Low reflectance measurements using the ‘VW’ technique

Application Note

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Introduction

Thin film anti-reflection (AR) coatings are designed to greatly reduce the light loss through the utilization of phase changes and the dependence of reflectivity on index of refraction. Using AR coating technology, a single coating can eliminate reflection at one wavelength, or a multi-layer coating can reduce reflections losses over a wider spectral range.

Due to their very nature, accurate measurement of the reflectance of AR coatings can prove challenging to say the least. Traditionally, such measurements have been difficult and time consuming to make using the double-reflection ‘VW’ technique. Single-reflection ‘VN’ based measurement methods have thus generally taken precedence regardless of their inherent limitations.

The ‘VW’ absolute specular reflectance accessory (SRA; Figure 1) is designed to measure ‘mirror-like’ reflectance from a sample surface, and has been described elsewhere[1]. The accessory uses a modification of the ‘VW’ configuration first described by Strong[2], which calculates absolute specular reflectance using a pair of matched mirrors to perform the calibration and measure the sample reflectance. The ‘VW’ absolute SRA described eliminates the need for expensive, perfectly matched reference mirrors by using one movable mirror for both the calibration and sample reflectance measurements.

The measurement of small and/or low reflectance samples requires a 'known mirror' correction (available as standard within the Cary WinUV software). The 'known mirror' technique involves the use of a high-reflectance reference mirror to reflect the first reflection of the beam onto the sample (which is positioned alongside it). The reflectance of the reference mirror is determined prior to measuring the sample(s), and the reflectance of the sample(s) can then be quickly and easily determined by correcting (in real time) for the 'known' reflectance of the reference mirror.

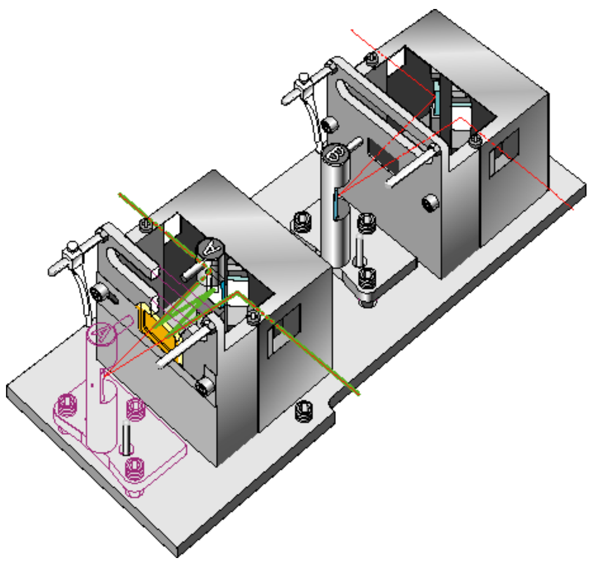

Figure 1. Optical diagram of the Cary VW absolute specular reflectance accessory

Agilent offers two high-performance UV-Vis-NIR spectrophotometers compatible with the 'VW' absolute SRA—the Cary 5000 and Cary 6000i. The former utilizes a lead sulphide (PbS) photocell for detection in the NIR, whilst the latter employs a state-of-the-art indium gallium arsenide (InGaAs) photodiode offering 100 times greater sensitivity than PbS.

The PbS photocell has a number of properties that make it suitable for use in the NIR, a wide operating range chief amongst them (750–3300 nm). Unfortunately, PbS also suffers from a number of limitations which are undesirable from a spectrophotometric point-of-view. Chief amongst these is linearity, noise/sensitivity, and detector homogeneity/spatial uniformity. Until recently, there has been little in the way of alternatives available.

Driven largely by consumer demand, recent years have borne witness to rapid technological advances in the telecommunications industry. Given the electromagnetic wavelengths used by the industry, these advances have included significant improvement in the area of NIR signal detection and detector technology. Not surprisingly, these advances in detector technology are equally applicable to NIR spectrophotometry. As such, an InGaAs UV-Vis-NIR spectrophotometer offers a number of significant performance advantages over its traditional PbS counterpart. Areas of specific interest include sensitivity, resolution, linearity and dynamic range—parameters of great importance to all practicing spectroscopists. Together, these performance improvements equate to better quality spectra (higher resolution and less noise), reduced analysis times and increased productivity. The Cary 6000i was used to collect all the spectra included in this report.

