

# Beam Profile Reflectometry – A novel and effective solution for coating quality control



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## Abstract

There are many techniques for measuring the thickness of transparent coatings deposited onto substrates, especially when the substrates are smooth and flat. The most common techniques are all optical in nature, relying on an analysis of light reflected back from the surface of the sample under test. In this paper, we describe Beam Profile Reflectometry (BPR), the technique employed in Nightingale-EOS Limited's *n*-Gauge™ metrology tool, and compare it with other more established techniques. It will be shown that BPR, as implemented in the *n*-Gauge™, offers several specific advantages over other techniques. In particular, it is able to make accurate measurements in cases where the substrate is warped or curved, and uniquely it is able to make deterministic measurements of the refractive index, *n*, of films in the absence of any prior information. This makes it especially useful for films whose composition may change as a result of process variation, such as the doped polymer, diamond-like carbon and metal oxide films often deployed in the medical device industry.

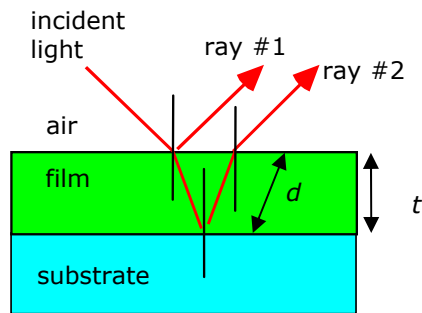
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## Introduction

Light reflected from a surface encodes a significant amount of information about the surface itself – its texture, orientation and composition. In many cases, especially where the surface is 'bare', it is sufficient to be able to measure its colour, roughness and form, which can be done using conventional machine vision or optical microscopy, or perhaps by using contact probes – a stylus, an indentation probe or a scanning probe microscope of some sort. However, when there is a coating upon the surface, more information is required. Contact probes can provide no useful information about the coating unless there is a 'step' where coated and uncoated surfaces can be compared side-by-side, and isolated measurements of colour yield little information because colour is a function both of composition and coating thickness.

There are nevertheless a range of optical techniques available to address this problem. They can be divided into two broad classes. One consists of 'surface imaging' techniques, such as confocal microscopy and white light interferometry, which locate each interface of the coating in isolation (i.e. the coating surface, the buried interface between the coating and substrate, and possibly any other buried interfaces in a multi-layer film), and measure the distance between them. These offer very high resolution images of the surface morphology, but suffer from low throughput because of the need to move the sample physically during the course of the measurement. The other category consists of 'interferometric' techniques, making use of the interference which occurs when light rays reflected from the various interfaces are brought together. In this category belong techniques such as spectrophotometry, ellipsometry and Beam Profile Reflectometry. These are high-throughput techniques well-suited to providing in-line quality control for coatings, but in the real world there are numerous challenges to overcome.



**Figure 1**

Interference between rays reflected at each interface.

Figure 1 shows the basic principle of light reflecting from a coated surface. On encountering the upper surface of the film, the light is split into a component which is reflected from the surface and a component which penetrates the surface and is reflected from the substrate. When this component exits the film, it is 'out of phase' with the first ray because it has travelled a physically further distance. When the two rays are brought together, for example by a lens, they will interfere either 'constructively', giving a bright fringe, or 'destructively', giving a dark fringe, depending upon the relationship between the path difference and the wavelength of the light. This relationship depends upon the wavelength of the light itself, the thickness of the film, and the angle of travel of the light within the film which determines how the path  $d$  differs from the thickness  $t$ . It also depends on the refractive indices,  $n$ , of both coating and the substrate materials, because these determine the relative amounts of light taking each path, the degree to which the path of the light is 'bent' within the film, and the degree to which the wavelength of the light is shortened while travelling through the film. Finally, it also depends on the polarisation of the light, which further affects the reflectance of each interface.

