

Optical characterisation of hybrid antireflective coatings using spectrophotometric and ellipsometric measurements

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A hybrid antireflective coating combining homogeneous layers and linear gradient refractive index layers has been deposited using different techniques. The samples were analysed by optical characterisation based on spectrophotometric and spectroscopic ellipsometric measurements under different angles of incidence in order to precisely characterise the coatings. The Lorentz-Lorenz model has been used for calculation of refractive index of material mixtures in gradient index layers as well as some homogenous parts of the coating. The obtained refractive index profiles have been compared with the targeted ones to detect errors in processes of deposition.

Key words: antireflective coating, optical characterisation, hybrid coating, reverse engineering, variable angle spectroscopic ellipsometry.

Copyrightposition

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1. Introduction

Optical characterisation methods are of utmost importance in analysis of coatings in thin films science and technology. They allow determination of refractive index, thickness of the coating and variation of refractive index through the depth of the coating (i.e. inhomogeneity). Numerous methods for optical characterisation of thin films have been developed. These methods usually start from modelling the optical behaviour of the sample through a set of parameters which represents some kind of initial approximation. The optimal values of these parameters are found by the minimisation of a merit function that quantifies the agreement between experimental measurements and the data simulated from the sample model. The optimisation procedure is normally carried out using numerical techniques. Some of these methods are based on analysis of spectrophotometric measurements¹, i. e. reflectance ($R(\lambda)$) and transmittance ($T(\lambda)$) as function of the wavelength (λ) in a certain spectral range, and some on analysis of ellipsometric Δ and Ψ functions^{2,3} or their combination^{4,5,6}. Spectroscopic ellipsometry has been shown to be a very sensitive technique with respect to the variation of the refractive index with the layer thickness⁷. A typical problem of these methods is the multiplicity of solutions, that is, the existence of different combination of parameters values that minimise the merit function. In order to avoid this problem it is useful to have more measurements of the same sample, like measurements

taken at different angles of incidence, to combine spectrophotometry and spectroscopic ellipsometry, or to include non-optical characterization methods⁸. This leads to a significant reduction of the solution multiplicity, thus facilitating the selection of the physically meaningful one.

The model, i.e. the refractive index profile found by optical characterisation, can be compared with the targeted profile that was aimed to be deposited. Analysis of the differences between the two is crucial in detecting the errors in the process of deposition and improves the manufacturing procedures.

Antireflective coatings belong to the most important coating designs because they reduce undesirable multiple reflections between different surfaces in optical systems that contribute to the intensity loss of the transmitted light and disturb observer's vision by creation of ghost images. For standard situations practical solutions are well known^{9,10}. As long as normal light incidence is required, the maximum principle¹¹ states that most feasible coating design will comprise only two dielectric coating materials with as high as possible difference in refractive indices. However, in the case of wide-angular or omnidirectional coatings it may be useful to introduce one or more additional materials or gradient index layers.

Some standard methods for the deposition of gradient layers by varying composition and refractive index in the direction of the film growth are sol-gel method^{12,13}, changing the composition of ternary compounds during growth of the layer by controlling the composition of the reactive gas¹⁴ and the co-deposition of two materials where the ratio of the individual fluxes of particles condensing at the substrate is varied¹⁵.

The antireflective coatings studied in this paper were prepared by three different co-deposition techniques: electron beam evaporation, radio-frequency magnetron-sputtering and ion

beam sputtering. Spectrophotometric and ellipsometric measurements under different angles of incidence were performed for optical characterisation of samples. The refractive index profiles thus obtained were compared with the targeted ones and conclusions about errors in the deposition processes and suggestions how to minimize them were proposed.

2. Design and deposition

The hybrid antireflective coating was designed to minimise the reflectance of a BK7 glass substrate in the wavelength range 480-680nm in the range of 0° - 50° of angle of incidence¹⁶. The hybrid coatings combine refractive index gradients (ramps) with layers of constant refractive index through the thickness of the film. The obtained hybrid design was refined according to the materials available for the different deposition techniques (see Fig. 1), and subsequently, a round-robin experiment of deposition with different techniques has been performed¹⁷. The studied samples were deposited by electron beam evaporation (EBE), radio frequency magnetron-sputtering (RFS) and ion beam sputtering (IBS). In all three techniques SiO_2 was used as the low index material, while high index materials were Nb_2O_5 , Ta_2O_5 and TiO_2 , respectively. In the case of IBS ramps are approximated and deposited as sequence of ultrathin layers with constant refractive indices. Some characteristics of each of the techniques can be found in Table 1.

