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FILM THICKNESS DETERMINATION BY GRAZING INCIDENCE DIFFRACTION

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Abstract

Thin films deposited via MOCVD (Metal Organic Chemical Vapour Deposition) are layers in the thickness range of a few nanometers to about ten micrometers. An understanding of the physics and chemistry of films is necessary for a better comprehension of the phenomena involved in the film deposition procedure and its optimisation. Together with the crystalline phase a parameter that must be determined is the thickness of the layer. In this work we present a method for the measurement of the film thickness. This procedure, based on diffraction intensity absorption of the X-rays, both incident and diffracted in passing through the layers, resulted quite simple, rapid and non-destructive.

The measurements were performed with X-rays impinging on the sample surface at glancing angles, from 1 to 20 degrees; the detection of diffraction intensities was obtained employing a thin film attachment that made data collection possible also in these non-focusing conditions. Thickness evaluation of thin films by this method, resulted quite satisfactory for thin films in the 0.5- 4 micrometer range. This procedure can be performed with the same set up for phase determination and offers an average thickness also for not very smooth films, or films grown on porous (rough) substrates, or amorphous films on polycrystalline substrates. The main limitations of this method are :

i) the film or the substrate must be polycrystalline
ii) the film density must be known
iii) crystallites in the film or in the substrate must be randomly distributed or the distribution function has to be introduced.

1 - Introduction

Thin film deposition techniques are becoming of crucial importance in many application fields; semiconductor devices and in general, electronic solid-state devices, are

all based on material structures created by thin film deposition. The understanding of the physics and chemistry of films, surfaces, interfaces and microstructures is necessary to establish deposition techniques and procedures with the aim to obtain the desired characteristics in the deposited materials.

Thin films are layers in the thickness range of a few nanometers to about ten micrometers; the thickness of the layer is obviously a fundamental parameter for the film characterisation and is particularly crucial in the case of multistrate structures.

In this work we present a procedure for the measurement of the thicknesses in multiple layers samples, obtained through a procedure based on X-ray diffraction at glancing angle incidence. The procedure resulted quite simple, rapid and non-destructive.

The samples here concerned as example are constituted by a substrate of alumina, covered by a thin layer of titanium oxide (rutile) and, successively, by a film of gallium oxide utilising the MOCVD (Metal Organic Chemical Vapour Deposition) technique; this device could be advantageously used as an oxygen-sensitive semiconductor material at temperatures of over 500°C for the peculiar electric properties of gallium oxide at high temperatures. The interposed layer of titanium oxide is concerned as a buffer layer to prevent the mobilisation of aluminium towards the surface in consideration of Al-Ga chemical affinity. The experimental MOCVD conditions and the choice of precursors are described elsewhere [1-2].

2 - Theoretical Considerations

The method is based on the diffraction intensity attenuation due to X-Ray absorption in passing through the layers.

Powder X-Ray diffraction analysis serves primarily to identify the structure of the deposited polycrystalline films; this can be done in an easy way comparing diffraction intensities with powder diffraction data banks such as JCPDS (Joint Committee on Powder Diffraction Standards). Anyway this procedure is often unsatisfactory for thin layers, because X-rays interact mainly with the substrate bulk and less with the surficial

film as a consequence of the little film volume affected in a θ-2θ geometry diffractometer.

Varying the incident angle, varies the effective volume.of the material that diffracts, and the most grazing is the incident beam, the higher is the transversal path in the sample; therefore the incoming X-ray beam can interact only with the surficial layer or partly with the substrate, and so on, up to the involvement of the whole sample.

This is well shown in Fig. 1, where at 1° the gallium oxide pattern, (relative to the surficial film), is the only one collected, and at 10° the alumina pattern, (relative to the substrate), has considerably grown too, while gallium oxide diffraction intensities decrease notably as a consequence of the different volumes concerned at increasing

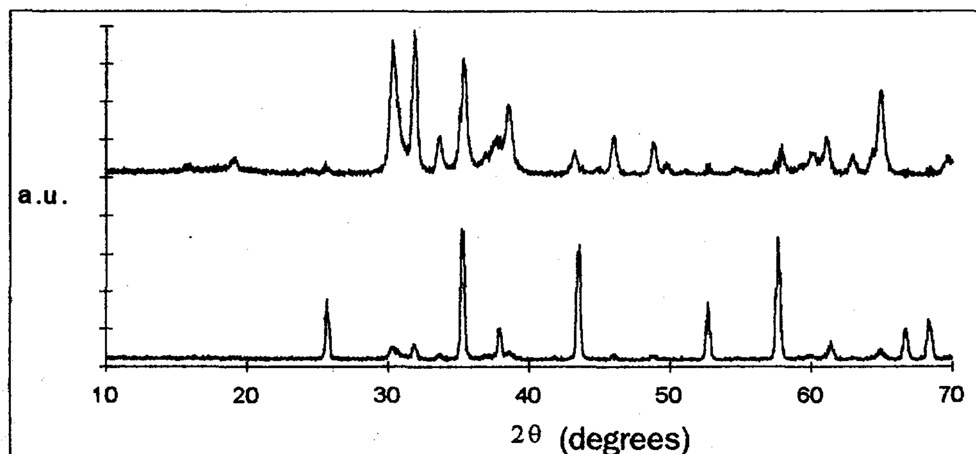

Figure 1 - Diffraction pattern of a layer of Ga_2O_3 on alumina substrate at 1° incidence angle (upper) showing mainly the bands relative to gallium oxide (JCPDS # 41-1103) and at 10° (lower) showing predominantly the alumina bands (JCPDS # 42-1468); a.u. means arbitrary units.

angles. For this reason the measurements were performed with X-rays impinging on sample surface at glancing angles, from 0.5 to 10 degrees, in any case at angles lower than 2θ; the detection of diffraction intensities is then obtained employing a thin film attachment that makes possible the data collection also in these non focusing conditions.

