important, because of its simplicity an easy interpretation of data, is the one reported by Manifacier *et al.* [17], this is a non destructive technique based on the envelopes of the spectrophotometric transmission measurement, it allows the extraction of the refractive index and thickness of a given thin film sample by using data obtained after the preparation of the sample. The maxima and minima of the transmission spectra produced by interference effects within the film form two envelopes, the bending of these lines gives information about the absorption while the thickness is introduced in the number of peaks.

The values of the optical constants of MgF_2 and ZnS thin films have been obtained with a classical oscillator treatment of the spectral transmission data [18], we have adjusted the experimental curves of these materials, prepared by thermal evaporation at the time that a study of the chemical composition was determined by Auger electron spectroscopy, the conclusion was that the optical properties were strongly dependent on evaporation conditions. With this method we have had the direct measurement of the refractive index dispersion in both materials, just after preparation in vacuum onto glass substrates. This technique makes use of a sum of oscillators that should explain characteristic features of the transmission spectrum; we only needed two oscillators, one in the high energy region to explain the minimum found in the UV region and another one in the low energy region to take account of the bending shown towards the IR region.

The detection of the two isoreflectance angles, θ_A and θ_B, is also possible by using ellipsometry [19, 20]; the polarized beam used in this technique allows the detection of phase shifts, and it has been stated that at these particular angles there is no phase shift, so the detection of the two angles is achieved in conditions of more sensitivity and

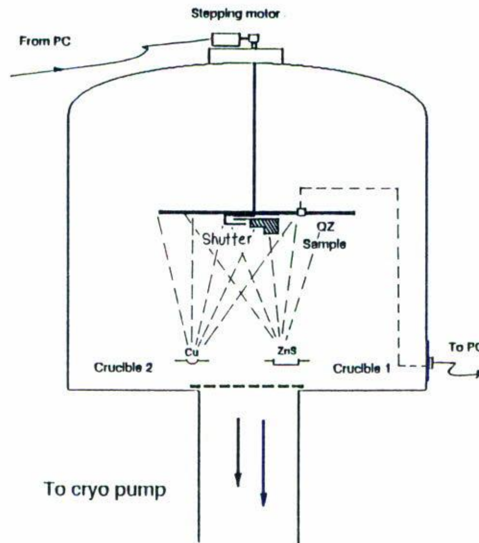

FIGURE 5. Experimental set up for the preparation of stepped dielectric thin-films. A quartz microbalance is controlled by a personal computer to command a stepping motor that moves a shutter to the desired positions once the programmed thickness is evaporated.

precision, and the interpretation of experimental data is also easier because of the two fixed points introduced by the same angles.

3. EXPERIMENTAL RESULTS

We describe here how we prepare the stepped samples and the experimental setup mounted in order to get experimental data that must be treated for the determination of the optical properties of the three types of materials.

a) Monitoring the growth of thin film samples

A typical vacuum chamber with thermal evaporation from two sources, equipped with a quartz microbalance and an optical monitor is used to prepare the stepped films. A computer controlled system is attached to the microbalance and is programmed to move a stepped motor that commands a shutter installed close to the substrate at the desired thickness values. It is possible to prepare a glass slide with four different steps and reproductibility is assured. In the case that this stepped dielectric film is to be an overcoating of a different absorbing sample, this one is also evaporated without breaking the vacuum regime. The characteristic angles will be detected always in the same angular position, whatever the substrate is, as long as the steps are reproduced. The working pressure is of the order of 10^{-6} mbar, the system is sketched in Fig. 5.

