National Institute of Standards & Technology

Certificate

Standard Reference Material 2535

Ellipsometric Parameters Δ and ψ and Derived Thickness and Refractive Index for a Silicon Dioxide Layer on Silicon

Serial No.

This Standard Reference Material (SRM) consists of a 76-mm (3-in) diameter silicon wafer on which a uniform silicon dioxide layer was grown. It is certified for the ellipsometric parameters delta, Δ, and psi, ψ, at the vacuum wavelength λ = 633.0 nm using the High-Accuracy Ellipsometer built at NIST [1]. The SRM is issued primarily to evaluate the accuracy of ellipsometers and may also be used as an aid in the calibration of various other optical thickness-monitoring instruments. Each SRM unit is individually measured; thus the certified values provided on page 3 apply only to the unit identified by the above serial number.

This SRM is also certified for derived values of the thicknesses and indices of refraction of both layers of a two-layer optical model of an oxide film on a single-crystal silicon substrate. The model consists of a homogeneous, isotropic, top layer separated from a homogeneous, isotropic substrate by a homogeneous isotropic interlayer. The substrate is characterized by a complex index of refraction, n_s; the interlayer is characterized by a thickness, t_i, and a real index of refraction, n_i; and the top layer is characterized by a thickness, t_f, and a real index of refraction, n_f. The bottom layer is interpreted as single-crystal silicon and the top layer as amorphous silicon dioxide. The physical interpretation of the interlayer is less straightforward, being a research topic, but the fit to the ellipsometric data is greatly improved by inclusion of the interlayer. Some applications may not be sensitive to the interlayer in the optical model in the same way as is ellipsometry. For this reason, the uncertainties stated on this certificate apply only when the SRMs are used in ellipsometric measurements.

The certified values along with noncertified supplemental information are given on page 3 of this certificate. The supplemental information includes values of Δ and ψ at 50°, 55°, 60°, 65°, 70°, and 75° angles of incidence that were calculated using the two-layer model. The thickness for a one-layer model of the oxide is also given.

It is not possible to determine n_s, t_i, n_i, t_f, and n_f from the measurements of Δ and ψ on a single wafer. Therefore, the derivation of the values reported in this certificate was carried out on a batch of wafers as described in Ref. [2] with the following exceptions: wafers with nominal 10-nm, 14-nm or 25-nm oxides may have been included in the data reduction. A more accurate value, 0.0156, was used for the imaginary part of n_s [3].

The SRM was fabricated in the Semiconductor Processing Research Laboratory by M.L. Miller and D.B. Novotny of the NIST Semiconductor Electronics Division. The ellipsometric measurements leading to certification were performed by B.J. Belzer of the NIST Semiconductor Electronics Division. Guidance on the statistical analysis was performed by M.C. Croarkin of the NIST Statistical Engineering Division.

The overall direction and coordination of the technical aspects of this SRM were performed by B.J. Belzer, N.V. Nguyen, D. Chandler-Horowitz, J.F. Marchiando, and D.L. Blackburn of the NIST Semiconductor Electronics Division.

The support aspects involved in the certification and issuance of this SRM were coordinated through the Standard Reference Materials Program by N.M. Trahey.

Gaithersburg, MD 20899
September 12, 1994
(Revision of certificate dated 4-15-92)

Thomas E. Gills, Chief
Standard Reference Materials Program

(over)

Cleaning and Handling: The SRM should be stored in the clean wafer container supplied, and should be handled only with proper wafer tweezers. Even when the SRM is kept in its clean container, a film forms on the surface of the oxide. This unwanted film forms rapidly, and can be removed by rinsing the SRM with reagent grade ethanol or isopropanol and then with deionized water. Both solvents, the alcohol and the water, can be blown off the wafer surface with ultra-clean nitrogen. The sample must then be permitted to stabilize in the atmosphere 20 to 30 minutes prior to continuing with the measurements. When the surface is prepared in this manner, the measurements are reproducible for several hours or more. If the SRM becomes excessively dirty from mishandling, improper storage, or any other reason, the wafer should then be cleaned using a semiconductor processing grade detergent and rinsed thoroughly with deionized water. No other cleaning method is recommended.

REFERENCES

[1] Candela, G.A., and Chandler Horowitz, D., An Ellipsometry System for High Accuracy Metrology of Thin Films; SPIE Vol. 480 Integrated Circuit Metrology II, p. 2 (1984).

