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Pitfalls in Thin-Film Optical Property Measurement

From the Archives: 2001 | Angus Macleod

Everyone in the optical coating industry has an interest in the optical properties of materials. They are especially used in the design of optical coatings and in the prediction of their performance. Going without them is rather like being at sea with no chart.

The two most important properties are refractive index, n, and extinction coefficient, k. These are two dimensionless numbers defined as:

$$n = \frac{\text{Velocity of light in free space}}{\text{Velocity of light in the material}}$$

$$\frac{k}{n} = \frac{1}{\text{number of wavelengths of path for amplitude to fall by } 1/e^{2\pi}}$$

This curious definition for k allows us to combine the two quantities into a complex refractive index (n-ik).

Measurement of the quantities n and k present us with some difficulty. They are virtually impossible to measure directly on a thin film and so we never, actually, do measure them. Instead, we measure some related properties, such as reflectance and transmittance, or the ellipsometric parameters, psi and delta. We construct a theoretical model of the thin film and we adjust its parameters until the theoretical performance matches, as well as it can, the measurements. We then adopt the adjusted values of the parameters as those of the film. Usually the closeness of fit between the calculated and measured performance is taken as an indication of the reliability of the process. It is not so much a measuring as a fitting process, and there are some pitfalls.

The pitfalls include poorly matched film and model behavior and inadequate, and even incorrect, measurements. It really does not matter how well the calculated results match the measured results. If the model is deficient, then the extracted parameters will be deficient. Similarly if the measurements are not correct, even a correct model will be adjusted so that its predictions are in error, and, once again, the parameters will be in error. If only we were going to use the extracted parameters to rebuild the input measurements this would all be of little consequence. But we use the parameters in predictions of performance of completely different films of the same material. To our ideas of accuracy of thin film parameters we can add the concept of stability. The extracted parameters are stable when errors in prediction are not significantly greater than those of the original measurements. We shall limit this discussion to dielectric layers with only slight absorption. Also, so that we know exactly the true nature of our films all results, even if described as measured, will actually be calculated.

The first example is a quite simple one. We have measurements of reflectance fringes of a thin dielectric film on glass. The film is slightly absorbing but the model that is being used for extraction of results is free of absorption. The fit of the actual and predicted reflectance results using the model is shown in Figure 1 and it is quite convincing.

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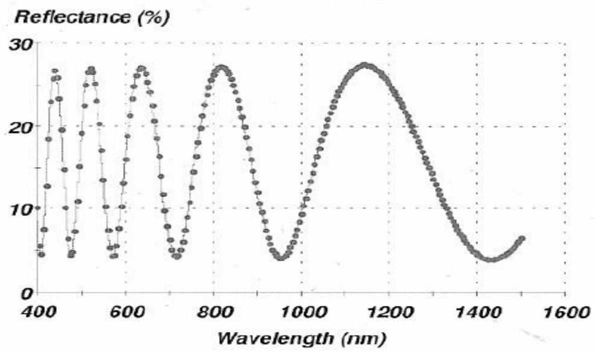

Figure 1. *The fit between the measurements (dotted) and the calculated results (solid curve) using the extracted parameters for the absorption-free model.*

Now we design an extended-zone high reflectance coating using this material as high index and SiO2 as low. The coating is produced and the comparison of measured transmittance and manufactured transmittance is shown in Figure 2. The difference is quite small and lends confidence that the operation has been successful.