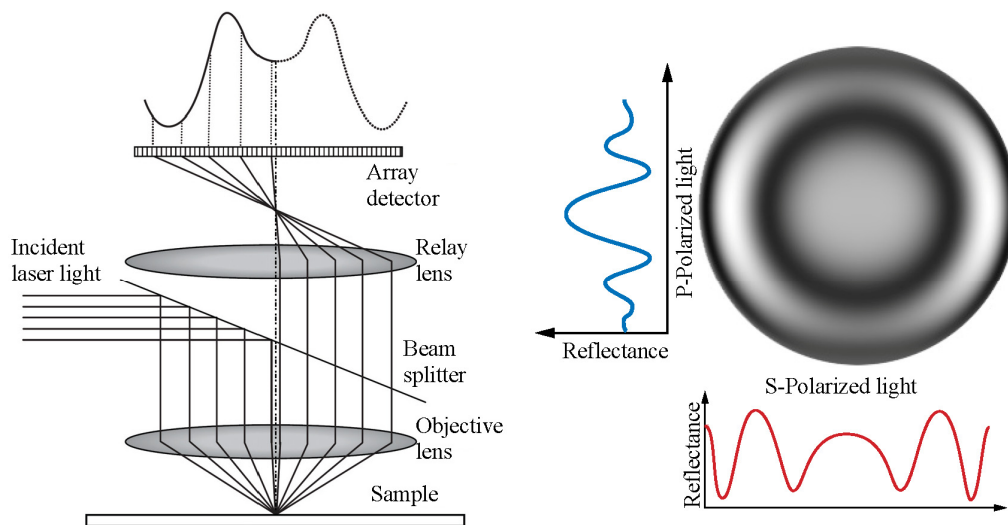
Each different measurement technique available works by measuring overall reflectance as a function of one or at most two of these factors, while holding the others constant. Traditionally, the surface is illuminated by white light at a constant angle-of-incidence and the reflectance is measured as a function of wavelength. In spectrophotometry, 'normal incidence' is used (i.e. the light is incident vertically downwards onto the sample), and the intensity of the reflected light is measured independently of polarisation. In ellipsometry, a steep angle-of-incidence (often  $\sim 60$ - $70$  degrees) is used, and the intensity and phase of the reflected light is measured as a function of wavelength and polarisation.

Uniquely, Beam Profile Reflectometry takes a different approach, keeping the wavelength fixed (using laser light) and measuring reflectance as a function of angle. It will be seen that this offers several significant advantages over other techniques.

### Beam Profile Reflectometry

BPR was first introduced by Therma-Wave Inc. in 1992 [1-2] as a technique for measuring thin films on silicon wafers. Prior to the introduction of BPR, measuring reflectance as a function of angle involved complex and expensive hardware arrangements where both the light source and detector needed to be moved each time a new angle was selected.

As illustrated in figure 2, BPR overcame this limitation by using a high-magnification lens to bring a collimated laser beam to a sharp focus. At the focal point, which is typically less than  $1\mu\text{m}$  across, light falls on the sample with the whole range of different angles-of-incidence through which the lens bends the light in order to achieve focus. After reflection, the lens re-collimates the reflected light and there is a one-to-one correspondence between the physical location of a ray of light within the recollimated beam and the angle at which that ray was reflected from the surface. It is therefore possible to measure reflectance as a function of angle of incidence for a wide range of angles (typically, for a  $\sim 100\times$  lens, a range of  $0$ - $60$  degrees) simultaneously, with a very short data acquisition time, using an apparatus with no moving parts.



**Figure 2:** Schematic representation of a Beam Profile Reflectometry (BPR) system

When the beam profile is viewed after reflection from a coated surface, a characteristic “bullseye” pattern is seen due to the pattern of light and dark fringes that form as a result of the interference between rays as shown in earlier in figure 1. The amplitude of the fringes depends only on the refractive indices of the materials in the filmstack. The period of the fringes is determined by the coating thickness. It is therefore possible to decouple the effects of thickness and refractive index and measure the two classes of parameter independently.

It can also be seen from figure 2 that the beam profile differs slightly depending on whether the cross-section is viewed horizontally or vertically. This is because of the dependence of the sample reflectance upon the polarisation of the incident light: the reflectance differs slightly for the two cases of  $s$  and  $p$  polarisation, where the ‘plane of incidence’ is respectively perpendicular and parallel to the polarisation. For unstrained films, both  $s$  and  $p$  signals contain essentially the same data, but if the film is strained – as is often the case for polymer or diamond-like carbon films – then the two diverge. This is because of strain-induced birefringence, which causes the  $p$ -polarised light to experience a slightly different refractive index from the  $s$ -polarised light. Because BPR measures both the  $s$  and  $p$  polarised components independently, it is able to quantify this refractive index difference and so measure the strain in the coating as well as the other parameters.

