Comparison of Reflection Methods for Determining Optical Constants with STAR GEM

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Abstract. A new optical accessory was developed which can measure both the absolute reflectance and transmittance as the incident angle is continuously varied in the wide range from 0.5° to 89.5°. Incident angle dependences of reflectance were measured for S- and P-polarizations of an aluminum mirror. Optical constants and their accuracy were determined.

1. Introduction

A number of experimental studies have been devised to determine optical constants, the index of refraction n and the extinction coefficient k, by means of reflection measurements. The optical constants of an isotropic medium can in principle be derived from a pair of reflection measurements at non-normal incidence. Of the nine reflection methods listed by Humphreys-Owen¹⁾, three are useful, Method D: the ratio R_p/R_s at two angles of incident, Method G: Brewster angle and R_s at that angle and Method H: Brewster angel and R_p/R_s at that angle, where R_p , R_s and R_p/R_s are defined as absolute reflectance of *p*-polarization, of *s*-polarization and their ratio, respectively, and where the Brewster angle is defined as that angle for which R_p is a minimum as function of incident angle. The methods except the Method G show an apparent fall in sensitivity at very low k, but the disadvantage of the Method G is a relatively poorer sensitivity and the need to normalize. The Method D and H have very high sensitivity at high k and measure only

the ratio. The disadvantage of the Method D is the need for one of these angles to be so near grazing. For example, Avery²⁾ chose the angles, 60° and 80°. No one method shows up overwhelmingly above the others.

There are three difficulties to realize Humphreys-

Owen's proposal, 1) the absolute reflectance measurement, 2) the reflection measurement as the incident angle is continuously varied around the Brewster angle and 3) the reflection measurement at grazing angle. For absolute reflectance measurement, the various methods, such as V-W, V-N, goniometer³⁾ and integrating-sphere methods⁴) have been developed. For the reflection measurement with continuously variable angle, the goniometer method and a Seagull® method are possible. Further for the reflection measurement at grazing angle, method satisfies us, because the no substantial sizes of a light source and a detector and some mirrors in the goniometer and the Seagull limit variable angle between 10 and 80 degree.

In order to fulfill the three requirements, a Geminated Ellipsoid Mirror (GEM®)⁵⁾ system has been designed and constructed for multiple optical property measurements. Scatter, Transmission And Reflection (STAR) using the GEM can be measured directly at arbitrary angles of incoming beam to a sample and outgoing beam from the sample. In particular, for specular samples, the absolute reflectance and transmittance can also be measured directly with high accuracy.

2. Description of the GEM

The signal and background measurements are made to obtain the transmittance. In both measurements the optical beams with and without a sample pass along the almost same path, namely, the path becomes two-fold as shown in Fig. 1(a). In this figure the signal measurement (TFB) is shown by a solid line and the background measurement (BFB) is shown by a dotted line. (These two abbreviations are as follows, for example in TFB, first T means a Transmission signal measurement, second F means a Front path from the source to the sample, and last B means a Back path from the sample to the detector. In BFB first B means a Background The second and measurement. third characters have the same means.) The ratio of the signal measurement to the background measurement cancels out the reflectance difference of the individual mirrors (omitted in Fig. 1) and the optical loss on the way from the light source to the detector. Consequently, we can obtain the absolute transmittance.



Fig.1 Symmetry X system for optical measurement. S is a light source and D is a detector.

We constructed the symmetry X system⁶⁾, as shown in Fig. 1(b), to make the path build two-fold for the reflectance measurement, we made the namely, two signal measurements (RFF and RBB) and two background measurements (BFB and BBF). (R in RFF and RBB means a Reflection signal measurement.) A character of "X" was put over the other "X" for measuring the reflectance as shown in Fig. 1(b). In addition to RM1 and RM2 mirrors mentioned below, we didn't need to remove the source, the detector and mirrors in the symmetry X system. The square root of the ratio of product of RFF and RBB to the product of BFB and BBF, which is equivalent to a geometric mean of two reflectance values, canceled out the reflectance difference of the individual mirrors (omitted in Fig. 1) and the optical loss on the way from the light source to the detector. Consequently, we obtained the absolute reflectance.⁶⁾



Fig. 2 Block diagram of STAR GEM

The new optical system comprised two ellipsoid mirrors⁵⁾ (E1 & E2) as shown in Fig.2. We geminated both mirrors as a focal point of the E1 mirror coincided with that of the E2 mirror and furthermore three focal points aligned. This line was an optical axis. A sample was placed on the common focal point (F0). An incoming beam rotating mirror (RM1) and an outgoing beam rotating mirror (RM2) were placed on two remaining focal points (F1 & F2). Two holes were opened at two intersections of the optical axis and the E1 & E2 mirrors. When the RM1 & RM2 mirrors were rotated in a correlating manner such as the symmetry X, the absolute reflectance and transmittance were measured at continuously variable angles. Figure 3 is a This photograph of the STAR GEM. dimension size was 370mm×270mm×150mm.



