EMITTANCE STANDARDS FOR IMPROVED RADIATION THERMOMETRY DURING THERMAL PROCESSING OF SILICON MATERIALS

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ABSTRACT

Temperature measurement remains a serious challenge for the thermal processing of semiconductor materials. For advanced device structures, uncertainties of less than 2 K ($k = 2$) in the range 600 °C to 1100 °C are required to achieve high productivity and quality. Common manufacturing practice to establish confidence in temperature determinations is to employ process-repeatable standards such as oxide growth rate and sheet electrical resistance measurements. Radiation thermometry methods are highly repeatable in process tools, but the spectral emittance of the wafer material must be known to infer the wafer temperature from observed spectral radiance temperature. Test wafers with or without imbedded thermocouples with specified surface conditions are useful to establish an emittance scale for production wafers, but presently there are no traceable standards suitable for industry-wide use.

To improve traceable in-tool temperature measurement metrology, we have launched a program to generate a reliable knowledge base of emittance for selected semiconductor materials. This new initiative is made possible by collaboration on three fronts: with industry having the capabilities to fabricate well characterized, stable materials that are compatible with tool processing environments; through new optical properties measurements capabilities at NIST to perform reference quality measurements in the temperature and wavelength regions of interest; and through validated optical models of specular thin-film substrate systems. The candidate materials are stacks of oxides, polysilicon or nitride on polished silicon substrates, which provide for spectral emissivities ranging from 0.2 to 0.95 in the near infrared wavelength region. The materials are fabricated at room temperature and can be optically characterized by traceable reflectance measurements methods. Using optical models with measured thin-film thicknesses and known optical properties, it is possible to make estimates of the material reflectance and high temperature emittance. However, to establish high confidence in the estimates, it is necessary to validate the models against high temperature emittance measurements. In this paper we will present work in progress on experimental room and high temperature radiative properties measurements and optical modeling of selected candidate materials.

1. INTRODUCTION

For the past six years, collaboration with industry has been key to the success of the NIST Rapid Thermal Processing (RTP) temperature project. The project’s interactions with the RTP industry were reinforced annually at the NIST Common Interest Group meetings. Critical feedback given to NIST aided in planning and implementing NIST temperature research goals. Since the beginning of these meetings, industrial partners have voiced a need for reduced temperature and emittance uncertainties by using emittance standards. The ideal suite of emittance standards would include a set of three to six silicon wafers with different coatings. These standards would exhibit reproducible emittance behavior with temperature and time at heating. A set of such standards with calibrated emittances versus temperature and wavelength would be invaluable in verifying in-situ emittance and temperature measurements. With the advent of the NIST high-temperature emittance facility, the possibility of assembling a practical emittance standard suite is being realized. At the RTP 2003 Meeting in Charleston, SC, representatives from the RTP industry discussed the need for emittance standards with measurements traceable to NIST and supported

NIST's efforts in this area [1].

2. THE EMITTANCE STANDARDS INITIATIVE

The industry-wide emittance standards initiative (ESI) outlined at RTP 2003 aimed at meeting the need for reliable radiative property values of semiconductor materials to support traceable RTP temperature measurements. Elements of the initiative were five-fold: invitation of participants, collection of samples, setup of equipment, measurement of samples, and reporting of results. RTP manufacturers and vendors were invited to participate in the initiative and to create a list of artifacts (along with the vendor name, model numbers, serial numbers, applicable temperature ranges, applicable wavelength ranges, and other relevant information). So far, four vendors have voluntarily submitted suites of potential emittance standards along with appropriate information about the wafers (diameter, coating material, coating thickness, doping material, type of polishing, type of processing). The wafers have been properly labeled and are being cut into 25.4 mm diameter samples. Temperature dependent emittance measurements of the ESI wafers will be performed at NIST. Emittance results will remain anonymous; however, NIST intends to prepare a blind summary of the results for presentation at the RTP 2004 Meeting in Portland, OR.

For the initial set of measurements, the temperature range will be restricted below 800 ºC. In the future, temperatures up to 1300 ºC may be possible. Two laser sources of wavelengths 0.9 µm and 0.95 µm have been employed in this first set. Depending on the vendor's applications, other wavelengths can be considered depending on the laser sources available. To fit the sample holder in the NIST high-temperature emittance facility, all samples are required to be about 25.4 mm diameter. Ideally, vendors desire to submit whole silicon wafers to be measured and returned. Currently, the facility is able to measure near-normal emittance, but directional emittance will be possible in the future. The high-temperature facility is designed to measure both transmittance and reflectance.

3. NIST ROOM-TEMPERATURE REFLECTANCE FACILITY

The emittance of a material is conveniently determined from its reflectance, which is turn can be used to validate optical models for estimating emittance at high temperatures. The Optical Technology Division of NIST maintains the national reference instruments for measurements of reflectance. Within the Division, the Spectrophotometry project is responsible for reflectance measurements at wavelengths from 250 nm to 2500 nm on non-fluorescent materials at room temperature. The reflecting properties of a material can be categorized as either specular (mirror-like), diffuse, or a combination of specular and diffuse. In addition, the results depend upon the geometry of the measurement. In general, the reflectance of a specular material is measured using a bi-directional geometry (incident and reflected radiant flux at fixed directions) while a diffuse material is measured using a directional-hemispherical geometry (incident or reflected radiant flux at a fixed direction, the other flux at all directions).