The volume irradiated by the impinging X-ray beam also depends on X-ray absorption in passing through the material because the incident beam is reduced in intensity as it penetrates. Generally speaking the emerging intensity I of an X-ray beam passing through a plate material of thickness d (cm), and linear absorption coefficient μ (cm^{-1}), is given by the well-known relationship :

$$I = I_0 \cdot \exp(-\mu d) \quad (1)$$

where I_0 is the incident X-ray intensity; multiplying and dividing μ by the density ρ of the material the latter expression becomes

$$I = I_0 \cdot \exp\left(-\left(\frac{\mu}{\rho}\right) \cdot \rho \cdot d\right) \quad (2)$$

in which the quantity (μ/ρ) is the mass absorption coefficient (dimensions of cm^2g^{-1}) and is independent of the state of the matter; (μ/ρ) varies with the mean atomic number of the sample, and with the wavelength of the X-ray beam, and can be found tabulated.

Now we consider a specimen in the form of a flat plate whose dimensions are much larger than the incident beam.

Let β be the angle between the X-ray beam and sample surface (see Fig.2), $\xi = 2\theta - \beta$ the angle between the sample surface and the emerging diffracted beam, where θ is the angle related to the reflecting planes through the Bragg equation [3]:

$$n\lambda = 2d\sin\theta \quad (3)$$

and A is the area of the incident beam.

Considering the volume element $dV = (A/\sin\beta)dx$ at depth x, let I_i be the intensity density of the incident X-ray beam; at depth x the attenuation factor is $\exp(-(\mu/\rho)\rho x/\sin\beta)$.

Then the contribution of the element dV is

$$dI = I_i \cdot \exp\left(-\left(\frac{\mu}{\rho}\right) \cdot \left(\frac{1}{\sin\beta}\right) \cdot \rho \cdot x\right) dV = I_i \cdot \left(\frac{A}{\sin\beta}\right) \exp\left(-\left(\frac{\mu}{\rho}\right) \cdot \left(\frac{1}{\sin\beta}\right) \cdot \rho \cdot x\right) dx \quad (4)$$

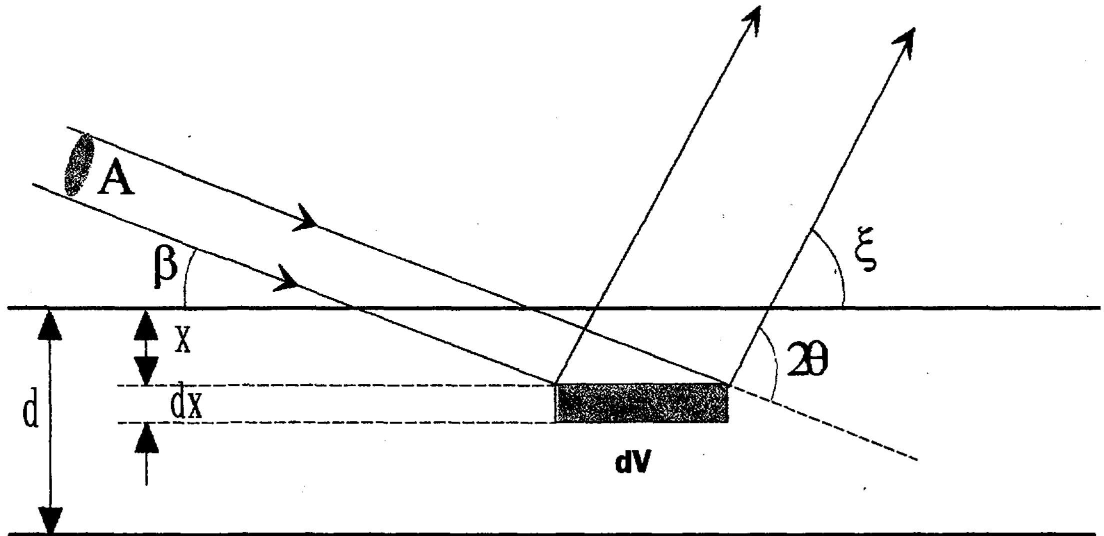

Figure 2 - Geometry of the diffraction system from a layer of thickness d and area much larger than the incident beam area A.

The correlated diffracted intensity $k_1 dI$ (where k_1 is the fraction of the beam which is diffracted by the film) will be attenuated along the exit path $x/\sin\xi$..

