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Application Note AN M12

Accurate Reflectance and Transmittance Measurements on Highly Refractive Materials

The accurate determination of absolute transmittance and reflectance values in the IR spectral range is a matter of increasing interest, e.g. for the development and specification of optical filters, mirrors or window glass, but also in several research fields. For both accurate reflectance and transmittance measurements, Bruker Optics provides dedicated accessories.

Fig. 1.

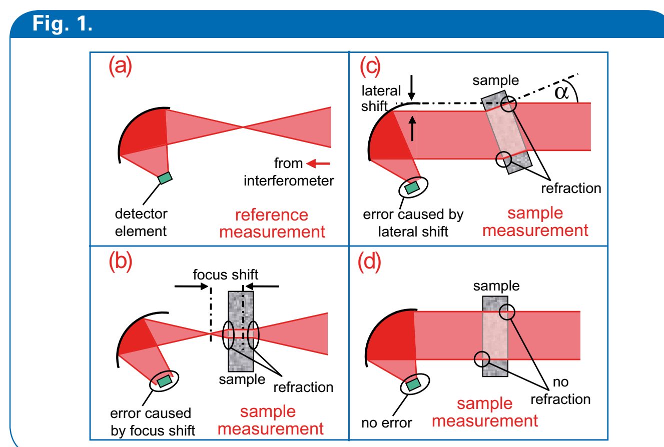

Fig. 1(a) and (b): The typical approach of standard transmission accessories and the associated error. 1 (c): oblique incidence causes an error due to a lateral beam shift. 1(d): Both error types can be overcome by using a parallel beam and perpendicular incidence.

The parallel beam transmission accessory A480

In standard transmission accessories the sample is usually placed in the focus of the IR-beam as indicated in fig. 1(a) and (b). Especially in the case of thick and highly refractive samples, refraction causes a shift of the IR-focus towards the detector, which is associated with an error in the transmittance values (see fig. 1(b)). For oblique incidence, refraction gives rise to an additional error caused by a lateral beam shift (fig. 1(c)). These inaccuracies can be overcome by measuring the sample in a parallel IR-beam with perpendicular incidence as suggested in fig. 1(d).

Fig. 2

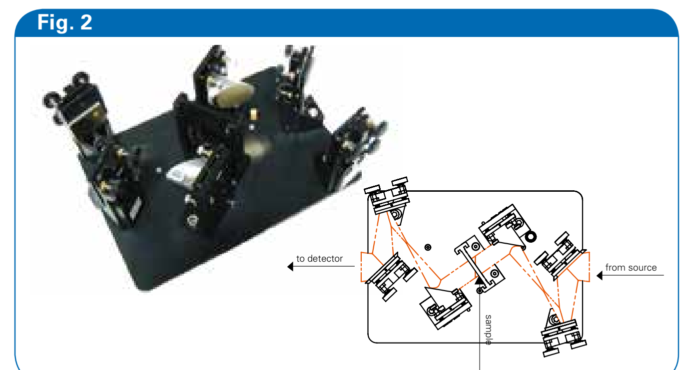

Fig. 2: Schematic beam path and photograph of the A480 parallel beam transmission accessory.

Exactly this concept is realized in the parallel beam transmission accessory A480, depicted in fig. 2. Especially for highly refractive (and thus reflective) samples (e.g. semiconductors) perpendicular incidence can create additional problems if no precautions are taken. IR light reflected by the sample, can lead to artefacts, caused by so-called double modulation. In Bruker Optics spectrometers such as the INVENIO R or the VERTEX 70v which base upon a RockSolid interferometer with cube corner mirrors, this problem is effectively solved by the insertion of a half-aperture (see fig. 3). But also in spectrometers using an interferometer with linear mirror movement and flat mirrors (e.g. the VERTEX80 series) the contribution of double modulation can be avoided. In this case the half of the intermediate focus is blocked by a knife edge aperture, resulting in accurate transmittance values as well (see accessory A480/8 for the VERTEX80 series).

