

Spectroscopic Ellipsometry Study of Liquid Crystal and Polymeric Thin Films in Visible and Near Infrared

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In order to be applied in photonic devices for telecom applications, soft materials, polymers and in particular liquid crystals, need to be optically characterised at the working wavelengths, typically in the Near IR region (NIR). Unfortunately, for most of these materials very few data are available in literature, especially in the NIR range. Moreover, because of the nature of these media, it is often necessary to make measurements on samples consisting of complicated multilayer stacks, including anisotropic, inhomogeneous, absorbing, and/or depolarising films. Recently, different ellipsometric techniques have been proposed to fulfil this task. However, in the case of such complicated sample structures, despite showing strong potentialities, ellipsometry seems to be very sensitive to the choice of a proper physical model and a good initial guess in the fitting procedure. In this work, we show the results of ellipsometric measurements, in the range from visible to NIR, for some liquid crystal and polymeric samples. We point out limits and potentialities of the technique and compare the obtained results with another experimental method, the m-lines spectroscopy, and/or with existing data in literature. The characterisation results of the analysed materials (the nematic liquid crystal 5CB, one commercial and one lab made optical polymer, and an Indium Tin Oxide film) are useful and interesting by themselves. However, more than in the provided data, we put the interest of the present analysis in the warnings about spectroscopic ellipsometry utilisation and eventually the necessity of complementary information.

I. Introduction

Soft matter application in devices and components for optical telecom networks is becoming a reality during last years. This is especially true for polymers, but also polymer based composites and liquid crystals (LC) can have niche market applications, as already demonstrated by a number of industrial projects and even products. Recently, developed polymers with fluorinated and chlorinated compounds [1] are expected to be the basic materials for passive parts of various optical telecom devices due to low optical losses (~ 0.5 dB/cm) and wide range of refractive index modulation ($1.4 \div 1.6$) as well in the visible as in the near IR (NIR) telecom bands. LC and composites made from polymers and LC can also play a role as active materials. Optical switches and Bragg grating wavelength filters based on LC [2, 3] are examples of such devices integrated in planar waveguide. Optical characterisation, in a wide spectral range, of newly developed materials, for photonic device application, is becoming a very important technical task. In particular, a very limited set of data regarding optical characterisation in the NIR region is available for LC and LC-polymer composites, even for standard and well known materials.

There are different methods for measuring the optical constants of solid and liquid materials. One of the most versatile, sensitive and well-established technique is Spectroscopic Ellipsometry (SE) [4]. SE and its further extensions, as the Generalised Ellipsometry (GE) and the Muller Matrix Ellipsometry (MME) [4], have been applied also to the systematic investigation of anisotropic thin (<10 μm) [5] and thick (<50 μm) [6] films of LC materials. However, ellipsometric measurements may pose serious problems of results reliability, due to the high complexity of usual LC containing samples. In fact, a typical LC cell is a multi-layer structure with its first layer formed by a very thick glass plate ($0.4 \div 1$ mm), and then it contains coating films, surfactant layers, inhomogeneous structures like the electrodes of Indium Tin Oxide (ITO). The presence of interface roughness and the strong anisotropy exhibited by LC, eventually with optical axis varying along the stratification direction, complete a picture not easy to analyse. On the other hand, ellipsometric data are sensitive to most of the optical and geometrical parameters of the sample under investigation; a meaningful data inversion is strictly dependent on the choice of a proper physical model; and, above all, the final multi-parameter fitting procedure may critically depend on a good initial guess. On these grounds, it appears reasonable supplementing ellipsometric data of complex structures with additional experimental information on the same sample, obtained with different and independent techniques.

In this paper, we present some results obtained by ellipsometric methods, in the range from visible to NIR, on LC samples and polymeric ones, and we discuss some difficulties of the technique, comparing it, in one case, with another experimental technique, the m-lines spectroscopy, and, when possible, with existing data in literature.