The Spectrophotometry project has reference and commercial instruments for measurements at both geometries. A schematic of the Spectral Tri-function Automated Reference Reflectometer (STARR) instrument is shown in Fig. 1. A goniometer is used for bi-directional reflectance measurements of specular materials. The expanded relative uncertainty (k = 2) for specular reflectance is 0.1 %. The integrating sphere is used for directional-hemispherical measurements of diffuse materials. The expanded relative uncertainty (k = 2) for diffuse reflectance factor is 0.4 %. A commercial spectrophotometer with an integrating sphere accessory using standards calibrated with STARR is available for more rapid measurements. The commercial instrument contributes approximately 0.1 % (k = 2) to the uncertainty [2].

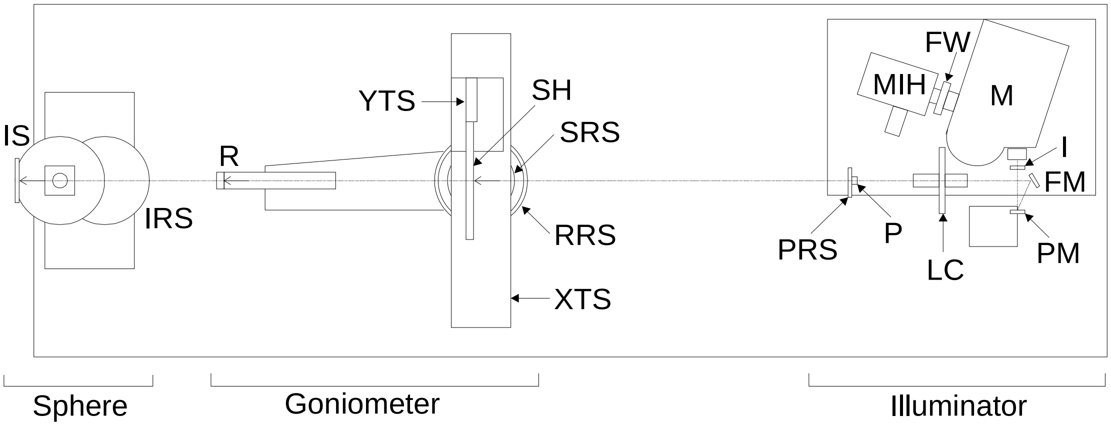

Figure 1. Schematic of STARR with the systems and components labeled. The line with the open arrows indicates the path of the illumination beam. The Illuminator system consists of the MIH – monochromator illuminator housing, FW – filter wheel, M – monochromator, I – iris, PM – parabolic mirror, FM – fold mirror, LC – light chopper, P – polarizer, and PRS – polarizer rotation stage. The Goniometer system consists of the SRS – sample rotation stage, RRS – receiver rotation stage, SH – sample holder, XTS – x-translation stage, YTS – y-translation stage, and R – receiver. The Sphere system consists of the IRS – integrating sphere rotation stage and IS – integrating sphere.

Because the samples for the ESI are specular reflectors, the goniometer of STARR is used to measure their reflectance at room temperature. Reflectance is determined using an absolute technique in which both the incident and reflected radiant fluxes are measured and the reflectance is simply the ratio of the fluxes. The sample is mounted on the goniometer with its front surface on the axis of rotation. The receiver rotates about this same axis. The incident radiant flux is measured by translating the sample out of the incident beam and rotating the receiver to collect the entire beam. The sample is then centered on the incident beam and rotated to the desired angle of illumination. The receiver is rotated to the specular direction and measures the reflected radiant flux. The reflectance is usually measured for polarizations of the incident radiant flux both parallel and perpendicular to the plane of illumination. Sample with sizes from 25 mm to 300 mm can be accommodated and the angle of illumination can range from 2.5° to 85°, depending upon the size of the sample.

For an opaque sample, and assuming equivalence between emittance and absorptance from Kirchhoff's Law, the reflectance ρ is related to the emmissivity ε by

$$\varepsilon(\theta';\lambda;T) = 1 - \rho(\theta;2\pi;\lambda;T) \ , \qquad (1)$$

where θ and θ' are the polar angles of the incident and exitent radiant flux, respectively, 2π indicates the reflected radiant flux is collected over the entire hemisphere above the sample, λ is the wavelength of the radiant flux, and T is the temperature of the sample. For specular samples, the reflected radiant flux is confined to one direction, $\theta' = \theta$, so Eq. (1) becomes

$$\varepsilon(\theta';\lambda;T) = 1 - \rho(\theta;\theta';\lambda;T) \ . \qquad (2)$$

Therefore, by measuring the specular reflectance of a sample at a specified illumination angle, the emittance of the sample at that same angle is determined.