3. Optical characterisation and numerical data evaluation

Due to the desired optical properties, there are no significant fringes with considerable amplitude in the structure of the spectra of hybrid AR coating in the visible range (see Fig. 2(a) for example). On the contrary, reflectance and transmittance is rather constant in most of the spectral

range of interest. This would make optical characterisation based solely on spectrophotometric data very difficult and ambiguous: lack of interferential fringes increases the correlation between refractive index and thickness, what is particularly critical in the present case, in which the aim is to find the profile of the refractive index through the coating thickness. Therefore, spectrophotometric measurements were combined with ellipsometric measurements. Spectrophotometric measurements were performed with a Perkin Elmer Lambda 900 spectrophotometer. A VN-attachment allowing absolute measurement of reflectance without moving the sample after the transmittance measurement has been used. Reflectance and transmittance in the spectral range 400-850 nm were measured in steps of 2 nm: R and T measured at angle of incidence of 6 degrees and R_s , R_p , T_s and T_p at 45 degrees. Measurements of spectra of ellipsometric Δ and Ψ functions were performed with a standard SENTECH SE800 ellipsometer with microspot (200 μm). Usage of microspot, together with thickness of the glass (2mm) results in measurements without the contribution of reflection from the back side of the substrate. Ellipsometric measurements in the same spectral range as R and T were taken at the angles of 50, 55 and 60 degrees, in 575 points per angle. These angles were chosen for the ellipsometric functions to present maximum amplitude of their interferential fringes. In the optimisation process $\cos\Delta$ and $\cos 2\Psi$ were considered. The bare substrates were characterised previously, by means of spectrophotometry.

For the optical characterisation of the samples we used general-purpose software¹⁸ that allows determination of the optimal value of a set of parameters defining the sample by fitting experimental spectra. Each sample has been represented as an in-depth inhomogeneous coating deposited onto a substrate with known optical constants. According to the design, some parts of the coating have a constant composition making constant refractive index layers, while others

show a linear variation in composition, corresponding to the ramps in the design. The inhomogeneity of a ramp is taken into account by dividing it into a given number of homogeneous sublayers, all with the same thickness. Each homogeneous layer and sublayer has been modelled as a mixture of the two materials of high (n_H) and low refractive index (n_L) with volume fractions f_H and f_L , being $f_H + f_L = 1$. For each sublayer the volume fraction of the high index material is given by:

$$f_{H-i} = f_{H-start} + \frac{f_{H-end} - f_{H-start}}{N_{sub}} \left(i - \frac{1}{2} \right). \quad (1)$$

Here f_{H-i} is the volume fraction of the material of high refractive index in the sublayer i ($i = 1, 2, 3, \dots, N_{sub}$), $f_{H-start}$ and f_{H-end} are the values of the volume fractions of high index material at the beginning and at the end of the ramp and N_{sub} is the number of sublayers. The optical constants for each mixture layer can be calculated using different mixing formula: Bruggeman¹⁹, Lorentz-Lorenz²⁰, Maxwell Garnett²¹ or a linear combination of refractive indices²². It has been suggested²³ that an appropriate description of the optical constants of the mixture in our case is given by the Lorentz-Lorenz formula:

$$\frac{\varepsilon_{eff} - 1}{\varepsilon_{eff} + 2} = f_L \frac{\varepsilon_L - 1}{\varepsilon_L + 2} + f_H \frac{\varepsilon_H - 1}{\varepsilon_H + 2} \quad (2)$$

where ε_{eff} , ε_H and ε_L are the effective dielectric functions of the mixture and the high and low index material, respectively. The optical constants of the high and low index materials can be used either from a data file or represented with a dispersion model.