In this instance, the 'VW' absolute specular reflectance technique was used to determine the reflectance of AR-coatings on various substrates. Using a high-performance and high-sensitivity spectrophotometer, a robust 'VW' accessory and known mirror correction, high-quality spectra were obtained quickly, with reflectance values of the order 0.2–0.02% R.

Instrumentation[3]

- Agilent Cary 6000i UV-Vis-NIR spectrophotometer
- 'VW' Absolute Specular Reflectance Accessory
- Extended Sample Compartment

Conditions

The 'VW' SRA was installed into the spectrophotometer and aligned[4]. UV-Vis-NIR spectra were, in general, acquired in the region from 200–1800 nm using appropriate baseline correction with a scan rate of 60 nm/min. The indicative instrumental parameters are shown in Table 1.

Table 1. Operational parameters

Condition	Setting
SBW (UV-Vis)	2 nm
Sample averaging time (UV-Vis)	1 s
Data interval (UV-Vis)	1 nm
Energy (UV-Vis)	3
Sample averaging time (NIR)	1 s
Data interval (NIR)	1 nm
Mode	Double beam
Slit height	Reduced
Pump speed	15 rpm

Near-normal (7°), absolute reflectance measurements were made using the 'VW' specular reflectance accessory (SRA). In each instance, the sample was positioned to the right of the reference mirror using a standard sample clip (supplied with the 'VW' accessory). The reference mirror was held in a similar manner.

'Known mirror' baseline correction was performed prior to the acquisition of sample spectra. This involved the acquisition of three baseline scans in order to: i) set 100% T; ii) set 0% T (particularly important when measuring samples with low reflectance); and, iii) provide the reference spectrum which forms the basis of the correction.

Discussion

Reference spectrum

The reflectance spectrum of the reference mirror was determined prior to sample measurement (Figure 2). A 'representative' full wavelength range spectrum of one of the samples (SF11 A) was also acquired (Figure 2), with the regions of very low reflectance around 532 and 1064 nm clearly evident.

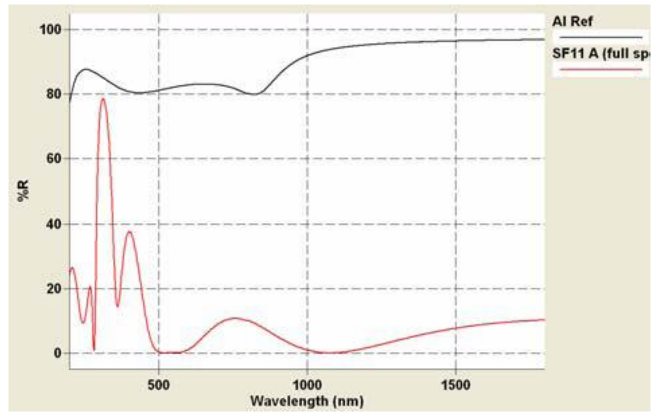

Figure 2. Absolute specular reflectance of quartz over-coated aluminum reference mirror and sample 'SF11A' (collected at 600 nm/min)

Near-normal reflectance measurements of low-R samples

Absolute specular reflectance spectra of all the samples measured can be seen in Figure 3. Using a scanning speed of 60 nm/min, each spectrum required less than 3 minutes to acquire. Based on the observed signal-to-noise level, a faster scan speed could be used without sacrificing spectral quality. No smoothing of any kind was applied to the spectra reported.