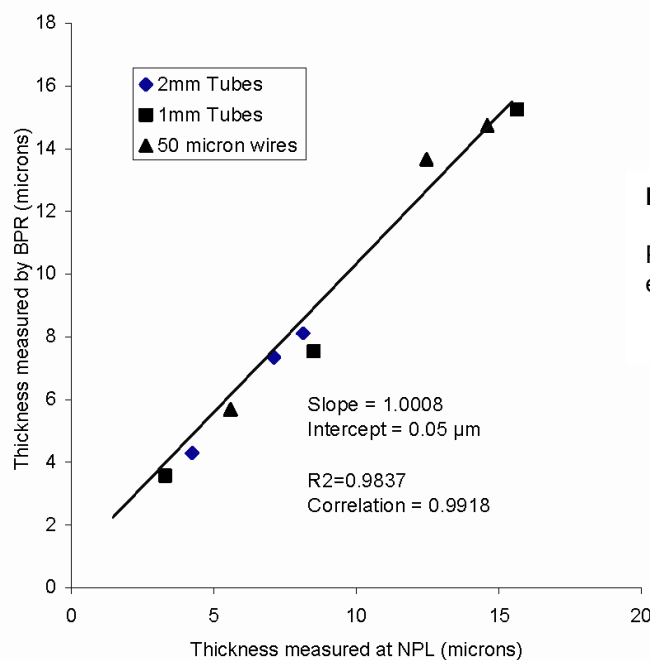
Note that all these measurements take place at a single wavelength, determined by the laser source employed. This is a big advantage over white-light techniques, because there is no need to take account of *optical dispersion* – the variation of refractive index with wavelength. This means that for each material in the filmstack there is only one value of refractive index to find (or at most two in the case of a birefringent film), and yet there are hundreds of independent data points in the raw data. This data richness enables direct, deterministic measurements of refractive index to be made, in contrast with spectral techniques which, as shall be shown later, are unable to do this and must rely instead on models and assumptions.

A further advantage of BPR is seen where the sample being measured is curved, warped or misaligned relative to the lens. In the simple case shown in figure 2, the sample is flat and aligned at right angles to the lens axis, leading to the simple and symmetrical fringe pattern shown. Most optical measurement techniques, including BPR itself as originally implemented in the semiconductor industry, rely on a high degree of such alignment. This often leads, in practice, to severe difficulties where the alignment is imperfect, and to solutions such as the sucking of warped silicon wafers onto a perfectly flat stage using a vacuum, with consequent risk of damage and contamination.



Nightingale-EOS has addressed this issue directly, by observing that where the sample is misaligned the fringe pattern shows characteristic distortion, enabling the misalignment to be identified and quantified. A proprietary data-analysis algorithm then models the fringe pattern taking full account of the misalignment, in effect allowing the sample's orientation to be measured along with the coating properties.

Where the surface is not only misaligned but also curved, there are further effects which need to be accounted for and to which, again, BPR is uniquely sensitive. Further details of the analysis involved are given elsewhere [3], but figure 3 gives an indication of the resulting capability of BPR by showing the results obtained over a range of samples with different coating thicknesses and curvatures, up to an extreme case of 15 $\mu\text{m}$  films deposited on 50 $\mu\text{m}$ -diameter wires. Extremely high (better than 99%) correlation was obtained relative to destructive measurements carried out by the UK's National Physical Laboratory [4].