Fig. 3 Photograph of STAR GEM

3. Results

A 0.633µm-wavelength He-Ne laser as a light source, a pyroelectric detector and a few quartz lenses were set outside the STAR GEM. A sample was an aluminum mirror of 30mm-diameter. A sample holder was settled on the rotation stage at the F0, stood both perpendicular to floor and parallel to the optical axis and had both the sample and a hole of the same diameter. Each origin of the angle of the RM1 and RM2 mirrors was the angle when each mirror faced each hole on the E1 & E2 mirrors. For the background measurement, the RM1 and RM2 mirrors turned around each focal point at the same direction. On the other hand, for the reflection signal measurement, they turned at the reverse direction. For an opaque sample the holder was also rotated in 180 degree around the F0, because we needed to make the signal and background measurements for the Back incidence (RBB and BBF) as wall as for the Front incidence (RFF and BFB).



Fig. 4 Absolute reflectance of an aluminum mirror.

The incident angle dependence of absolute reflectance spectra of the aluminum mirror for both polarizations is shown in Fig. 4. The incident angle changed continuously between 0.5 degree and 89.5 degree. For the measurement at the angle more than 80 degree, we needed to rotate the sample holder in 20 degree clockwise for the Front incidence and in 20 degree counterclockwise for the Back incidence.

4. Discussions

Our method is based on the premise that any R vs. Φ curve, in the absence of interference effect, is uniquely determined by the pair of constants *n* and *k* which are related to the reflectance by the generalized Fresnel relation equations⁷⁾,

$$R_{s} = \frac{(a - \cos \Phi)^{2} + b^{2}}{(a + \cos \Phi)^{2} + b^{2}},$$
 (Eq. 1)

$$R_p = R_s \times \frac{(a - \sin\Phi\tan\Phi)^2 + b^2}{(a + \sin\Phi\tan\Phi)^2 + b^2},$$
(Eq. 2)

$$a^{2} = \frac{1}{2} \left[\left\{ \left(n^{2} - k^{2} - \sin \Phi^{2} \right)^{2} + 4n^{2} k^{2} \right\}^{1/2} + \left(n^{2} - k^{2} - \sin \Phi^{2} \right) \right],$$
(Eq. 3)

$$b^{2} = \frac{1}{2} \left[\left\{ \left(n^{2} - k^{2} - \sin \Phi^{2} \right)^{2} + 4n^{2}k^{2} \right\}^{1/2} - \left(n^{2} - k^{2} - \sin \Phi^{2} \right) \right].$$
(Eq. 4)

Since these equations can't be solved explicitly for n and k, various means have been devised to obtain the solution, however, all are essentially the same since they represent simultaneous solution of the Fresnel equations. On the other hand, an exact analysis of the Brewster condition was made by Humphreys- Owen¹⁾, which led to explicit equations applicable to methods including Brewster angle, θ_B ,

$$2\left(\left(n^{2}+k^{2}\right)^{2}+n^{2}-k^{2}\right)x^{3}+\left(n^{2}+k^{2}\right)^{2}\left(\left(n^{2}+k^{2}\right)^{2}-3\right)x^{2}-2\left(n^{2}+k^{2}\right)^{4}x+\left(n^{2}+k^{2}\right)^{4}=0, \text{ (Eq. 5)}$$

where $x = \sin \theta_{\rm B}^2$.

The absolute reflectance of the aluminum mirror was successfully measured in the wide range from 0.5° to 89.5° . The $R_{\rm s}$ was

monotonically increasing. The R_p was at first decreasing with increasing incident angle, reached the minimum at 79.7° and was abruptly increasing. The Brewster angle was

 $\theta_{\rm B} = 79.7^{\circ} \text{ and } R_{\rm p}(79.7) = 0.725 \pm 0.002 \text{ and} R_{\rm s}(79.7) = 0.975 \pm 0.006. At <math>\Phi = 0.5^{\circ}$, $R_{\rm s}$ was equal to $R_{\rm p}$ and its value was 0.903 ± 0.008 . At $\Phi = 89.5^{\circ}$, $R_{\rm s}$ was 0.993 ± 0.002 and $R_{\rm p}$ was 0.947 ± 0.004 . At $\Phi = 45^{\circ}$, $R_{\rm s}$ was 0.929 ± 0.004 and $R_{\rm p}$ was 0.870 ± 0.004 . We succeeded in demonstrating a previously unnoticed property of the Fresnel equations at $\Phi = 45^{\circ}$, $R_{\rm s}^2(45) = R_{\rm p}(45)$. According Avery, at $\Phi = 60^{\circ}$, $R_{\rm s}$ was 0.946 ± 0.006 and $R_{\rm p}$ was 0.825 ± 0.003 .

Table 1 Optical constants and Errors of Al mirror

| | n | k |
|----------|-------------------|-------------------|
| Method | 0.808 ± 0.040 | 5.605±0.006 |
| D' | | |
| Method G | 1.184±0.285 | 5.535±0.066 |
| Method | 0.871 ± 0.007 | 5.594 ± 0.001 |
| H' | | |

The value of R_p was used instead of the ratio of R_p/R_s in the calculations of the Method D and H, because we can directly measure the absolute reflectance by the STAR GEM. We compared the optical constants and its errors obtained from the calculations using Eq. 1 to Eq. 5. The results are summarized in Table 1. The errors in the Method H' were the smallest. For materials of high *k*, the Method H' was more accurate.

5. Conclusion

We developed the STAR GEM system, which can measure scatter, transmission and reflection at arbitrary incoming and outgoing angle. The incident angle dependence of the absolute reflectance of the aluminum mirror was measured in the wide range. It is clear that the optical constants obtained from the pair of the Brewster angle and R_p at that angle are the most accurate.

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