In the hypothesis that all the layer would be affected by the X-ray beam, that is the layer is a thin film, we can integrate over the thickness d:

$$I_1 = \int_0^d k_1 \cdot \frac{A}{\sin\beta} \cdot I_i \cdot \exp\left(-\left(\frac{\mu}{\rho}\right)_1 \cdot \left(\frac{1}{\sin\beta} + \frac{1}{\sin\xi}\right) \cdot \rho_1 \cdot x\right) \cdot dx \tag{5}$$

where I_1 is the total intensity (cps) of the diffraction peak at the Bragg angle θ and will be correlated to the measured area of the corresponding peak at 2θ in the experimental diffraction pattern; $(\mu/\rho)_1$, ρ_1 and d are the mass absorption coefficient, density and thickness of the film respectively.

Replacing $k_1 A I_i = K_1$ the integration leads to:

$$I_1 = \frac{K_1}{\sin\beta} \cdot \frac{1 - \exp\left(-\left(\frac{\mu}{\rho}\right)_1 \cdot \left(\frac{1}{\sin\beta} + \frac{1}{\sin\xi}\right) \cdot \rho_1 \cdot d\right)}{\left(\frac{\mu}{\rho}\right)_1 \cdot \left(\frac{1}{\sin\beta} + \frac{1}{\sin\xi}\right) \cdot \rho_1} \tag{6}$$

In a similar way, the diffraction intensity due to the second layer is given by:

$$I_2 = \frac{K_2}{\sin\beta} \cdot \frac{1-\exp\left(-\left(\frac{\mu}{\rho}\right)_2 \cdot \left(\frac{1}{\sin\beta}+\frac{1}{\sin\xi}\right)\cdot \rho_2 \cdot d_2\right)}{\left(\frac{\mu}{\rho}\right)_2 \cdot \left(\frac{1}{\sin\beta}+\frac{1}{\sin\xi}\right)\cdot \rho_2} \cdot \exp\left(-\left(\frac{\mu}{\rho}\right)_1 \cdot \left(\frac{1}{\sin\beta}+\frac{1}{\sin\xi}\right)\cdot \rho_1 \cdot d_1\right) \quad (7)$$

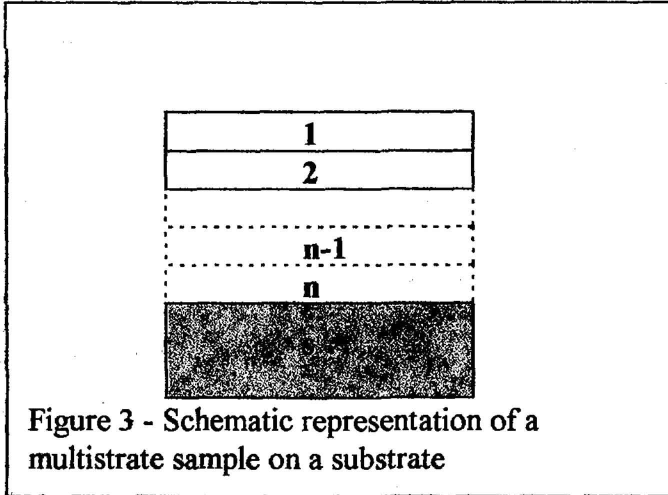

Figure 3 - Schematic representation of a multistrate sample on a substrate

In general, putting $K_n = k_n A$ I_i the diffraction intensity due to the nth layer adsorbed by the n-1 upper layers (Fig. 3) is given by :

$$I_n = \frac{K_n}{\sin\beta} \cdot \frac{1-\exp\left(-\left(\frac{\mu}{\rho}\right)_n \cdot \left(\frac{1}{\sin\beta}+\frac{1}{\sin\xi}\right)\cdot \rho_n \cdot d_n\right)}{\left(\frac{\mu}{\rho}\right)_n \cdot \left(\frac{1}{\sin\beta}+\frac{1}{\sin\xi}\right)\cdot \rho_n} \cdot \prod_{i=1}^{n-1} \exp\left(-\left(\frac{\mu}{\rho}\right)_i \cdot \left(\frac{1}{\sin\beta}+\frac{1}{\sin\xi}\right)\cdot \rho_i \cdot d\right) \quad (8)$$

The intensity I_n depends on the n thickness parameters d_n and on a parameter K_n as factor scale, while the absorption coefficients $(\mu/\rho)_n$'s and the densities ρ_n's are evaluated from literature. When the intensity from the substrate is considered, then the previous equation becomes (putting $d_n \rightarrow \infty$):

$$I_s = \frac{K_s}{\sin\beta} \cdot \frac{\prod_{i=1}^{n} \exp\left(-\left(\frac{\mu}{\rho}\right)_i \cdot \left(\frac{1}{\sin\beta} + \frac{1}{\sin\xi}\right) \cdot \rho_i \cdot d_i\right)}{\left(\frac{\mu}{\rho}\right)_s \cdot \left(\frac{1}{\sin\beta} + \frac{1}{\sin\xi}\right) \cdot \rho_s} \qquad (9)$$

where subscript "s" refers to the substrate.

In the example of two layers system, the theoretical trends of Eq. 6 (referring to the surficial layer), Eq. 7 (referring to the second layer) and Eq. 9 for n=2 (referring to the substrate) versus the incidence X-ray angle, are shown in Fig.4.