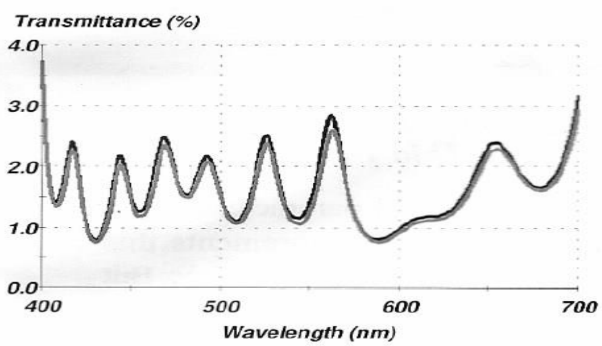

Figure 2. *Comparison of the transmittances of a theoretical design using the derived material constants and the actual ones. The results using the derived values are slightly higher but there is very little difference.*

Comparison of the reflectance curves, Figure 3, tells a very different story. Two serious mistakes were made in this procedure. First, it is very difficult to detect layer absorption in reflectance measurements on single layers. It is shown much more clearly in transmission. Second, a reflecting coating should never be tested using only transmission measurements.

\We can consider a second example. A record of the transmittance of a high-index film is shown in Figure 4. The optical constants of the film were extracted using a homogeneous and absorbing layer model. The correspondence is impressive. The extracted values of n and k are shown in Figure 5. One would be forgiven for concluding that these values could be relied upon. Unfortunately, the film used to generate the input transmittance was an absorption-free inhomogeneous layer. The outer and inner indices of that layer are also shown in Figure 5 and there is quite good agreement with the extracted homogeneous n. The derived extinction coefficient shown rising towards longer wavelengths is completely spurious. Comparison of reflectance values, Figure 6, show a discrepancy.

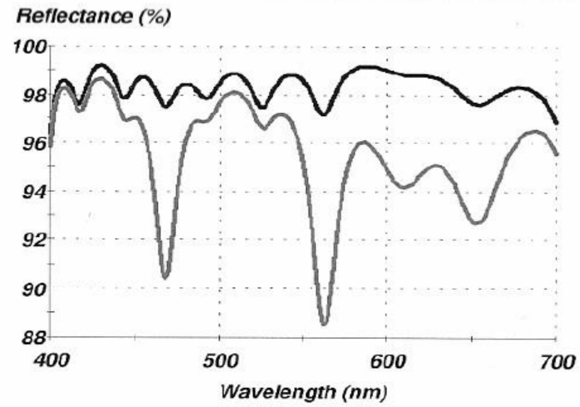

Figure 3. *The reflectance curves show a large drop due to the absorption in the high-index layer. The upper curve is the designed curve. The lower curve shows the coating with the true absorption in the high-index layer.*

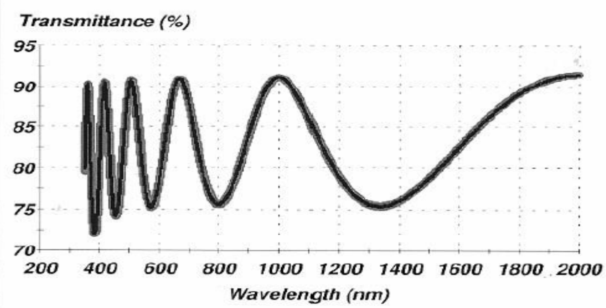

Figure 4. *The transmittance of a high-index film. The purple curve represents the performance data used for the extraction of optical constants and the black superimposed curve is a recalculation of the input results using the derived optical constants.*

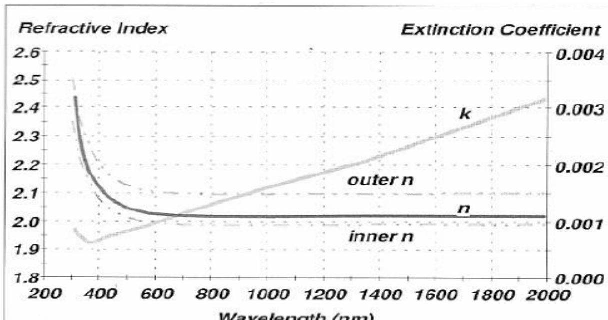

Figure 5. *The values of n and k extracted from the transmission curve in Figure 4. The two dashed lines mark the inner and outer indices of the absorption-free inhomogeneous layer used to generate the original results. The thickness of the inhomogeneous film was set at 490.14nm. The derived thickness was 494.69nm.*