This way of modelling permits to represent the sample through a limited set of parameters: volume fraction and thickness for each layer and parameters defining the dispersion model for each material. The software enables fixing these parameters to a given value or to optimize them within some limits. Furthermore, it is possible to establish links between different parameters,

allowing imposing the continuity of the volume fraction at the interface of different layers. The optimisation of the parameters is carried out by the minimisation of a merit function that is the chi-square estimator χ^2 :

$$\chi^2 = \frac{1}{N - m - 1} \sum_{j=1}^{N_s} \sum_{k=1}^{N_j} \left(\frac{y_k^j - y^j(x_k; P_1, \dots, P_m)}{\sigma_k^j} \right)^2$$

where N is the total number of experimental data points, N_s is the number of measured spectra, each one containing N_j experimental data points, y_k^j represents measured values at the wavelength x_k with associated experimental error σ_k^j , $y_j(x_k; P_1, \dots, P_m)$ is the corresponding value calculated using standard thin film computation algorithms²⁴ and P_1, \dots, P_m are the m parameters being optimized. It must be highlighted that this merit function permits considering different magnitudes simultaneously in the same optimisation procedure (like spectrophotometric and ellipsometric measurements) since the quantities being added are dimensionless. The minimisation of the merit function is carried out using the Downhill-Simplex algorithm²⁵.

The original targeted designs have been taken as starting designs for optical characterisation defining the initial values of the parameters to be optimised. Each ramp in IOF and IST sample was divided into 8 sublayers. In this way, only starting and ending volume fractions of one material, as well as the thickness of the ramp, were optimized. Initially, limits to thickness of RFS and IBS samples were set to 3% of the design's thickness. In the case of EBE sample, higher errors are expected due to the fact that rates of deposition were controlled only by quartz monitor, but also due to high deposition rates compared with other two techniques and expected instabilities of these rates. Therefore, the limits to thickness were set to 6%. Refractive indices of the pure materials obtained from the measurements of the single material layers were used. In the next step, and in order to improve the data fits, the optimisation of the optical

constants of the materials by using dispersion formulas was allowed. For the case of the RFS sample, spectrophotometric measurements indicated the presence of absorption ($R+T<1$). Since in RFS high index material data file absorption was neglected, it was included in optimisation by a dispersion formula for the extinction coefficient

4. Results and discussion

The final models of refractive index profiles that are obtained in the process of optical characterisation, together with the original designs that were used as starting models, are shown in Fig. 1 for all three samples. In Table 2 numerical values of the materials Cauchy parameters and refractive indices are presented. The spectral characteristics of the models, compared with the measured spectra, are given in Fig. 2, 3 and 4. Figure parts (a) and (b) present R and T measurements, respectively, shown in the scale 0-1. Ellipsometric functions $\cos\Delta$ and $\cos 2\Psi$ are presented in figure parts (c) and (d).

Tables 3, 4, and 5 show the comparison of designs and models, in the sense of discrepancies of thicknesses and refractive indices. Errors are calculated as the absolute value of the difference of the parameter value in the model and design, divided by the parameter value in the design. The errors are given in percents.

Generally speaking, the characterisation procedure led to a remarkable agreement between the simulated and experimental data and a model close to the initial design. Differences between the obtained model and the initial design can be explained in terms of deviations in the deposition process. When only spectrophotometric measurements are used for optical characterization, R and T fits of the similar quality are obtained, but with other refractive index

profiles which are not consistent with the ellipsometry data. In this way, the complementary use of ellipsometric data results in a rather meaningful physical picture.

The optimisation of the refractive indices of SiO₂ of EBE and RFS samples, modelled with the Cauchy dispersion formula, enabled significant improvement of function of merit. On the contrary, no improvement was achieved by optimising the dispersion parameters of high index materials (except the need to include absorption in Ta₂O₅ of RFS as described above) or SiO₂ of IBS, regardless the used dispersion model. The differences to the data file refractive indices of EBE and RFS samples determined from samples with single layer of pure silica are between 1% and 2%, the higher corresponding to the RFS sample. Such difference could be explained by different conditions during the process of deposition (different pressure or temperature of the substrate) or difference in growth of the material when it is deposited directly to the bare substrate or to the pre-evaporated coating. Besides, for the RFS sample a more probable explanation could be contamination with the high index material. In fact, the found difference in SiO₂ refractive index would correspond to 5% volume fraction of Ta₂O₅ inclusions. The origin of these inclusions could be that SiO₂ target becomes contaminated by Ta₂O₅ while the latter is sputtered. Another possible reason could be as follows: all of the time during the deposition both sources must be kept running. So, at pure material deposition the other source is still running, although at extremely low power. This low power mode can be instable, depending of the process history. It should be possible to avoid this effect by running only one source for pure materials switching the other one off completely and protecting it from contamination with a closed shutter. It can also be reduced by using higher low-power limits. The refractive index of the layer that was supposed to be pure silica in the model for IBS sample is higher than could be expected for this material (Table 5). The reason for this, similar as for the case of SiO₂ of

