Regular Reflectance and Transmittance Measurements of Transmissive Materials Using a STAR GEM® Optical Accessory

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Abstract. *So far, five methods for the measurement of absolute reflectance have been developed. All these methods are analyzed and compared with particular emphasis on application to transmissive materials, such as windows and filters. Four of the methods are commonly used: V-W, V-N, goniometer, and integrating sphere method; the fifth, new developed method, is a STAR GEM method which is expected to achieve the greatest accuracy in the measurement of regular reflectance and transmittance. This is because beams, spatially shifted due to multi-reflections inside the material, can be directed to a detector by automatic adjusting of an output mirror angle. The absorptance calculated from reflectance and transmittance measured by the STAR GEM is compared with the absolute absorptance measured by a laser calorimeter. Measurement results from these two methods agree well each other.*

Keywords: STAR GEM, Symmetry X, absolute reflectance, laser calorimeter

1. Introduction

Light incident on a material can interact with it in five main ways: it can be absorbed, transmitted either regularly or diffusely, and reflected either regularly or diffusely. In this paper we will consider that a sample under test is specular, and in the other paper we will discuss the measurements of diffuse transmittance and reflectance [1]. Most commonly the transmittance (T) and reflectance (R) are measured by a spectrophotometer. The absorptance (A) can be then calculated from equation $A=1-T-R$. On the other hand, the absolute absorptance can be directly measured by a laser calorimeter.

Although the techniques of the absolute reflectance measurement are discussed in many reference books [2], they do not pay so much attention to the regular transmittance measurements. All spectrophotometers and contemporary accessories on the market have been developed according to the idea that it is possible to separately measure transmittance and reflectance. However, when the absorptance is calculated from $1-T-R$ and optical constants of a sample are evaluated from solutions of simultaneous equations for refractive index and extinction coefficient using measured values of T and R, the accuracy of the results becomes worse. A few researchers discussed the measurement methods of T and R with the equal accuracy. [3, 4]

Up to now, four methods for the measurement of absolute reflectance have been developed: V-W, V-N, a goniometer and an integrating sphere methods. For a transmissive sample the additional beams, coming from the back surface reflections, are displaced with respect to the beam reflected from the front surface. Displacement depends on the incident angle, on the refractive index of the sample, and the sample thickness. So far, in the V-W, V-N and the goniometer accessories no solution employing a fine adjustment of the mirror angle in front of a detector was introduced. Usually only an averaging sphere is used to reduce sensitivity to alignment, but the spatial nonuniformity of the response of the integrating sphere makes the solution of the displacement more complicated.

2. Description of a STAR GEM

The fifth method is a STAR GEM. The STAR GEM is an acronym of Scatter, Transmission, and Absolute Reflection measurements using a Geminated Ellipsoid Mirror. The STAR GEM has been developed on the basis of two ideas in AIST. The first idea is the Symmetry X to measure the absolute reflectance [4]. For the transmittance measurement, an optical path of a transmission overlaps that of a background and the ratio gives us the absolute transmittance. The Symmetry X was inferred from the transmittance measurement. We make two reflection measurements at the same angle of incidence from a front side (RFF) and a back side of the sample (RBB) and we also make two background measurements at the same angle from a front side (BFB) and a back side (BBF), so these four paths overlap everywhere. The shape of the four paths is like a "X" over the other "X". A geometric mean of two reflectance values gives us the absolute reflectance.

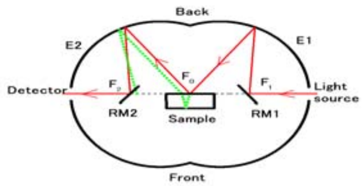

Fig.1 Structure of GEM and beams

The second idea is a Geminated Ellipsoid Mirror (GEM). The GEM consists of two equivalent ellipsoids of revolution (E1 and E2). A focus of the E1 coincides with that of the E2 and this common focus (F_0) aligns with two remaining foci (F_1 and F_2) in Fig. 1. A sample is placed at F_0 and two rotating plane mirrors (RM1 and RM2) are placed at F_1 and F_2, respectively. The RM1 and RM2 can rotate by each stepping motor independently. The STAR GEM Type 1 is shown in Fig.2 and its size is 198mm×166mm×120mm. A sample holder has two equivalent holes and a sample is attached to the one hole. Six modes in Fig. 3 are carried out for measurements of both absolute reflectance and transmittance according to the Symmetry X. As a result, reflectance and transmittance can be measured with the equal accuracy. An incident angle to a sample can be changed continuously from 0 to 90 degrees. The STAR GEM can be used not only with an outside light source and a detector, but also with an installation into a sample compartment of a commercial spectrophotometer without any change of the spectrophotometer itself.

