New developments in Spectroscopic Ellipsometry for Nano Sciences

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ABSTRACT

Spectroscopic Ellipsometry (SE) is an optical technique, non destructive, to characterize the complex reflectivity of surfaces and layers from the deep UV (190nm) to the mid InfraRed (20µ). SE does not need any reference surface like interferometry nor reference materials or reference beam like in absorption spectroscopy (IRRAS): this is an absolute technique often called first principal technique.
A new optical configuration to avoid the beam reflected from the back face of the sample has been developed particularly for Infra Red application, the patented idea will be disclosed.
The layer thicknesses become thinner in the nano-sciences and in the case of surface preparation.
Spectroscopic Ellipsometry is the technique of choice to characterize not only the thickness with a fraction of angstrom sensitivity and the roughness but also the molecular composition of layers at the nano-scale or single mono molecular.
A new application of SE is in imaging of coated planar surfaces. The weakness of the technique is in its spatial resolution (several µ) particularly because of the incidence angle and limited divergence.
The kinetics can be measured for the full spectrum in real time with resolution of 0.1sec in the case of In-situ real time deposition or growth.
Several examples of applications will be given using different wavelengths and different configurations of SE.

Keyword : Spectroscopic Ellipsometry, Infra-Red Ellipsometry, Imaging Ellipsometry.

1 INTRODUCTION

Spectroscopic Ellipsometry has long been recognized as a powerful technique to characterize thin films and complex multilayers. The first spectroscopic ellipsometer was commercially available in 1983 [1] .

In the present paper, we use the last generation of SOPRA ellipsometers to show the potentialities of this technique for the characterization of materials and structures [2], [3].

The first application presented in this paper is the characterization of an OLED structure, which is composed of a multiplayer stack. Multi-angle ellipsometry measurements are used to extract accurately optical indices and thicknesses.

Infrared ellipsometry allows molecular bound detection but also resistivity measurements of metallic films [4]. Complete structure can be control with production oriented instrument.

Spectroscopic Ellipsometry Imager (SEI) is a also a very powerful approach to perform measurments with a good lateral resolution down to 10 µm on the sample. One example taken obtained on a pattern wafer will be presented.

2 OLED CHARACTERISATION

Organic light-emitting diodes (OLEDs) based on conjugated polymers have attracted much attention during the last decade, due to their possible application in large area flat panel display A schematic OLED structure is reported in Figure 1. Each of these layers can be characterized in term of thickness, refractive index and extinction coefficient by ellipsometry technique.

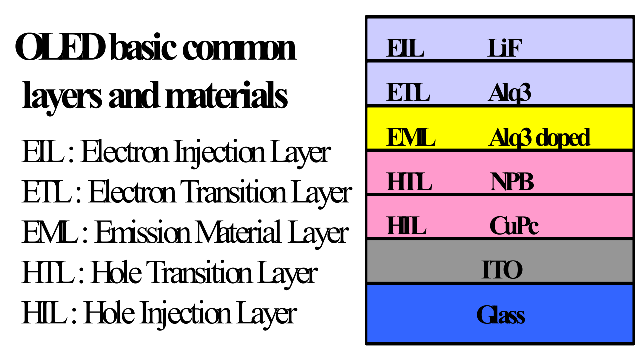

Figure 1 : Schematic diagram an standard OLED structure

2.1 Characterization of ITO layers

The bottom ITO layer is used as conductive and transparent electrode for the OLED structure. For this layer,

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infrared spectroscopic ellipsometry is the technique of choice to measure at the same time the layer thickness and its conductivity in a non-contact, non-destructive way. Indeed, the Drude law can be used to describe the free-carrier absorption effects that can be detected on the absorption coefficient in the infrared region. The free carrier concentration can be deduced [5]. In Figure 2 we have reported the experimental and simulated ellipsometric curves obtained on an ITO electrode layer on glass. The adjustment is made at the same time on the layer thickness and the optical index of the ITO layer using a Drude model. In this way the refractive index and the absorption coefficient are deduced independently.