RFS, could be inclusions of TiO_2 . In fact, the obtained refractive index of this layer corresponds to silica with 4% of volume fraction of titania. For co-deposition with the IBS technique a specially prepared zone target has been used²⁶. The mismatch in dependency of the refractive indices against the target position, leading to co-sputtering of both materials instead of only one of them, can originate from a slightly broadened ion beam²⁷. An additional effect may arise from the contamination of the non-sputtered side of the target with the actual coating material.

Regarding thicknesses, they remain within the expected errors of 6% for EBE and 3% for RFS and IBS. Only the third ramp of EBE model and the second one together with Ta_2O_5 layer of RFS model are out of these error ranges, improving the fits significantly. From the Fig. 1(a) it can be seen that the model of EBE sample gives ramps ending with higher refractive index compared to the design. This indicates that the rate of deposition of Nb_2O_5 was higher than expected, i.e. it was not well calibrated. On the contrary, the thickness of the pure Nb_2O_5 layer is as expected because it was controlled by quartz crystal monitor and not by time of deposition as in the case of ramps. In the case of RFS only the total rate of deposition, when both materials were evaporated simultaneously is known. Therefore, it is possible that the rate of deposition of Ta_2O_5 was not well estimated or suffers from larger deviations which resulted in the error in the thickness of the pure Ta_2O_5 layer. Other experiments showed similar effects at mixtures with a high fraction of Ta_2O_5 . It seems that the otherwise stable deposition rates vary significantly for the higher refractive material mixtures. The origin of this behaviour is still under the study.

Finally, it must be mentioned that other effective medium theories were tested to describe the optical constants of mixtures. Thus, using Bruggeman formula, merit functions were about 50% higher than those obtained by Lorentz-Lorenz, indicating that the latter is the proper choice for this kind of materials and deposition conditions being used.

5. Conclusions

Optical characterisation has been successfully applied for the analysis of hybrid antireflective coatings. It has been shown that the combination of spectrophotometric and ellipsometric measurements at different angles of incidence is the proper choice for the characterisation of systems like antireflective coatings, where the optical performance lacks details (fringes) in the reflected and transmitted spectra. The hybrid designs consisting of ramps of refractive index variation through depth of the film and layers of constant refractive index have been represented with appropriate models. The models had optimisation parameters which number and range were maximally controlled in the process of optimisation. Thus, simple and as realistic solutions as possible were obtained. For calculations of refractive indices of mixture materials the Lorentz-Lorenz model, as the most appropriate one, has been used.

The resulting models were helpful for determining the errors in deposition processes of each of the utilised deposition techniques, and this was the main goal. Thus, it has been found that the thicknesses of the coatings were controlled mainly within the expected accuracy. The main problem in deposition of the studied samples is control of the desired refractive index by means of better determination of deposition rate of high index material or by avoiding the undesired co-deposition when only the low index material is supposed to be deposited.

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Tab.1. Expected deviations in thickness and in refractive indices correspond to mixture layers.

Tab. 2: The dispersion formula for the refractive index was $n(\lambda)=a_0+a_1/\lambda^2$ and for the extinction coefficient $k(\lambda)=k_0\cdot\exp(k_1/\lambda)$ where λ is the wavelength in nanometers.

Tab.3. Refractive indices correspond to the starting refractive index of the layers. Only the thickness d_{model} of the third ramp (D) is more than 6% higher than the thickness of the original design d_{design} . Average error in refractive index is 2.7%. Error of Nb_2O_5 refractive index is 0 because it was fixed. When allowed to optimize, the quality of the fit did not improve.

Tab.4. Refractive indices correspond to the starting refractive index of the layers. The thickness of the second ramp B d_{model} is 8% higher than the thickness of the original design d_{design} and the thickness of the Ta_2O_5 layer is 27% higher (11 nm). Thicknesses of the other layers are within 3% of error to the starting thickness. Average error in refractive index is 2.2%. Error of Ta_2O_5 refractive index is 0 because it was fixed. When allowed to optimize, the quality of the fit did not improve.