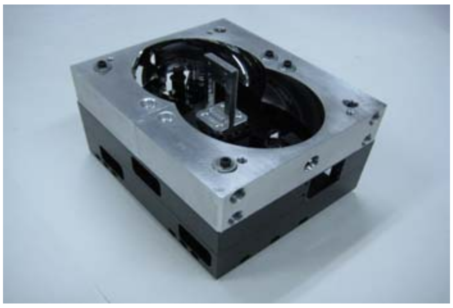

Fig. 2 STAR GEM Type 1

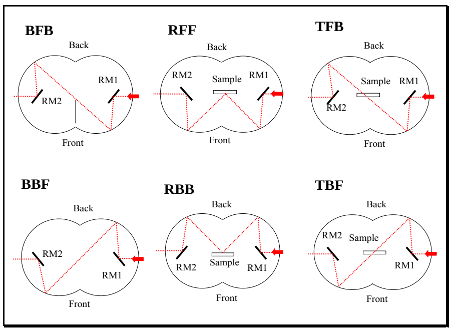

Fig. 3 Six modes for absolute reflectance and transmittance measurements

3. Experimental details

The STAR GEM optical device was inserted inside a sample compartment of the Varian FTIR spectrophotometer (FTS 7000e). The FTS 7000e was configured with a ceramic lamp, a Ge/KBr beam splitter and a Deuterated Tri-Glycine Sulfate (DTGS) detector for the spectral measurements in the region from 1.6 µm to 25 µm. The IR beam was focused onto the RM1 mirror. STAR GEM with an additional lens was also inserted inside a sample compartment of a Shimadzu grating spectrophotometer (SolidSpec 3700). The SolidSpec 3700 was configured with two lamps: tungsten, and deuterium D2 and with three detectors: a photomultiplier (PMT), an InGaAs detector, and a cooled PbS detector. An averaging sphere was used in front of the detectors for the spectral measurements in the region from 0.24 μm to 2.6 μm. The lens, which focused the original beam from the SolidSpec 3700 onto the RM1, was settled near the entrance port of the STAR GEM.

In order to directly measure absolute absorptance, we prepared and used a laser calorimeter [5]. An addenda was made of aluminum foil. A platinum thin film resistor as a thermometer and an electric resistor as a heater were folded in the addenda and a sample was attached to the addenda with a small amount of grease to increase thermal contact. The addenda was hung using thin nylon thread inside a vacuum chamber with an entrance and an exit windows for a laser beam. The laser was a diode-pumped frequency-doubled, Nd:YAG laser that emitted an output beam at a 532 nm wavelength. The maximal output power was 100 mW. At first, the sample was irradiated by laser beam for more than ten minutes and the temperature increment induced by light absorption was measured. It was very important to take care of the addenda, which must not be directly irradiated by laser beam and also must not be irradiated by scattered laser light from the chamber's windows and the sample. Secondly, the temperature increment induced by Joule heating was measured. The absolute absorptance was obtained from the two temperature increments and the measurement of laser power at the sample.

Spectral and calorimetric measurements for the same fused quartz disk were carried out. Its size was 10 mm diameter and 1 mm thickness.

4. Results and Discussions

For a transmissive sample, the first beam is reflected from an incident surface and focuses at F_2 (red solid line in Fig. 1). The additional beams coming from the back surface reflections and focus near F_2 (green dotted line in Fig. 1). The angle of the RM2 mirror, which directs all beams reflected from the sample under test to the detector, depends on the incident angle, the refractive index of the sample and the sample thickness. The calculation result of this angle difference is shown in Fig. 4 (the thickness is 1 mm and the index is 1.457). This result means that the Symmetry X is not sufficient for the transmissive sample. The suitable measurement procedure should always search a rotation angle (ϕ_0) of RM2 for the maximum signal intensity at each mode in Fig. 3. Angle of the RM2 mirror is rotated by 0.1 degree between ϕ–10 and ϕ+10 degrees, where ϕ is the expected angle from the Symmetry X, and then the spectral measurements at ϕ_0 are done.