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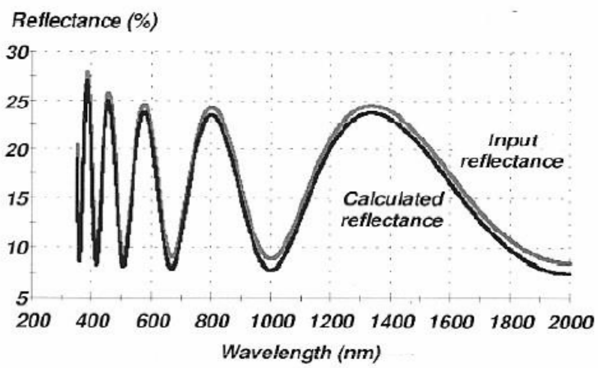

Figure 6. Comparing input reflectance (upper curve) with calculated reflectance (lower curve) shows a discrepancy.

Thin film measurements are often made on films deposited on silicon disks. The disks are readily available with a very good optical polish and the fringes are quite pronounced especially when high-index films like tantalum oxide are involved. However, this technique, too, suffers from problems. Figure 7 shows the slow variation of minimum reflectance with film index when the film is a good antireflection coating for the substrate. Small errors in the measurement of reflectance translate into much larger errors in extracted index near the minimum of the curve.

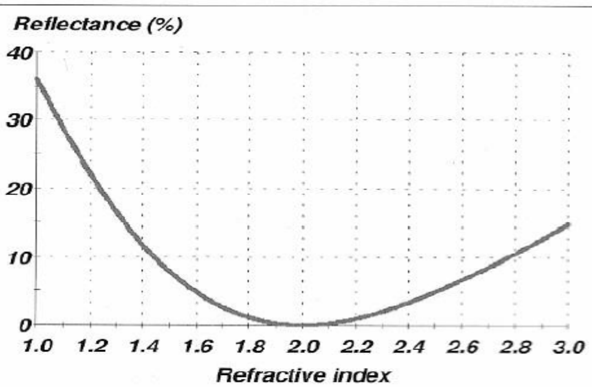

Figure 7. The reflectance of the fringe minimum as a function of the refractive index of the film. The film is supposed homogeneous and free from absorption and it is deposited on a wafer of material, such as silicon, with refractive index 4.00.

Absolute precision in measurement of transmittance or reflectance is impossible. There are always slight errors in the calibration of the instrument. Let us imagine that the error in a particular case is around 0.1% absolute. From Figure 7 it is quite easy to calculate the resulting error in determining the index of the film from the reflectance measurement. This is shown in Figure 8. Not only is the error in index much larger near the reflectance minimum but it is easy to see from Figure 7 that there are always two solutions for refractive index given the fringe minimum. These two solutions merge at the minimum and in that vicinity it is difficult to decide which solution is correct. It is good policy to avoid the antireflection condition in the film specimens used for optical constant derivation. In fact, for thin film materials for the visible region, glass is a rather better substrate for optical constant derivation giving improved results over the whole range of refractive indices.

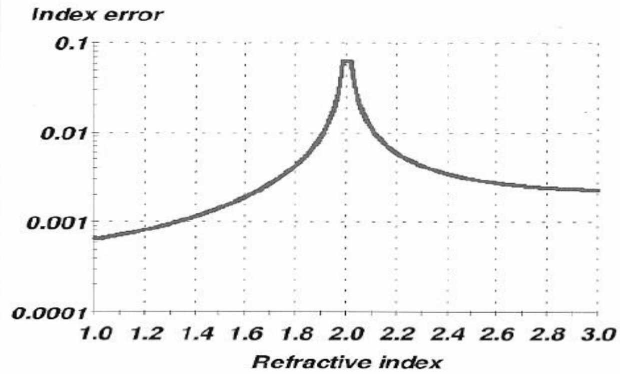

Figure 8. The absolute error in refractive index caused by an error of ±0.1% absolute in determining the reflectance at the minimum of the thin film fringe when deposited on a high-index substrate with refractive index 4.0, such as silicon.

