INFRARED EMITTANCE MEASUREMENTS AT NIST

Leonard M. Hanssen, Benjamin K. Tsai, and Sergey N. Mekhontsev
NIST
100 Bureau Dr., Stop 8442
Gaithersburg, MD 20899-8442

A new capability for the measurement of the temperature-dependent emittance of specular samples in the near infrared spectral region has been developed in NIST's Infrared Spectrophotometry Laboratory to provide emittance measurements and standards for a broad range of applications including rapid thermal processing (RTP). Our approach employs the indirect measurement of reflectance and transmittance measurements to obtain emittance. A vacuum goniometer system controls the sample environment and measurement geometry. The main system, including the sample, is contained in a vacuum chamber that enables characterization of materials otherwise susceptible to oxidation. Details of the lasers, sources, detectors, and other optics in the system are given. The system has initially been used to characterize the spectral emittance (by reflectance) of a variety of semiconductor wafer samples including bare silicon and silicon substrates coated with SiO_2, Si_3N_4, and polysilicon films. The spectral range for these measurements is from 600 nm to 1100 nm, where Si is opaque; the temperature range is ambient to 800 °C. The results are analyzed and compared with those predicted by several models from the literature.

INTRODUCTION

For emittance (emissivity) characterization, the indirect method of reflectance and transmittance measurement has been implemented using a vacuum goniometer. The goniometer has both vacuum- and inert atmosphere capabilities to eliminate oxidation at these temperatures. The research will support industry's needs for emittance data in semiconductor manufacturing by rapid thermal processing (RTP). Selective measurements will be performed to eliminate gaps in the existing experimental and theoretical knowledge of the optical properties of silicon.

MEASUREMENT METHOD AND INSTRUMENTATION

For the indirect measurement of spectral emittance of a material, the spectral reflectance, ρ, and transmittance, τ, of the sample must be measured. Typically this is done by comparison with the reflectance and transmittance of a known reference under the same conditions. Then the emittance can be expressed as

$$\varepsilon = 1 - \rho - \tau \,. \qquad (1)$$

For opaque samples, $\tau = 0$ and $\varepsilon = 1 - \rho$. Parts of the system are contained in a vacuum/inert-gas chamber that enables characterization of materials susceptible to oxidation. Vacuum electrical and water feedthroughs are used to provide electricity for the heaters and water cooling. A broad-band source (a stabilized quartz-halogen lamp with gold reflector and fiber-optic bundle) and three diode lasers (900 nm, 950 nm, and 1550 nm) are available as radiation sources. Using mirrors, the light is coupled in and out of the chamber through a window. A Glan-Thompson polarizer with a 20 mm clear aperture is used for polarization-resolved measurements. A schematic of the goniometer is shown in Figure 1. External Si (2 each) and InGaAs detectors can be used in both reflectance and transmittance modes. These detectors have narrow band filters centered at the diode laser wavelengths noted above. In addition, a double monochromator with a Si or InGaAs detector is used. The overall spectral range of the monochromator and detectors is 700 nm to 2300 nm. The light is mechanically chopped and detected using a lock-in amplifier. A LabVIEW [1] program is used for the data acquisition.

A cold reference and hot sample are mounted on a horizontal stage, which in turn is mounted on a rotation stage, allowing for angle-dependent measurements of the reflected and transmitted beams. The alignment of the sample during heating was monitored by a quadrant detector and controlled and adjusted by the sample

rotation stage and a linear stage for sample tilt adjustment. In addition to the undoped Si and Au, several other standards are available, including Pt-coated-Si. For the reflectance measurements, the set of filtered detectors and the monochromator are mounted on a movable external plate.

Two sample heaters are used: a ring heater for transparent samples (requiring measurement of both reflectance and transmittance) and a "solid" heater for opaque (requiring only reflectance measurement). Both heaters employ a platinum-ceramic element and have temperature ranges from 100 ºC to 1000 ºC. They are shielded from the chamber by a water-cooled shroud. Thermocouples are used for temperature monitoring of the heaters and samples. The heaters use a DC-power supply and are Proportional-Integral-Derivative (PID) controlled by means of a custom LabVIEW program. The measurement is fully automated for a fixed angle of incidence.

A scroll pump is used to provide an oil-free vacuum; by flushing the vacuum chamber with a purge gas, an oxygen level below 20 µmol/mol (20 ppm by volume) is achievable. This process is used to obtain a sample surface equilibrium during heating and measurement.

