Broadband Interferometry - a non-contact optical method for measuring the thickness of transparent thin films and coatings

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Abstract

An introduction to thin film interference and how this can be used to measure the thickness of thin films. The importance of refractive index to determine physical layer thickness. What degree of accuracy is possible, and what are the main determinants?

What happens if there is more than one layer? Description of a practical off-line instrument. What materials can be measured, and how many layers? What materials can’t be measured?

Adapting the instrument for in-line use. Why there are performance issues due to line speed, and how these will be overcome. Use of the system in explosive atmospheres.

Introduction

“Fringe counting” has long been employed to estimate small changes in film thickness. Recent advances in modern optics and electronics have made it possible to automate this and produce a thin film measurement system which is fast, accurate and easy to use. Furthermore by using broad-spectrum (or “white” light) illumination, it is often possible to measure the thickness of several layers of transparent material simultaneously. This paper briefly outlines the theory behind this technique, assesses its accuracy and discusses its areas of application. Finally it addresses the instrument’s suitability as an in-line tool, why it can be used in high solvent concentration (explosive) situations and explains why the line speed is limited today but will shortly be increased.

Thin Film Interference

When coherent monochromatic light is shone onto a transparent thin film suspended in air, it is reflected off both the upper surface (R1) and the lower surface (R2). Since light path R2 is longer than R1 there is a phase shift $\Delta \delta$ between the two paths, determined by:  

$$\Delta \delta = n \cdot (2d \cos \phi)$$

where $n$ is the refractive index of the film, $d$ is its physical thickness and $\phi$ is the angle to the normal of the light path inside the film.
If the path difference between R1 and R2 is a whole number of wavelengths, one might expect to observe an intensity maximum. However when R1 is reflected off the upper surface of the film, it undergoes a phase shift of $\pi$, whereas R2 is internally reflected off the lower surface and suffers no phase shift. This means that an observer will see an interference pattern with a series of intensity minima described by:

$$2n d \cos \phi = m \lambda$$

And intensity maxima described by

$$2n d \cos \phi = (m + 1/2) \lambda$$

where $\lambda$ is the wavelength of the monochromatic light, and m is the number of fringes observed. In practice one can use this phenomenon to estimate changes in the thickness of a thin film by using an extended source, to give fringes of equal inclination. These will be observed as a series of concentric circular rings (often called Michelson Rings) centered on the point where the normal from the surface of the film meets the eye or objective lens. As the film is moved, changes in its thickness will cause the fringes to move, either by emanating from the center spot, or by disappearing into it. By counting the number of fringes which appear or disappear in this way, $\Delta m$, it is possible to estimate the change in thickness of the film, from:

$$2n \Delta d \cos \phi = \Delta m \lambda$$

But $\cos \phi = 1$ at the normal, therefore change in thickness,

$$\Delta d = \Delta m \lambda/2n$$

There are many traditional interferometers using variations of this technique (Michelson, Fabry-Perot, etc), but all are relatively slow and cumbersome. The use of modern optics and electronics speeds up this technique and makes it much easier to use, by using a broadband source (usually in the visible and near infra-red part of the spectrum) coupled with a miniature spectrometer and a computer for converting the optical results into thickness data.

**Broadband Thin Film Interference**

Returning to Fig 1, if the incident light is actually a broadband or white-light source, then the path difference between R1 and R2, expressed in multiples of $\lambda$, will change depending on the wavelength of light, and this gives
rise to the multi-coloured or “rainbow” pattern of interference fringes, similar to the effect seen when a rainwater puddle has a drop of oil on it.

If this interference pattern is detected using a spectrometer, it makes sense to represent it as a plot of reflectance (signal intensity) versus wavelength. The following figure shows an example of the interference spectra of two films, whose optical thickness are 1µm (micrometer, or micron) and 2µm:

![Fig 2 Interference Spectra of 1µm and 2µm layers (optical thickness)](image)

These interference spectra can be described by the following simplified formula:

\[
I(\lambda) = A + B \cdot \cos\left(2\pi \cdot \frac{\Delta r}{\lambda} + \Delta\delta\right)
\]

where:
- \(I(\lambda)\) is the reflectance intensity of interference spectrum
- \(A\) is the factor representing the intensity of the two light rays
- \(B\) is the amplitude of the cosine function
- \(\lambda\) is the wavelength
- \(\Delta r\) is the optical path difference
- \(\Delta\delta\) is the phase shift of the two light rays.

Now, the maxima of these two interference spectra correspond to the condition where

\[
\cos\left(2\pi \cdot \frac{\Delta r}{\lambda} + \Delta\delta\right) = 1
\]

which means that

\[
[2\pi \cdot \Delta r / \lambda + \Delta\delta] = m \cdot 2\pi
\]

where \(m\) is an integer.