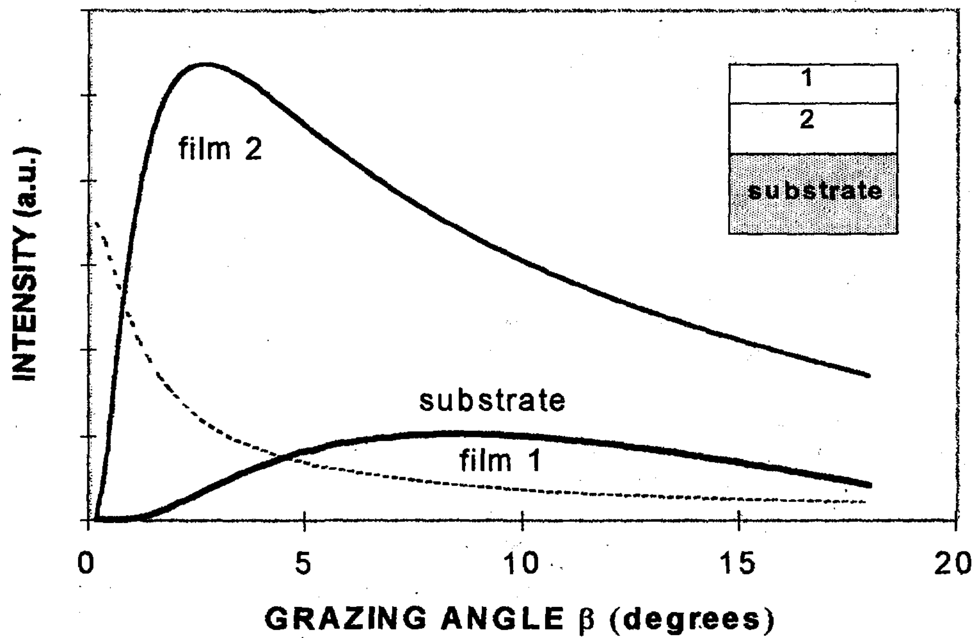

Figure 4 - Theoretical trend of intensities vs. grazing angle β for a two layer system.

For the simplest system constituted by a film on a substrate the intensities I_1 and I_S depend on the film thickness parameter d and on the parameters K_1 and K_S respectively

as factor scale, while the absorption coefficients (μ/ρ)'s and the densities ρs are evaluated from literature [4].

In this way two separate evaluations of the same parameter d can be obtained, one from Eq.(6) and one from Eq.(9). This also makes possible the estimation of the thickness of an amorphous film on a polycrystalline substrate using exclusively expression (9) or the estimation of the thickness of a polycrystalline film on an amorphous substrate using only expression (6).

Now, from the comparison between the theoretical trend using Eq.(6) or Eq.(9), and the measured peak areas obtained varying the glancing angle (β), and utilising the method of minimum least squares fit, the thickness of the layer concerned is determined. A mathematical function called the reduced chi-squared (χ_{red}) is used as a measure of the goodness of the fit [5]; χ_{red} should be close to unity, but if the fitting function is not appropriate for describing the data, or if starting parameters are not reasonably close to correct values, the deviations will be larger and χ_{red} will give a value much greater than unity.

Generally speaking each parameter d_n can be evaluated separately both from the equation corresponding to the I_n intensity and from the equation corresponding to I_{n+1} intensity. This also makes possible the estimation of the thickness d_n of an amorphous film on a polycrystalline one using exclusively the I_{n+1} intensity or the estimation of the thickness d_n of a polycrystalline film on an amorphous substrate using only I_n intensity.

3 - Formulae Application

3.1 - Mass absorption coefficients

The mass absorption coefficient μ/ρ is an atomic property of each element independent, for practical purposes, of state of chemical or physical aggregation. It is a function only of wavelength and atomic number. Some values of (μ/ρ) are reported in Table 1 for elements with Z=1 to 83.

Table 1 - Mass absorption coefficients μ/ρ (cm^2/g) of the elements Z=1 to 83 for the wavelengths of Cu kα 1.5418 Å and Mo kα 0.7107 Å

El	Cu kα	Mo kα	El	Cu kα	Mo kα
H	0.435	0.380	**Cr**	260	31.1
He	0.383	0.207	**Mn**	285	34.7
Li	0.716	0.217	**Fe**	308	38.5
Be	1.50	0.298	**Co**	313	42.5
B	2.39	0.392	**Ni**	45.7	46.6
C	4.60	0.625	**Cu**	52.9	50.9
N	7.52	0.916	**Zn**	60.3	55.4
O	11.5	1.31	**Ga**	67.9	60.1
F	16.4	1.80	**Ge**	75.6	64.8
Ne	22.0	2.47	**As**	83.4	69.7
Na	30.1	3.21	**Se**	91.4	74.7
Mg	38.6	4.11	**Br**	99.6	79.8
Al	48.6	5.16	**Kr**	108	84.9
Si	60.6	6.44	**Rb**	117	90.0
P	74.1	7.89	**Sr**	125	95.0
S	89.1	9.55	**Y**	134	100
Cl	106	11.4	**Zr**	143	15.9
A	123	13.5	**Nb**	153	17.1
K	143	15.8	**Mo**	162	18.4
Ca	162	18.3	**Tc**	172	19.7
Sc	184	21.1	**Ru**	183	21.1
Ti	208	24.2	**Rh**	194	22.6
V	233	27.5	**Pd**	206	24.1