II. Measurement techniques

1. Ellipsometry

Ellipsometry application to anisotropic media (Generalised Ellipsometry) requires the adoption of the 2×2 Jones matrix formalism. In this way it is possible to generalise the Standard

Ellipsometry parameters, the relative phase change, Δ , and the relative amplitude change, Ψ , suffered by incident light after transmission or reflection by a sample, to the case when a change occurs in light polarisation. The six GE parameters are linked to the Jones matrices of reflected (\mathbf{J}^r) or transmitted (\mathbf{J}^t) beam through the following equations:

$$\begin{aligned} \tan \Psi \cdot e^{i\Delta} &= \frac{J_{pp}}{J_{ss}} \\ \tan \Psi_{sp} \cdot e^{i\Delta_{sp}} &= \frac{J_{sp}}{J_{ss}} \\ \tan \Psi_{ps} \cdot e^{i\Delta_{ps}} &= \frac{J_{ps}}{J_{pp}} \end{aligned} \quad (1)$$

where $\mathbf{J} = \mathbf{J}^r$, \mathbf{J}^t and $\mathbf{J}^r = \begin{pmatrix} r_{pp} & r_{sp} \\ r_{ps} & r_{ss} \end{pmatrix}$, $\mathbf{J}^t = \begin{pmatrix} t_{pp} & t_{sp} \\ t_{ps} & t_{ss} \end{pmatrix}$ with r_{pp} , r_{ss} , r_{ps} , r_{sp} (t_{pp} , t_{ss} , t_{ps} , t_{sp})

representing the reflection (transmission) coefficients for p -, s -, and cross-polarisations, respectively. In the isotropic case of Standard Ellipsometry the Jones matrices become diagonal, Equations 1 reduce to the first one and only two parameters are meaningful. We remark that off-diagonal elements vanish also for anisotropic media in highly symmetric configuration, i.e. when the optical axis is oriented parallel or perpendicular to the plane of incidence [7].

However, the Jones matrix formalism is based on the assumption of a completely polarised light. When a significant amount of reflected or transmitted light becomes depolarised, it may be necessary to introduce the Mueller matrix representation. In this scheme, a 4×4 matrix connects the Stokes vectors representing the input and the output beam, as described in Equation 2:

$$\begin{bmatrix} S_0 \\ S_1 \\ S_2 \\ S_3 \end{bmatrix}_{OUT} = \begin{bmatrix} M_{11} & M_{12} & M_{13} & M_{14} \\ M_{21} & M_{22} & M_{23} & M_{24} \\ M_{31} & M_{32} & M_{33} & M_{34} \\ M_{41} & M_{42} & M_{43} & M_{44} \end{bmatrix} \cdot \begin{bmatrix} S_0 \\ S_1 \\ S_2 \\ S_3 \end{bmatrix}_{IN} \quad (2)$$

In our measurements, we used a Variable Angle Spectroscopic Ellipsometer (VASE[®]) from J. A. Woollam Company [8]. This instrument is equipped with a rotating analyser that incorporates an adjustable compensator before the sample. Through it all four Stokes parameters can be produced for the input beam; instead, due to the absence of a retarder after the sample, only the first three parameters of the output beam can be measured. Thus, our instrument provides access to the first three rows of MM elements, each one normalized to the M_{11} element.

Fortunately, for most materials it is not necessary to independently measure all 16 parameters, because of the symmetries in the Mueller matrix. Also in case of anisotropic, depolarising media, with non-zero off-diagonal elements, our eleven elements can give a complete description of the optical properties of the sample [9, 10].

In order to get a large amount of data, the GE as well as the MME parameters were measured as a function of both angle of incidence and wavelength, in the spectral range from 300 to 1700 nm; in one case we also performed measurements for different sample orientation. All data were simultaneously analysed using WVASE32[™] software to determine the sample refractive index and eventually thickness, by means of a multiple fitting procedure. This procedure ends providing a numerical value for the discrepancy between experimental and

generated data, called Mean Square Error (MSE), which we used to check the goodness of the fit. Matching the fit generated data with the experimental ones and obtaining reliable quantitative results generally requires a proper optical model for the studied sample, always consisting of a stack of different layers. Thus, the main possible drawbacks in SE, GE and MME analysis could be the dependence of final results on: 1) accuracy of the optical model, 2) starting points in fitting procedures, 3) statistical correlation between fit parameters.

2. *M-lines spectroscopy*

A very common technique for thin film characterisation in waveguide configuration, namely the m-lines spectroscopy, is based on different and very well known physical principles [11, 12]. We used it as an alternative technique in our experimental characterisations, not only for complementing ellipsometric data but also for cross-checking them, whenever possible. The m-lines typical experimental set up that we used is shown in Figure 1.