Now, take two different wavelengths at which the function has a maximum value, and calculate the difference between them, which can be represented as:

\[
[2\pi \cdot \Delta r / \lambda_2 + \Delta\delta] - [2\pi \cdot \Delta r / \lambda_1 + \Delta\delta] = 2\pi[m_2 - m_1]
\]

or

\[
\Delta r \left(\frac{1}{\lambda_2} - \frac{1}{\lambda_1}\right) = m_2 - m_1
\]

The optical path difference \(\Delta r\) (also called optical film thickness) is the product of the physical film thickness \(d\) and the refractive index \(n\), for light which is normal to the film:

\[
\Delta r = 2n \cdot d
\]

Therefore

\[
d = \frac{|m_2 - m_1|}{2n \cdot \left(\frac{1}{\lambda_2} - \frac{1}{\lambda_1}\right)}
\]

or

\[
d = \frac{|m_2 - m_1| \cdot \lambda_2 \cdot \lambda_1}{2n \cdot (\lambda_2 - \lambda_1)}
\]
Now consider the example spectrum shown of the 2µm layer in Fig 2. Between 400nm and 800nm there are 5 whole wavelengths, so \([m_2 - m_1] = 5, \lambda_1 = 0.4, \lambda_2 = 0.8\) (in microns)

\[
d = \frac{5 \times 0.4 \times 0.8}{2 \times n \times (0.8 - 0.4)}
\]

\[
d = \frac{1.6}{0.8} = \frac{2}{n} \text{ µm}
\]

in the case where \(n=1\), this gives the optical thickness, \(d_o\)

so

\[
d_o = 2 \text{ µm}
\]

and a quick look at the 1µm spectrum shows that there are 2½ wavelengths between 400nm and 800nm, so this film is half as thick as the other.

In order to compute the physical film thickness from the measured optical film thickness, one needs to know the refractive index, \(n\). Now \(n\) actually varies with wavelength, and this property is called dispersion. For highly accurate results, or for measurements on materials where the dispersion is significant over the range of wavelengths used to make the measurement, one needs to take account of it by using a special polynomial formula, called the Cauchy dispersion formula, which gives the following dispersion correction:

\[
n(\lambda) = n_0 + \frac{B}{\lambda^2} + \frac{C}{(\lambda^2 \times \lambda^2)}
\]

where:

- \(n(\lambda)\) Dispersion
- \(n_0\) Polynomial constant
- \(B, C\) Polynomial factor
- \(\lambda\) Wavelength

Clearly, in order to use this equation one needs to know the polynomial constant \(n_0\), and the Cauchy \(B\) and \(C\) coefficients. If these are not known, then one can simply use \(n(\lambda) \approx n_0\) which is a good approximation for most applications. If the broadband interferometer being used will allow the user to select the wavelength range over which the measurement is made, it is possible to estimate the error due to dispersion by measuring the same sample at the same point using two or more different ranges of wavelengths, with a single fixed value of refractive index. The results for physical film thickness will show small variations due to dispersion.

**Thickness Calculation & Measurement Accuracy**

One method used to calculate film thickness uses a Fast Fourier Transform (FFT) technique, which is considerably faster and more accurate than the manual method outlined above. It has benefits over other methods in giving an absolute result without requiring calibration, and is able to resolve the complex waveforms generated by multiple-layer interference into their constituent components. The FFT algorithm simply transforms the intensity/wavelength function into a relative intensity/film thickness function (Fig 3):
As can be seen from the figure above, the resolved FFT peak has a distribution which is due partly to variations in the target film thickness over the area where the measurement is being taken, partly due to the optical quality of the target film which affects the quality of the light signal, and partly due to electrical noise in the equipment. The mean film thickness to be determined corresponds to the centroid of the resolved FFT peak and this can be calculated arithmetically with the help of another algorithm specially developed for this purpose.

So, how accurate is the instrument? The four main areas to consider are, the accuracy of the spectrometer, the accuracy of the FFT thickness calculation, the precision of the sample under test, and the accuracy of the refractive index conversion from optical thickness to physical thickness.

A typical high quality spectrometer is the Zeiss MCS-UV/NIR, which has 1024 pixel elements and a spectral range of 190nm – 1020nm, so its pixel dispersion is approximately 0.8nm per pixel (i.e. each pixel is responding to a light band of 0.8nm in width). Furthermore the Zeiss specification states that absolute wavelength accuracy is less than 0.3nm, and repeatability is better than 0.1nm. Applying a FFT function to a resolved peak similar to that shown in Fig 3, and then calculating the centroid is a numerical process which can be done to a precision well within the possible error from the spectrometer.

