

Evaluation of the Cary Absolute Specular Reflectance accessory for the measurement of optical constants of thin films

Application Note

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Introduction

The optical constants of thin absorbing films can be determined using photometry, in which the transmittance (T) and the reflectance from the front (R) and rear (R^1) of the substrate-film system are measured as a function of the wavelength of incident unpolarized light. This information and the film thickness (t) can be used to obtain the dispersion, $n(\lambda)$ and the extinction coefficient, $k(\lambda)$. From these a variety of useful quantities, such as the dielectric function and absorption coefficient can be calculated.

The method of determining the optical constants from photometric data which adequately overcomes the difficulties of missing or multiple solutions is discussed in detail by McPhedran et al.⁽¹⁾

Using a Cary 2400 spectrophotometer, we routinely measure R , R^1 and T for wavelengths between 200 nm and 3000 nm (the instrument has a range of 185 nm to 3152 nm).

To measure film and substrate transmittance is straightforward: the baseline is recorded, the instrument is zeroed, then the slide is placed in the sample beam and the resultant spectrum is recorded. The effect of finite substrate thickness is removed by calculation⁽¹⁾, assuming that the optical properties of the substrate material are known. For transmittance measurements, it is not our usual practice to place an uncoated slide in the reference beam.



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The calculation of optical constants assumes that the film is of homogeneous composition and uniform thickness and the films we measure approximate this ideal well. This means that the reflectance is predominantly specular and so we do not normally require an integrating reflectance accessory which would limit the wavelength range and reduce the signal to noise ratio.

Recently a new Cary absolute specular reflectance accessory (SRA) was released. The design was evaluated in our laboratory and this paper is a preliminary report on the results.

Figure 1 is an optical schematic of the SRA. Note that:

- i. The sample beam undergoes three reflections from aluminum-coated mirrors in the reference, or "V", position;
- ii. The reference beam symmetrically undergoes three reflections.

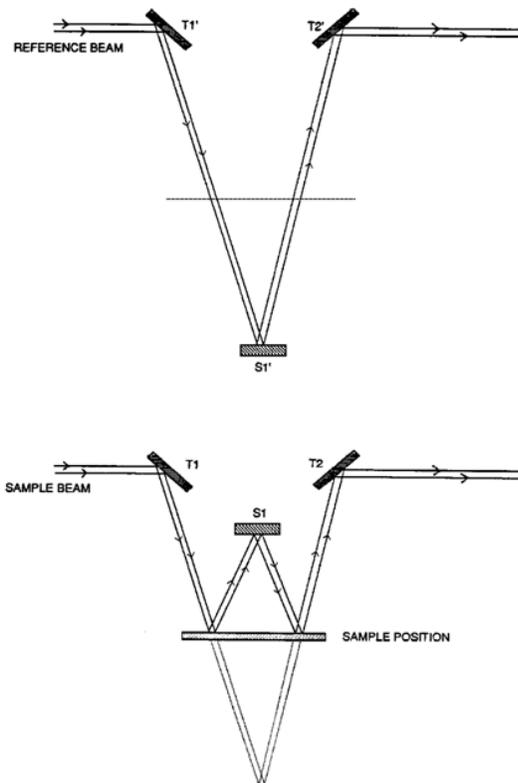


Figure 1. The optical design of the Cary absolute specular reflectance accessory (SRA)

Aluminum is used as a coating for the SRA mirrors because it has a uniformly high reflectance in the UV-Vis-NIR. Despite this, the light losses can be quite large after several reflections, particularly around 820 nm where the aluminum absorbs significantly. The reflectivity of the three mirror design is indicated in Figure 2.

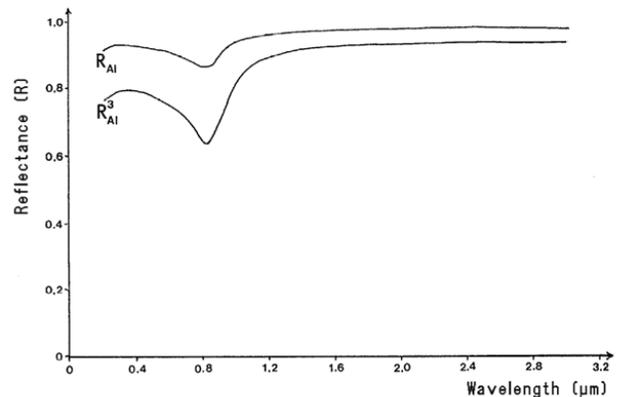


Figure 2. The cumulative reflectivity effects of aluminum surfaces

Optical performance

It is convenient to use polished wafers of single crystal silicon as a standard when assessing a reflectance instrument such as this one. Although evaporated metal films such as aluminum or silver have a higher reflectance, the value for such films can significantly depend on deposition and post-deposition conditions. Thus in practice it is preferable to use a single crystal material. Uncertainties deriving from the presence of an oxide layer and surface roughness have a major effect on the normal incidence reflectance only in the UV, so comparison with the literature is reasonable.