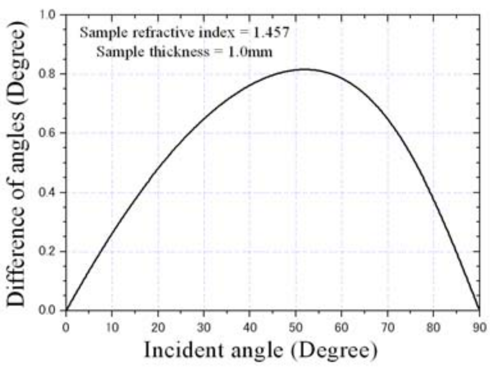

Fig. 4 Difference of RM2 rotation angles for reflection beams from a front and a back surfaces

The automatic measurement procedure using the STAR GEM is as follows: (1) the sample holder is rotated by 180 degrees, (2) the background measurement of a BFB mode is carried out for an empty hole, (3) the sample holder is raised up, (4) the sample measurements of a TFB and an RFF modes are carried out for a sample, (5) the sample holder is rotated by 180 degrees, (6) the sample measurements of an RBB and a TBF modes are carried out for the sample, (7) the sample holder is held down, (8) the background measurement of a BBF mode is carried out for the empty hole. The maximum signal is found by the adjustment of the RM2 rotation angle for each measurement.

Figure 5(a), (b) and (c) are the measured spectra of reflectance and transmittance and the calculated spectrum of absorptance. The abscissa is a logarithmic scale. Green and red curves are spectra measured by the grating and the FTIR spectrophotometers, respectively, and black solid circles are spectral values calculated using the optical constants from the handbook [6]. In these measurements the incident angle was 10 degrees. The difference between the optimized RM2 angle and the RM2 angle expected from the Symmetry X was 0.1 degree. This difference is about half of the value estimated from Fig. 4 and it is reasonable. When the spectrophotometers were changed, each spectrum measured using the STAR GEM agrees well with the corresponding spectrum in the overlapping wavelength region and the difference is less than 0.1 %. Each measured spectrum also agrees well with the calculated spectrum. In the visible region from 0.3 µm to 1.38 µm the absorptance spectrum is less than ±0.05 % and the noise level of the measured spectrum is estimated to be ±0.2 %, so that the sample didn't absorb light. The transmittance and reflectance measured using the STAR GEM are independent each other and have the same accuracy of the measurement.