Tab.5. Thickness of each layer is within 3% of error. Thicknesses of 10 layers have reached their minimum/maximum allowed value. There was no improvement to the fit when absorption was introduced. Average error in refractive index is 1.1%.

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Fig 1. The final models of refractive index profiles and the original designs that were used as starting models.

Fig. 2. The spectral characteristics of the models compared with the measured spectra of the sample deposited by electron beam evaporation. The back side of the substrate remained uncoated.

Fig. 3. The spectral characteristics of the models compared with the measured spectra of the sample deposited by radio-frequency sputtering. The back side of the substrate remained uncoated.

Fig. 4. The spectral characteristics of the models compared with the measured spectra of the sample deposited ion beam sputtering. The back side of the substrate remained uncoated.

Table 1. Characteristics of the deposition techniques

Deposition technique	Rate of deposition (nm/s)	Thickness control	Optical monitoring	Expected deviation in thickness	Expected deviation in refractive index
EBE	1.4 nm/s	time	no	6 %	0.6 %
RFS	0.14 nm/s	time	no	1.5 %	0.25%
IBS	0.035 nm/s	optical thickness determination	yes	2%	0.2%

Table 2. Dispersion parameters and material refractive indices

material	a0	a1	k0	k1	n(570nm)	k(570nm)
Nb ₂ O ₅	data file determined from single layer				2.28377	0
Ta ₂ O ₅	data file determined from single layer		0.00033232	0.012652	2.12494	3.2339e-4
TiO ₂	data file determined from single layer				2.40776	0
SiO ₂ EBE	1.4703	2790.5	0	0	1.47892	0
SiO ₂ RFS	1.4852	3524.8	0	0	1.49606	0
SiO ₂ IBS	datafile determined from single layer				1.49917	0

Table 3. EBE discrepancies from the design

layer	d_{design} (nm)	d_{model} (nm)	err_d (%)	n_{design} at 570nm	n_{model} at 570nm	err_n (%) at 570nm
A	124.05	118.68	4.33	1.598	1.615	1.07
B	112.89	106.17	5.95	2.111	2.239	6.00
C	35.91	33.75	6.00	1.598	1.595	0.21
D	87.15	96.72	10.98	2.111	2.273	7.67
E (Nb ₂ O ₅)	75.46	72.91	3.38	2.284	2.284	0
F (SiO ₂)	95.55	101.00	5.70	1.463	1.479	1.06

Table 4. RFS discrepancies from the design

layer	d_{design} (nm)	d_{model} (nm)	err_d (%)	n_{design} at 570nm	n_{model} at 570nm	err_n at 570nm
A	129.72	126.97	2.12	1.600	1.657	3.55
B	90.05	97.28	8.03	2.125	2.125	0.00
C	76.97	75.81	1.51	1.600	1.594	0.21
D	96.39	96.42	0.04	1.600	1.594	0.21
E (Ta_2O_5)	41.44	52.66	27.08	2.125	2.125	0
F (SiO_2)	97.45	97.50	0.06	1.469	1.496	1.84

Table 5. IBS discrepancies from the design

layer	d_{design} (nm)	d_{model} (nm)	err_d (%)	n_{design} at 570nm	n_{model} at 570nm	err_n (%) at 570nm
A1	31.84	32.67	2.61	1.59973	1.586	0.90
A2	8.61	8.80	2.21	1.67153	1.663	0.49
A3	10.61	10.82	1.98	1.74209	1.771	1.65
A4	13.9	14.32	3.00	1.81438	1.838	1.34
A5	17.22	17.74	3.00	1.88494	1.889	0.26
A6	18.27	18.71	2.41	1.957	1.934	1.16
A7	16.29	15.80	3.00	2.0276	2.019	0.42
B1	34.72	35.76	3.00	2.10046	2.126	1.21
B2	13.57	13.84	1.99	2.03673	2.047	0.47
B3	13.7	13.29	3.00	1.97523	1.961	0.76
B4	13.34	13.74	3.00	1.91215	1.914	0.09
B5	12.12	12.25	1.07	1.84959	1.879	1.60
B6	8.94	9.13	2.13	1.78677	1.815	1.64
B7	6.43	6.50	1.09	1.72466	1.755	1.74
B8	4.7	4.56	3.00	1.66177	1.691	1.75
C	78.47	76.12	3.00	1.59973	1.612	0.74
D1	13.39	12.99	2.99	1.70051	1.728	1.61
D2	19.54	18.95	3.00	1.80259	1.836	1.87
D3	21	21.14	0.71	1.90392	1.928	1.27

