Problems posed by scattering transmissive materials for accurate transmittance and reflectance measurements

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ABSTRACT

The characterization of the spectral transmittance and reflectance of windows and other optical components is a basic and important measurement. In principal, the measurements are relatively straightforward. However, even with an ideal high-accuracy measurement system, the sample's scattering properties can render the measurement results inaccurate or easily misinterpreted. The effects of low levels of scatter from specular transmissive samples on optical property measurements are demonstrated in the infrared with specular and hemispherical detection instrumentation. Complete measurement of the reflected, transmitted, and scattered light from these samples is demonstrated in the infrared using a center-mount integrating sphere.

1. INTRODUCTION

The characterization of the specular or regular transmittance and reflectance of samples is generally performed using light that is incident within a narrow angular range and then collected over a similar range. It is generally assumed that the samples do not scatter significantly outside the output collection range (by significant we mean greater or equal to that represented by the uncertainty of the measurement). When samples that scatter a significant amount are measured, then most absolute and relative measurement devices will only record the 'specular' component of the reflected or transmitted light. If one requires the total reflectance or transmittance, for example to determine the absorptance or emittance of the material, then the results will be in error. Furthermore, some samples, due to inherent characteristics from the fabrication process (e.g. in growth or in polishing), will unavoidably exhibit some scatter, even though their prime function is as a regular transmitting or reflecting element. Over a broad spectral range the amount of scatter may vary considerably due to wavelength dependence.

The effects described in this paper have been observed before for a different spectral region.[1,2] Attempts have been made to deal with the problems of scatter, as in the case of haze transmittance measurements on transparent plastics, for which an ASTM method was developed.[3] However, it is easy to find many discussions of transmittance and reflectance measurements of transmissive materials, some of which deal with scattering, including review papers and book chapters, which do not contain any reference to or hint of the problems demonstrated and discussed herein. There are so many that it would not be fair to single out any particular paper(s) for criticism. Even the ASTM method does not specifically mention the problems we describe, but attempts to deal with them by specifying elements of the measurement geometry in great detail to foster consistency and agreement between instruments. Rather, it is the purpose of this paper is to remind the reader about the issue of scatter and the problems surrounding it.

For accurate characterization there is a need for (a) a general practice or check of the level of scatter in samples, and (b) for measurement instrumentation designed to characterize and account for the scattered light in transmittance and reflectance measurement situations. Instrumentation such as total integrated scatter (TIS), integrating spheres, and bi-directional scatter distribution function (BRDF) instruments, can be used to help in the evaluation of sample scatter and distinguishing it from absorption.

We examine the problems posed by samples which are not diffusers, but scatter to a degree which is significant for accurate transmittance and / or reflectance measurement. Two types of scattering transmissive samples are illustrated by example in Section 2: a volume scattering sample (ZnS) and a surface scattering sample (Si, polished on one side). A system for measuring all components of the scattered light is described in Section 3, and demonstrated for the ZnS and Si samples.

Part of the SPIE Conference on Optical Diagnostic Methods for Inorganic Transmissive Materials
San Diego, California • July 1998
SPIE Vol. 3425 • 0277-786X/98/$10.00

2. MEASUREMENT EXAMPLES OF SAMPLES WITH SCATTERING

The instrumentation used to perform the specular and directional hemispherical measurements is described elsewhere in detail.[4,5,6] It consists of a Fourier Transform (FT) Spectrophotometer and an integrating sphere accessory (with sample wall-mount) designed for both regular (specular) and directional hemispherical absolute reflectance (DHR) and transmittance (DHT) measurements. The beam geometry is 8° incidence with an f/5 cone, with additional capabilities of normal incidence for sorting out regular from diffuse components. The detector used for the results presented here is a LN_2-cooled Hg:Cd:Te (MCT) detector with a cut off at 18 μm. The absolute calibration for the measurement of the diffuse components of the reflected and transmitted light is described in Reference 5. The estimated expanded uncertainty for specular measurements is 0.003 and for the DH (2π) measurements is 0.015.

