

## New Optical Measurements Realized by Oblique Incidence

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**Abstract.** *An optical equipment has been developed, which is originated with two mirrors shaped like an ellipsoid of revolution. Firstly it is found that the pseudo Brewster angle for an absorbing substrate becomes small with increasing a nonabsorbing film thickness. The thickness of a dielectric thin film can be measured on a metal substrate in the visible region. Secondly a plane-parallel plate serves as an interferometer and the coefficient of finesse for an S-polarized plane wave increases with increasing an incident angle. Thus the refractive index of a thin film on the plate can be measured in the millimeter wave region.*

**Keywords:** STAR GEM, GAEA, pseudo Brewster angle, absolute reflectance

### 1. Introduction

Many optical measurements have been carried out at oblique incidence. An ellipsometer is one method to be put to practical use and can measure the thickness and refractive index ( $\bar{n} = n + ik$ ) of thin films on the substrate. We have also been interested in the development of new methods using oblique incidence. We find in optics books two remarkable phenomena, the Brewster angle for *P*-polarized light and the reflectance increasing towards unity for *S*-polarized light, as the incident angle increases.

Firstly when a material is non-absorbing, the absolute reflectance ( $R_p$ ) for *P*-polarized light becomes zero at a certain angle of incidence, which is called the Brewster angle ( $\theta_B$ ). When a material is absorbing,  $R_p$  doesn't reach zero, but the angle for which it is a minimum is called the pseudo Brewster angle ( $\theta_B'$ ). [1] What happens as the non-absorbing film thickness increases on the absorbing substrate? We carefully investigated the change of  $\theta_B'$  and  $R_p$  with increasing the film thickness on the substrate.

Secondly the intensity of the interaction between the material and incident light is considered to be proportional to the ratio of optical path to wavelength ( $\lambda$ ) of incident light,  $nd/\lambda$ , where  $n$  and  $d$  are refractive index and thickness of the material, respectively. As a result, optical measurements of a thin film become difficult for a long wavelength such as a millimeter wave. The reflectance ( $R_s$ ) of all materials for *S*-polarized light monotonically increases with increasing an incident angle. We prepared a thin film on a substrate. The thickness of the substrate is the same order as the wavelength, but that of the film is one thousandth as long as the wavelength. The multiple reflections inside the sample increase at a grazing angle of incidence, so that the intensity of the interaction enlarges. We succeeded in measuring the refractive index of the thin and low-dielectric film in the millimeter wave region.

Concerning an absolute reflectance measurement method, various methods, such as V-N, goniometer and integrating-sphere methods have been developed. Concerning an optical measurement method with continuously changing angle, the goniometer and a Seagull methods are possible. However the goniometer method satisfies both requirements, it is actually difficult for us to measure absolute reflectance. It makes the goniometer so large both to attain the satisfactory repeatability for the movements of the detector between sample and background measurements and to be able to measure reflectance at a grazing angle of

incidence, that its diameter is larger than 1m.

A GEM (Geminated Ellipsoid Mirror) system has been designed to fulfill two requirements mentioned above. A STAR GEM (Scattering, Transmission and Absolute Reflection using the GEM) can make many optical measurements directly at arbitrary angles of incoming beam to a sample and of outgoing beam from the sample.[2] The STAR GEM is so small that it can be incorporated into a sample compartment of commercial Fourier-transform infrared (FT-IR) spectrometers..

## 2. STAR GEM

The basic idea of the GEM is that the light emitted from a focal point of the ellipsoid mirror is concentrated to the other focal point. The GEM consisted mainly of two ellipsoids of revolution (E1 and E2) as shown in Fig.1. A focal point of the E1 was joined to that of the E2 and this common focal point (F0) aligned with two remaining focal points (F1 and F2). A sample was placed on F0 and two rotating plane mirrors (RM1 and RM2) were placed on F1 and F2, respectively. When RM1 and RM2 were rotated correlatively with the same direction, the incident angle dependence of absolute transmittance was measured. When RM1 and RM2 were rotated correlatively with the opposite direction, the incident angle dependence of absolute reflectance was measured. When RM1 was fixed and RM2 were rotated independently, the angular resolution of scattering from the sample was measured. The STAR GEM is shown in Fig.2 and its dimension size was 198mm×166mm×182mm.