Fig. 3

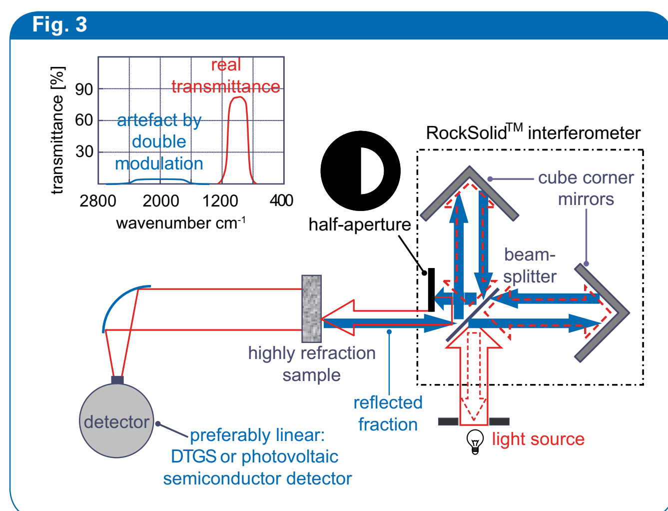

Fig. 3: Schematic illustration how doubly modulated light (blue arrows) is blocked by inserting a half-aperture in between RockSolid interferometer and sample compartment. Without precautions double modulation can cause artefacts such as the simulation in the upper left corner shows for the case of a bandpass filter.

As a first example fig. 4 shows the transmittance of a bandpass filter measured with the A480 accessory. Since outside of the spectral window between 2600-3000 cm^{-1} transmittance should vanish, this sample is well suited to check the accuracy in case of zero transmittance. The zero transmittance of the bandpass filter can be impressively reproduced with a deviation ≤ 0.01 % (fig. 4).

In order to verify the accuracy of the A480 accessory regarding absolute transmittance values different from zero, one needs a sample the real transmittance of which is known as exactly as possible. For this purpose we chose the semiconductor Germanium: the refraction index is experimentally well determined to be $n_{Ge} \approx 4.003$ at 1000 cm^{-1} [1, 2]. Using the Fresnel equations including multiple internal reflections, this corresponds to a theoretical transmittance

Fig. 4, 5 and Fig. 6

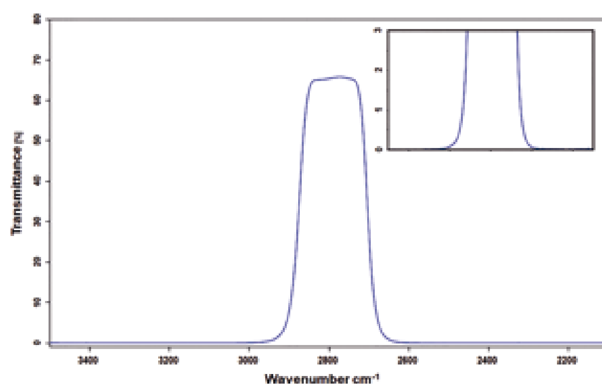

Fig. 4: Transmittance of a bandpass filter, measured with the A480 accessory, using the INVENIO R and a DTGS detector. As can be seen in the inset, outside of the transmission range the nominal value of zero is well reached.

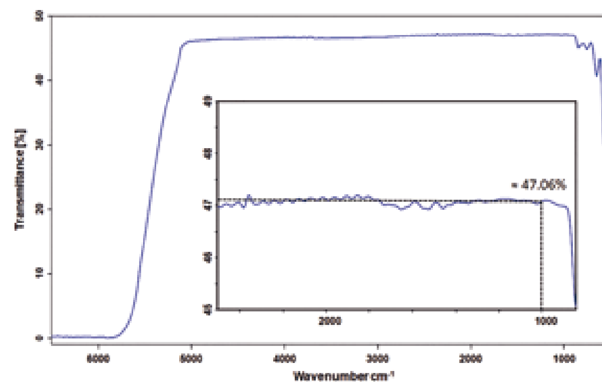

Fig. 5: Transmittance of a double sided polished Germanium sample, measured with the A480 accessory, using the INVENIO R and a DTGS detector. Because the sample is quite thick (6 mm), no interference fringes appear for the applied spectral resolution of 4 cm^{-1}. The inset shows that in the mid infrared (1000 cm^{-1}) the theoretical transmittance is virtually reproduced.

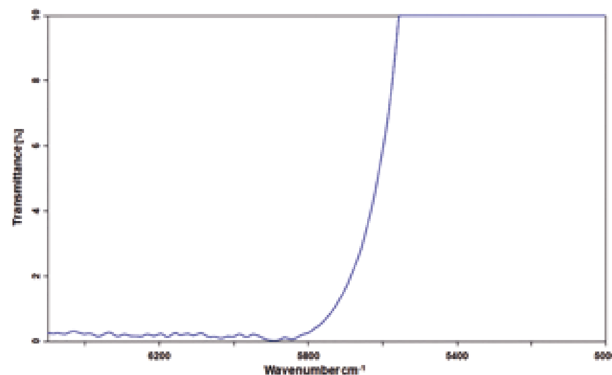

Fig. 6: Transmittance of Germanium, measured with the A480 accessory, using the INVENIO R. As predicted by theory the transmittance above the bandgap is found to be zero with a deviation < 0.3 %.

of ≈ 47.03 % at 1000 cm^{-1}. The experimental results for Germanium (depicted in fig. 5) agree well with these predictions.