Views of the reflected and transmitted light flux that contributes to the reflectance and transmittance measurement of (a) specular and (b) diffuse samples are shown in Figure 1. For transmitting specular samples, a collection of the transmitted and reflected flux requires a detector positioned at an appropriate angle and sized to collect all the multiply reflected components as shown in Figure 1 (a). For a diffuse transmitting sample with a characteristic scattering length much less than the sample width and on the order of or greater than the sample thickness, a hemispherical collection of the light reflected or transmitted in Figure 1 (b) is required. Most instrumentation is designed to accurately perform measurements for samples of the types shown in Figure 1. The results of measurements of the flux components shown in Figure 1 (a) and (b) are described in this Section.

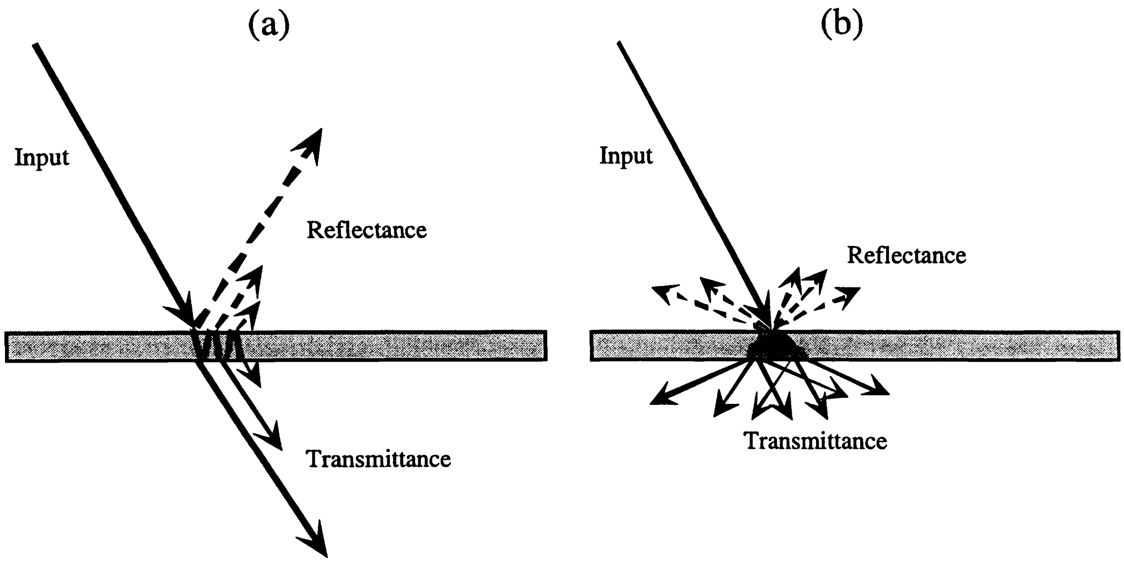

Figure 1. Light collected in reflectance and transmittance measurement geometries for (a) specular and (b) diffuse samples.

In the infrared a number of window and optical component materials are used which exhibit some scatter at shorter wavelengths. They may have other advantages such as spectral coverage and stability to various environments. First we examine a material which exhibits volume scattering: ZnS. The measured specular transmittance and reflectance of a ZnS 38 mm diameter by 3 mm thick window are shown in Figure 2. Note the increasing transmittance and reflectance with wavelength. This is a characteristic signature of scattered light loss.

Given these results, a logical step is to measure the directional hemispherical properties of the sample using an integrating sphere or conic mirror device. The results of such measurements are also shown in Figure 1 labeled with '2π' (referring to hemispherical collection). At longer wavelengths the specular and 2π results merge indicating the absence of scatter. The ZnS sample, then, is a diffuser at one end of the spectrum and a specular material at the other. Except for structure at 6 μm, the DHR is fairly flat below the absorption edge, whereas the DHT exhibits a slight increase with wavelength, indicative of some residual scatter loss. This can be seen more clearly in Figure 3.

The indirectly measured absorptance results, obtained by subtracting the sum of the transmittance and reflectance from 1, are shown in Figure 3. The results are a measure of the true absorptance in the absence of scatter. However in the presence of scatter additional information is required to obtain the correct absorptance. From the curves in Figure 3 it can be seen that the integrating sphere measurement accounts for most, but not all of the reflected and transmitted light. At shorter wavelengths there is an increasing amount of flux that is unaccounted for (i.e. non-zero absorptance).