**Figure 3**

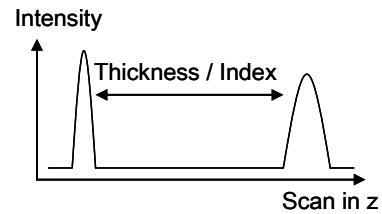
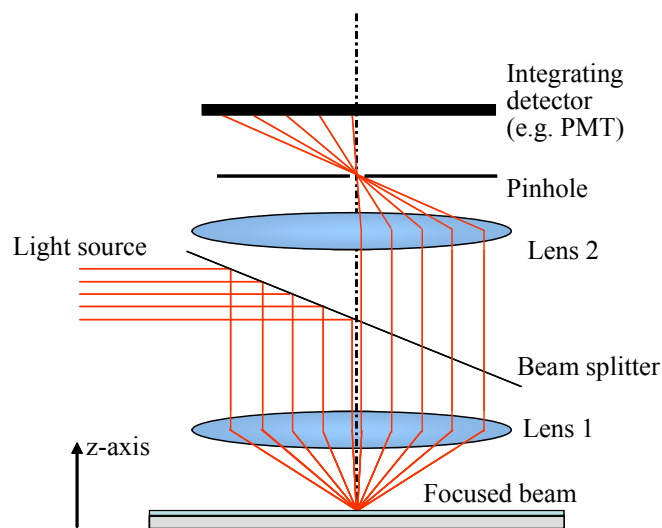
Results obtained from NPL evaluation on curved samples.

### Alternative techniques

We now compare and contrast BPR with some of the other techniques which are currently in use, especially in the medical devices sector. A particularly relevant review article was published in the November/December 2008 edition of Medical Device Technology magazine by A. Forster, T. Neudeck and S. Blatcher of the PA Consulting Group [5], but that article pre-dates the availability of BPR for non-semiconductor applications and so does not mention it. Here the alternative techniques will be briefly described and the contrasts made more explicit.

### Confocal Microscopy

A schematic diagram of a confocal microscope arrangement is given in figure 4. By a cursory comparison between this and figure 2, it can be seen that the basic optical layout of a confocal microscope is almost identical to that of a BPR system. The only significant difference is that whereas BPR uses an array detector to measure the characteristics of a reflected beam profile over thousands of discrete pixels, a confocal microscope has a single integrating detector such as a photomultiplier tube located behind a pinhole. Commercial confocal microscopes often also have a goniometer arrangement, not shown here, to enable the location of the beam focus to be scanned rapidly over the sample surface in a raster pattern, so enabling an image to be formed.



**Figure 4**

Principle of confocal measurement of film thickness

Operationally, however, the methods are completely different. Confocal microscopy is a technique for imaging a particular 'slice' of a sample by excluding light which originates outside the slice. Its original application, when it was first developed in the 1950s, was for imaging extended translucent objects such as biological cells. With a conventional microscope, getting a sharp image of such an object is problematic because light from parts of the sample which are out-of-focus is always superimposed on light from the part which is in focus, preventing a clear image of the latter from being formed. In a confocal microscope, light from the out-of-focus parts of the sample is excluded, enabling a sharp image of a particular slice through the sample to be obtained. In the case of biological cells, it is then possible to construct a full three-dimensional image of the sample by creating a succession of these 'slices' through different parts of the sample.

Referring to figure 4, the method works because when light emanates (by reflection or fluorescence) from a surface located at the focal point of the main objective lens, the same light will also be brought by the relay lens to focus at the pinhole. This light passes through the pinhole, unattenuated, to fall on the detector. However, light which is out-of-focus below the objective will equally be out-of-focus at the pinhole, and so will fail to pass through and be heavily attenuated. Typically, an image of the focal plane is constructed by using a laser beam as a source and scanning it through a matrix of pixels to build up the image. An alternative approach is to use white light as the source and to have a matrix of separate pinholes to build up the image.

In either case, to use this technique for film-thickness measurement it is necessary to image separately the upper and lower interfaces of the film and calculate the distance between them. This leads immediately to two complications:

Firstly, isolating the two surfaces from one another is difficult to do in practice. In contrast to a biological sample with similar brightness throughout its volume, for a transparent film on an opaque substrate the brightness of the image from the lower interface is so much greater than that from the top interface that even the pinhole makes it difficult to decouple them entirely. Moreover, spherical aberration in the image of the lower interface, introduced by the presence of the film, limits the ultimate sharpness with which the lower interface can be resolved.

