Witness sample preparation for measuring antireflection coatings

Ronald R. Willey

Willey Optical, Consultants, 13039 Cedar Street, Charlevoix, Michigan 49720, USA (ron@willeyoptical.com)

Measurement of antireflection coating of witness samples from across the worldwide industry has been shown to have excess variability from a sampling taken for the OSA Topical Meeting on Optical Interference Coatings: Measurement Problem. Various sample preparation techniques have been discussed with their limitations, and a preferred technique is recommended with its justification, calibration procedures, and limitations. The common practice of grinding the second side to reduce its reflection is less than satisfactory. One recommended practice is to paint the polished second side, which reduces its reflection to almost zero. A method to evaluate the suitability of given paints is also described.   © 2013 Optical Society of America

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1. Introduction

The results of the OSA Topical Meeting on Optical Interference Coatings: Measurement Problem were presented by Duparre [1] at the Optical Interference Coating meeting in Whistler, Canada. The problem was to determine the reflectance of a broadband antireflection (BBAR) coating in the spectral region from 400 to 700 nm at an angle of incidence near 0°. Identical samples were prepared and sent to all of the participants.

Figure 1 shows examples of three typical designs of AR coatings that might require the techniques discussed here. The subject AR coating in [1] was intermediate between the two 4-layer design curves shown and might be typical of the AR coatings used in many optical instruments. V-Coats are usually the most critical and may have specifications for less than 0.05% reflectance (R) at the design wavelength. BBARs would typically have somewhat less stringent specifications, but measurement errors of $\pm 0.1\% R$ are likely to cause problems with satisfying the specifications. The Optical Interference Coatings (OIC) report [1] showed discrepancies significantly greater than $\pm 0.1\% R$.

2. Problem

The amount of variation (several tenths of a percent) in the results was deemed surprisingly broad by many in the audience. It appeared that the major contributing factor was the treatment of the second surface reflection, which in many cases still added to the values that were expected to be only those from the first surface reflection. Hayton and Jenkins [2] and also Synowicki [3] have discussed similar problems in ellipsometric measurements and their suggested solutions. The ellipsometric problem they have discussed can lead to very erroneous results in measurements of index of refraction if not dealt with properly. The investigations included various commercially available tapes and even a putty ("Blu-tack" [2]) that could be made to have optical contact to remove the specular reflection from the second surface. However, their acceptance of scattered reflectance from ground or painted surfaces appears to be more tolerant than the cases discussed in this paper. There could potentially be other causes
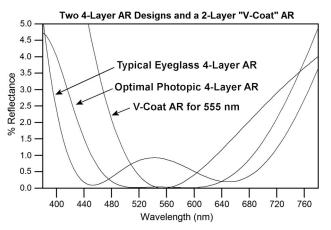
Fig. 1. AR coating examples: typical 4-layer design for eyeglasses, a 4-layer design to optimize photopic response, and a 2-layer V-Coat for AR at 555 nm.

of error such as dust, etc., but it is assumed that the contributors to the report in [1] would have been careful to avoid such things in this "contest" environment.

As illustrated in Fig. 2(a), one approach is to measure the witness sample, including the second side reflection, which is typically greater than 4% over this spectral region. Reflection from the second side could then be removed mathematically if the index and dispersion of the substrate are well known. This adds a small level of complexity to the data reduction process and also the possibility of errors in the estimate of the second surface contribution. A second approach is illustrated in Fig. 2(b), which is to coarse grind the second side of the sample so that the light reflected from the back surface is scattered widely, and hopefully, very little of the light scattered from the surface gets back into the spectrophotometer beam that reaches the detector. The suitability of this approach will be somewhat dependent on the F-number of the instrument, and there is a risk that there may still be some significant flux that reaches the detector. Some organizations choose to further blacken the ground surface with a black marking pen or other media as shown in Fig. 2(c) to reduce the reflection from this side. This still may not reduce
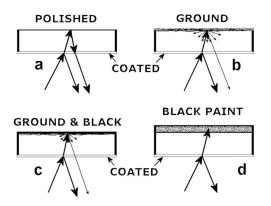

Fig. 2. Various means of dealing with second side reflections for measuring an AR coating on the first side.

the detector signal from the second side to zero. This can be evidenced by the observation that the eye can still see some reflection from the second side, and that the eye is likely to be viewing at an even smaller acceptance angle (F-number) than that of the spectrophotometer.

