



## Thickness Analysis of natural oxide film on microscopic Si pattern

### Introduction

The MSV-5000 series microscopic spectrophotometer is for transmission/reflection measurement in a wide wavelength range from ultraviolet to near-infrared. It allows the measurement of the area of as small as 10  $\mu\text{m}$  diameter and the built-in high-resolution camera enables to observe the samples precisely to determine the area to be measured. This instrument is most suitable to measure the minute samples or samples having microstructure. This time, the sample on which Si patterns of 35  $\mu\text{m}$  widths are lined up on Ti substrate with 14  $\mu\text{m}$  intervals was measured as a microstructure sample. Actually, the thickness of  $\text{SiO}_2$  formed upon Si was analyzed from the obtained reflectance spectrum, because Si is easily oxidized in the air to form thin oxide film of  $\text{SiO}_2$ .

**Keywords** microscope, silicon, absolute reflectance

### Measurement System

MSV-5200 Microscopic spectrophotometer

VWML-791 [Multi-Layer Analysis] program

**Sample:** Si and Si oxide film on the Ti substrate

### Measurement condition

UV/Vis spectral bandwidth:	5.0 nm
NIR spectral bandwidth:	20.0 nm
Scan speed:	100 nm/min
Response:	Slow
Data interval:	0.5 nm
Cassegrain objective mirror:	16x
Incidence angle:	23°
IN aperture:	10 $\mu\text{m}\phi$
OUT aperture:	10 $\mu\text{m}\phi$

### Measurement

1. Baseline: Al vapor-deposited mirror as a reference is used for baseline measurement.
2. Measurement area: The sample is observed by the high-resolution camera to determine the measurement area (Fig. 1). The red spot in Fig. 1 shows the size and position of detected light.
3. Sample measurement: The reflectance spectrum is measured.
4. Transforming into absolute reflectance: The absolute reflectance spectrum of the sample is calculated by multiplying the obtained relative reflectance by the absolute reflectance of Al vapor-deposited mirror.

### Analysis

Reflectance(R) is expressed by the equation of refractive index of the film ( $n_i$ ), extinction coefficient ( $k_i$ ), the angle of incidence ( $q_i$ ), wavelength ( $\lambda$ ) and the film thickness ( $d_i$ ). This time, optical constants of Si and  $\text{SiO}_2$  are used from the literature value. and then the film thickness of  $\text{SiO}_2$  is estimated by using [Multi-Layer Analysis] program by fitting the calculated reflectance spectrum to the measured one to make the thickness value reasonable.

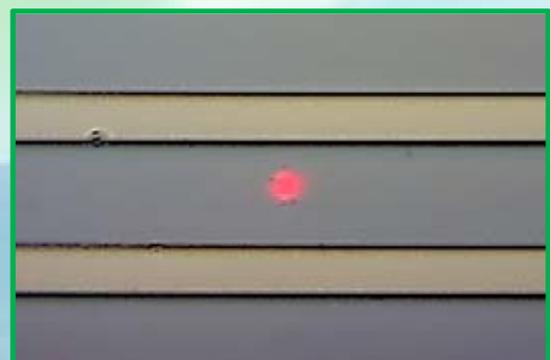
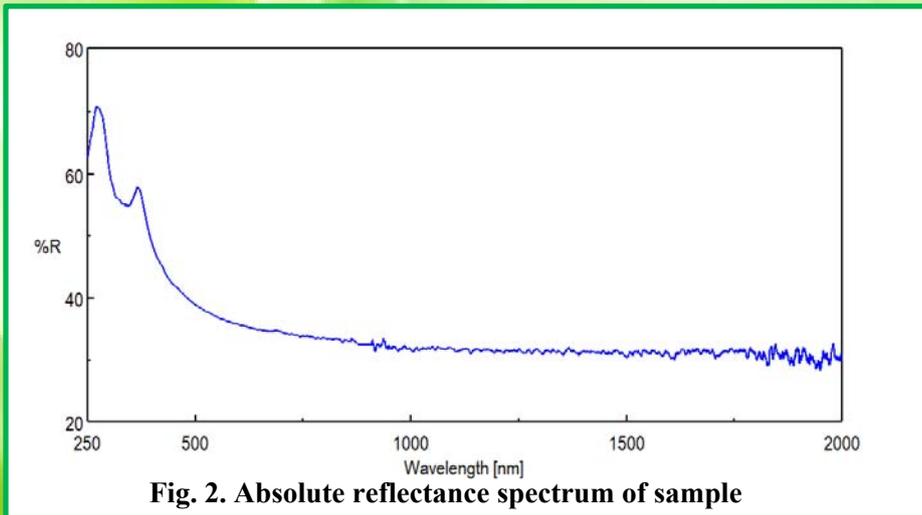