El	Cu kα	Mo kα	El	Cu kα	Mo kα
Ag	218	25.8	**Yb**	146	84.5
Cd	231	27.5	**Lu**	153	88.2
In	243	29.3	**Hf**	159	91.7
Sn	256	31.1	**Ta**	166	95.4
Sb	270	33.1	**W**	172	99.1
Te	282	35.0	**Re**	179	103
I	294	37.1	**Os**	186	106
Xe	306	39.2	**Ir**	193	110
Cs	318	41.3	**Pt**	200	113
Ba	330	43.5	**Au**	208	115
La	341	45.8	**Hg**	216	117
Ce	352	48.2	**Tl**	224	119
Pr	363	50.7	**Pb**	232	120
Nd	374	53.2	**Bi**	240	120
Pm	386	55.9			
Sm	397	58.6			
Eu	425	61.5			
Gd	439	64.4			
Tb	273	67.5			
Dy	286	70.6			
Ho	128	73.9			
Er	134	77.3			
Tm	140	80.8			

μ/ρ for a compound, solution or mixture of elements A, B, C,.. in weight fractions W_A, W_B, W_C,.., is readily calculated from the values of the constituents as [6]:

$$(\mu/\rho)_{ABC...} = \Sigma\, W_i(\mu/\rho)_i = W_A(\mu/\rho)_A + W_B(\mu/\rho)_B + ...$$

In the case of the compound TiO_2 where $W_{ti} = 0.6$ and $W_O = 0.4$, taking $(\mu/\rho)_{Ti}$ and $(\mu/\rho)_O$ from the table 1 for $\lambda = 1.5418$ Å (CuKα) we obtain

$$(\mu/\rho)_{TiO2} = 0.6*208+0.4*11.5 = 129\ (cm^2/g)$$

3.2 - X-ray absorption limits

Our theoretical considerations imply that the film upperstanding the substrate does not reach the limit of infinitely thick layer.

The criterion for defining an infinitely thick layer is somewhat arbitrary and depends on our definition of what is a negligible contribute to the total intensity. For our aim we define infinite thickness as that for which the contribution to the total intensity from the back is 1% about that the intensity contributed from the front side.

Then by using the expression (4) :

$$\frac{dI_{x=0}}{dI_{x=d}} = \frac{I_i \cdot A \cdot dx}{I_i \cdot A \cdot \exp\left[-\left(\frac{\mu}{\rho}\right)\cdot\left(\frac{1}{\sin\beta}+\frac{1}{\sin\xi}\right)\cdot\rho\cdot t\right]dx} =$$

$$= \exp\left[\left(\frac{\mu}{\rho}\right)\cdot\left(\frac{1}{\sin\beta}+\frac{1}{\sin\xi}\right)\cdot\rho\cdot t\right] = 100$$

or

$$t = \frac{\ln 100}{\mu\cdot\left(\frac{1}{\sin\beta}+\frac{1}{\sin\xi}\right)} \qquad (10)$$

Considering different substances, for typical values of $2\theta \approx 30°$ the relative infinite thickness is reported in Fig. 5 as a function of the grazing angle β.