Figure 3 shows the reflectance of a mechanically polished <100> oriented silicon wafer as measured by the SRA, in the wavelength region around 800 nm. Also shown are the reflectance values determined by Aspnes and Studna⁽³⁾, from measurements using spectroscopic ellipsometry.

The region around 800 nm is problematic for two reasons:

1. The changeover from the photomultiplier to the PbS detector occurs at 800 nm. This means that both detectors are operating at the edge of their region of best performance. Hence, with low light signals, the signal to noise ratio and photometric linearity of the PbS detector in particular will be impaired.
2. This is also the region where the light loss due to absorption in the SRA mirrors is greatest, as discussed before and shown in Figure 2.

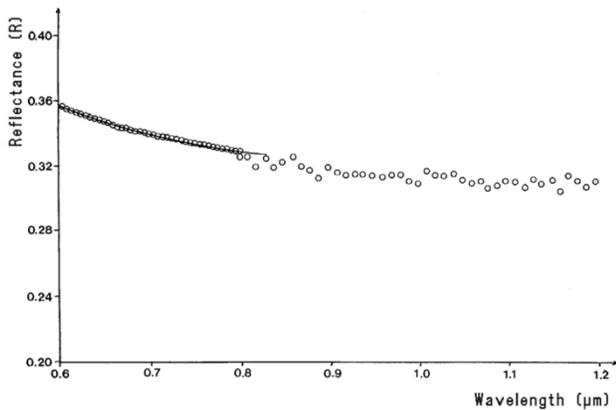


Figure 3. Reflectance of single crystal <100> silicon Circles represent data obtained using the SRA

Alignment tests

It is important to determine the effect of misalignment of the sample in a specular reflectance accessory. This was performed in two ways.

In the first instance, a B270 glass slide, 2 mm thick, with a freshly deposited aluminum film was rotated about a vertical axis by using shims inserted under either end. This geometry is shown in Figure 4.

Figure 5 shows the decrease in reflectance relative to zero rotation for both accessories. As is illustrated, measurements using the accessory are sensitive to rotation.

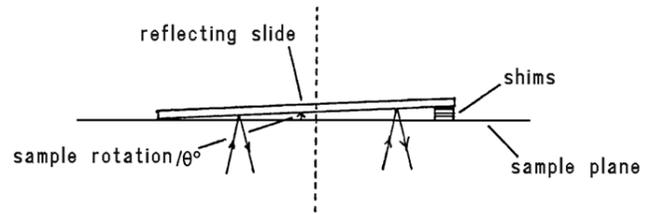


Figure 4. The geometry employed to determine the sensitivity of the reflectance to rotation of the sample

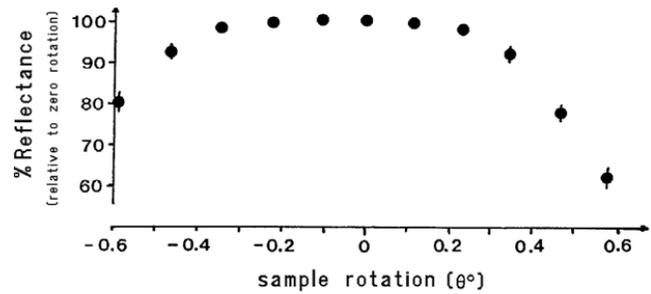


Figure 5. Reflectance of the slide as a function of the angle of rotation, relative to zero rotation

It should be noted that:

- i. The reflectance is unaffected by rotations of up to 0.2° in either direction.
- ii. The SRA can be readily aligned by the user, thus any asymmetry can be eliminated.

The second alignment test involved a displacement of the reflecting surface from the sample plane by varying amounts, as shown in Figure 6.

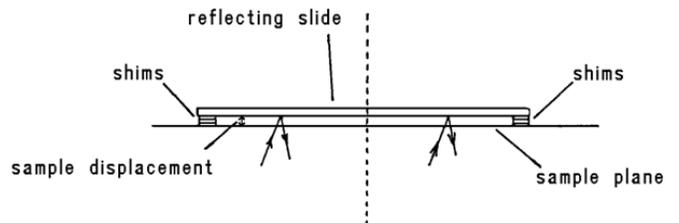


Figure 6. The geometry employed to determine the sensitivity of the reflectance to displacement of the sample away from the sample plane

This is particularly important in our work, because we have a need to measure the reflectance of thin films from the rear of the film, which results in a 0.5 mm displacement of the most reflecting interface from the sample plane.

The measured reflectance of the aluminum coated slide varied by less than 1.5% for displacements of the slide of up to 1 mm.

Conclusions

Preliminary tests on the new Cary SRA indicate that it has excellent signal to noise performance, particularly in the wavelength region where aluminum absorbs: above and near 800 nm.

The symmetry of the front and rear beam path means that it is possible to store baseline correction values over the entire UV-Vis-NIR range of wavelengths.

The sample mounting configuration offers two key advantages to the user:

- i. The ability to measure smaller samples.
- ii. The front loading of the sample ensures greater visibility and less risk of sample mismounting or damage.

The sensitivity to misalignment of sample is not likely to cause a problem and the mirror alignment can be easily carried out by the user.

References

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