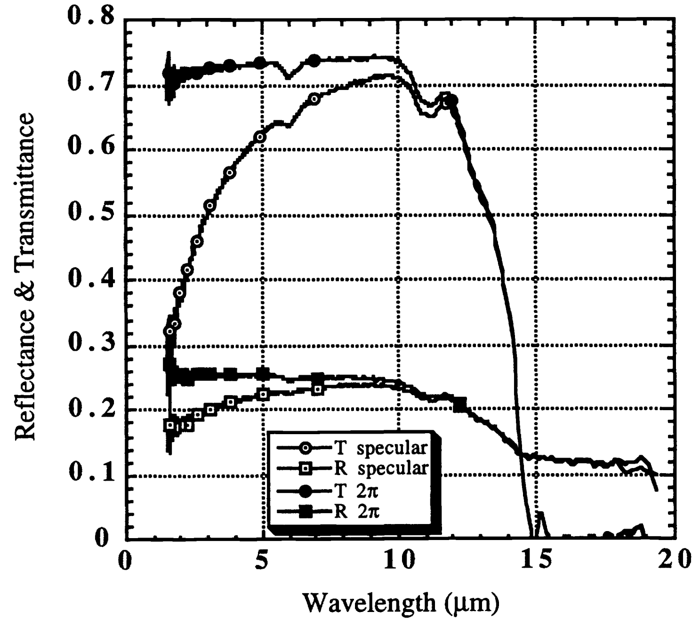

Figure 2. Specular and diffuse (2π) transmittance and reflectance measurements of a ZnS window.

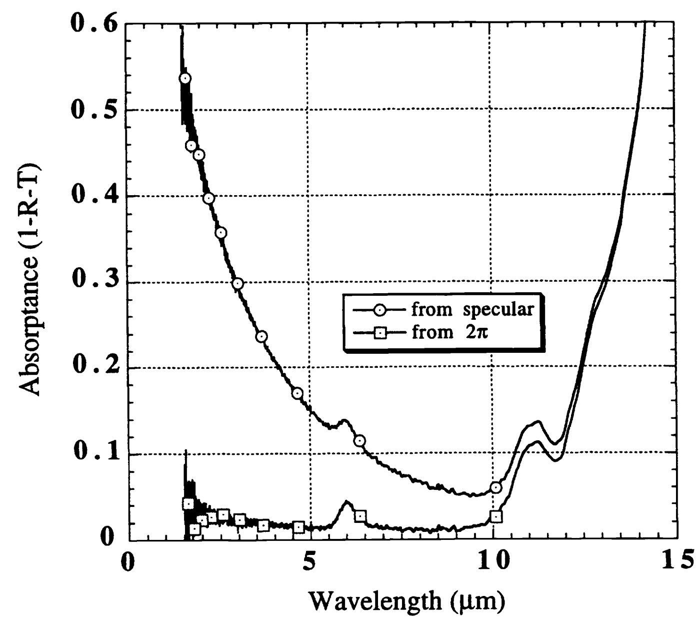

Figure 3. Absorptance of a ZnS window calculated from specular and diffuse (2π) transmittance and reflectance.

Another type of infrared window that scatters is one whose surface is imperfect to some degree. As an example of a surface scattering sample, we examine a single-side-polished Si wafer, 25 mm in diameter and 0.5 mm thick. The results of specular and diffuse reflectance and transmittance measurements are shown in Figure 4. The Si window is oriented with the smooth side facing the input beam in the reflectance measurement. At short wavelengths the specular transmittance is 0, but increases with wavelength as the scattered component is reduced. This means that the specular reflectance measurement includes only the front surface reflection at 2 μm, but contains an increasing contribution from the back surface reflections as the wavelength is increased.

The directional hemispherical reflectance and transmittance measurement results in Figure 4 are quite different in character from the specular results. Overall the results are much closer to, but still less than, what one would expect from a Si sample without scatter. Another feature of the 2π results is the increased depth of the absorption structure relative to a specular sample of the same thickness. This indicates greater effective path lengths (or 'thickness) through the sample due to the scatter.