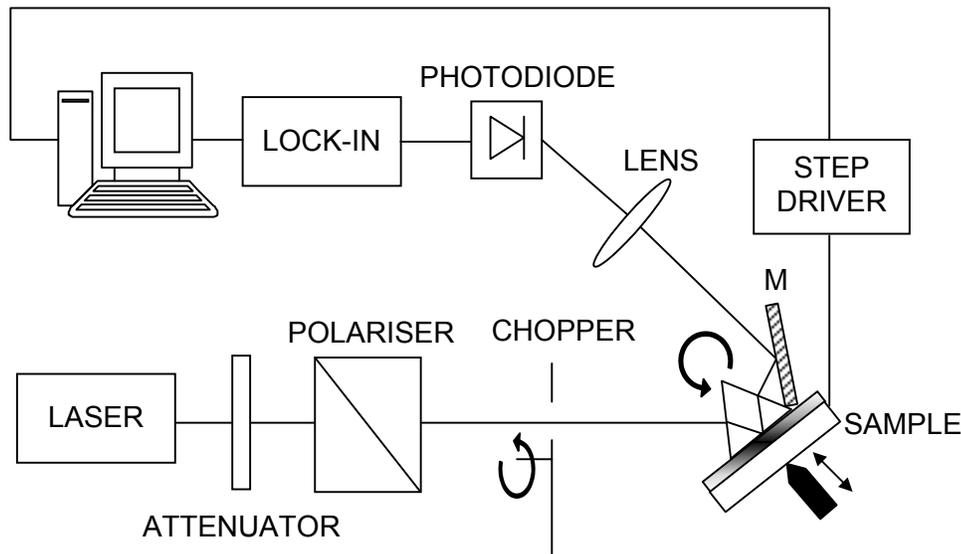


Figure 1. M-lines set-up.

A thin sample is pressed against the base of high index SF6 prism to make a thin air gap of about 0.1 μm . A laser beam, controlled in both power (by Attenuator) and polarisation (by Polariser), impinges on the sample through the prism and can be coupled to the guided modes of the film due to the “optical tunnelling” effect, if the angle of incidence fulfils the condition of mode exciting:

$$\sin \gamma = n_p \sin \left[\alpha - \arcsin \frac{\beta}{kn_p} \right] \quad (3)$$

where γ is the angle of incidence on the prism, n_p the refractive index of prism, α the angle between the base and the input face of the prism, β the effective index of a guided mode, k the beam wavenumber in vacuum. For making measurements at variable incidence angle, the sample is put on a motorized rotating stage together with the prism and the mirror M . This mounting allows the light beam, after reflection at prism base-film interface, to emerge from the output

face at a fixed direction. Thus, a simple detection scheme is enough, with a collecting lens and a fixed photodiode, avoiding the need for a rotating arm and/or re-positioning and re-calibration. The γ values, corresponding to excited modes, are measured in correspondence of the minima of the reflected power. Finally, the effective index of the guided modes is obtained and used to calculate the optical constants of the waveguide core and substrate materials by means of a simple analysis software. This set-up was automated with LabVIEW™ 6.1 software and provided an accuracy for γ measurements of $\pm 0.01^\circ$.

III. Results and discussion

1. Sample 1

The first sample investigated is a 12.4 μm thick layer of a nematic LC (5CB provided by Merck) sandwiched between 0.4 mm thick silica glasses. The LC film was homeotropically aligned by using a very thin layer of DMOAP surfactant¹. In this simple geometry, we could consider the off-diagonal elements of Jones matrices to be vanishing [7] and use SE technique in reflection. Due to the absence of any resonances in the examined spectral range, we can describe the ordinary and extraordinary indices of the nematic by using the simplest 2-parameter Cauchy formula (where the subscripts *o* and *e* stand for ordinary and extraordinary, respectively):

$$n_{o,e} = a_{o,e} + b_{o,e}\lambda^{-2} \quad (4)$$

Trials made with the three parameter Cauchy formula did not give any significant difference in the obtained dispersion curves; furthermore, the third fitted parameter was always quite strongly correlated with the previous ones, especially the second one, decreasing accordingly the degree of confidence in the fit.

Starting from our experimental data we tried out different fit procedures. In the first case (fit 1), the film thickness d was held fixed to the above-mentioned value, previously obtained by empty cell measurements. Starting guess values for the fit parameters, $a_{o,e}$ and $b_{o,e}$, were found by a special routine of WVASE32™ software, namely a grid-based value pre-selection. This feature resulted to be extremely useful, a too far initial guess often producing unreliable results.