4. NIST HIGH-TEMPERATURE EMITTANCE FACILITY

Measurement instrumentation and methodology for temperature dependent emittance of solid materials are under development in the NIST Fourier Transform Spectrophotometry Laboratory [3]. The effort is directed to support US industrial needs for emittance data and standards for a broad range of applications including RTP. A vacuum goniometer (VGEM) for reflectance and transmittance measurement has been developed for the characterization of the emittance of RTP samples, including coated wafers used in RTP [4]. This system uses the indirect method of reflectance and transmittance measurement to obtain emittance data. Several requirements specific to RTP needs have been addressed in this system: (1) the susceptibility of several materials of interest to oxidation at elevated temperatures requires the use of a chamber that can be evacuated or filled with a high purity neutral gas; (2) much interest is in emittance at specific wavelengths such as 900 nm and 950 nm, so diode laser sources and filtered detectors can be used to reduce the effects of sample emitted light on the detectors in reflectance and transmittance mode measurements; (3) a monochromator is used to provide spectral complement to the single wavelength laser sources; and (4) since the samples of interest can be provided in a low scatter form (specular polished wafers), hemispherical detection (e.g. using an integrating sphere) is not required and a goniometer arrangement can provide absolute reflectance and transmittance results.

This system makes use of a vacuum chamber that encloses the sample and the main portion of the measurement system. It can be operated under moderate vacuum or flushed with an inert gas to enable measurement of samples susceptible to oxidation. Light for measurement is brought via optics or fibers to window entrance and exit ports. The goniometer inside the chamber consists of the sample heater assembly and motion stages that enable absolute reflectance and transmittance measurements for non-scattering samples. Sample stages shown in Figure 2 allow sample rotation for alignment and angle change, and translation for cold reference and empty sample measurements. For relative spectral measurements a halogen lamp source and external monochromator (or set of narrow band filters) are used. The spectral range of the monochromator/detectors is continuous from 900 nm to 2300 nm.

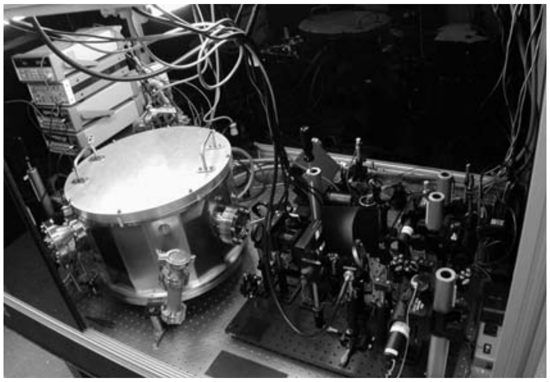

Figure 2. Photograph of the measurement system for the VGEM consisting of a source/selection portion, goniometer for reflectance and transmittance, and detector portion. Both broadband and laser diode sources are used. The detection system includes a monochromator, filtered and unfiltered detectors. Fiber optics are used for both source input and monochromator output.

The emittance measurement procedure follows these steps: (1) alignment of the sample and detector units using position sensitive detectors for a given incidence angle; (2) absolute absorptance measurement ($\alpha = 1 - \rho - \tau$) using a laser source (in collimated or converging beam geometry); (3) relative spectral absorptance measurement using the halogen lamp and monochromator (in converging beam geometry); (4) measurement of the sample temperature t_0 using the filter radiometer (in converging beam geometry) which will be checked against thin-film thermocouple instrumented test wafers; and (5) spectral directional emittance at temperature t_0 and given incidence angle is equal to the spectral directional absorptance. For both transmitted and reflected light, a set of detectors mounted on a translation stage is used. Alignment of the sample and detection optics will likely be the primary factor in setting a limit on the emittance measurement accuracy. Lasers and quadrant detectors provide high resolution monitoring of the sample position and orientation as stages are moved. The sample rotation stage and detector vertical stage will be used to maintain alignment for all measurements. The second detector (Si or InGaAs) of the set will be used for measurements with a laser source. The third will be an optical fiber bundle connected to the external monochromator. And the last filtered detector will provide radiance measurement for the sample temperature determination.

5. THIN-FILM OPTICAL MODELS

Existing optical property models have been surveyed and chosen to cover the spectral region of interest in a recent paper [5, 6]. Radiative properties of multilayer structures were determined using thin-film optics and were compared with measurements of selected silicon samples using the NIST spectrophotometer in the wavelength region from 0.5 µm to 1 µm. The excellent agreement between the measured and calculated reflectance suggests that the expressions for the model of refractive index of silicon is valid at room temperature. It can also be inferred that the assumptions about the optical properties of silicon dioxide and silicon nitride are appropriate at room temperature. Models have been developed to predict the spectral radiative properties of silicon at elevated temperatures. Results from these models will be compared with the measurements from the high-temperature emittance facility at NIST and further improvement on the models will be made.

6. FUTURE WORK

We have completed two parts of the five-fold emittance standards initiative at NIST. At the present, we are finishing up the setup of the equipment and the sample wafers. In the next few months, we expect to produce data at elevated temperatures for comparison. Our goal is to present preliminary emittance data of the standards as a function of temperature and the comparison of the different emittance suites at RTP 2004.

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