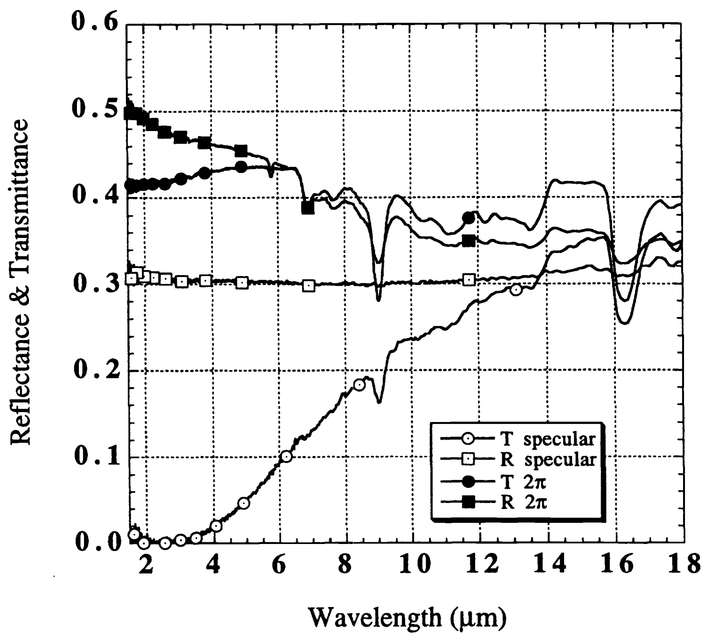

Figure 4. Specular and diffuse transmittance and reflectance measurements of a single-side polished Si wafer.

The indirectly measured absorptance of the Si sample from the specular and 2π measurement results is shown in Figure 5. As with the volume scatter ZnS result, the apparent absorptance that should be near 0 at shorter wavelengths[1] for a specular Si sample, decreases from a very large number at 2 μm to more reasonable values at 16 μm to 18 μm. Most of the absorptance structure beyond 6 μm that is apparent in the 2π measurement is only faintly discernable in the specular result. Yet, as in the case of the ZnS window, the 2π result has a significant (≥ 0.10) component of the total flux unaccounted for below 6.5 μm. (i.e. non-zero apparent absorptance).

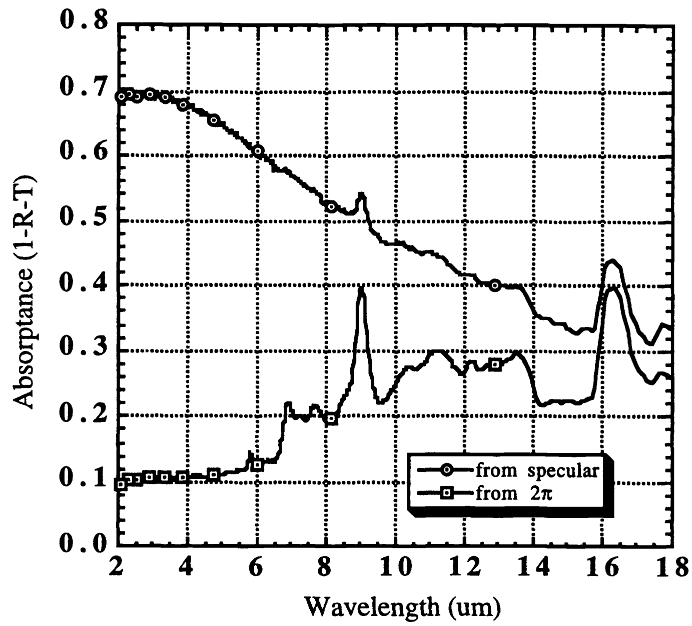

Figure 5. Absorptance of a Si wafer calculated from specular and diffuse (2π) transmittance and reflectance measurement results.