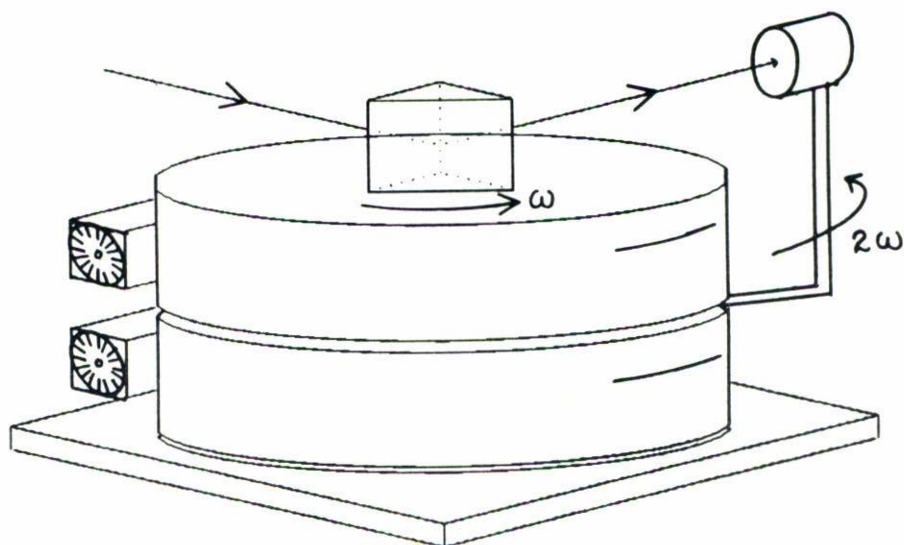

FIGURE 6. Diagrammatic scheme of the double rotatory stage for the angular scanning reflectance measurements, at different wavelengths. The overall system is controlled through a personal computer.

b) Angular scanning system and detection of θ_A and θ_B

The evaporated samples are analyzed on a rotation stage where a microcomputer commands two stepping motors, that independently move the sample or the detector, it is possible to program different movements, synchronous or goniometric or to fix one of the motors while the other is sweeping a given interval, etc. In the case of internal reflection the goniometric movement is the most convenient, the detector has an angular velocity two times that of the sample in order to receive the reflected signal through the prism all the time. The diagram of this set up is shown in Fig. 6.

c) Determination of the refractive index and thickness of dielectric thin films

We present here the case of a stepped dielectric thin film, the sequence of the steps is 1, 2 and $4d_o$ as in Fig. 2a, the characteristic angles θ_A and θ_B are easily detected, either by transmission or reflection, both in p–polarization while θ_A may also be observed in s–polarization. (Fig. 7). By using Eqs. (1) to (3) we can determine the refractive index and thickness of the step of the sample.

d) Extension to the case of weakly absorbing materials

There are two possibilities of treating weakly absorbing materials, that being still transparent present a complex refractive index, even though the imaginary part is small compared to the absolute value of the real part, it is possible to use the same technique described in [6], where the sample is overcoated with a stepped dielectric film; as in this case the system is not completely opaque, the effects of multiple reflections may be taken into

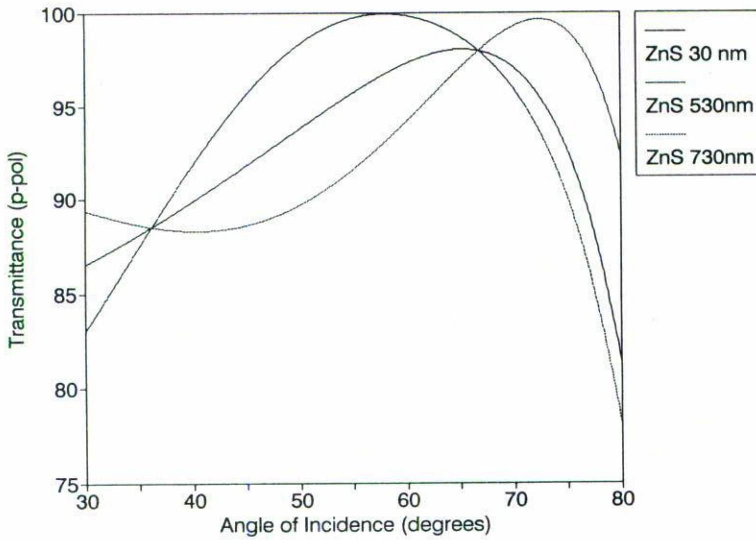

FIGURE 7. Experimental detection of θ_A and θ_B in a stepped dielectric thin-film (ZnS).