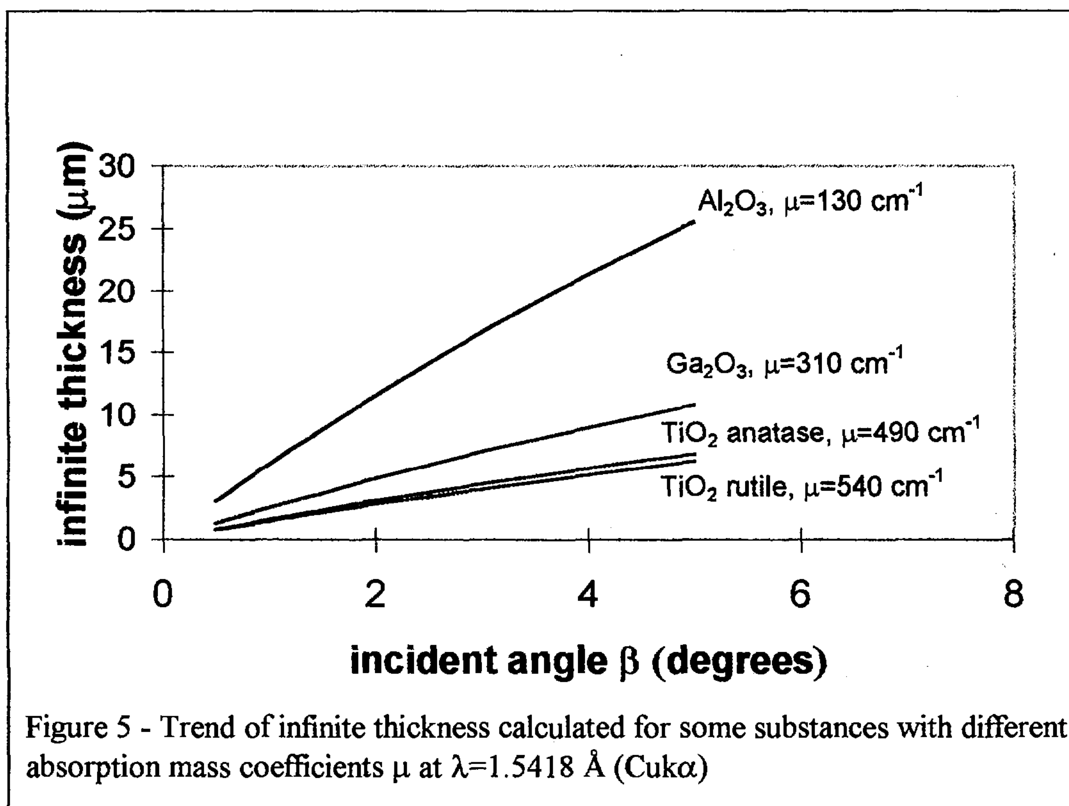

Figure 5 - Trend of infinite thickness calculated for some substances with different absorption mass coefficients μ at λ=1.5418 Å (Cukα)

The higher is μ the higher is the useful minimum angle β. Of course if more than one layer are concerned, the useful minimum angle β is not easy to be evaluated.
From practical point of view it could be estimated when the highest reflections from the substrate become detectable.

3.3 - Geometry constrictions

Figure 6 is a schematic perspective of the optical arrangement, where the focal spot is viewed longitudinally. The axial (δ) divergence of the primary beam is limited by the dimensions of the direct-beam aperture.

Our theoretical considerations require that both the impinging beam and the escaping diffracted one pass throughout the surface of the specimen, the limit condition is displayed in Fig. 6.

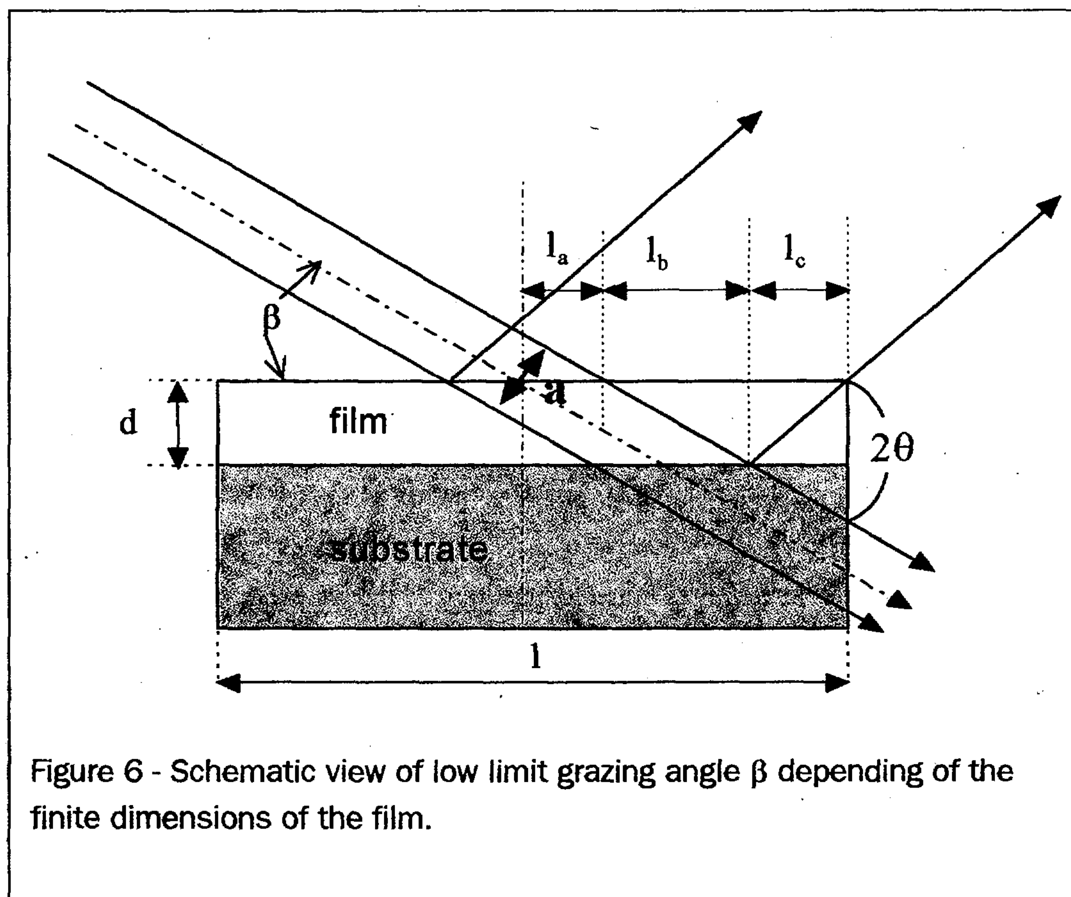

Figure 6 - Schematic view of low limit grazing angle β depending of the finite dimensions of the film.