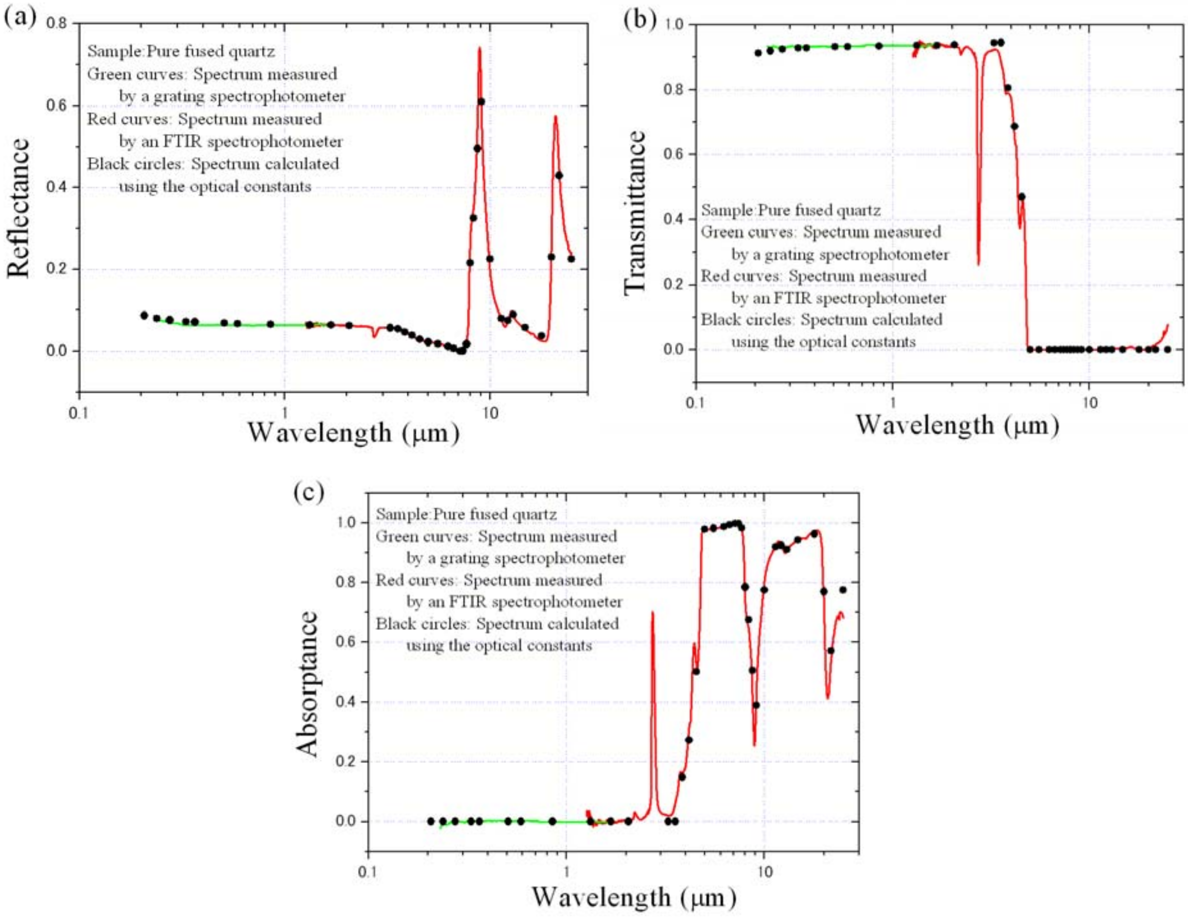

Fig. 5(a), (b) and (c) Reflectance, transmittance and absorptance spectra of a pure fused quartz

The temperature increments of the same fused quartz measured by the laser calorimeter are shown in Fig. 6. Two black curves in Fig. 6(a) are temperature variations without laser irradiation measured two times and two red curves are temperature increments measured with laser irradiation of 29.5 mW power. Figure 6(b) is temperature increments induced by Joule heating from 0.04 mW to 0.25 mW. The noise level of the laser calorimeter is estimated to be

±0.03 %. The absolute absorptance of 1mm thick fused quartz at 532 nm measured by the laser calorimeter was 0.00±0.000.

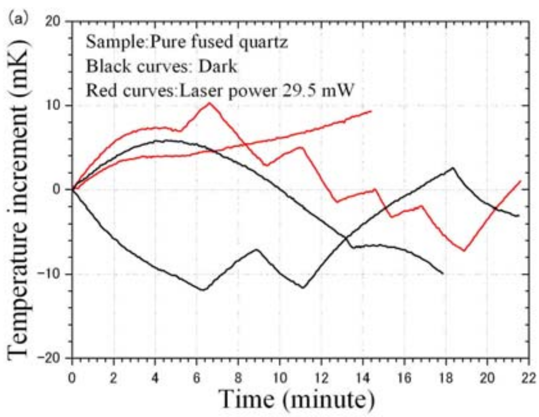

Fig. 6(a) Black curves are temperature variations at dark and red curves are temperature increment by laser irradiation.

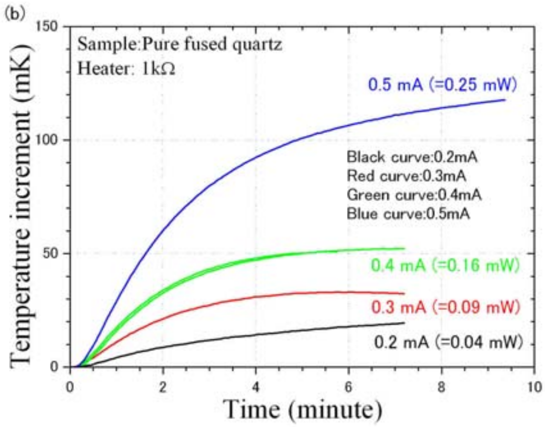

Fig. 6(b) Temperature increment by Joule heating depending on the current.

5. Conclusions

The Symmetry X is an innovative idea which was employed in the STAR GEM optical device for the absolute reflectance and transmittance measurement. In order to evaluate the accuracy of the STAR GEM, the absorptance calculated from reflectance and transmittance measured by the STAR GEM was compared with the absolute absorptance measured by the laser calorimeter at the wavelength 532 nm. Both absorptance measurements were carried out for the same sample made from fused quartz and results show good agreement. As it was analyzed above, measurements by means of STAR GEM optical device for transmissive materials need to find the maximum signal intensity. This procedure was fully automated in the STAR GEM optical device and therefore it can be used as a new innovative accessory of a FTIR or grating spectrophotometer for fully automated and precise optical measurement of absolute reflectance and transmittance of materials.

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