Other approaches to eliminate second surface reflection include coating a witness substrate that has a large enough wedge so that the second side reflection never enters and mixes with the measured beam. This approach could also be done by having a plane-parallel witness part that is oiled with an index matching fluid to a wedge to create the same geometry. The wedge would have a similar index to the witness and the oil would have an index nearly the same. An opaque black glass with the same index as the substrate (and the oil) could be oiled to the witness to cause the backreflection to disappear.

3. Solution

Another approach that is recommended is shown in Fig. 2(d), where the back surface of a flat "both-sides-polished" witness piece is painted with what amounts to an index matching fluid filled with an appropriate black pigment such as carbon black (this would be black paint). In this case, all of the light reaching the rear surface enters the vehicle of the black paint, which has an index near that of the glass, thus producing **very little** reflection at the interface. The light that enters the paint on the second surface is absorbed by the black pigment, thus **only** the front surface reflection is measured. The residual reflection from the interface between the paint and the substrate can be calculated from the Fresnel reflection equation in Eq. (1).

$$\%R = 100 \times ((n_s - n_p) \div (n_s + n_p))^2. \qquad (1)$$

Here n_s is the index of the substrate and n_p is the index of the paint. If the substrate index n_s were 1.52 and the paint vehicle index n_p (when dry, cured) were 1.4265, then the normal incidence reflection would be 0.10%. With n_p equal to 1.4899, reflection would be only 0.01%. Any paint with an index closer than 1.4899 to the 1.52 substrate index would have no measurable reflection. Also, a piece such as that in Fig. 2(d) could also be oiled to a plane–parallel witness part in order to avoid repeated painting of witnesses.

The question then is, "What is the index of the black paint that is used?" A commercially available refractometer that covers the index range of the substrate might be used to measure the index of the paint, but there may be some difficulties with this. It is desirable for the paint to be dry when measured in order to get a realistic value of the index as it would be used. Commercially available refractometers are primarily used to measure liquids, which are later removed from the instrument totally. This removal might be more difficult with dried paint. If a refractometer were used, some attention might need

to be paid to the spectral range being observed with respect to the range of interest.

An alternate method to assess the index of the paint is illustrated in Fig. 3. A right-angle prism with a known index such as BK7 is painted on separate areas on one of the short polished sides with each of the paints to be compared, as shown in the bottom view in Fig. 3. When the paint vehicles in contact with the glass in all the areas have reached a reasonably dry condition, they can be compared for index of refraction.

A simple comparison can be made by viewing the painted areas through the unpainted short side of the prism from the direction labeled "Input Light" in Fig. 3. At any large angle with the horizontal, these painted areas should appear totally black. As the viewing angle diminishes, the painted area with the largest index difference from the substrate disappears first due to the occurrence of total internal reflection (TIR) when the critical angle is reached at the prism–paint interface and thus, the light is not absorbed by the paint. The last area to disappear as the angle diminishes is the one with the best index match with the substrate. These observations could be made effectively with just room light or more rigorously with lasers or monochromators.

The critical angle at which TIR occurs as viewed from inside the prism is arcsine (n_p/n_s). The angle of the incident light on the prism in Fig. 3 is "i" and the refracted light is at an angle "r." In this case, from Snell's law, $r = $ arcsine $((\sin i)/n_s)$. For paint with index 1.4265, r would be 69.8° (see Fig. 3) and the second side normal reflection per Eq. (1)
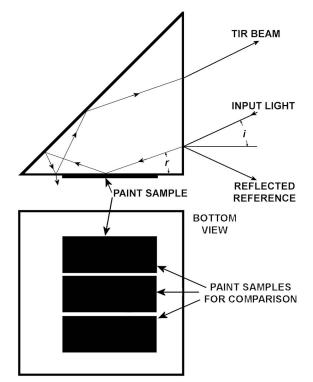
would therefore, be 0.10%. For paint with index 1.6193, the second side reflection would **also** be 0.10%, but not measured by this TIR procedure with a lower index prism. For paint with index 1.4899, the critical angle would be 78.6° and the second side reflection would be 0.01%. For paint with index 1.5507, the second side reflection would also be 0.01%.