Generated and experimental data, shown in Figure 2, are in good agreement apart a high frequency and small amplitude interference oscillation pattern, present in the generated curves that was never observed in actual measurements. These oscillations were finally recognised as an artefact of the simulation software, due to the glass plate expected interference. Lack of coherence of the lamp light does never allow this interference to be actually observed for a geometric path difference in the millimetre range (twice the glass plate thickness).

A second fit (fit 2) was made including also the film thickness d amongst the fitted parameters. We obtained a fitted value for the thickness $d = 12.5 \mu\text{m}$, in good agreement with the empty cell value, and slightly different values for the a and b Cauchy parameters (Table 1).

¹ The presence of the DMOAP film had a negligible influence on our sample measurements. Further measurements made just on a treated glass led to the determination of the DMOAP thickness corresponding to one molecular layer.

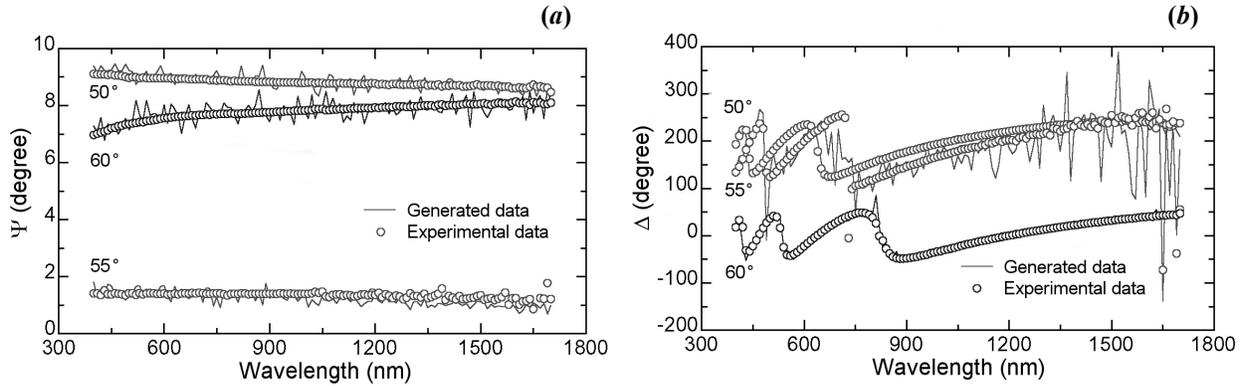


Figure 2a-b. Spectroscopic ellipsometric measurements for the 5CB liquid crystal sample: Ψ (a) and Δ (b) experimental data and respective generated curves (fit 1) for 50°, 55° and 60° incidence angles.

Our experimental curves for ordinary and extraordinary refractive indices and the ones calculated on the base of data provided by Karat and Madhusudana [13] are shown in Figure 3: a quite good agreement with the literature is clearly obtained by fit 2, especially in the visible region. The curve displacement in the NIR region is not surprising. In fact, using our spectroscopic ellipsometer we obtained experimental data from the visible up to the NIR and we paid attention to take equally spaced data points in the whole range. On the contrary, the Karat and Madhusudana reference curve is only an extrapolation, built from refractive index values measured at three wavelengths in the visible region, through a Cauchy dispersion model (Equation 4). Obviously, small errors in the data points can result in a quite large uncertainty at the extrapolated end of the spectrum.

In order to verify our assumptions in the choice of the ellipsometric technique (small depolarisation, zero off-diagonal elements) and at the same time to check the reliability of our fit results, we made an independent measurement of the birefringence Δn , by using transmission MME [10], also releasing the assumption of perfect homeotropic alignment of the LC film, and performing measurements at different sample orientation. Fit generated and experimental curves were in good accordance also in this case, and results were quite close to the previous ones (Table 1). The obtained dispersion curves for the birefringence are shown in Figure 4: fit 2 curve is almost coincident to the literature data, and very close to the one calculated from the MM transmission fit; fit 1 curve is again a little lower, with a relative displacement around 1÷2%.

	n_e		n_o		Δn	
	a	b (μm^2)	a	b (μm^2)	a	b (μm^2)
Fit 1	1.6325 ± 0.0005	0.0164 ± 0.0002	1.5011 ± 0.0002	0.0068 ± 0.0001	(0.1314 ± 0.0007)	(0.0096 ± 0.0003)
Fit 2	1.6452 ± 0.0003	0.0193 ± 0.0002	1.5124 ± 0.0002	0.0086 ± 0.0001	(0.1328 ± 0.0005)	(0.0107 ± 0.0003)
Fit MM	-	-	-	-	0.1348 ± 0.0002	0.01007 ± 0.00006
Karat and Madhusudana	1.637 ± 0.002	0.0222 ± 0.0006	1.50673 ± 0.00008	0.01090 ± 0.00003	(0.130 ± 0.002)	(0.0113 ± 0.0006)

Table 1. Comparison among 5CB dispersion Cauchy parameters obtained from different ellipsometric measurements and fit procedures and published data [13]. Parentheses in Δn values indicate that were obtained by difference of the respective extraordinary and ordinary indices.