Above the Germanium bandgap at ≈ 5800 cm^{-1} the transmittance should be zero. Once again this is experimentally well confirmed as shown in fig. 6.

The absolute reflectance accessory A519

In standard reflection accessories the sample reflectance is usually determined as the ratio of the single channel spectrum reflected by the sample and the single channel spectrum reflected by a reference mirror. This procedure is adequate for many applications, since all prominent spectral features will appear, but concerning absolute reflectance values one has to accept a certain error. The resulting absolute values would only be correct if the reflectance of the reference mirror was exactly 100 %. In fact the real reflectance of typical (gold coated) mirrors is rather in the order of 99 % and even shows a weak (flat) spectral dependence. Further deviations can occur due to scratches or adsorbed surface species (resp. dust). In general there are two approaches to avoid this source of error:
First, using a calibrated reference mirror, the error can be eliminated by accounting for the real mirror reflectance. This procedure can even be automated with a suitable OPUS macro. However, many users dislike this approach because calibrated mirrors are costly and have to be handled with extreme care in order to avoid subsequent reflectance changes. If accurate absolute values are of prime importance, the mirror should even be regularly recalibrated, to observe possible reflectance changes on longer timescales.

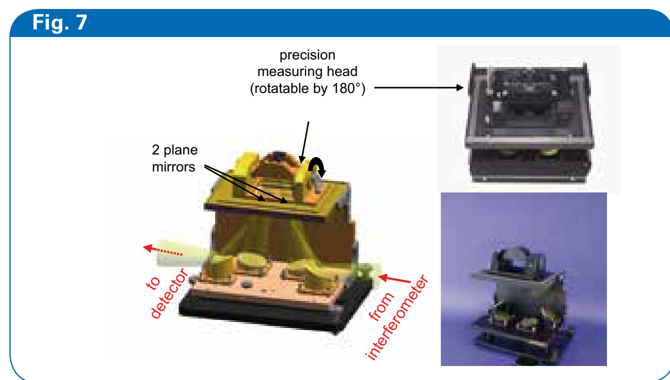

Fig. 7: Schematic beam path and photograph of the A519 absolute reflectance accessory."

To overcome the above mentioned uncertainties, Bruker Optics developed the absolute reflectance accessory A519 (see fig. 7) which does not rely on the usage of a calibrated mirror.

This is possible because the reference mirror is also part of the beam path for the sample measurement. Therefore, by taking the ratio of sample and reference measurement, the reflectance of the mirror is eliminated and does not influence the result. The accessory can be switched between two positions for reference and sample measurement with a high precision measuring head which is rotatable by 180° and includes the reference mirror.

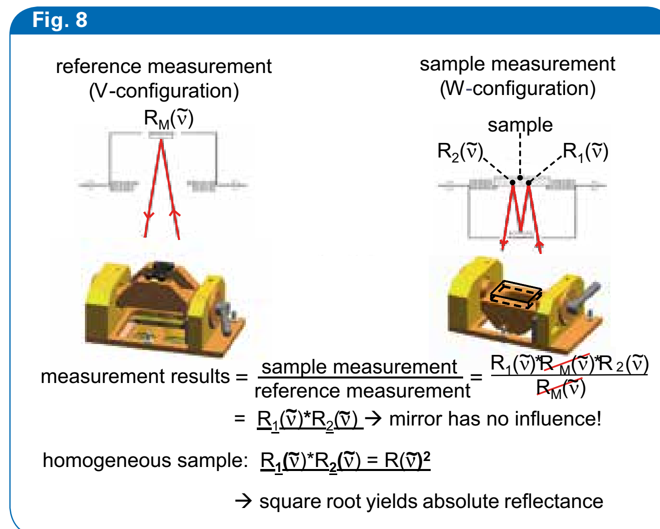

Fig. 8: Principle of the absolute reflectance accessory A519. Because the reference mirror (reflectance R_M) is part of the beam path in both reference and sample measurement, it has no influence on the result R^2 (respectively $R_1 * R_2$ for inhomogeneous samples).

As depicted in fig. 8, for the sample measurement the infrared beam is reflected twice at two different sample spots (distance 22 mm; angle of incidence: 11°). For homogeneous samples the result is just the square R^2 of the sample reflectance. The absolute reflectance is then simply determined by computing the square root of the measurement result via software. For inhomogeneous samples this procedure yields the average absolute reflectance $R_{1/2}$.
In order to check the accuracy of the A519 accessory we used another semiconductor, namely Silicon. Using a refraction index of n_{Si} = 3.417 [2] the theoretical reflectance of Silicon should be ≈ 46.09 % at 2000 cm^{-1}.