Upgrades currently underway include mounting the detectors and a fiber-output on a vertical stage to allow rapid remote selection of the detector. For quasi-simultaneous measurement of both reflectance and transmittance, the detector stage will also be mounted on a rotation stage to enable measurements as a function of incidence angle. In addition, a pyrometer for temperature measurement is planned.

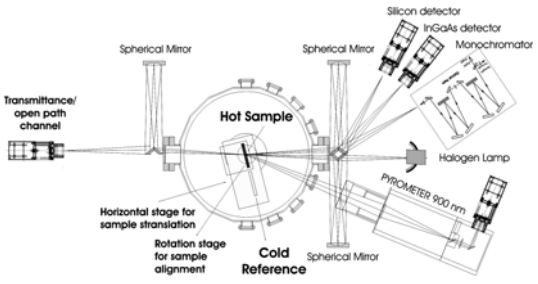

Figure 1. Schematic of the vacuum goniometer system

VALIDATION TESTS

Several validation tests have been performed. The stability of the system output including the effects of source, detector and electronics has been measured. The stability of a halogen source using a Si detector with a 905 nm filter is shown in Figure 2.

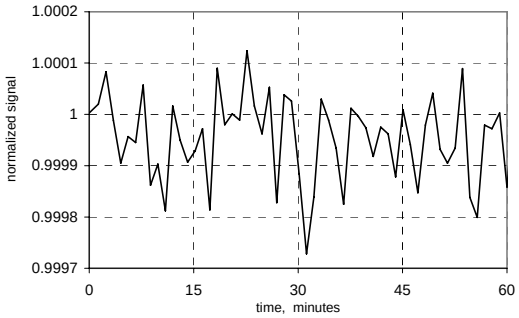

Figure 2. Stability of halogen source, detector, and electronics, measured with a 905 nm filter radiometer

The stability, beam shape, and power (between 15 mW and 40 mW) and spectral profile of the diode lasers have also been measured. The results for the laser sources were not completely satisfactory. Efforts are currently underway to improve the laser performance in the future. Here we will restrict our discussion to the halogen source. The monochromator was calibrated using Ar emission lines. The sample temperature stability was found to be better than 0.04 K, using our PID-control

For an evaluation of the measurement accuracy of the system, we measured the relative reflectance of Si and Au samples which have been previously calibrated to a relative expanded uncertainty of 0.2 % ($k = 2$), which becomes 0.3 % for the reflectance ratio [2]. Table 1 shows the results for two wavelengths (of the filter radiometers). The ratios obtained are in good agreement with the calibration values obtained elsewhere at NIST.

Table 1. Comparison of measured data with certificate data

Wavelength nm	Si/Au ratio, Measured	Si/Au ratio, Certificate	Ratio Measured to Certificate
905 P polarized	0.3298	0.3294	1.0012
950 P polarized	0.3284	0.3273	1.0034
905 S polarized	0.3297	0.3299	0.9994
950 S polarized	0.3287	0.3280	1.0021

RESULTS

For validation of the operation of the new system, several tests have been performed. A number of <100> silicon wafers with and without coatings have been provided by various RTP instrument manufacturers and vendors. From these samples, 1 inch diameter coupons have been cut for measurement. The coating materials and thicknesses are provided by the manufacturers. The angle of incidence for the measurements is 2.7°. The measurements were carried out in nitrogen or argon. The oxygen level of the purge gas was monitored and remained below 20 ppm.

Figure 3 shows the spectral reflectance of a <100> silicon sample with a 200 nm SiO_2 layer and a 110 nm polysilicon layer for several temperatures between 20 °C and 800 °C. The interference due to the transparent coating results in an emissivity of 1 near 850 nm. The dependence of the spectral reflectance at 800 °C on changes in the coating layer thicknesses is shown in Figure 4. As in Figure 3, interference effects dominate the spectral behavior.

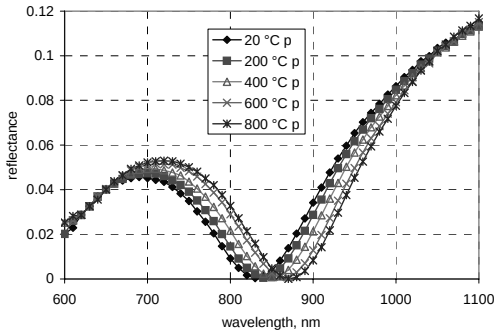

Figure 3. Temperature dependent spectral reflectance of a <100> silicon sample with a 200 nm SiO_2 layer and a 110 nm polysilicon layer.