Secondly, and more seriously, the measured distance moved by the sample stage in going from one interface to the other must be multiplied by the film refractive index in order to obtain the actual film thickness value. The confocal microscope, however, provides no way to measure this index and its value must be assumed. If anything happens to change the index (e.g. different drug loading in a drug-eluting film) then this will be invisible to the technique and the result will be an error in the thickness measurement obtained.

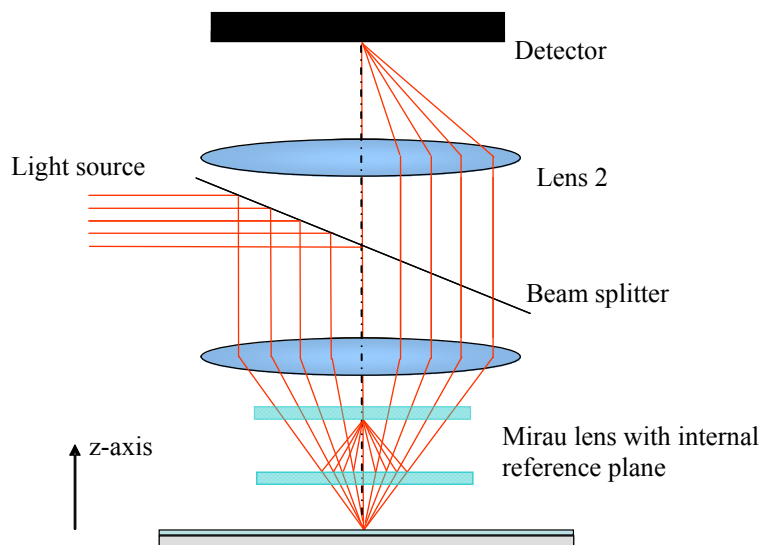


BPR is immune to these issues because (a) it explicitly combines light from all interfaces to form a single interference pattern, hence sidestepping the problem of how to decouple one interface from another, and (b) it makes a direct measurement of the refractive index at each measured point, so film index variations do not lead to thickness errors but rather can be identified, and therefore controlled, in their own right.

### Scanning White Light Interferometry

Scanning white-light interferometry has many things in common with confocal microscopy, in that its principal function is to form an image of a particular surface of a sample. Like confocal microscopy, it can only perform film-thickness measurements by imaging each interface in isolation; like confocal microscopy, it runs into difficulties when the relative brightness of different interfaces differs greatly, and like confocal microscopy it is dependent upon an externally-supplied value for the refractive index in order to obtain a thickness value. The use of white light exacerbates this problem further, because of the dependence of refractive index upon wavelength – in effect, multiple values of refractive index must be externally supplied.

Only the technique used for forming the image of each interface is different. In a white-light interferometer (see figure 5), interference patterns are formed between light from an interface and light from a fixed reference surface within the tool itself. The interference patterns obtained are processed numerically in order to construct an image of the surface under investigation. While the technique is generally quicker and often more robust than confocal microscopy, the resolution with which interfaces can be separated is not so good and there is typically a lower film-thickness limit of  $\sim 2\mu\text{m}$  when using this technique [6].



**Figure 5**

Schematic diagram of a white-light interferometer

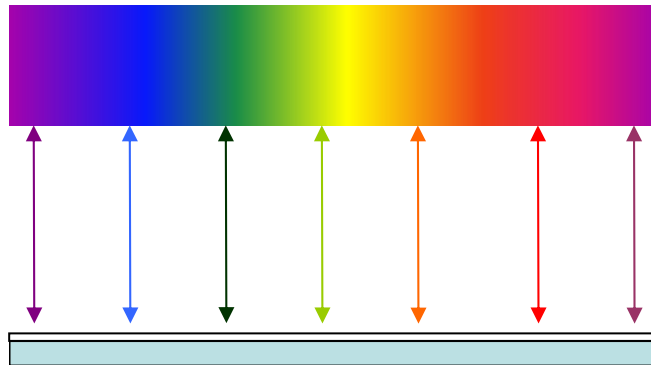
BPR is immune to these issues for the same reasons as it is immune to the issues faced by confocal microscopy.

These two techniques have some capability on tilted, curved or otherwise misaligned samples, in that they can image the surface and so deduce its orientation. However, the orientation is not immediately apparent from a single measurement in isolation, and so making the necessary corrections is a relatively slow process.