Our final example is of a film that is more complicated in its makeup than the model that is being used. Figure 9 shows a set of transmission fringes for a film that are quite obviously misshapen in the vicinity of 700nm. Such peculiar behavior affecting only a small number f fringes is a sure sign of a periodic variation of properties through the thickness of the film. The usual cause in practice is a lack of control during the deposition of the film. A controller, such as gas flow, may be faulty or a too frequent manual adjustment of process variables may be the culprit. In fact, the results of Figure 9 were derived from a film with a small sinusoidal variation of refractive index but practical variations can be much less regular. The extraction of the optical parameters using normal techniques and an absorbing film model with simple monotonic inhomogeneity leads to quite strange results like those of Figure 10. Such results cannot be reliably used in performance prediction and, indeed, the correct course of action when a film like this appears is to improve the control of the process.

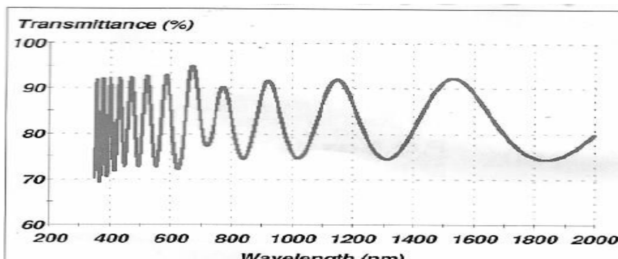

Figure 9. Misshapen fringes like these are a sure sign of an inhomogeneity that is more complicated than the usual models can accommodate. They are usually a sign of lack of control during deposition. These shown here are due to a small cyclic variation of index through the film. This film has no absorption and so the reflectance results are the inverse of the transmittance.

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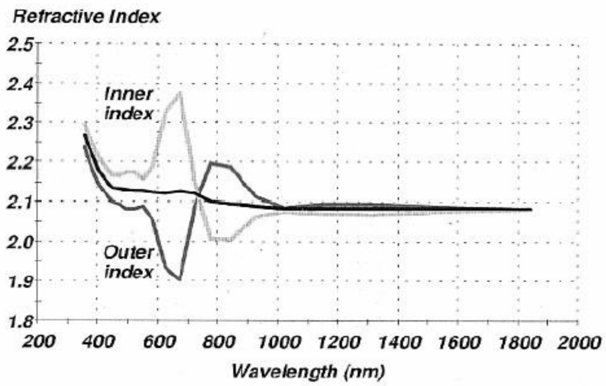

Figure 10. *Any attempt to extract the parameters of the film leads to very distorted results in the vicinity of the misshapen fringes. Here the apparent inhomogeneity varies rapidly from negative to positive. Note that when the wavelength is rather larger than the period of the disturbance through the film the results are relatively well behaved.*

These examples are all theoretical. The results were generated from known film models. But the messages are ones that are essentially practical. We often seek reassurance that our extracted parameters are valid by comparing calculated and measured performance. A good fit is taken as positive encouragement, if not absolute confirmation. The assumption that a good fit indicates reliability is an extremely dangerous one. After all, we have adjusted the parameters of the model so that we get the best possible fit. We should always try to use as much information as possible about the film and we should always try to have more than one film sample, preferably with different thicknesses. We should be suspicious of any predictions made on the basis of the extracted parameters until we have had a chance to check them in practice. Measurement accuracy is of primary importance. A spectrometer that is used by many people and belongs to none is almost certain to be badly calibrated. The achievement of accuracies as good as 0.1% absolute in reflectance and transmittance requires great attention to detail and meticulous maintenance. It is very important to have any accurate measuring instrument under the care and control of one dedicated individual.

Originally Published in Coating Materials News, Volume 11, Issue 3 - September, 2001.

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