If R is the radius of the goniometer circle, δ is the beam divergence in degrees, a is the beam aperture on the sample with half projection l_a on the film surface, l_b and l_c are defined in Fig. 6 and depend on the film thickness d, then

$$a = R \cdot \delta \cdot \frac{\pi}{180}$$

$$l_a = \frac{a}{2 \cdot sin\beta}$$

$$l_b = d \cdot tg^{-1}\beta$$

$$l_c = d \cdot tg^{-1}(2\vartheta - \beta)$$

and the constraint

$$l_a + l_b + l_c = \frac{l}{2}$$

must be fulfilled.

In table 2 the calculated low limit grazing angles β vs film thickness d as a function of instrumental divergence δ and sample length l for typical values of 2θ ≈ 30° are reported.

Table 2 - Calculated low limit grazing angles β

Sample length (mm)*thickness (μm)	limit grazing angle β with divergence slit of:		
	1/6°	1/12°	1/30°
10*1	2.4°	1.2°	0.5°
10*10	2.5°	1.3°	0.6°
20*1	1.2°	0.6°	0.2°
20*10	1.3°	0.7°	0.3°

The limit grazing angle β strongly depends both from slit divergence and from sample length but is practically insensitive to the film thickness.

4 - Experimental

A PHILIPS X'Pert Diffraction System on a PW3710 Generator was employed for diffraction measurements; the system is equipped with a Thin Film Attachment that permits the analysis of polycrystalline thin film . $Cu_{K\alpha}$ radiation was employed.

The samples were also investigated using a PHILIPS XL 40 Scanning Electron Microscope (SEM) equipped with a PV9900 EDAX system for microanalysis. The samples were embedded in araldite resin and lapped with emery paper to obtain a transversal section.

5 - Results and Discussion

To illustrate the theoretical considerations, considerations on some samples are reported. Samples belonging to series T consisted of alumina substrates covered by films of TiO_2, samples G consisted of alumina covered by a film of Ga_2O_3 and sample GT consisted of alumina covered by a multilayer Ga_2O_3/TiO_2.

i) Titania films were deposited at a temperature of 420°C via MOCVD and resulted polycrystalline in the anatase phase [1]. After annealing in air at 900°C for three hours an anatase-rutile mixture appeared in the films and after three hours at 1100°C a single-phased rutile was the only one obtained.

ii) Gallium oxide films were deposited at 470°C gave amorphous layers and after annealing in air at 1000°C for three hours the films became completely polycrystalline[2].

iii) The gallium oxide/titanium oxide multistrate was deposited at the conditions above reported for i) and ii) sequentially.

The calculated thickness values and the corresponding χ_{red} for monolayers are reported in table 3; for one TiO_2 film it is possible to have two values of thickness due to the different phases investigated as the titania layers were analysed before and after the annealing procedure.

The thicknesses of anatase films resulted notably greater than the corresponding rutile values; this is not only explained by the different density of the two phases (anatase density 3.84, rutile density 4.26), but it seems more probable that anatase films have a density considerably higher than the bulk anatase density; if the thickness could be obtained in a different way with good accuracy, our investigation could be employed for density measurement of thin films.

TABLE 3

Sample	Film phase	Film thickness (μm)	χ_{red}
1T	Titanium oxide, anatase	3.14	1.56
1T	Titanium oxide, rutile	2.56	1.32
2T	Titanium oxide, anatase	2.93	2.22
2T	Titanium oxide, rutile	2.11	0.96
3T	Titanium oxide, anatase	2.68	1.84
3T	Titanium oxide, rutile	1.95	1.16
4G	Gallium oxide	0.56	0.71

The comparison between X-ray and SEM data on the annealed films, is reported in table 4; a satisfactory agreement was obtained with deviations within 25%.

TABLE 4

Sample	Calculated thickness (μm)	SEM thickness (μm)	% variation
1T (rutile)	2.56	2.57	0.4
2T (rutile)	2.11	1.94	8.0
3T (rutile)	1.95	2.30	17.9
4G	0.56	0.42	25.0

In the immediate future we intend to study in more detail the density of the investigated films, with the aim of obtaining a correction of thickness values that takes into account the real film density.

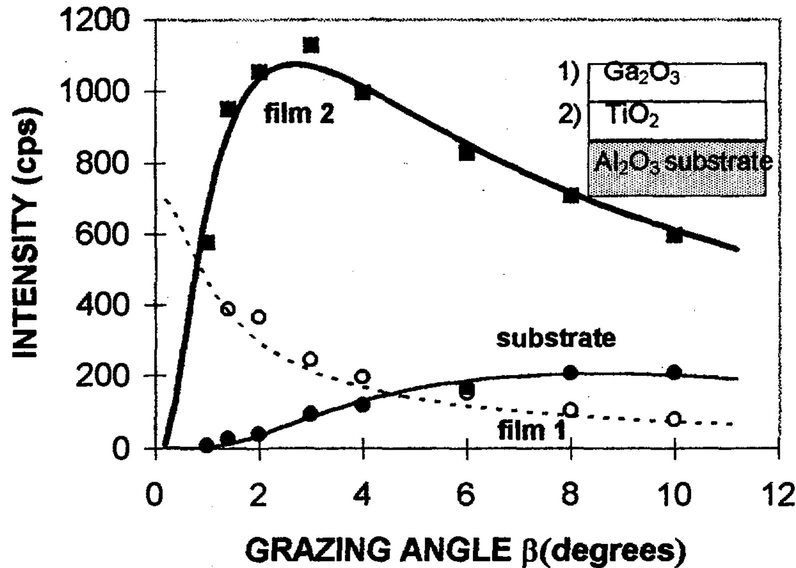

Figure 7 - Theoretical and calculated intensity patterns as a function of grazing angle β of a multilayer (Ga_2O_3 and TiO_2 thin films) on an alumina substrate, sample 1GT.