In order to get light to the target sample and back, a fibre-optic cable is used, and this will typically have a diameter of around 1 mm at the probe end. So the fibres detecting the interference pattern and taking it to the spectrometer are sampling data from a large area, relative to the thickness of the film under test. In most polymer coating applications the variation in film or coating thickness over this area is likely to be rather more than one nanometer.

To determine an accurate physical thickness, the user needs an accurate refractive index. With many coatings this is known to the third decimal place, but some polymers are notorious for having a variation in refractive index due to the extrusion process. For example PET has a wide variation in refractive index between 1.58 and 1.64. Proprietary versions go even higher - the refractive index of Mylar is between 1.640 and 1.670.

In addition, as mentioned in the previous section, to get the most accurate accurate physical thickness requires the Cauchy dispersion coefficients, as the refractive index will change with wavelength. To get an idea of the effect of dispersion for some typical coatings, five samples of coated polymers were tested by measuring their coating thickness at the same test point using different ranges of wavelengths. The results are summarized in the table below:

<table>
<thead>
<tr>
<th>Example Coating</th>
<th>Wavelength Range</th>
<th>Dev from</th>
<th>Delta %</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>400-700nm</td>
<td>700-1000nm</td>
<td>Mean µm</td>
</tr>
<tr>
<td>UV cured hardcoat on polycarbonate</td>
<td>7.08</td>
<td>6.86</td>
<td>0.11</td>
</tr>
<tr>
<td>Dipped hardcoat on polycarbonate</td>
<td>15.13</td>
<td>14.65</td>
<td>0.24</td>
</tr>
<tr>
<td>Unspecified coating on PE</td>
<td>13.47</td>
<td>13.37</td>
<td>0.05</td>
</tr>
<tr>
<td>Hardcoating on PET</td>
<td>7.56</td>
<td>7.35</td>
<td>0.11</td>
</tr>
<tr>
<td>Hardcoating on multilayer film</td>
<td>5.46</td>
<td>5.34</td>
<td>0.06</td>
</tr>
</tbody>
</table>

*Table 1 Examples of Typical Errors in Physical Thickness when Effects of Dispersion are Ignored*

In summary, although the instrument is capable of accuracy of measurement to less than one nanometer, in most applications its absolute accuracy will be limited by the quality of optical data on the product under test. Given consistent assumptions about refractive index and a fixed range of wavelengths used for measurement, its repeatability will be excellent and certainly better than 0.1%.
Measuring Multi-Layer Film Thickness

If the film has a coating or if a second film is welded to it, and providing that the refractive index of each layer is different, there will be two internally-reflected paths of light (R2 and R3). Both of these will interfere with the reference path R1 as well as each other. Again, the shift in phase between the paths will be proportional to the additional thickness of material travelled, and to its refractive index, and this information will be present in the resulting interference pattern.

In this way each extra layer of material will generate additional interference patterns with every other layer, and these will contain information about their optical thickness. However as layers are added, the complexity of the resulting interference pattern rises exponentially, and it becomes much harder to distinguish the signals. Despite this, the FFT technique allows us to calculate the thickness of each layer simultaneously, provided that the resultant interference pattern has a strong enough component to be resolved by the spectrometer.

The double layer structure shown in Fig 4 will generate an interference pattern similar to that shown in Fig 5, which is a classic two-layer interference pattern. In theory one would expect to see a more complex pattern caused by the three possible sets of interference, but in practice the third set is often too weak to detect accurately. The pattern shown below was generated by a 1µm coating on a 12µm film, but actually corresponds to the 1µm coating and the 13µm overall film thickness. By separate evaluation of both FFT peaks and then subtraction, the thickness of the substrate layer can be determined, if required.
A Practical Off-Line Instrument

Any instrument has four essential elements – light source, fibre-optic cable, spectrometer and computer. In this example the light source (upper unit), is linked to the sample under test (not shown here) by one arm of a bifurcated or “Y” shaped fibre-optic cable. The end of the fibre-optic cable (or “probe”) is simply placed onto the test sample (it does not need to touch it), and the reflected light is transmitted back up the other arm of the cable and into the spectrometer (lower unit). The spectrometer captures the interference pattern and converts it into digital data, which is then sent to the PC for FFT analysis, peak search and calculation, and then display. A typical display might look like:

Fig 6 Instrument with PC showing Fibre-Optic Cable which carries light to and from the sample under test

Fig 7 Typical Display Showing 20µm Film with 3µm coating
This particular instrument actually has two independent FFT calculators, so it can display the results of two separate layer thickness calculations simultaneously. Here, on the left hand side we are measuring the coating thickness, and on the right hand side the overall thickness, of a nominal 20µm film with 3µm coating.