3. TOTAL SCATTER (4π) MEASUREMENT

After characterization of a number of nearly specular transmissive samples with low or moderate levels of scattering, it becomes apparent that neither specular nor hemispherical (2π) measurements are sufficient to collect all the flux not absorbed in the sample. In the case of strong scattering such as is shown in Figure 1 (b), if a sample is placed against a collecting port of an integrating sphere, then as long as the port size is larger than the input spot size, all the light, including the scattered component will be collected. With a short characteristic scatter length, light which reaches the edge of the port or sample will have undergone a large number of scattering events and have become an infinitesimal fraction of the total flux. However, when the characteristic scatter length is comparable to the dimensions of the sample, a single scatter event can result in light reaching the port or sample edge. This is illustrated in the diagrams of light passing through volume (a) and surface (b) scattering samples in Figure 6.

In the case of volume scatter, light scattered inside the sample will have components contributing to reflectance and transmittance measurement as well as components channeled to the sample edges both directly and indirectly via total internal reflection at the sample surfaces. Additional scatter events, as shown in Figure 6 (a), can result in light exiting the sample near the edges and possibly not being collected by a finite size port. Light leaving by the edges of a sample will not be collected in either specular or 2π measurement systems. Another problem posed by this situation is that light reflected back towards the center of the sample, or off the port surroundings or mounting hardware, can return to the central region of the sample and ultimately contribute to a specular or 2π measurement.

A similar situation occurs for samples with surface scattering, as shown in Figure 6 (b). Here the scatter events take place at the sample surface, but the results are nearly the same as for the low level volume scatter case. Again, after a scatter event some light can be trapped inside the sample and channeled to the outside edges. Secondary scatter events can result in light exiting the sample anywhere over its entire surface.

Many implications follow from these observations. For these types of samples or materials, the entire sample surface finish and shape becomes important, including the sample edges. The channeling effects are greater for higher index materials with correspondingly smaller critical angles inside the sample. Also, during measurement, the manner in which the samples are mounted and the optical properties and geometry of the materials used must be carefully considered. Finally, in order to obtain optical property information about the samples, ray tracing and modeling of the measurement system and sample may be required.

