Keywords: Thin films, thickness and refractive index characterization
Methods: Spectral reflectometry
Application: Multilayer thin film thickness and refractive index analysis

Spectral reflectometry technique for measuring bilayers system optical parameters

Prepared by: Edgars Nitiss

Spectral reflectometry is one of the most widely used techniques in science and industry for measuring the optical parameters such as thickness and refractive index of thin films. By employing a simple setup for collecting thin film reflected light spectra it is possible to measure thin films with optical thickness of micron order. As will be shown in the application note, spectral reflectometry can be used for measuring thickness of a system that consists of two layers.

Background

Thin layers are applied more and more frequently as optical components in novel optoelectronic devices either as slab waveguides or coatings etc. In most cases it is very important to estimate the linear optical parameters and thicknesses of the layers. These parameters are determined experimentally. Commonly optical noncontact methods are preferred since they avoid mechanical interaction with the layer. Here we will describe a simple and fast method for measuring bilayer system optical parameters in the low absorbance part of the sample transmittance or reflectance spectrum. In this approach the thicknesses and refractive indexes are determined by separating the interference fringes caused by each layer. Thus a mandatory requirement in order to have at least one exact solution of problem is that the layer thicknesses must be comparable to light wavelength to have more than one interference fringe in the spectrum. The fringes are then separated by applying Savitzky – Golay (SG) smoothing filters with two different orders of polynomial fits and window lengths. Eventually the wavelength values which correspond to the extreme points in the spectrum are obtained. For finding the solution of thicknesses and refractive indexes these values are then approximated using standard deviation of thicknesses as the penalty parameter.

Experimental conditions

For the testing of the method a single layer and bilayer samples were made. For single layer thin film a polymer sample, polymer was spin-coated from a chloroform solution onto a high refractive index ITO covered glass slide (SPI Supplies, ITO thickness 15 – 30 nm). This polymer films were coated at different thicknesses by changing the concentration of the chloroform solution. Bilayer system was obtained by covering an ITO glass slide carrying the polymer film with another bare ITO glass slide. Afterwards both slides are squeezed together in the sample holder. On the micron scale the surface of the polymer sample, especially at slide edges, is rather rough causing the formation of an air gap between the surface of the polymer and second ITO layer thus a bilayer system in the micron scale is created. For the spectral reflectometry measurements we used a simple experimental configuration. The light from a light source (*OceanOptics* Mini-D2-GS) is transmitted through a reflection probe (*OceanOptics* QR200-7-VIS-NI) to the sample. The reflected light form the sample is then collected and transmitted to the spectrometer (*OceanOptics* HR4000CG-UV-NIR).

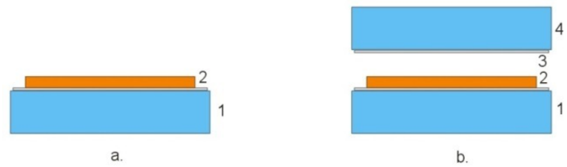

Fig. 1. Sample geometry a) single layer thin film sample: 1- ITO coated glass, 2 - polymer. b) bilayer sample: 1, 4 - ITO coated glass, 2 - polymer, 3 - air gap.

Results

From a bilayer sample such as shown in Fig. 1 the collected light intensity spectral dependence or the raw data are shown as “Raw data” in Fig. 2. It is clearly evident that the spectral intensity is governed by interference effects in two layers with different optical path lengths. In the reflected light spectrum the wavelength separation of fringes caused by the thicker layer should be smaller and for the thinner ones larger. Thus it is assumed that the long period fringes are caused by interference in the thin film, but the short period ones caused by the interference in the air layer. The optical parameters for each of the layers one can obtain by separation of the interference fringes. To have interference fringes that can clearly be separated by SG filtering the layers must have optical path lengths that differ at least a couple of times.