D4	18.12	17.72	2.21	2.0046	1.995	0.48
D5	14.63	14.28	2.39	2.10476	2.078	1.23
D6	15.88	15.60	1.7	2.20701	2.239	1.42
D7	17.49	17.56	2.09	2.30851	2.322	0.60
E (TiO ₂)	14.84	14.81	0.13	2.40957	2.389	0.87
F (SiO ₂)	99.86	101.71	1.85	1.50033	1.525	1.64

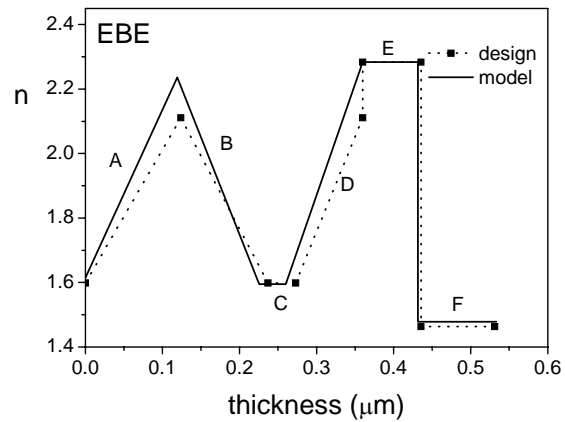


Fig. 1.a)

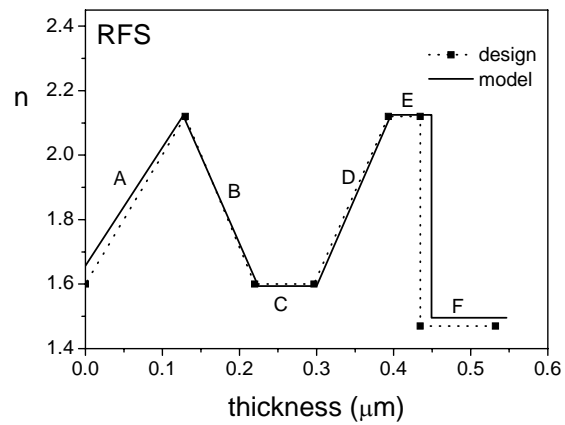


Fig. 1.b)

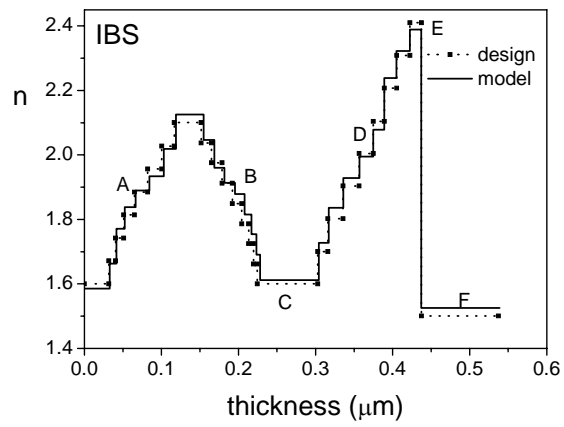


Fig. 1.c)

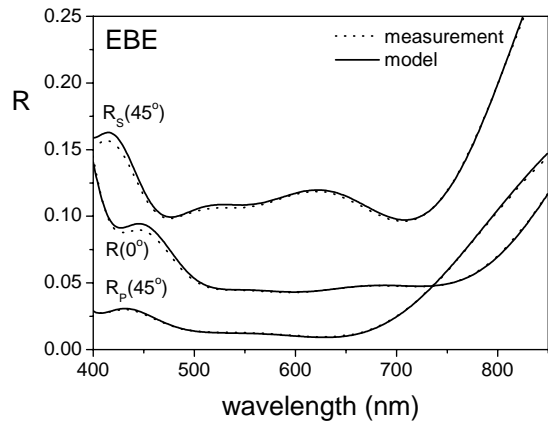


Fig. 2.a)