## Spectrophotometry

This is conceptually the simplest technique for coating thickness measurement in that it uses a simple white-light source at normal incidence, and sometimes can be accomplished without using a lens (for example, a fibre optic bundle can be used to deliver the light and collect the reflected rays). Both Forster et al [5] and Flaherty & O'Connor [7] report some success in applying it to the challenging application of drug-eluting films on stents, though it is known to be a time-consuming process to collect data of sufficient quality.



**Figure 6**

Basic concept of spectrophotometry

As mentioned in the Introduction, spectrophotometry falls into the same class of techniques as BPR, but reflectance is measured as a function of wavelength at a constant angle. The substantive differences between spectrophotometry and BPR, and the reasons why BPR is generally the more accurate technique, can be summarized as follows:

- 1) For the vast majority of materials, the refractive index varies as a function of the wavelength. In spectrophotometry, however, each data point corresponds to a different wavelength and therefore a different refractive index. Therefore, the number of unknown parameters always exceeds the number of independent data points available, and a deterministic measurement cannot be made. To remedy this situation, a 'dispersion model' is used to parameterize the variation of index with wavelength, but this relies on numerous *a priori* assumptions. Ultimately the method is reduced to depending on an externally-supplied refractive index estimate, just as the previous techniques were seen to be.
- 2) Using a laser source, it is a very simple matter to ensure that one is working at a single wavelength. In spectrophotometry, however, use of a single angle-of-incidence is not possible in practice. Any lens used to image the surface being measured causes the light to be incident at a range of angles, and the higher the magnification of the lens the larger the range of angles involved. As a spectrophotometer has no way of resolving the individual angles, it is forced to integrate over them. This introduces loss-of-resolution, and for very thick films (>5 $\mu\text{m}$  for example) the data can become completely 'washed-out' unless a very low magnification lens is used. This in turn limits the spatial resolution of the measurement (increasing the 'spot size') and makes it harder to home in on particular parts of the sample.
- 3) If the sample is tilted, spectrophotometry lacks the capability of seeing this and so any tilt will simply cause an error in the measurement – for example, reducing the overall amount of light reaching the detector and getting confused for roughness or absorption in the film.
- 4) As it does not take account of the polarization of the light, spectrophotometry cannot be used to determine strain within the film. On the contrary, strain, if present, will result in a further loss of accuracy of which the user will be unaware.



Even so, there are cases where the combination of BPR and spectrophotometry can be very powerful. For example, the variation of refractive index with wavelength may itself contain useful information about film composition, and the correlation may be strongest at wavelengths other than the BPR laser wavelength: by combining the two techniques, BPR can be used to make an accurate measurement of the coating thickness together with a direct determination of the refractive index at the laser wavelength, hence reducing the number of assumptions needed to model the film properties at other wavelengths. The design of the Nightingale-EOS n-Gauge™ includes an upgrade path to add a spectrophotometer to the unit at a later time, enabling such combination measurements to be made.

### Spectroscopic Ellipsometry (SE)

SE is an extension of spectrophotometry that takes account of phase differences between different polarizations of light, usually at a fairly high angle of incidence ( $\sim 65^\circ$ ). Unlike basic spectrophotometry, it gathers two pieces of data (amplitude and phase) per wavelength and is often thought of as an absolute technique for refractive index characterization.

This is true in the case of bulk substrates, but not for thin films. This is because a full characterization of the film properties involves finding both the real and imaginary parts of the refractive index, i.e. two unknowns per wavelength, plus the film thickness. Even with two independent data points per wavelength, the technique still provides fewer data points than unknowns and is ultimately unable to provide a deterministic measurement. Moreover, like basic spectrophotometry it lacks the ability to detect film strain, and yet can be more severely affected by it. In head-to-head evaluations between BPR and SE in the semiconductor industry, thicknesses obtained by SE from strained films were found to be inaccurate by up to 15%.

On tilted or curved surfaces, SE presents particular challenges because of the difficulty of capturing the reflected beam if it has been reflected at an unexpected angle. The technique is not known to have been implemented for medical devices or other samples with complex shape, instead being mainly used on flat silicon wafers where these effects can be neglected.

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