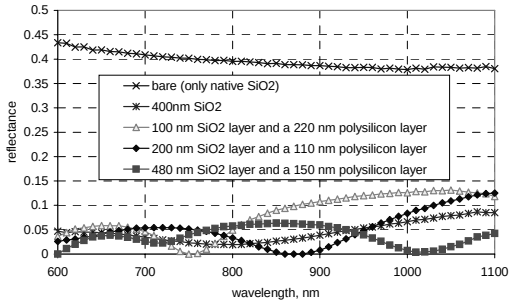

Figure 4. Spectral reflectance of different samples at 800°C (s-polarization)

Figure 5 shows the effect of cycling on the reflectance of a Si sample with a 480 nm SiO_2-layer and 150 nm polysilicon-layer. Measurements were made at the two wavelengths, 905 nm and 950 nm for the p-polarization. The curves of heating and cooling do not repeat exactly. After the measurement, a visible change of the surface was observable. The reason of this change is not clearly identified at the moment. It may be a change in the oxide layer thickness [3].

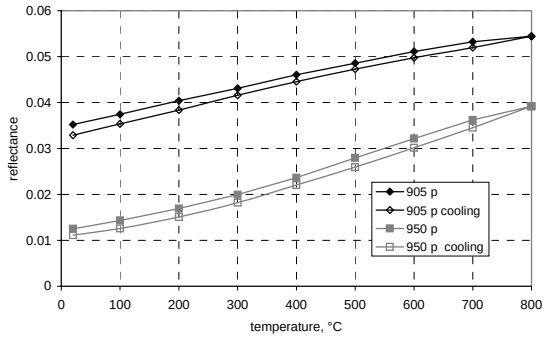

Figure 5. Effect of cycling on the reflectance of a Si sample with 480 nm SiO_2-layer and 150 nm polysilicon-layer

COMPARISON WITH NUMERICAL DATA

One short term goal of our research is to provide data to fill in the gaps in the existing knowledge of the optical properties of silicon to enable/confirm numerical modeling. Figure 6 shows measured data for a silicon sample with a Si_3N_4-layer compared with calculated values using different layer thicknesses at 600 °C using the model described in Reference [4]. Calculations using the different thicknesses allow the determination of the actual thickness, which in this case is 128 µm from Fig. 6. The agreement within the wavelength range from 700 nm to 1000 nm is very good.

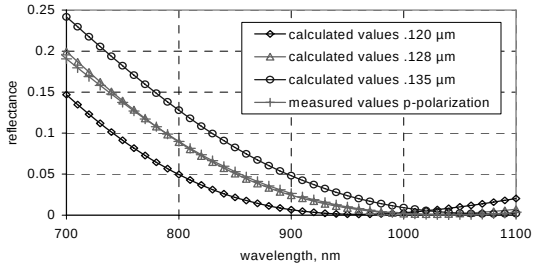

Figure 6. Comparison of measured and calculated values of a Si sample with a Si_3N_4-layer at 600 °C.

SUMMARY AND FUTURE DIRECTIONS

A new vacuum goniometer system has been developed to complement the capabilities of the existing systems for emissivity characterization at NIST. Its high temperature measurement capability expands our capacity to meet the growing needs of the semiconductor industry. The current results on Si and coated-Si samples are consistent with calibration and modeling results.

In the near term, our efforts will be directed at improving the temperature measurement accuracy and expanding the spectral and temperature ranges. Changes and contamination of the sample surface have to be discussed with industry experts and evaluated. We will perform a thorough comparison of the measured data with existing data in the literature and with theoretical models. Also measurement comparisons will be made with other spectrophotometric facilities at NIST and elsewhere.

REFERENCES

[1] Certain commercial equipment, instruments, or materials are identified in this paper to specify the experimental procedure adequately. Such identification is not intended to imply recommendation or endorsement by the National Institute of Standards and Technology, nor is it intended to imply that the materials or equipment identified are necessarily the best available for the purpose.

[2] E.A. Early, "Room temperature hemispherical and bi-directional reflectances," 11th IEEE International Conference on Advanced Thermal Processing of Semiconductors – RTP2003, pp. 133-136 (2003).

[3] J. Sik, J. Hora, and J. Humlicek, "Optical functions of silicon at high temperatures," Journal of Applied Physics **84**, pp. 6291-6298 (1998).

[4] B.J. Lee and Z.M. Zhang, "Development of experimentally validated optical property models for silicon and related materials," 11th IEEE International Conference on Advanced Thermal Processing of Semiconductors – RTP2003, pp. 143-150 (2003).