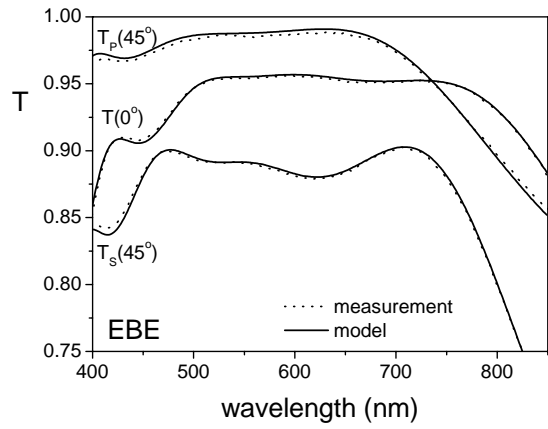


Fig. 2.b)

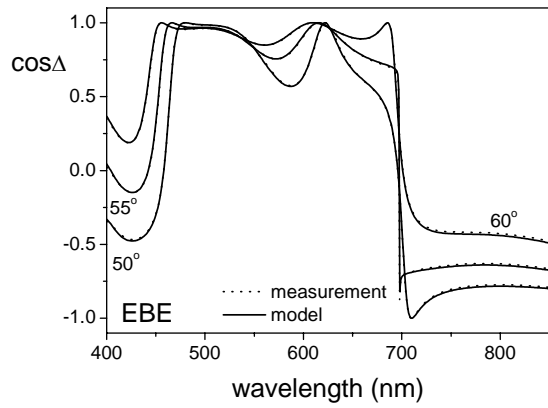


Fig. 2.c)

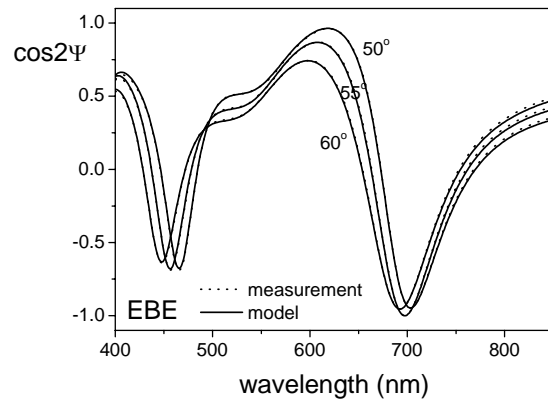


Fig. 2.d)

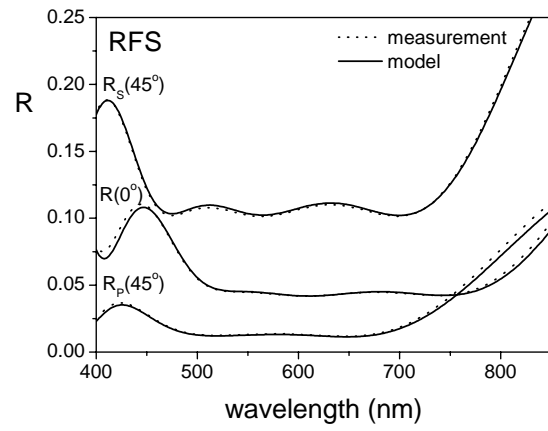


Fig. 3.a)

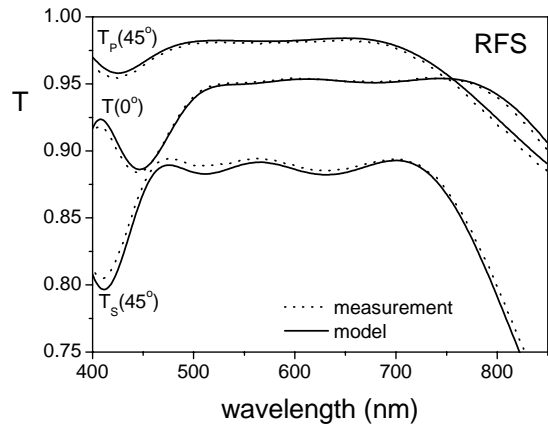


Fig. 3.b)

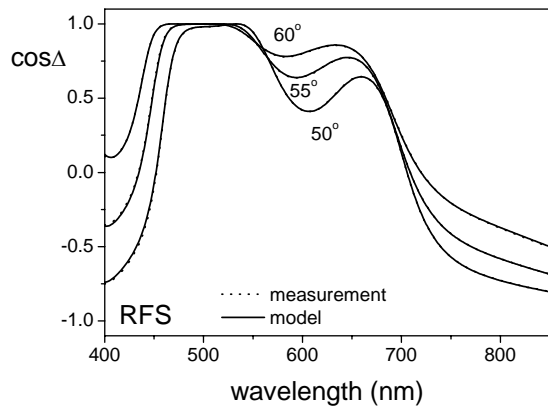


Fig. 3.c)