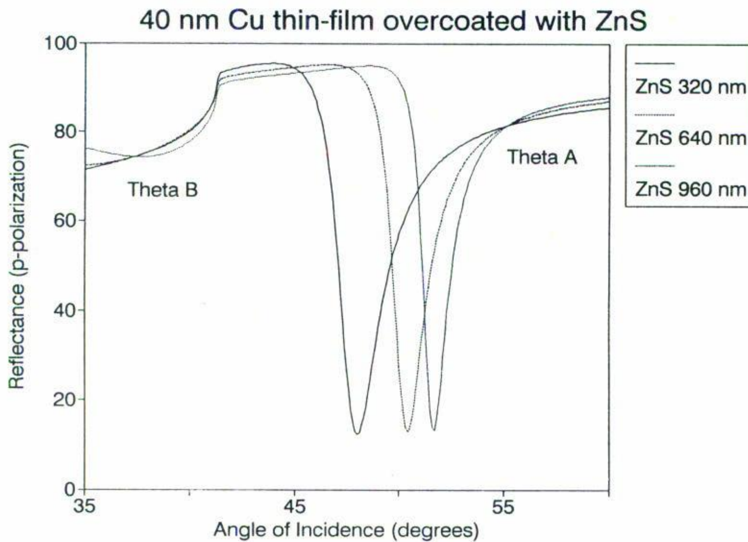

FIGURE 8. Attenuated total reflection signal of a Cu thin-film overcoated with a stepped ZnS film.

account at the moment of the interpretation of the experimental measurements and we should consider this effect in the inversion procedure. But we can also prepare a stepped film of this weakly absorbing material, following the study presented in [10], the detection of the characteristic angles θ_A and θ_B is still possible, in a diffuse region whose extension is related to the absorption of the material. A study on the quantification of the absorption coefficient using this method seems to be interesting.

e) Determination of the complex refractive index and thickness of metallic thin-film over-coated with dielectric stepped films

In the case of the optical characterization of evaporated Cu thin films, we prepare a 35 nm thick sample overcoated with a stepped ZnS where $d_0 = 160$ nm. In the experimental response obtained from the setup previously described we clearly observe the characteristic angles θ_A, θ_B and θ_C, as well as the minima of the attenuated reflectance measurements for every step. The corresponding resonance shifts are well resolved too, and we also note the presence of a guided mode due to the thicker dielectric step (Fig. 8). From this experimental information we can deduct the following values for $\lambda = 632.8$ nm, $n = 0.253$, $k = 3.468$ and $d = 34.2$ nm. These values, as expected, are different to those reported in the literature [21], because sample preparation in different laboratories always present differences with each other and also because in our case we are dealing with clean surfaces.

4. APPLICATIONS AND FUTURE WORK

a) Experimental results

Among the possible applications of this method, beside the determination of the optical properties and thickness of all kind of material in the form of thin films already mentioned, there is also the possibility of calibrating a quartz microbalance by comparing the input and output data to the experimental values obtained in dielectric films or the study of natural overlayers formed with ageing and exposure of metallic surfaces to the ambient.

Thickness monitoring during the evaporation process is performed mainly by using a quartz microbalance; this method requires the input of some information on the material, such as the density or the acoustic impedance or tool factors due to the relative position between the sample and the quartz sensor, and the resulting values converted to thickness depend also on the vacuum pressure and substrate temperature during the evaporation. The most relevant of these parameters is the density, typically obtained from handbooks, these data correspond to the bulk, while actually, the thin film may be porous and shows a rough surface [22], yielding a lower packing density. The microbalance is in fact informing us of a kind of weight thickness d_W.