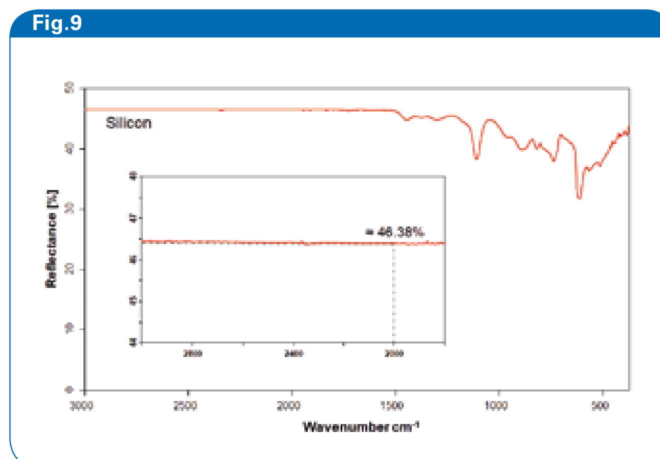

Fig. 9: Absolute reflectance of a thick, double sided polished Silicon sample, measured with the A519 accessory, using the INVENIO R and a DTGS detector.

Fig. 9 shows the absolute reflectance of a Silicon sample, determined with the A519 accessory. The observed small deviation of ≈ 0.3 % is not necessarily a measurement error but could also result from a slight error in the used refraction index. Already increasing n_{Si} by 0.02 to $n_{Si}=3.44$ would result in a theoretical reflectance of 46.4 %. A useful test method which is independent from the published refraction index and even combines the two discussed accessories A480 and A519 is as follows: It is well known from semiconductor physics that above 1800 cm^{-1} Silicon does not absorb at all because the infrared active lattice vibrations are all situated lower in energy. Since the investigated sample had a high surface quality, scattering losses can be neglected and therefore the sum of transmittance and reflectance should be 100 %. Fig. 10 gives an overview of Si transmittance measured with A480, Si reflectance measured with A519 and the sum of both. Finally fig. 11 shows that in the non absorbance region above 1800 cm^{-1} the sum of reflectance and transmittance is very close to the theoretical value of 100 %.

In general the accuracy of both discussed accessories depends on the investigated sample. As can be seen on the examples, by using optimized components, typical accuracies better than 0.25 % can be reached.

References:

[1] "Handbook of Optics", 2nd Edition, Vol. II (McGraw-Hill,1995)
[2] Online database of "Korth Kristalle GmbH"; www.korth.de

Fig. 10 and Fig. 11

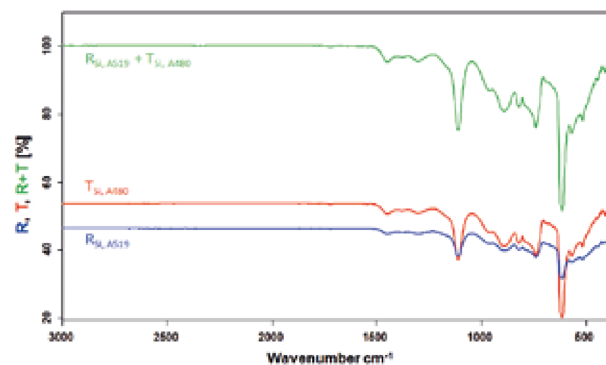

Fig. 10: Si Reflectance (blue; measured with A519), Si transmittance (red; measured with A480) and the sum of both (green). All spectra were recorded using a INVENIO R and a DTGS detector.

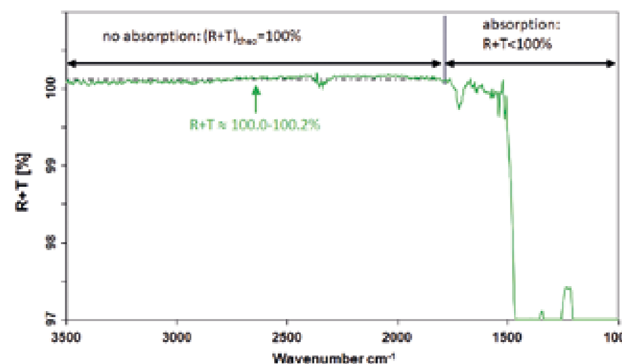

Fig. 11: Sum of Silicon transmittance and reflectance measured with both of the discussed high precision accessories, using a INVENIO R and a DTGS detector. In the non absorbance region above 1800 cm^{-1} the theoretical value of 100 % is reached in very good approximation.

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