In Figure 3, the extinction coefficients measured on two ITO layers with and without thermal annealing are reported. The annealing effect at 450°C is easily detected. The different physical parameters of the ITO layers can be easily deduced as reported in Table I.

Sample	Thickness (nm)	Conductivity (S/m)	Resistivity (mΩ/cm)
As deposited	162.2	1.67E+5	0.60
Annealed 450°C	169.1	3.96E+5	0.25

Table I: Physical parameters deduced from Fig 3

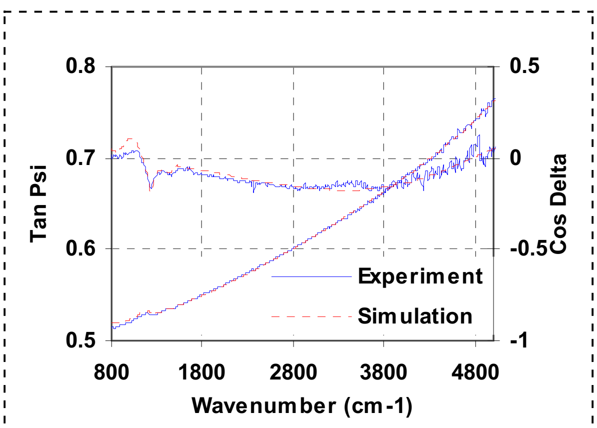


Figure 2 : Measurement of a ITO layer on glass in the infrared range. The layer thickness is 162nm

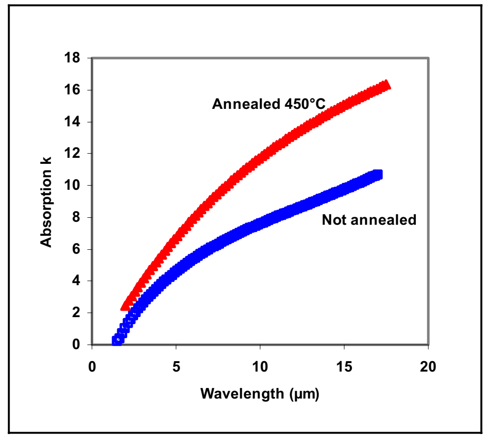


Figure 3 : Measured extinction coefficient of two ITO layer with and without thermal annealing

2.2 Characterization of CuPc layers

Absorption bands in complex organic materials, like CuPc need to be carefully characterized in the visible range to predict the emission properties of the display. In this respect, variable angle spectroscopic ellipsometry is the technique of choice to measure independently the refractive index and the absorption coefficient of these films. In figure 4 some experimental curves obtained on a hole injection layer (CuPc on glass) are reported with the simulations. Three incidence angles have been used at 65, 70 and 75°. From these data, the optical indices of the CuPc film and its thickness are deduced for each wavelength independently has shown in Fig. 5, and the accuracy of this extraction can be estimated (generally lower than ±0.02 on n and k).