For the collected raw data at first we apply SG filter with a 701 points wide window length to extract the interference fringe corresponding multiple internal reflections in the thin film. For the sake of clarity SG filter with such parameters will be referred as 1st SG filter. From the filtered data three extreme points are found. Using these points, layer thickness and refractive indexes at three light frequencies are calculated applying the point by point dispersion relation. The calculated thickness of the thin film is 0.73±0.01 μm using the point by point method.

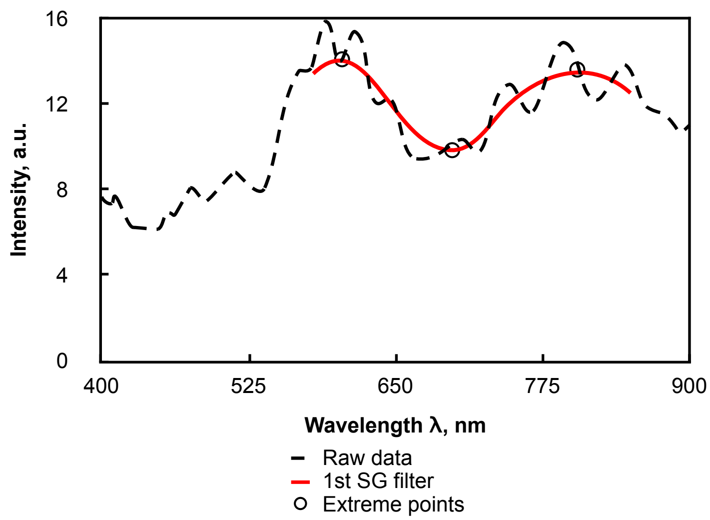

Fig. 2. The collected light intensity spectral dependence and smoothed data with 1st SG filter

If the SG filtered values are subtracted from the raw data then the shorter period interference fringes can be separated. We will call these values the "Filtered raw data". The shorter period interference fringes or the filtered raw data in the spectral range from 710 to 870 nm are shown in Fig. 3. After application of SG filter of window length 101 points (2nd SG filter) the wavelengths which correspond to the extreme points in the reflected spectra can be found. From these points the optical parameters of the air layer can be calculated giving layer thickness of 5.55±0.29 µm when using point by point method.

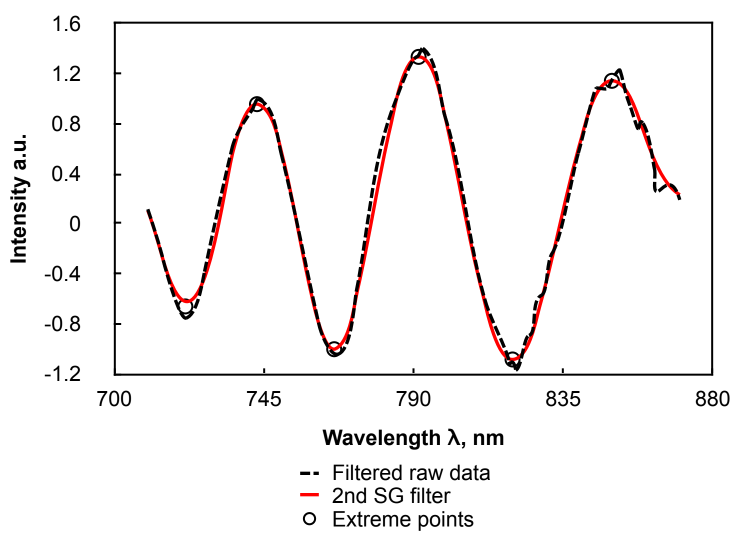

Fig. 3. The filtered raw data and smoothed values with 2nd SG filter

Conclusions

In this application note it has been shown that the spectral reflectometry is simple to use and overall a precise tool for estimating the thicknesses of a two layer system. However, tTo have interference fringes that can clearly be separated by SG filtering the layers, the optical path lengths should differ at least a couple of times.

References

1. E. Nitiss, R. Usans, M. Rutkis, Simple method for measuring bilayer system optical parameters, *SPIE Proceedings,* 8430, 84301C (2012), DOI: 10.1117/12.922317