Four black paints were tested, which were at least as good as this 0.01% case: ACE Hardware Rust Stop, Black Satin, 17073, and three Rust-Oleum products, 253365 Gloss Black, Acrylic Lacquer; 249844 Satin Canyon Black, "Ultra Cover;" and 252465 Semi Gloss Black, Automotive Enamel.

4. Discussion

It does not matter *per se* whether the paint is flat, gloss, or in-between, since the interface with the substrate would be "glossy" in any case. The painted area would need to be opaque in the spectral region of interest, which could be tested by measuring the transmittance of the second-side-painted test sample for an AR coating over the wavelength region of interest.

If more quantitative measurements are desired, then the angle between a laser and the plane of the painted area can be measured as illustrated in Fig. 3. Critical angle is when the TIR beam that has fallen **only on the painted area** fades away and/or reappears as the angle of incidence is varied, plus or minus. This angle can be used to calculate the index of the paint (after taking account of the refraction at the prism's entrance face using Snell's law). Note that the laser input and the TIR beam are parallel in this geometry (the prism in this orientation is a retro-reflector), but the intensity of the TIR beam is reduced because there is no TIR at the second and third reflections after the painted test area. This intensity would be an order of magnitude less than that of the reflected reference from the uncoated entrance face of the prism. For this reason, the use of a laser might be preferred because of its high brightness.

There could be a potential problem if the test substrate has an index much higher than the ~1.52 index of the paint. The reflection at the paint interface would still be 0.1% or less for substrates with index up to 1.6193. At index 1.66235, reflection would be 0.2%; at 1.69617, it would be 0.3%; at 1.72525, it would be 0.4%; and at a substrate index of 1.751318, it would reflect 0.5%. These reflections could be subtracted from the measured values, or one could coat a witness chip of ~1.52 index and compare the results against a calculation of what the coating should reflect with a chip of ~1.52 index. There are some index matching fluids with a higher index that might be used to advantage. Also, high-index prisms could be used as in Fig. 3 to search for paints with a higher index.

There is another very minor factor to be considered. The black pigment in the paint will also show some very small amount of scattering, which can be seen under very bright light, like that from a laser or
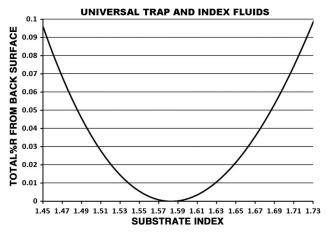

Fig. 3. Geometry for comparing the indices of paint samples by observing the critical angle for TIR.

Fig. 4. Residual %R back-surface reflection from a universal light trap oiled to a witness substrate with a given index of refraction.

sunlight. The amount of this scattering is unknown at this time, but could be worked out with proper measurements using an integrating sphere and/or a total integrated scatter instrument.

Another idea is to make a "universal light trap" for the second side reflection to be used on a wide variety of witness substrate indices. If a universal light trap glass were made from BaF_3 glass with index 1.583, for example, it could be used with witness parts with index 1.45 through 1.73 as seen in Fig. 4 without total second side reflection of more than 0.10% at the extreme indices (and much less for substrates with index nearer 1.583). BaF_3 glass is suggested here because it has the best durability properties and lower dispersion as compared with other glasses near that index. The trap could be a wedge, black paint, or any other type of trap made

from 1.583 index glass. The index matching fluid would be chosen as closest to the square root of the product of the substrate index and the 1.583 index of the glass from a set of 19 fluids from Cargille [4] in 0.01 index increments from 1.460 to 1.640. The %R from each interface with the fluid would be approximately equal, and they would be as low as practically possible via this configuration choice. The trap would be oiled to the witness and the %R spectrum would be measured. If the best possible estimate of %R is needed, the measured values could be further reduced by %R counts from Fig. 4 for the specific substrate index involved.

5. Conclusion

It appears that many organizations in the industry might benefit from the procedures described here, i.e., to paint the polished second side of a reflection witness chip or oil it to a universal reflection trap instead of roughening the side. As the requirement for AR coatings become more demanding, these or related techniques may become essential.

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