This result confirms that liquid crystal samples, provided they are in the simple homeotropic geometry, can be optically characterised also by the fastest SE, with no significant loss of accuracy. This is a good example of cross-checking the goodness of a fit by means of independent measurements.

Finally, we want to recall that refractive indices, hence also the birefringence, in nematic materials are very sensitive to the temperature, the more the closer the clearing point, T_c , is. Furthermore, next to T_c , the more meaningful dependence of the optical constants is on the physical parameter $\Delta T = T - T_c$ rather than on the absolute temperature T . This should be clear especially considering that a small impurity content in the sample can induce relatively large variations of T_c . Thus, we paid attention in Figures 3-4 to compare data in reference [13] obtained at the same ΔT , actually 5 °K, at which we performed our measurements. However, our samples were not controlled in temperature better than the whole clean room was, that is up to $\pm 1^\circ\text{K}$.

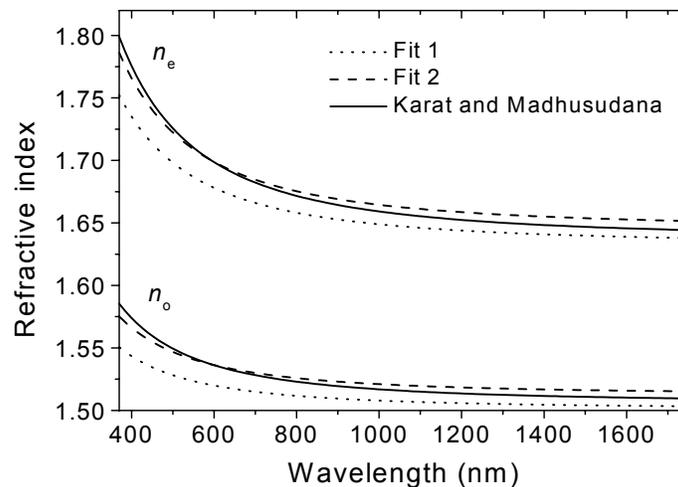


Figure 3. Ordinary and extraordinary refractive index dispersion for 5CB liquid crystal: the curves refer to two fitting procedures and one literature data set.

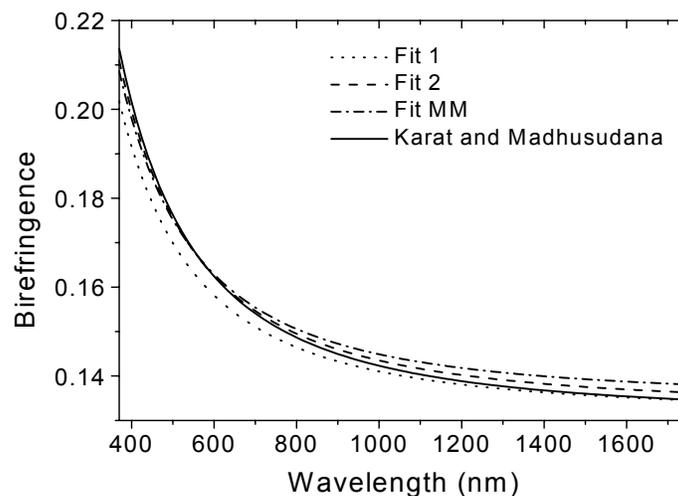


Figure 4. Birefringence dispersion for 5CB LC: the curves refer to three fitting procedures and one literature data set.