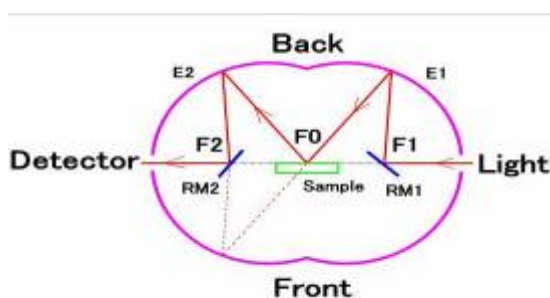


Fig. 1 Block diagram of a GEM

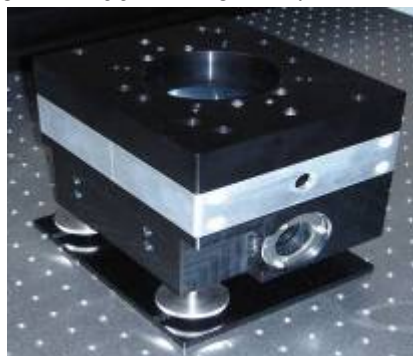


Fig. 2 Photograph of a STAR GEM

## 3. Theoretical works

### 3.1 Pseudo Brewster angle method

The pseudo Brewster angle ( $\theta_B'$ ) increases and also its minimum  $R_p$  at  $\theta_B'$  increases, as  $k$  increases with  $n$  held constant. On the contrary, the  $\theta_B'$  decreases but its minimum  $R_p$  at  $\theta_B'$  increases, as  $n$  decreases with  $k$  held constant. A metal is usually absorbing and has high  $k$ , on the other hand a dielectric is usually non-absorbing and has low  $n$ . The  $\theta_B'$  of the metal is larger and usually different from the  $\theta_B$  of the dielectric. A model for the calculation is a magnesium fluoride ( $MgF_2$ ) film evaporated on an aluminum (Al) mirror. This sample is usual and  $MgF_2$  is a good material to protect the mirror. The refractive indexes of  $MgF_2$  and Al are  $1.38+i0$  and  $0.871+i5.594$ , respectively, for the light of  $0.633 \mu m$  He-Ne laser. The film thickness dependences of  $\theta_B'$  and  $R_p$  are carefully calculated. These results are shown in Fig. 3 and 4. When the film thickness is larger than  $\lambda/4$ , another effects (multi-reflection etc.) will be added to the changing of the pseudo Brewster angle, so that the results becomes complex. The calculated results are shown less than 100nm film thickness

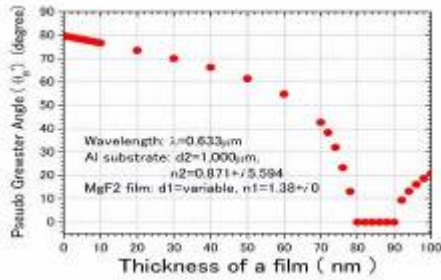


Fig. 3 Pseudo Brewster angle vs. film thickness.

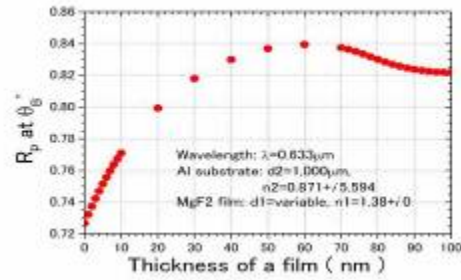


Fig.4 Minimum reflectance at  $\theta_B'$  vs film thickness.

### 3.2 Grazing Angle Etalon (GAEA) method

A plane-parallel plate (etalon) serves as an interferometer of multiple waves obtained by division of incident-beam amplitude and generates all of the reflected and transmitted beams. The incremental phase difference between the interfering beams is constant and is the same as  $\Delta\phi = 4\pi nd \nu \cos(\theta')/c$ , where  $\theta'$ ,  $\nu$  and  $c$  are the angle inside the plate, frequency and light velocity, respectively. The reflected intensity  $I_r$  can be computed by summing the successive reflected beams.

$$R_s = \frac{I_r}{I_0} = \frac{F \sin^2(\Delta\phi/2)}{1 + F \sin^2(\Delta\phi/2)} \quad \text{Eq. 1}$$

where  $I_0$  is the incident intensity and  $F = [2\sqrt{R}/(1-R)]^2$ .  $F$  is the coefficient of finesse of the system and is a function of the surface reflectivity only. The value of  $F$  will have a large impact on the shape of the intensity pattern. A minimum of reflected intensity will occur when  $\Delta\phi/2 = m\pi$ , where  $m$  is an integer. The fringe pattern will be very dark bands on a uniform bright background. The reflectance ( $R_s$ ) is plotted as a function of frequency in Fig. 5 and 6 for two values of reflectance, 0.2 and 0.9, where  $n$  and  $d$  are 3.449 and 0.7 mm, respectively. As the incident angle increases, the reflectance of all materials for the  $S$ -polarized light increase monotonically, so that the dark bands in the fringes becomes narrower. When the increase of the thickness  $d$  is 2% with  $n$  and  $\theta'$  held constant, the dark band shifts toward lower frequency as shown by dotted curve in Fig. 5 and 6. When this increase of the thickness is due to the fabricated film, this GAEA (Grazing Angle Etalon) method serves as the measurement method of the thickness and refractive indices of thin films on the substrate.