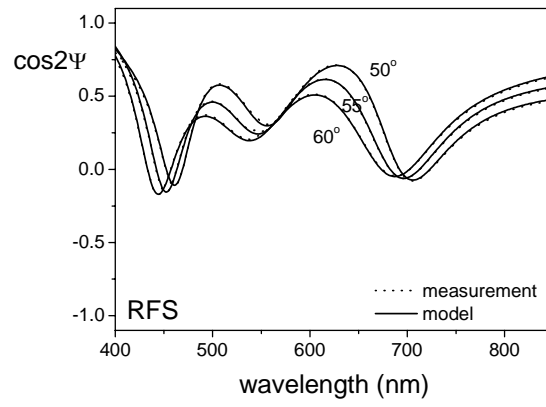


Fig. 3.d)

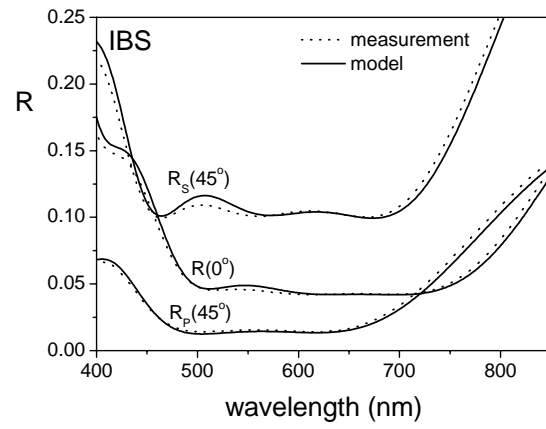


Fig. 4.a)

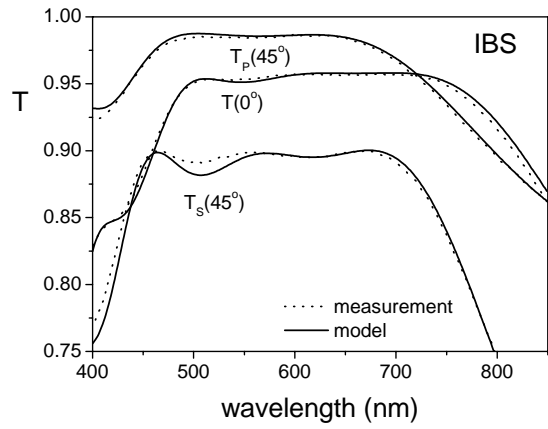


Fig. 4.b)

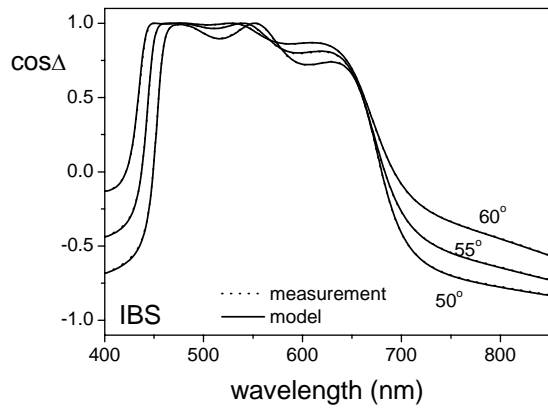


Fig. 4.c)

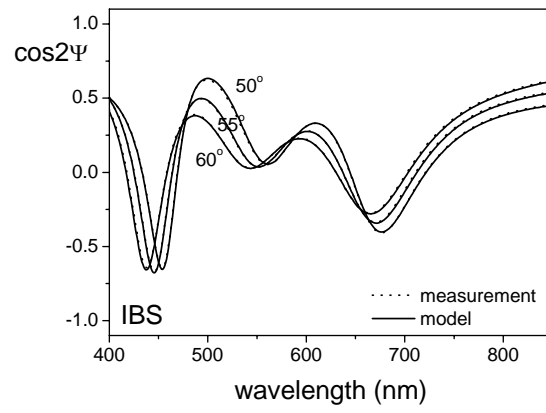


Fig. 4.d)