2. Sample 2

The second characterised sample was a polymeric waveguide made by a layer of Poly(4-Chloro Styrene co-Glycidyl Methachrylate), known as CS-GMA, with a 75/25 w/w ratio of the two polymeric components, spin-coated on a fused silica substrate² In the ellipsometric analysis, we used the isotropic model and fitted the two Cauchy parameters a and b and the thickness d of the waveguide polymeric core. Measurements by the m-lines technique were carried out at six wavelengths: 514, 532, 633, 671, 1282 and 1550 nm and approximated by the 2-parameter Cauchy formula. The same value for the thickness, $d = 13 \pm 1 \mu\text{m}$, was obtained by the two methods. The dispersion curves obtained from ellipsometry and m-lines measurements and corresponding fits are presented in Figure 5. The two curves are clearly different, outside the errors, but obviously show a similar behaviour. We have a possible reason for the origin of this discrepancy both in the thickness non-uniformity of the sample and in the different spot size of the light beam used in the two techniques. In fact, the large error in the measured thickness was only due to the sample non-uniformity, both techniques being inherently much more precise than that. On the other hand, the waist of the beam used in our ellipsometric measurements was of the order of 3÷5 mm, while in the m-lines set-up it was of the order of 0.1÷0.2 mm. Thus, ellipsometry explicitly “sees” the sample non-uniformity that may lead to significant light depolarisation, hence to a large uncertainty in the final result [10]; on the contrary, the m-lines technique gives results related to the actual thickness of the sample in the illuminated region. As a consequence, we believe that, in this case, m-lines results are more reliable than ellipsometric ones, even though they are much more time consuming and limited by the use of a small number of single wavelength light sources.

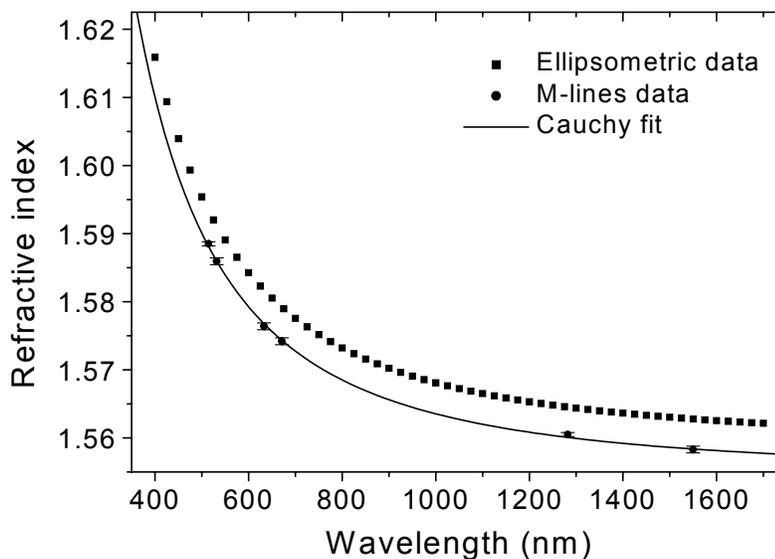


Figure 5. Results of ellipsometry and m-lines optical characterisation of CS-GMA polymer planar waveguide.

² This material was first synthesized by Prof. Hult's group at KTH, Stockholm, during a joint project with our group in Naples [2].

3. Sample 3

We also have characterized a planar waveguide manufactured by the company TeraHertz Photonics (UK). This waveguide is a multilayer stack, formed by a glass substrate, a 20 nm thick ITO film, a 6 μm thick polymeric buffer, and on top a 3 μm polymeric core.

First, we applied the m-lines technique and we were able, as expected, to get a complete information about thickness ($\pm 0.1 \mu\text{m}$) and refractive index values (± 0.0002). As an example, we show in Figure 6 the angular spectrum of the TE modes for red light.

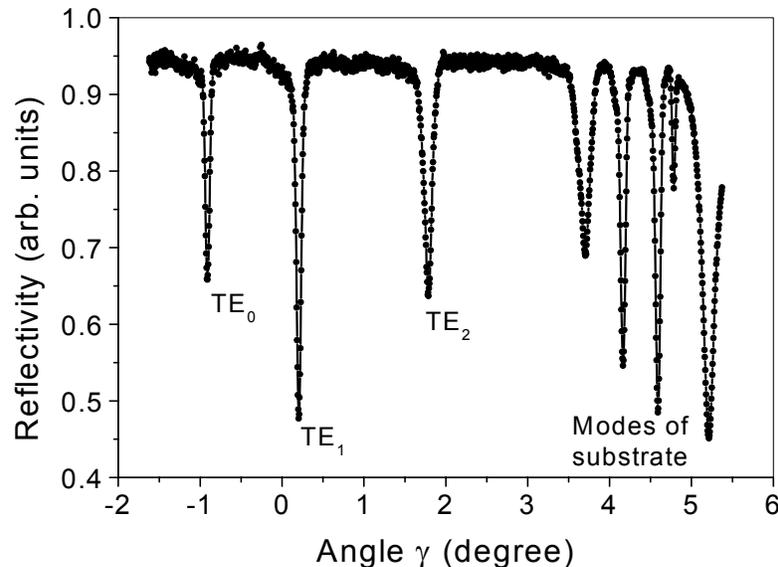


Figure 6. Angular spectrum of TE guided modes obtained by m-lines method for the TeraHertz sample ($\lambda = 633 \text{ nm}$)