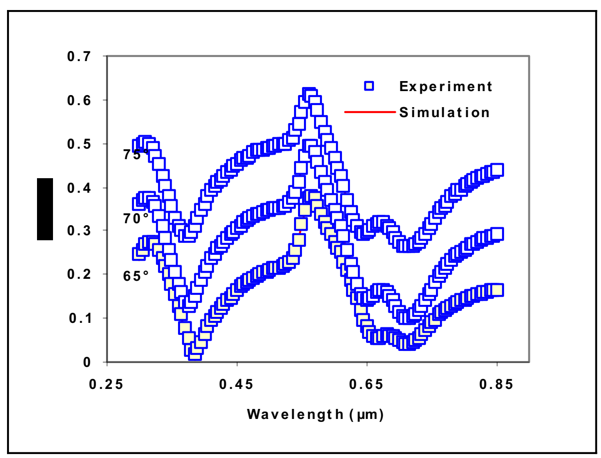


Figure 4 : Variable angle SE measurement on CuPc/Glass sample

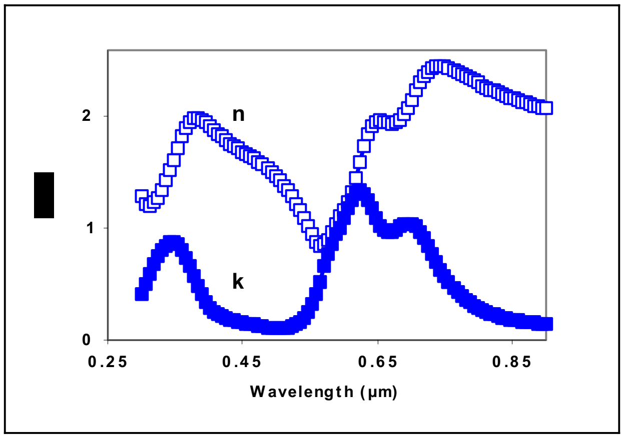


Figure 5 : Optical indices of the CuPc layer of Figure 4.

When all the materials have been carefully characterized, SE can control the complete OLED structure and the thickness of the different layer can be deduced independently. The same equipment and analysis method can be used to characterize polymer films.

3 Low K dielectric materials

Reduction of the CD dimensions in the IC's induces an increase of the circuit density and consequently of the RC parameters between isolated contacts. To reduce this effect, SiO_2 is replaced by new low K dielectric materials.. Si-O-C-H films deposited by plasma enhanced chemical vapor deposition (PECVD) have shown the formation of nano-sized voids due to Si-CH_3 and OH related bonds included in the film [6]. These features are controlling the dielectric properties of the films and must then be measured accurately.

Infrared ellipsometry controls thickness and chemical properties of thin films at the same time. The properties of the substrates, roughness and interfaces are taken into account in the analysis model. As shown in Figure 7, different absorption peaks can be detected in the infrared region in addition to the O-Si-O bounds classically detected on silica. A well defined peak around 1270cm^{-1} associated to Si-CH_3 bounds is also measured and two other peaks at 2350cm^{-1} and 3000cm-1 associated to the water absorbed by the films. We have used these measurements to extract the optical indices of different samples as shown in Figure 9. The relative concentration of carbon incorporated in the film can be calculated normalizing to the peak area of the O-Si-O stretch using:

$$Relative\_carbon\_concentration(\%) = \frac{A_c}{A_o + A_c}$$

where A_o and A_c are the peak areas of the Si-O stretching vibration mode at 1040cm^{-1} and the Si-CH_3 vibration mode at 1270cm^{-1} respectively. A summary of the results is reported in Table II.

These characterizations are obtained on the SOPRA IRSE 3000 which is an automatic spectroscopic Ellipsometer covers the mid Infrared range from 600 cm-1 to 7000 cm-1 (1.4 µm to 16.6 µm).

Main hardware components are reported on the following schematic (Fig. 6.):