**Limitations**

The principal limitation of this technology, is that it will only work with materials which will generate an interference pattern. Returning to Fig 1, this means that top and bottom surfaces of any film must be smooth, and there must be nothing inside it to scatter the light. The film must be transparent, so that light can be reflected off its lower surface. In practice it can be coloured, as this only absorbs light in part of the useful wavelength range. It cannot measure coatings laid directly on rough surfaces such as paper, but can measure for example the thickness of a polymer film laminated onto paper. It works well with most coatings on most polymer films, coatings on polycarbonate, coatings on smooth sheet metal and coatings on sheet glass.

A second limitation is in the range of film and coating thickness that this kind of instrument can measure. In general, the range can be altered by selection of an appropriate light source and spectrometer. An instrument using the Zeiss spectrometer referred to earlier with a standard halogen source and FFT-based thickness algorithm will operate from around 0.5µm to 100µm physical thickness. Using a UV source can extend the range and measure thin layers down to 0.1µm in some cases.

A third limitation is in the number of layers which can be measured. This is determined by the reflectivity of the different layers, and the need to get enough light reflected back to create an interference pattern. In practice with a good optical film it is possible to detect up to 6 thickness peaks. Relating these to layers within the film requires a detailed knowledge of the structure. Also, these results give a good measure of optical thickness, but the calculation to give an accurate physical thickness is complicated because each layer will have a different refractive index.

Finally, many people want to know if it is possible to measure one or more layers of material present in a co-extruded film. The answer is that measurement is only possible where there is a reflection from the relevant layer surfaces, which means that the adjacent layers must have different refractive indices. For this to be the case, they must be different materials, for example a PP-PE copolymer next to PE. A co-extruded film with all layers identical will simply resemble a single-layer film.

**New Developments for In-line Use**

One of the key benefits of a measurement system based on this technique is that the user gets an accurate and direct result, and no calibration is necessary. As was discussed earlier, the absolute accuracy of the instrument is better than 1 nm, which is good enough for all practical applications in the coating of polymer films. The optical properties of the film are being measured directly, and used to generate a physical measurement. Also, the measurement is not sensitive to the distance between the probe and the target film, so long as there is sufficient light to give a strong interference pattern. So can this technique be used for in-line measurement?

Today the answer is yes – but only at relatively low line speeds. It takes time for the instrument to collect a spectrum for analysis. This time depends on the brightness of the light source, the reflectivity of the target material, the distance of the probe to the target material and the sensitivity of the spectrometer. If there is not enough light, the data collected by the spectrometer gets converted to a weak electrical signal which is drowned out by electrical noise in the rest of the instrument. To get the best results the spectrometer must be set up to collect as much light as possible without saturating (or overloading) itself. This time period is known as the integration time, and today it is typically measured in tens of milliseconds.

If the target sample moves during this time, the interference pattern detected will change, due to changes in the thickness of the film, and the quality of the resolved thickness peak will degrade. If the changes are significant enough, we will lose the resolved peak altogether. A good analogy is with the blurring of a photograph when trying
to capture a fast-moving object with slow photographic film. Tests done to date indicate that it is possible to measure the thickness of a good optical quality film or film and coating moving at up to 50 meters per minute.

In order to increase the acceptable line speed, the integration time must be reduced. As developments in optics continue rapidly, there are already more powerful light sources and more sensitive spectrometers available, which have the promise of allowing a significant reduction in integration time and hence increase in achievable line speed to 300 meters per minute or more.

For some applications, an important aspect of the system is that it can be used in explosive atmospheres. The main instrument can be located many meters from the point of measurement. Only the fibre cable carrying the light from the instrument needs to be mounted at the point of measurement. There are no active elements within the fiber cable and so no sources of ignition. Such capability is not available with most other forms of sensor.

**Conclusion**

The broadband interferometer offers a fast and accurate non-contact method of measuring transparent film and coating thickness off-line. It inherently produces a value for the film’s optical thickness, and this can be converted to actual physical thickness providing that the refractive index is known. Simultaneous measurement of several layers in a multi-layer film is also possible.

The technique is fast enough to be used in-line, on relatively slow processes today but will shortly be available as an in-line tool for faster process speeds.

Finally, due to the passive nature of the sensor, it can be used in high solvent concentration or explosive atmospheres without special precautions.

**Acknowledgements**

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