Fig. 1. Observation figure of measurement position



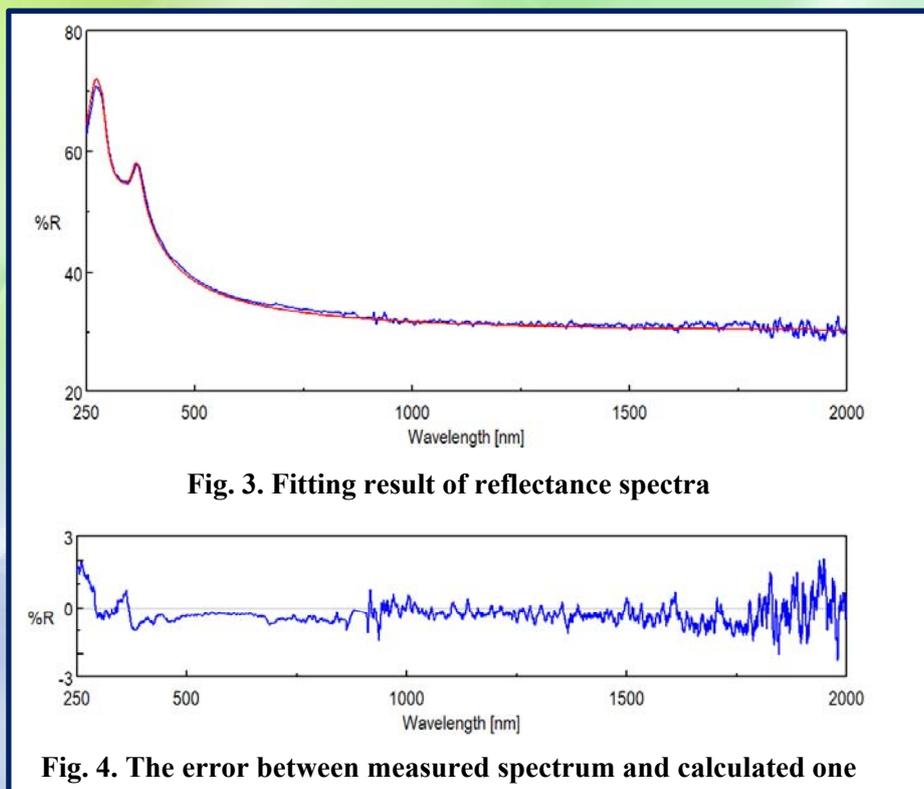
### Measurement Results

Measured absolute reflectance spectrum is shown in Fig. 2. MSV-5000 series adopts the confocal optical system, which enables the measurement eliminating the influence of back side reflection. In the range over 1100 nm where the light passes through Si, the spectrum would not be influenced by the back side reflection.



### Analysis Results

The result of fitting the reflectance spectra using [Multi-Layer Analysis] program is shown in Fig. 3. The error between measured spectrum and calculated one was within 2% (Fig. 3) and the film thickness of SiO<sub>2</sub> was calculated to be 7.6 nm.





**[Integrating sphere photoelectric spectroscopy]**

**Measurement/analysis system**

- V-750/760/770/780 UV/Vis spectrophotometer
- ISV-922/ISN-923/ISN-901i Integrating sphere unit
- VWWQ-789 Chromaticity/turbidity measurement program
- Rectangular cell, 10 mm, 20 mm, 30 mm and 50 mm



**Procedure**

1. Standard solution is prepared from neat standard sample diluted by purified water. Blank sample is purified water filtrated using 0.2 μm membrane filter.
2. Turbidity calibration curve is created from the measurement results of blank sample and standard solution prepared in 1. Firstly, standard white plate is mounted in integrating sphere and total light transmittance ( $T_t$ ) is measured, and then the plate is removed and diffuse transmittance ( $T_d$ ) is measured.
3.  $T_t$  and  $T_d$  of sample water at 660 nm is measured, and turbidity is calculated from the results and calibration curve.