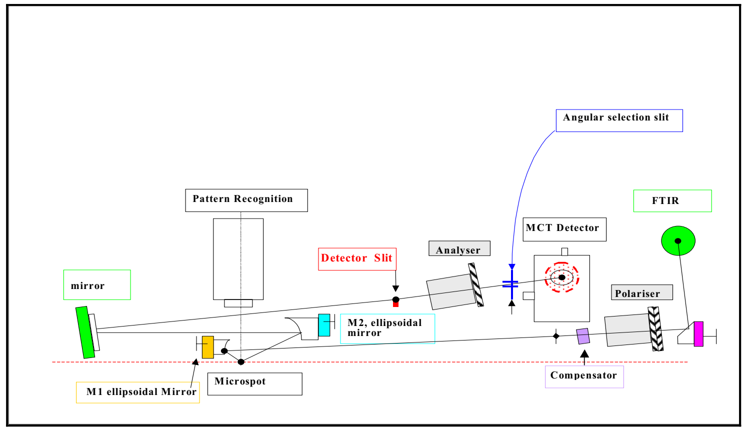


Fig 6: Mounting Schematic

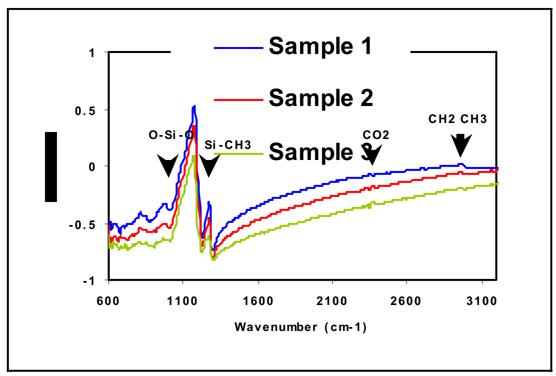


Fig 7: SE measurements on three Si-O-H-C low K dielectric with variable C content. Different absorption bounds are easily detected.

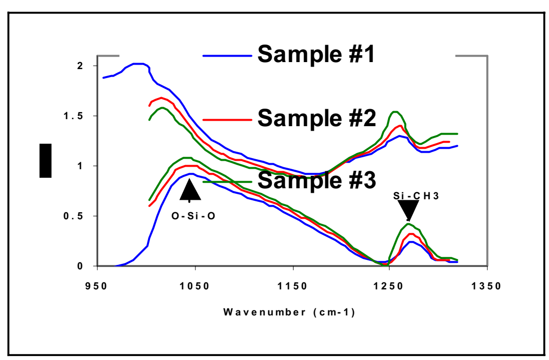


Fig 8: Measured optical indices of the Si-O-C-H films of Fig 7. The thickness of the films is between 300 and 500nm

	A_0	A_c	Carbon (%)
Sample 1	119.4	8.3	6.5
Sample 2	132.5	10.4	7.3
Sample 3	150.7	15.6	9.4

Table II: Carbon content of the Si-O-C-H low K dielectrics of Fig.7 & 8

4 IMAGING ELLIPSOMETRY

4.1 Introduction

In conventional ellipsometers the light beam is typically a few mm in diameter but can be reduced down to 20µm using a microspot by focusing the measuring beam [7]. This kind of system is used for in line analysis in semiconductor production. The time to obtain an image of the surface of the sample is nevertheless too long to study rapidly varying phenomena. It is the reason why a Spectroscopic Ellipsometer Imager (SEI) was developed. With this instrument we measure a full image of the surface of the samples with a spatial resolution of 10µm, or better, at a rate of a few wavelengths per image. The total image covers an area of 0.6x2mm with 480x780 pixels.

4.2 Experimental results

The spatial resolution and anamorphous effect of our new instrument have been evaluated by imaging a 200x200µm target (Fig 9). The raw gray scale image obtained at 577nm and 45° of polariser angle of this target shows an important (but expected) anamorphous effect. Taking into account the angle of incidence of the imaging measurement we can expect a reduction of Y scale of a factor of 3. An intensity profile along the line A of Figure 9.a shows the spatial resolution obtained with our instrument (cf. Figure 9.b). It is found around 10µm with some diffraction effects detectable on sharp boundaries. The edges of the patterns diffract the light, which cannot be collected in the 2° limited aperture angle of collection. In addition, due to the diffraction limit of 15µm, the pixels at edges of image are not correctly illuminated.

On Figure 10 we have reported the first SEI observation on a patterned SiO2/Si sample. As shown in the figure, each pixel contains a complete spectroscopic spectrum of the tanψ and cosΔ parameter. Zones with large SiO2 thickness around 105nm can be easily detected (red areas of tan ψ parameter at 436nm for example). Analysis treatment of such a set of images can be made as follows: First, different kinds of areas can be detected using pattern recognition or colour filtering on one well contrasted image. Then one structural model is attributed to each type of area. Standard regressions using the same model can then be applied to all the pixels of one area assuming that the structural parameters are continuous inside the area. Images of the different structural parameters can finally be deduced with the appropriate models. This technique or fuzzy logic or neural network can be applied for determination of complex multilayer structures corresponding to various different zones and models. Multilayer model can be applied and at the difference of single wavelength ellipsometer, no shadow zone nor cycle problem occur.