We propose a simple method to separate both the refractive index and the weight thickness on transparent thin films, by preparing the sample in a stepped form and using scanning angle transmission or reflectometry to detect θ_A and θ_B and convert the data through Eqs. (1) to (3). In this way real values of n and d are obtained and may be used to correct the input density data and the tooling factor in the quartz microbalance to have reliable output in every evaporated material [23].

As the optical properties of very thin films are strongly dependent on the thickness, specially in porous materials or composite media, the knowledge of this parameter is of great importance.

The optical properties of adsorbates or absorbates, films that grow naturally over metallic surfaces exposed to the ambient is also a possible application of the above mentioned technique. Once the stepped overcoat is evaporated on the metallic sample, it acts as a protective treatment, while a similar exposed sample may show a shift in the ATR

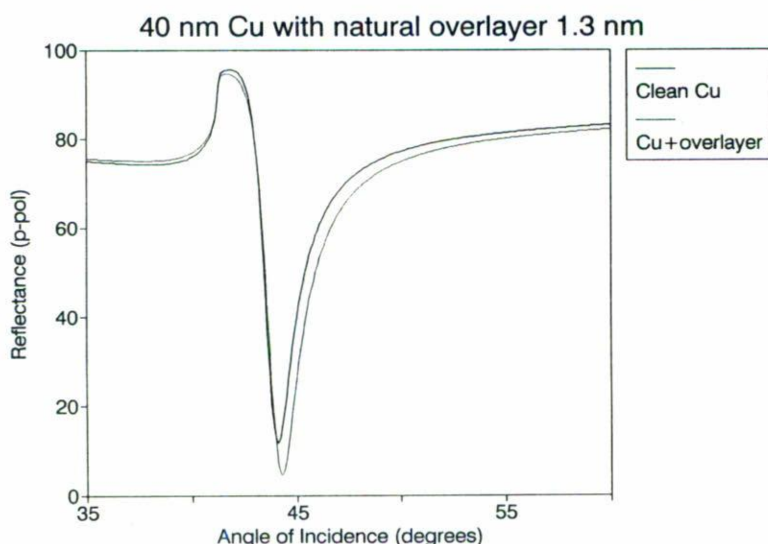

FIGURE 9. Study of the formation of a natural overlayer onto the surface of evaporated Cu. The knowledge of the optical constants of clean Cu allows the determination of those of the overlayer.

signal, informing about the growth of a natural very thin overlayer. So, from the already known complex refractive index and thickness of the clean metallic surface, it is possible to extract the optical parameters of the natural overlayer with the same inversion routine. The study of Cu protected by steps of ZnS, and the subsequent modification of a non covered part of the Cu surface has been the object of another work, (Fig. 9) where we have found values for the natural overlayer of $n = 0.66$. $k = 1.77$ and $d = 1.3$ nm [24].

From this system, Cu/ZnS, we also have to study whether in reality the overcoating acts as a protection against ambient modification or if the presence of these two materials in the same interface may react chemically producing a very thin transition film or a physical interconnection of rough surfaces of porous materials, in such cases, the treatment must be that of a composite material. The study of these possibilities is another opening of this technique [25].

b) Interpretation of results: real vs. ideal models

Up to now, in the interpretation of experimental results, we have considered ideal models where films are homogeneous and isotropic media, that both films and substrates present always smooth surfaces. But in reality, we must make quantitative analysis on the porosity and surface roughness of the evaporated thin films and make the corrections introduced by this effects in the determination of the optical properties of materials.

Some evidences are found when calibration of a quartz microbalance is performed [23], while the surface roughness introduces small absorption peaks in the reflection spectrum of thin film samples, that is the case of systems that include a granular layer such as CaF_2/Ag and Ag/CaF_2 [26]; a quantitative analysis of these effects in metallic and dielectric surfaces are achieved by using the ATR technique, by fixing the angle of resonance in the incoming

light and moving a sensor to detect the radiation of the metal- dielectric interface in the hypotenuse of the prism, in the inverse process where surface plasma waves (SPW) couple the surface roughness and is converted to light, the typical surface roughness have rms features of the order 0.5–1.5 nm [27].

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