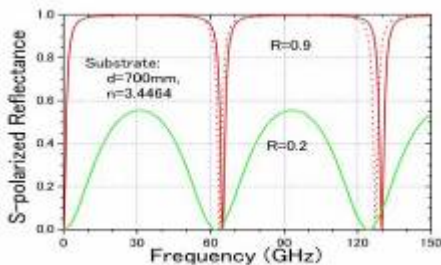


Fig. 5 S polarized reflectance from an etalon

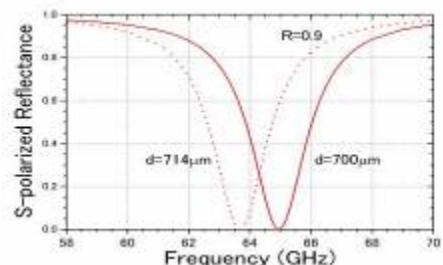


Fig.6 An enlargement of Fig. 5 around a valley

## 4. Results and Discussions

### 4.1. Measurements of Pseudo Brewster angle

A light source is a 0.633  $\mu\text{m}$ -wavelength He-Ne laser and a detector is an Si-photodiode. Samples are a bare and an  $\text{MgF}_2$  coated aluminum mirrors on the market. In

order to make an absolute reflectance measurement, we need to measure light power of background and signal measurements from a front and a back incidence, that is, we need four times measurements (BFB, BBF, RFF and RBB) not two times. For the background measurement (BFB and BBF), the RM1 and RM2 mirrors turned around each focal point at the same direction. On the other hand, for the signal measurements (RFF and RBB), they turned at the reverse direction. For an opaque sample the sample was also rotated in 180 degrees around the F0. The angle dependence of  $P$ -polarized reflectance of a bare and a  $MgF_2$  film coated mirrors is shown in Fig. 7. The incident angle changed from 0.5 to 89.5 degrees continuously.

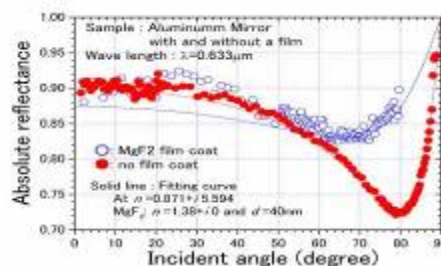


Fig. 7  $P$ -polarized absolute reflectance of a coated and a bare Al mirrors

The pseudo Brewster angles of a bare and a  $MgF_2$  film coated aluminum mirrors are 79.7 and 66.1 degrees, respectively. The reflectance values of two mirrors are 0.727 and 0.830 at each  $\theta_B'$ . The thickness of  $MgF_2$  film is obtained 40nm from Fig. 3 and 4. This value is near 50nm of the real thickness. We measured the thickness of the same  $MgF_2$  mirror using an ellipsometer and obtained 210 nm thickness.

#### 4.2. Measurements of Grazing Angle Etalon

A sample was a non-doped silicon wafer 700  $\mu m$  thick, 100 mm in diameter coated on both sides with 2  $\mu m$  thin  $SiO_2$  films. Thickness of the silicon substrate was selected to obtain one valley in the measured frequency range. Half of the film areas was removed side by side of the wafer using hydrogen fluoride acid.

The reflectance spectra for  $S$ -polarized waves are shown in Fig. 8. The incident angle is 80 degrees. The valleys shift from right to left with increasing the film thickness, that is, 66.403, 66.324, and 66.203 GHz. We can obtain the refractive index of  $SiO_2$  film to be  $2.014 \pm 0.192$  from these valleys. This value is near the value from the paper.[3]

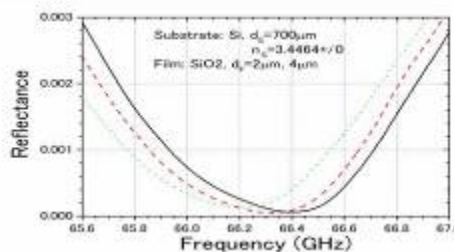


Fig. 8 Reflectance spectra of an etalon

#### 5. Acknowledgements

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#### 6. References

- [1] M. Bass, E.W.van Stryland, D.R. Williams, and W.L.Wolfe, "Handbook of Optics" vol. 1, (McGRAW-HILL, New York 1995) pp.5.1-5.30
- [2] E.Kawate, Measurement Science Review, **5**, (2005), pp1-5
- [3] J. W. Lamb, Int. J. Infrared and Millimeter Waves, **17**, (1996), pp1997-2034