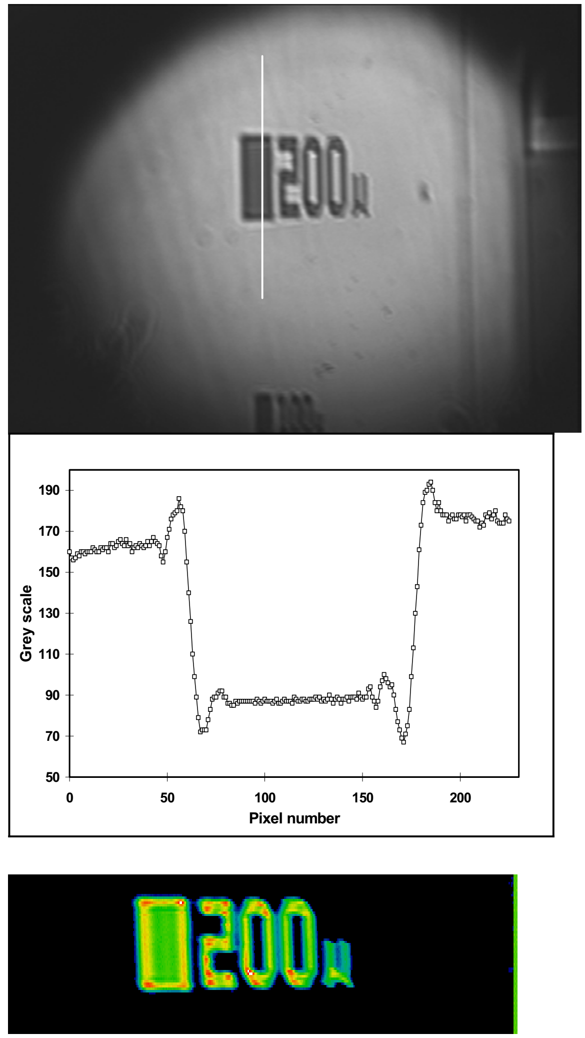
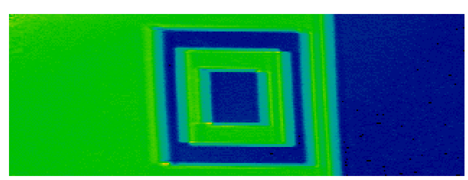


Fig 9: Image of 200 µm squared box on silicon substrate at λ = 577 nm



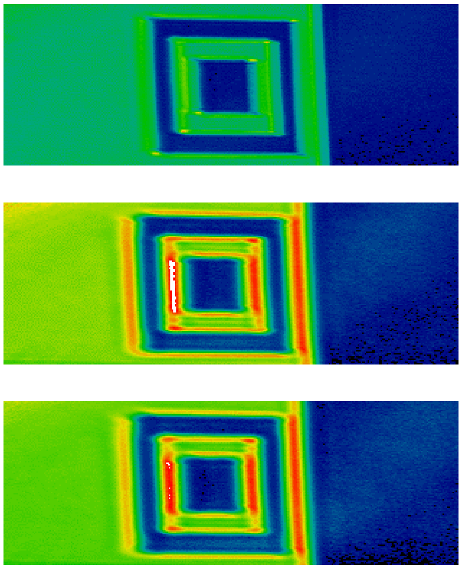


Fig 10: Cos Delta values obtained for 4 differents wavelengths

5 CONCLUSION

These different applications show clearly that spectroscopic ellipsometry is a powerful technique to characterize extremely thin layers in term of thickness but also indices. The possiblility to perform measurements on a very broad spectral range from 137 up to 20 µm is also extremely powerful, depending of the application. In mid Infra Red range, molecules signatures will be seen and if the material is conducting, using some appropriate model like Drude model, one can extract electrical parameters like mobility and conductivity of the layers.

Imaging ellipsometry is a very promising technique to overpass the limit of lateral resolution to reach 10 µm resolution and in a next future up to 1 µm. We can see applications of this technique not only in microelectronic but also in biotechnology field.

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