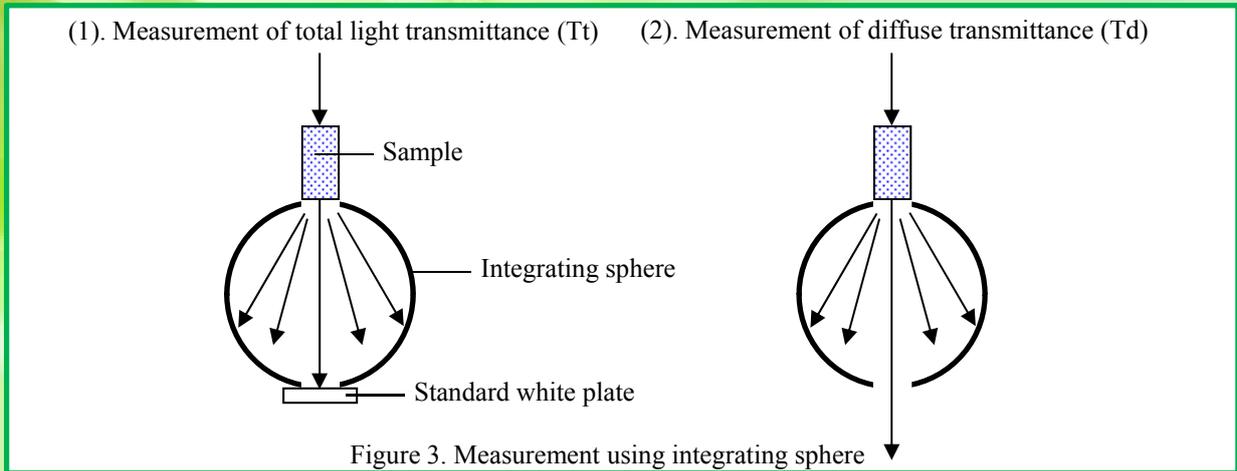


Figure 3. Measurement using integrating sphere

**Calibration curve**

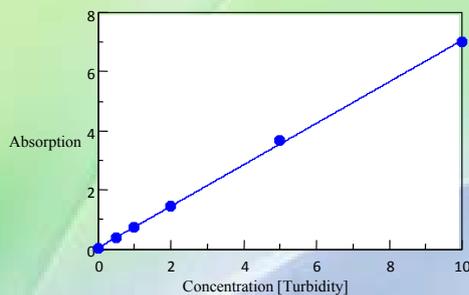


Figure 4. Turbidity calibration curve (integrating sphere photoelectric spectroscopy)

Table 4. Turbidity calibration curve (Integrating sphere photoelectric spectroscopy)

Concentration [turbidity]	Td/Tt x 100	Quantitative value [turbidity]
0	0.004	-0.05
0.5	0.389	0.50
1	0.726	0.98
2	1.435	1.99
5	3.666	5.17
10	7.003	9.92

Standard solutions with turbidity at 0, 0.5, 1, 2, 5, 10 degree were measured using 20 mm light pathlength cell and the results are shown in Table 4. From the calibration curve, the standard deviation is calculated as 0.08(6) degree, detection limit as 0.28 and quantitation limit as 0.86 degree. \*2)

50 mm light pathlength rectangular cell is recommend to be used for the analysis of low turbidity sample with concentration close to or less than quantitation limit .

**Calibration curve information:  $y = 0.7018x + 0.0397$   $R^2 = 0.9997$**



# Transmittance and Reflectance Measurement System for Minute Lenses

## Introduction

Small type lens is widely used in various products as smart phone , tablet PC etc.,. This note show the transmittance and reflectance measurement for 1-mm-diameter lenses used in mobile-phone cameras.



MSV-5200 UV/Vis/NIR Microspectrophotometer

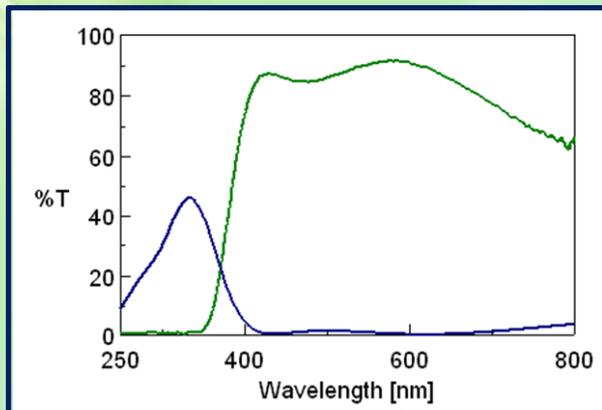


Integrating sphere for MSV-5200

For samples that refract light such as lenses, the transmittance can be measured using an integrating sphere. The reflectance can also be measured if the aperture size is sufficiently reduced so that the area being analyzed can be considered to be flat.



1-mm-diameter mobile-phone camera lens



System	Model No.	Product name	Code	Remarks
Instrument	MSV-5200-16-ST	UV/Vis/NIR Microspectrophotometer	6973-J008A	Wavelength range: 200 to 2700 nm Supplied with 16x Cassegrain objective, converging mirrors, and manual stage.
Optional accessories	MISP-552	Integrating sphere for MSV-5200		Wavelength range: 250 to 2000 nm Only for the dedicated manual stage for the integrating sphere. Not applicable for diffuse transmittance or reflectance measurements. Normal (non-diffuse) reflectance measurements can be performed using the reflectance optical path.
Description	Available models are the MSV-5100 (200 to 900 nm) and MSV-5300 (200 to 1600 nm). The type of integrating sphere varies depending on the instrument model, and determines the wavelength range that can be used.			