As an example of a multistrate we report in Fig. 7 the fits performed on a Ga_2O_3/TiO_2 multilayer on an alumina substrate. Ga_2O_3 thickness can be evaluated from its diffraction bands utilising Eq. (6) or from TiO_2 diffraction bands utilising Eq. (7) or from Al_2O_3 diffraction bands utilising Eq. (9). Analogously TiO_2 thickness can be evaluated from its diffraction bands utilising Eq. (7) or from Al_2O_3 diffraction bands utilising Eq. (9). The experimental intensity data can be obtained by peak area integration of bands of interest in the X-ray pattern as shown in Fig.7, or by Ω scan at fixed 2θ.

The results of performed fittings are reported in table 5 for the sample 1GT constituted by a Ga_2O_3 film grown on a TiO_2 film deposited on an alumina substrate ; the final thickness will be evaluated as a weighed average of the corresponding data.

TABLE 5

Sample	Film phase	calculated on phase :	Film thickness ±σ (μm)	χred
1GT : Gallium oxide/ rutile/ alumina(substrate)	Gallium oxide	Gallium oxide	0.42 ± 0.03	0.95
	Gallium oxide	rutile	0.56 ± 0.05	7.31
	Gallium oxide	Alumina	0.35 ± 0.03	2.93
	rutile	rutile	1.57 ± 0.14	7.31
	rutile	Alumina	1.52 ± 0.14	2.93

Our theoretical considerations are directly applicable to materials with no preferred orientations ; really films grown by MOCVD technique in particular conditions like lower deposition temperatures and higher thickness, can show some preferential growth ; moreover the substrate itself can show some orientation. In these cases we can hypothesize a Gaussian distribution function of the oriented planes, so the intensity relationship can be improved by introducing a corrective function defined as

$$f_c = 1 + \frac{k}{\sigma} \cdot \exp\left[-\frac{1}{2}\left(\frac{\beta - \beta_m}{\sigma} \right)^2 \right] \tag{11}$$

where the second term of the expression represents a continuos function describing the probability that from a measured distribution with a mean value β_m and a standard deviation σ the value of a random observation would be β.

The function f_c is then included in the fitting procedure, decreasing in this way the degrees of freedom. Only in the case of the substrate of alumina, we can evaluate *a priori* the corrective factor f_c measuring directly the substrate itself before depositing the films. The theoretical intensity trend I_s from Eq. 9 with n=0 is then modified by the empirical function (11) ; the factor f_c is obtained fitting f_c * I_s with the experimental

points (see fig. 8) ; this corrective function f_c for alumina substrates will be then employed in all calculations when the alumina substrate is concerned.

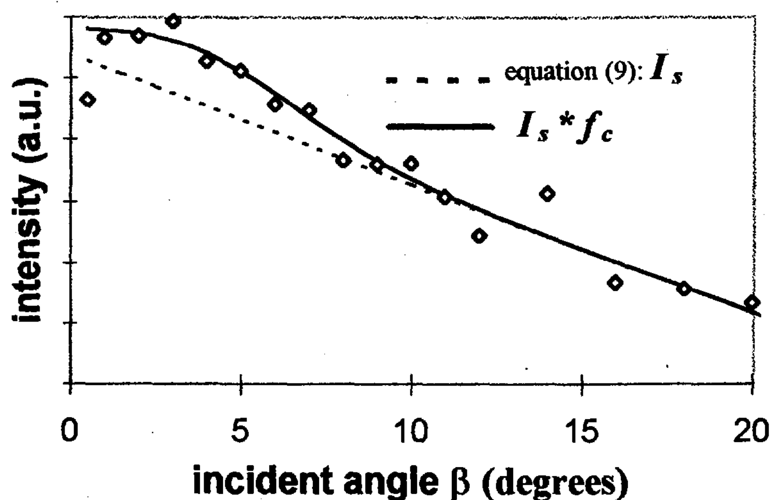

Figure 8. The theoretical intensity trend for alumina substrate (dotted line) is modified by the empirical fuction f_c (pleine line) to fit the experimental points.

6 - Conclusions

Thickness evaluation of thin films by our method, based on diffraction intensity attenuation as a function of X-ray beam incident angle, resulted quite satisfactorily for thin films in the 0.5 to 4 μm range. The procedure is rapid and non destructive.

The main limitation of this method is that either the film or the substrate should be polycrystalline and its nature must be carefully accomplished ; secondly we need to know the density value of thin films and maybe this is the main problem to be resolved and will be object of future investigations.

7 - Acknowledgements

We gratefully acknowledge the contribution of Dr. Pierino Zanella for helpful discussion.

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