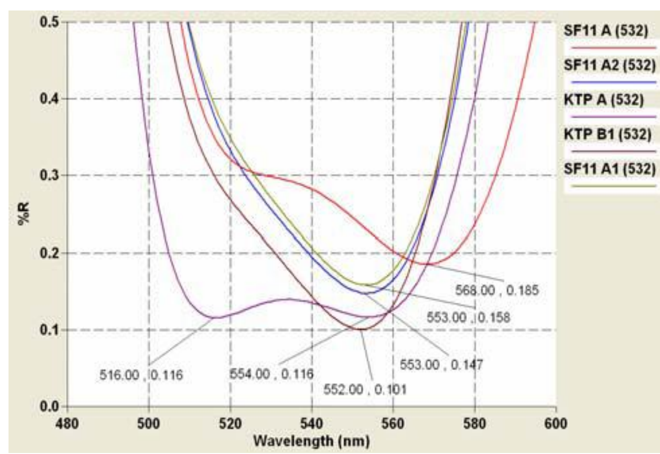

Figure 3. Absolute specular reflectance spectra of low-R samples using 'known mirror' correction (Top–visible region; bottom–NIR region)

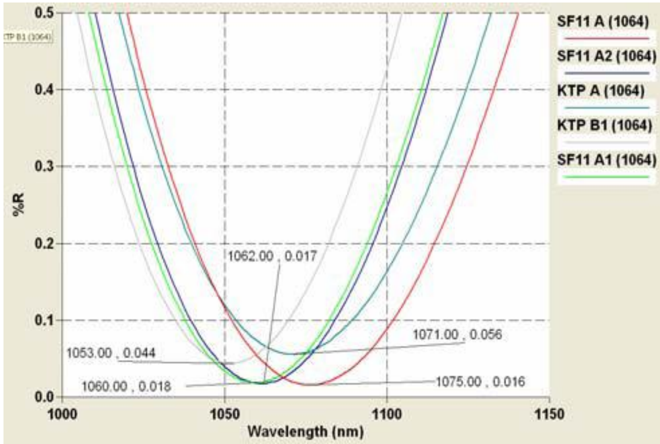

Figure 3. continued from previous page

The results obtained demonstrate the efficacy of the 'VW'technique for the measurement of low-reflectance samples. Compared to the single-reflection 'VN' technique, the 'VW'provides a more robust and less alignment susceptible means of measurement. 'VN' accessories are known to 'flip' the beam orientation between the sample and reference measurements, which makes them very susceptible to accessory stability and alignment, spectrometer alignment, variation in source intensity, and variation in detector sensitivity across the detector surface. In contrast, the 'VW' method does not flip the beam and is thus not susceptible to the same problems. The 'VW' technique is thus accepted to be the more robust (and accurate) of the two. The only major limitation of the 'VW' technique is that it requires a larger sample surface due to the two reflections from the sample surface. However, as described above, this can be easily overcome through the use of 'known mirror' correction

Conclusion

The efficacy of Agilent's Cary 6000i UV-Vis-NIR spectrophotometer has been demonstrated for high-performance, high-precision optical measurements.

Near-normal reflection measurements of low reflectance samples have been easily (and quickly) made using the 'VW' technique with 'known mirror' correction. Furthermore, the maximum scan time for any of the spectra presented was less than 3 minutes, with half the scans requiring less than this. This clearly demonstrates one of the major advantages of InGaAs over PbS speed. Reduced noise, higher light throughput, greater sensitivity, and better resolution combine to provide faster acquisition times than have traditionally been achievable with PbS. This in turn equates to reduced cost of analysis and greater productivity.

References

1. Hind, A.R. and Soebekti R., 'The deep ultraviolet spectroscopic properties of a next-generation photoresist', UV Application Note 82, www.agilent.com.

2. Strong, J., 'Procedures in Experimental Physics', 1st Ed., Prentice-Hall, Inc., New York, 1938, 376.

3. Part numbers :

Product	Part Number
Agilent Cary 6000i UV-Vis-NIR Spectrophotometer	0010079400
'VW' Specular Reflectance Accessory	0010043800
Extended Sample Compartment	0010079800
Cary WinUV Analysis Pack Software	8510195000

4. Cary WinUV Software, 'Cary Help' online help, Version 3.0

www.agilent.com/chem

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