We also tried to characterise the same waveguide by spectroscopic ellipsometry, but we failed to get a reasonable fit of the experimental data. We believe that this difficulty is due to the complex structure of the ITO layer, which is absorbing and non homogeneous, as we will discuss in the following. This problem appeared only in the ellipsometric analysis and not in the m-lines measurements, because in the latter technique the presence of the thick buffer totally prevents the evanescent tail of the guided wave to propagate through the ITO film.

For its wide use in opto-electronic devices, ITO film characterisation is presently of great interest and a number of ellipsometric investigations have been performed on it [14, 15]. Due to the described failure, to get a deep insight in this problem we have decided to analyse a simpler stack, namely a commercial ITO coated glass, provided by Merck..

4. Sample 4

Our sample was made by a 100 nm thick ITO film coated on a BK7 glass substrate; like for every standard ITO coated glasses, also in our case a thin oxide film was interposed in order to prevent ion migration.

We made a spectroscopic ellipsometric analysis and fitted the experimental data using three different optical models, based on two absorbing Lorentz oscillators. In the first, simplest

approach (fit 1), we assumed homogeneity for the ITO film and we could not obtain a good fit. This was not surprising, because, as already shown in reference [14], there are strong indications that ITO mass density, microstructure and film continuity, degree of oxidation, and hence also its optical properties, are generally varying along the stratification direction. Then, our second approach (fit 2) was to use the Bruggeman Effective Medium Approximation (EMA) model [16]. We don't want to go into details for this model that, in few words, assumes a linear variation of the optical parameters with the film depth. In this way, we obtained two different spectral curves for each of the optical parameters to be measured, namely the refractive index and the extinction coefficient. The two curves are shown in Figure 7a, one representing the values at the air-ITO interface (top) and the other the ones at the ITO-SiO₂ interface (bottom). Although much improved with respect to the first approach, the obtained fit was not yet satisfactory, in terms of final obtained MSE. Thus, we finally considered a third approach (fit 3) in which the ITO layer was divided in two sub-layers, in each of which we applied an EMA model. The final fit was very good; the whole analysed stack was: 1. BK7 glass (1 mm, not fitted), SiO₂ (25.7 nm, fitted), ITO (95.9 nm, fitted) and a layer of surface roughness (5.2 nm, fitted); the results for the ITO layer are shown in Figures 7b and 8.

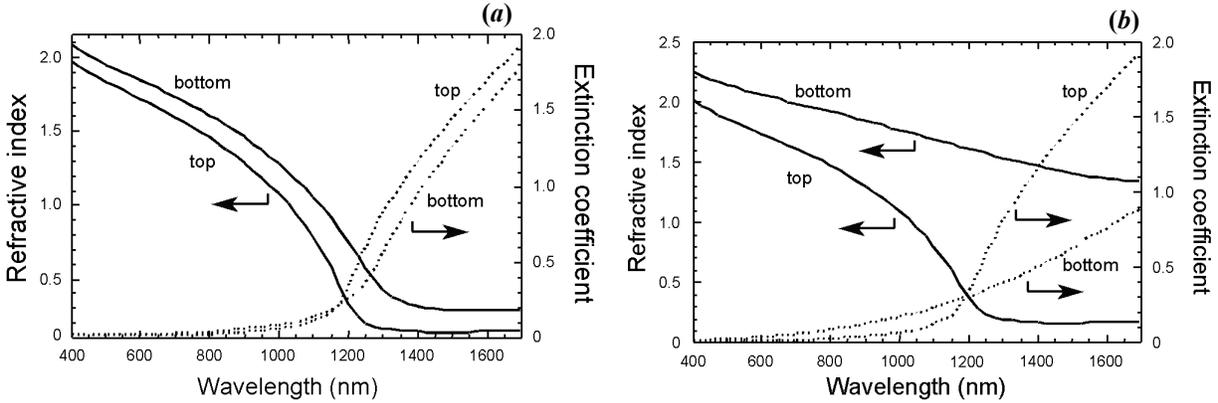


Figure 7a-b. ITO layer refractive index and extinction coefficient versus wavelength for fit 2 (a) and fit 3 (b).