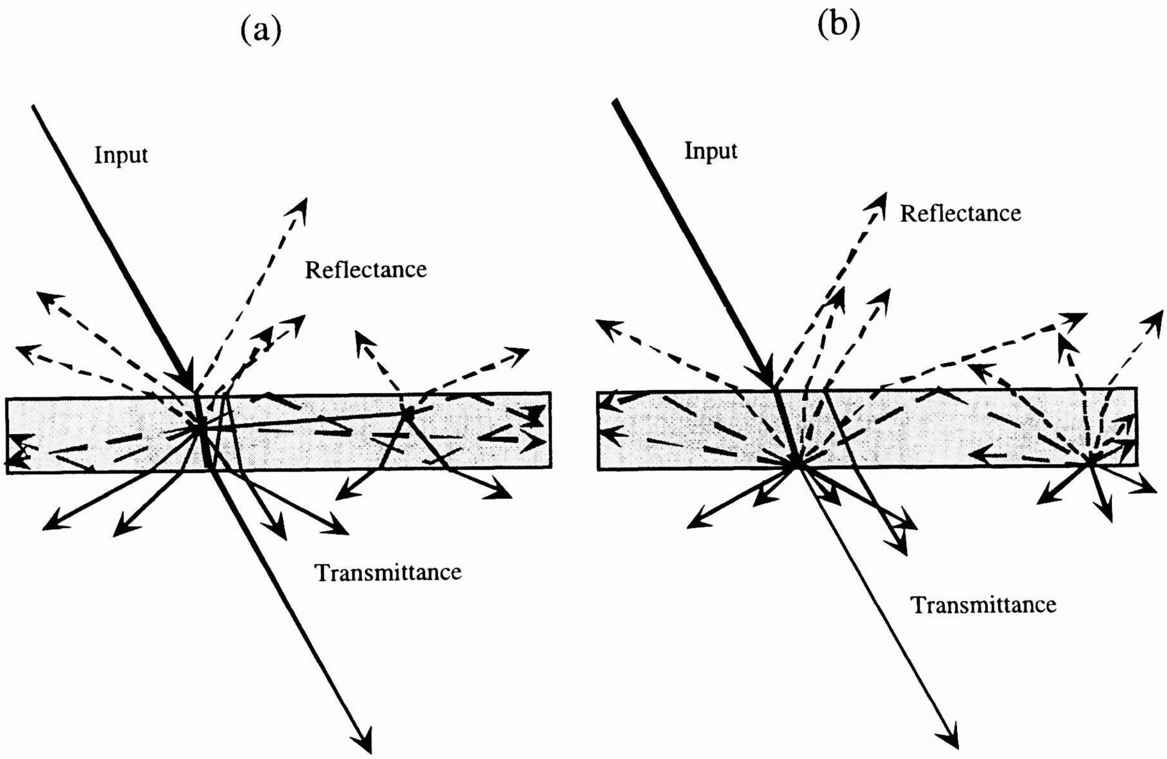

Figure 6. Effects of volume (a) and surface (b) scattering on reflected and transmitted light. The solid arrows indicate contributions to transmittance, short dashed arrows to reflectance, and long dashed arrows to light channeled to the sample edges.

Other measurement devices may prove useful in the analysis and characterization of the types of samples we have illustrated. These include BSDF (bi-directional scatter distribution function) measurement systems, which can completely characterize in detail the distribution of all the scattered light including that transmitted and reflected. Some modifications to particular instruments might be necessary to enable the detector to view all locations on the sample including the edges.

Another useful device that we have employed is a center-mount integrating sphere. Although primarily intended for the measurement of reflectance of opaque samples with an adjustable angle of incidence, it can also be used to measure the absorptance of transmissive materials. For transmissive materials, measurements with a center-mount sphere design can be described as '4π' measurements because all light leaving the sample, even from the edges, will be captured in the sphere and contribute to the measurement.

A schematic of the center-mount sphere used for the infrared is shown in Figure 7. The sphere is 20 cm in diameter and is coated with a SiC blasted aluminum surface, over-coated with gold.[7] Top (a) and side (b) views are shown. In the top view, the sample mount is held by a post to center the sample in the sphere. The post is mounted on a rotatable plug with which the angle of incidence on the sample can be varied. The sample mount for the measurements on the transmissive samples is a flat-jawed alligator clip covered with a diffuse gold coating. The relative surface area covered by the clamp is very small. The detector views the back of a baffle that blocks it from seeing the sample directly. View (b) is from the side, looking directly along the sample post towards the baffle. The specularly reflected and transmitted light are denoted by arrows. As indicated in Figure 7 (b), the sphere can be tilted to perform the reference measurement. In the sample measurement, both the reflected and transmitted light are measured together, so that the absorptance can be obtained by subtracting the results of a single measurement (ratioed by a reference measurement and corrected by calibration with a diffuse gold standard)[5] from 1.