[2] Candela, G.A., Chandler-Horowitz, D., Marchiando, J.F., Novotny, D.B., Belzer, B.J., and Croarkin, M.C., Preparation and Certification of SRM-2530, Ellipsometric Parameters Δ and ψ and Derived Thickness and Refractive Index of a Silicon Dioxide Layer on Silicon; NIST Special Publication 260-109 (1988).

[3] Geist, J., Schaefer, A.R., Song, J-F., Wang Y.H., and Zalewski, E.F., An Accurate Value for the Absorption Coefficient of Silicon at 633 nm; J. Res. NIST **95**, 549-558 (1990).

ADDENDUM
TO
CERTIFICATE
STANDARD REFERENCE MATERIAL 2530 SERIES

The Standard Reference Materials (SRMs) described in the Certificate of Analysis accompanying this Addendum are fabricated, measured, and certified as described in NIST Special Publication 260-109 (SP 260-109) with the following exceptions:

1) The SRMs are now fabricated without any lithography following the growth of the thermal oxide on the 76.2-mm diameter silicon substrate. This is in response to customer requests for access to the center of the wafer, and to NIST experience that some of the lithographic steps cause marginal decreases in oxide quality.

2) The SRMs are now measured and certified over a 5-mm-diameter area at the center of the wafer instead of over two larger areas in windows on each side of the center of the wafer. Each SRM is measured twice: for the first measurement, the SRM is aligned on the vacuum chuck so that the wafer flat is vertical and the center of the laser beam is concentric with the center of the wafer to within 1-mm. For the second measurement, the SRM is aligned in the same way except that the wafer flat is horizontal. If the two measurements differ by more than 0.10-nm, the wafer is rejected. Otherwise, the mean of the two measurements is reported as the certified thickness within a 5-mm-diameter circle at the center of the wafer.

3) For the purposes of the data analysis, a lot of wafers is defined to be a set of at least three batches of wafers satisfying the following conditions: each batch of wafers consists of at least five wafers with oxides grown in a furnace at the same time. A lot, then, consists of batches of wafers with nominal 50-, 100-, and 200-nm oxides, where all of the batches were grown at the same temperature with the same gas-flow conditions in the same furnace. Batches of wafers with other thicknesses may be included in a lot.

Some applications may not be sensitive to the interlayer in the optical model of the SRMs in the same way as is ellipsometry. In those cases,

$$t_0 = t_f + t_i/2 \pm (\sigma_f^2 + \sigma_i^2/4 + \delta_i^2)^{0.5}$$

should be used as the oxide film thickness. In this equation, σ_f and σ_i represent the uncertainties stated after t_f and t_i on the Certificate of Analysis, and $\delta_i = t_i$ represents an uncertainty chosen to cover alternate physical interpretations of the interlayer. The former two uncertainties, which are based on replication and propagation of bounds for a number of small uncorrelated errors through the equations describing the ellipsometric measurement and analysis, are quite different from the latter uncertainty, which is based on physical arguments.

The plausibility of the uncertainty associated with calculations of thickness based on ellipsometric determinations of the parameters Δ and ψ identified for the SRM 2530 series has been tested by means other than ellipsometry. A measurement of the thickness of the oxide in a specimen SRM has been carried out with transmission electron microscopy (TEM). The TEM image included the entire thickness of the oxide and an even greater thickness of the silicon substrate and provided sufficient resolution to image the silicon lattice planes for use as a length standard. The reported oxide thickness resulting from this measurement was 102.8-nm. The corresponding certified value, calculated and reported in accordance with the information given in the certificate, is 103.7 ± 1.2 nm. The 0.9-nm difference between these values falls within the estimated uncertainty for the NIST certified values.

In addition, work comparing measurements made with the NIST High-Accuracy Ellipsometer and a NIST profilometer was carried out and reported in Ref. [1]. Examination of the data presented in this reference shows agreement between the two methods to within 3-nm for specimen SRMs having oxides nominally 50-nm thick (the ``on-chrome'' profilometry data agree with the ellipsometrically derived data to within 1 nm). The NIST Precision Engineering Division has provided unpublished data that demonstrates the estimated uncertainty for these profilometer measurements to be ± 3.8 nm, expressed in the same manner as the estimated uncertainty stated in the previous paragraph.

REFERENCE

[1] Candela, G.A., Chandler-Horowitz, D., Novotny, D.B., Vorburger, T.V., and Giauque, C.H.W., Film Thickness and Refractive Index Standard Reference Material Calibrated by Ellipsometry and Profilometry, Proc. SPIE **661**, 402-407 (1986).