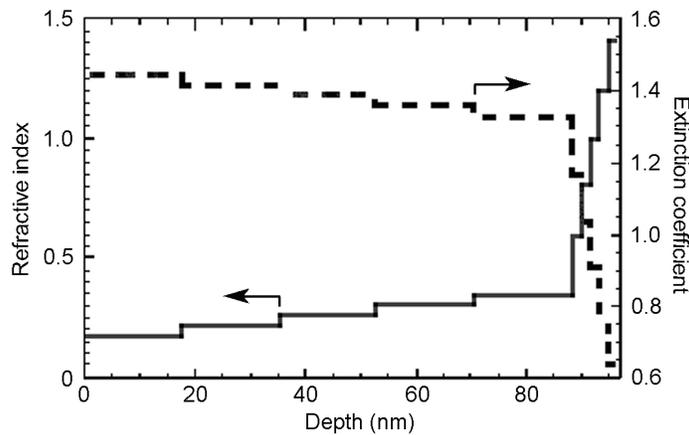


Figure 8. Optical constants profile for the ITO layer at $\lambda=1500$ nm (fit 3). Depth=0 corresponds to air-ITO interface.

Figure 7b is the analogous of Figure 7a, for this last approach, while in Figure 8 we show the behaviour of the refractive index and the extinction coefficient as a function of the film depth, at a given wavelength (1500 nm): it is evident from the figure that the first sub-layer is much larger than the second one. From these results, we can draw some interesting conclusions about the ITO film structure and optical properties. We confirm the results and observations in [14] about the influence of the ITO microstructure on the conductivity and hence the extinction coefficient. We highlight that the steepest parameter variation happens in a thin layer close to the substrate, whose interaction with the ITO film during sputtering and post-processing (e.g. annealing) is likely the major responsible for the observed non-homogeneity, also in the optical properties. Differences and also apparent contradictions between results reported in [15] and present work (and [14]) should be ascribed to the different preparation and processing techniques for the ITO films, mainly resulting in a different absorption behaviour at NIR wavelengths. However, also retaining the simplifying assumption of film homogeneity in [15], even in presence of opposite indications, could have played a role. Then, in our opinion, a strong warning about the overall process of fabrication and characterisation of ITO films should be put to all the end-users of this widely exploited material, when using it in photonic devices, especially in the NIR region.

IV. Conclusions

The problem of optical characterisation of organic thin film, polymers and liquid crystals, from visible to the NIR range has been afforded using spectroscopic ellipsometry and complementing it with the m-lines technique. We have presented the obtained results for a small number of representative samples, including the widely used 5CB liquid crystal, two low-losses optical polymers, one of which was especially developed by us for use as a passive waveguide material, a commercial made ITO film. We have discussed the encountered problems and possible ways to afford them, especially when complex samples, say multi-layered, strongly anisotropic or non-homogeneous, are dealt with. We want to underline that spectroscopic ellipsometry has proved to be a very powerful, sensitive, and fast technique also for this kind of samples and will be more and more employed for the optical characterisation, also at telecom wavelengths, of photonic materials, including polymeric composites and nano-structured materials. However, a correct use of ellipsometric results demands a great care in establishing the best appropriate optical model for the analysed sample, together with a proper consideration of the hardware and software tools exploited. At the end of our analysis, we believe that complementing ellipsometry with guided-wave techniques, like the m-lines (or also the half-leaky wave, best suited for multi-layered materials), whenever possible, could be of a great help for the above mentioned task. These techniques can be even more accurate than ellipsometry but the relative set-ups have generally the drawback of being based on single wavelength light sources. Thus, they are much more time consuming but mainly they cannot provide reliable results when the material under analysis has a resonance close to the wavelength of interest.

Finally, we want to stress the importance of the above warnings about results utilisation, recalling our short discussion in the previous section on sample 4, the ITO film. Different investigations made on different samples of the “same” material with the “same” technique lead to results that can be either similar or quite different, according to the sample preparation procedure, the actual set-up of the hardware, and the way the software analysis is performed.

V. Acknowledgements

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