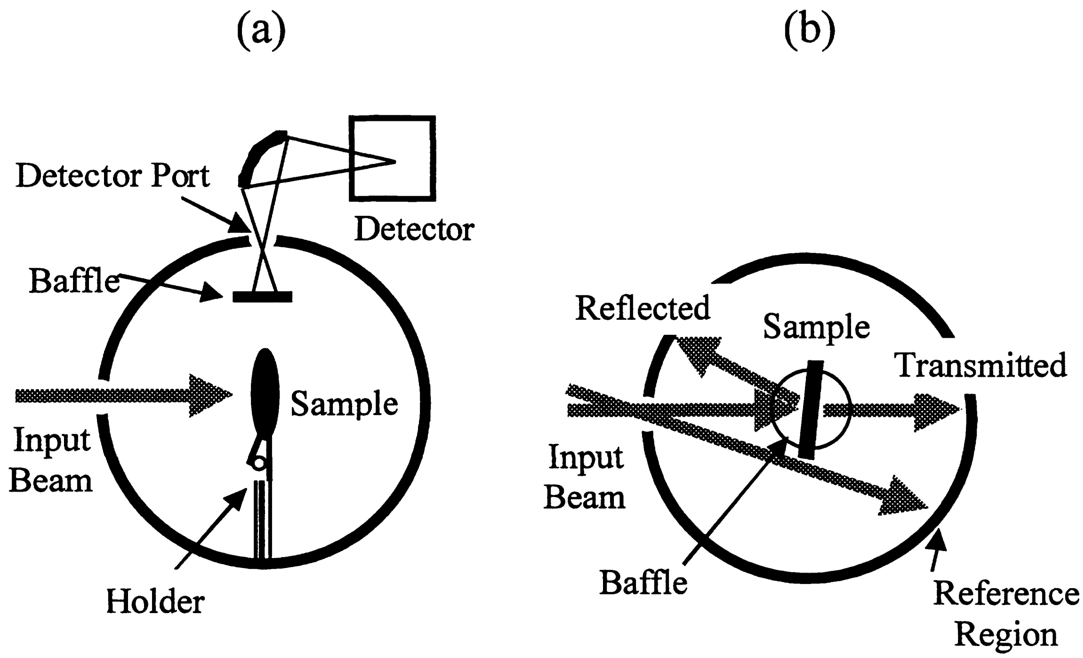

Figure 7. Top (a) and side (b) views of the center-mount integrating sphere for 4π total measurement.

The integrating sphere was used to perform 4π measurements on both the ZnS and single-side polished Si samples. The center-mount integrating sphere has not undergone an extensive characterization to determine correction coefficients for specular and diffuse components, which has been done with the wall-mount sphere used for the 2π measurements.[2] This includes entrance port loss for diffusely reflected light. For the wall-mount sphere, the port loss is approximately 1%; for the center-mount sphere the loss is approximately 4% (the port presents a solid angle 4 times larger in the center-mount case). Errors such as the entrance port loss are only indirectly corrected for by calibration with the standard. Hence, the estimated expanded uncertainty for the 4π measurements of absorptance is 0.05. Primarily, this is an uncertainty of the absolute baseline (0 absorptance level) of the measurement. Because of the potential variation of the amount of port loss and the variation in spatial uniformity of the sphere throughput,[5] the baseline could vary over a ±0.05 range. However, the relative uncertainty in comparing portions of the spectrum is much smaller. The detector used with the 4π sphere is a narrow band MCT that cuts off at 13 µm. Some fine structure below 7 µm in the spectra presented here is due to atmospheric absorption unrelated to the sample.

A comparison of the absorptance spectra obtained by the three measurement geometries (specular, 2π and 4π) of the ZnS sample is shown in Figure 8. At longer wavelengths, the 2π and 4π results match well. As shorter wavelengths are approached, the 4π absorptance curve is significantly closer to 0 than the 2π curve. The small absorption structure at 3 µm may be due to water on the sample or in the sphere. The dashed line is shown to indicate the baseline in the absence of the water structure. This comparison indicates that the 4π system nearly completely measures and accounts for all the light reflected, transmitted or scattered from the sample.

The Si sample was also measured using the 4π geometry center-mount integrating sphere. The resulting absorptance spectrum is compared with those obtained from the specular and 2π geometries in Figure 9. The 4π absorptance values at short wavelengths are within the measurement uncertainty of 0, the expected value. At longer wavelengths there is also agreement with the 2π geometry sphere results. Most of the structure due to atmospheric absorption

For the 4π geometry, the values of absorptance near 0 at the shortest wavelengths and the agreement with the 2π results at longer wavelengths, lead us to two conclusions: (a) that the light flux which is unaccounted for in the 2π results is not absorbed, but is scattered and leaves by or near the edges of the sample, and (b) that the 4π measurement system is capable of capturing all the light exiting from the sample. Further characterization of the center-mount sphere will reduce the measurement uncertainties and is expected to result in measured absorptance levels closer to 0 in the appropriate spectral regions for the examples shown.

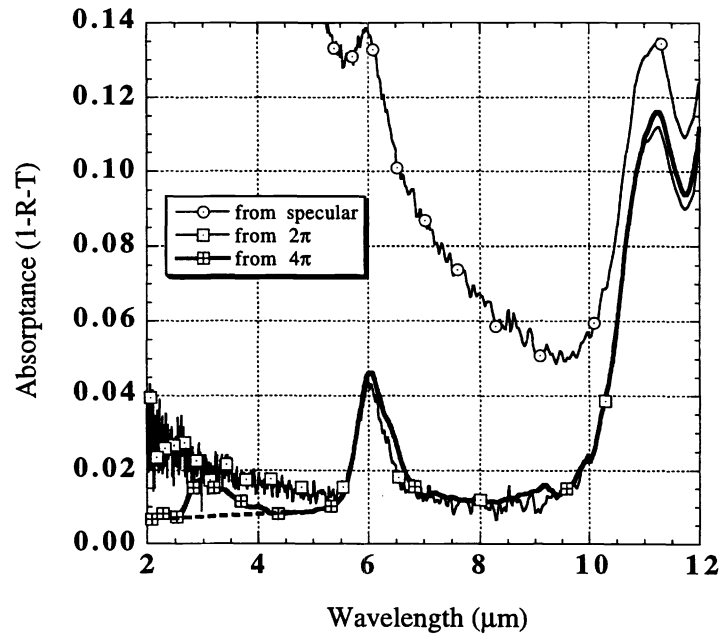

Figure 8. Comparison of absorptance spectra of a ZnS wafer, determined from specular, 2π and 4π measurement systems.

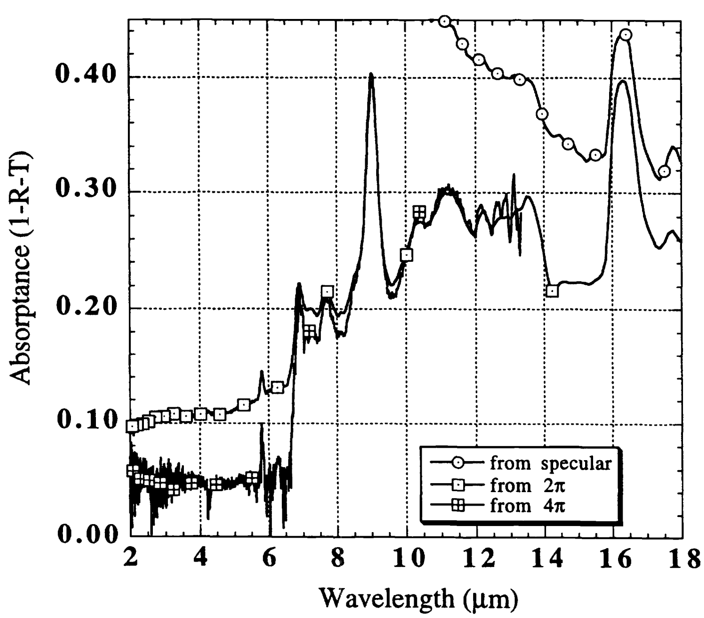

Figure 9. Comparison absorptance results of Si wafer determined from specular, 2π and 4π measurement systems.

4. CONCLUSIONS

The reader is reminded of the importance of evaluating samples, intended for specular reflectance and transmittance characterization, for the presence of optical scatter. If scatter is present, either from the sample surface or volume, then there will be limitations to the accuracy and applicability of the measurement results. This is due to channeling effects that lead to light exiting at or near the sample's edges. These limitations will depend on the type of measurement instrumentation used. Even hemispherical measurement systems, such as wall-mount integrating spheres, may not be able to account for all the light flux that was incident on the sample. On the other hand, 4π measurement systems such as a center-mount integrating sphere can be used to collect and account for all the flux for the determination of the sample absorptance.

For samples with low levels of scatter, the usefulness of 'reflectance' and 'transmittance' measurement results depend on precise constraints and accompanying descriptions of the input beam geometry, sample size, shape and condition, sample mounting, and detection geometry (because all these factors will have an effect on the measurement results). Depending on the information required, measurements of the scattering profile using BSDF systems and modeling of the scatter within the sample may be necessary.

5. REFERENCES

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[7] Both integrating spheres used for the measurements presented in this paper were manufactured by Labsphere, Inc. of North Sutton, NH. The mention of manufacturers and model names is intended solely for the purpose of technical information useful to the reader and in no way should be construed as an endorsement of the named manufacturer or product.