National Institute of Standards & Technology

Certificate

Standard Reference Material® 2547

Silicon Resistivity Standard - 200 ohm·cm Level

This Standard Reference Material (SRM) is intended primarily for use in the determination of sheet resistance and bulk resistivity using the DC four-point probe method [1,2]. SRM 2547 is a nominal 100 mm diameter float zone n-type silicon wafer with (111) crystallographic orientation, doped with phosphorus by the Neutron Transmutation Doping (NTD) process. Wafer preparation was performed utilizing a simultaneous two-sided lapping process with a nominal 7 µm aluminum oxide abrasive.

This wafer unit was individually measured. The certified sheet resistance and resistivity values at the wafer's center are given in Table 1a and the sheet resistance is given at 60° increments on the radii of 5 mm and 10 mm circles in Table 1b. The stated uncertainty values for this unit are derived from a statistical analysis of the entire lot of wafers produced at the 200 ohm•cm resistivity level. A summary of these uncertainties is given in Table 2.

Previous silicon resistivity SRMs have also been used as de facto standards for calibration of noncontact (eddy current) instruments and/or with other material systems such as compound semiconductors. This SRM has similar utility, but it must be emphasized that NIST has rigorously investigated the DC four-point method on bulk silicon only. This SRM cannot be used as a thickness reference standard.

Expiration of Certification: The certification of this SRM is deemed to be indefinite, provided the SRM is handled and stored in accordance with the Care and Handling and Cautions to User sections of this certificate. However, the certification will be nullified if the SRM is damaged, contaminated, or modified. It is possible to take several thousand individual probe measurements before performance degradation occurs. Any degradation due to probe damage is evidenced by a shift in measured resistivity and a significant increase in measurement standard deviation. When such damage occurs, the certification is invalidated and the SRM should no longer be used.

The doped bulk silicon material was supplied by Topsil Semiconductor Materials A/S, Frederikssund, Denmark.

Statistical guidance and analysis were provided by M.C. Croarkin of the NIST Statistical Engineering Division.

Resistivity measurements for this SRM were performed by D.R. Ricks and J.M. Thomas of the NIST Semiconductor Electronics Division.

The coordination and overall direction of this SRM were provided by J.R. Ehrstein of the NIST Semiconductor Electronics Division.

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

David G. Seiler, Chief
Semiconductor Electronics Division

Thomas E. Gills, Chief
Standard Reference Materials Program

Gaithersburg, MD 20899
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See Certificate Revision History on Last Page

Table 1a. Certified Values and Expanded Uncertainties at Center of SRM 2547

Sheet Resistance* (Center) (Corrected to 23 °C)	Resistivity (Corrected to 23 °C)
ohms $^{+5.7}_{-7.8}$ ohms	ohms $^{+0.36}_{-0.58}$ ohms

Table 1b. Certified Values and Expanded Uncertainties on the 5 mm and 10 mm Radius Measurement Circles of SRM 2547

Sheet Resistance* (Corrected to 23 °C)		
Angle	5 mm Radius	10 mm Radius
0° ± 3°	ohms $^{+7.0}_{-9.0}$ ohms	ohms $^{+7.0}_{-9.0}$ ohms
60° ± 3°	ohms $^{+7.0}_{-9.0}$ ohms	ohms $^{+7.0}_{-9.0}$ ohms
120° ± 3°	ohms $^{+7.0}_{-9.0}$ ohms	ohms $^{+7.0}_{-9.0}$ ohms
180° ± 3°	ohms $^{+7.0}_{-9.0}$ ohms	ohms $^{+7.0}_{-9.0}$ ohms
240° ± 3°	ohms $^{+7.0}_{-9.0}$ ohms	ohms $^{+7.0}_{-9.0}$ ohms
300° ± 3°	ohms $^{+7.0}_{-9.0}$ ohms	ohms $^{+7.0}_{-9.0}$ ohms

* Values for sheet resistance may be converted to resistivity values by multiplying by the wafer thickness.

Certified Value Uncertainties: The uncertainties for the certified values were calculated in accordance with the ISO and NIST Guides [3]. The sources of uncertainty are identified below, with a more detailed explanation given in Reference [4]. It is assumed that rectangular distributions underlie all Type B uncertainty determinations; for a rectangular distribution, the variance is one-third of the half-width. The combined uncertainty is obtained from the square-root of the sum of the Type A and Type B variances. The expanded uncertainty is obtained by multiplying this combined uncertainty by a coverage factor; following NIST guidelines, a coverage factor of two is used, and the expanded uncertainty is written as a 2 σ value. There are 26 effective degrees of freedom for the uncertainty of center averages and 66 effective degrees of freedom for the uncertainty of individual measurements.

The following components of uncertainty were determined by Type A evaluations: 1) electrical measurement process precision (short term) was estimated from a pooling of the wafer center measurement variances of all the wafers (approximately 125) in this batch with comparable variances from pre- and post-certification control experiments; 2) day to day and longer term process variability was obtained from comparison of pre- and post-certification control experiment values and from check wafer data taken during the course of the certification; 3) the effect of using a single four-point probe, out of all possible probes, for certification was estimated from the control experiments which utilized multiple probes; 4) an unusual effect whereby the measured resistivity tends to decrease modestly for wafers that are reprobed in a time interval on the order of hours to days; (this effect is responsible for the assymetric uncertainty interval given in the tables). The combined variance from the above sources applies both to sheet resistance and to resistivity values. An additional term for the typical variation of thickness values for individual wafer units is included in the uncertainty for resistivity values, but not in that for sheet resistance. (Note: the electrical measurement process precision contributes only one-sixth as much to the uncertainty of the average (of 6 measurements) as it does to the uncertainty of the individual measurements.)

Type B determinations were made for the following sources of uncertainty that affect both the resistivity and the sheet resistance values: 1) digital voltmeter and standard resistor calibration uncertainty; 2) uncertainty in the measurement of wafer temperature that affects correction of the measured values from ambient temperature to 23 °C. An additional source evaluated by Type B determination that affects only the resistivity value: uncertainty of transfer of thickness scale from gauge blocks through the thickness tool to the wafer.

Table 2. Summary of Uncertainty for Individual Measurement Values

	Sheet Resistance	Resistivity
Total Type A Uncertainty	3.2 ohms	0.20 ohm•cm
Total Type B Uncertainty	1.4 ohms	0.10 ohm•cm
Combined Uncertainty (1σ)	+3.5 ohms -4.5 ohms	+0.22 ohm•cm -0.29 ohm•cm
Expanded Uncertainty (2σ)	+7.0 ohms -9.0 ohms	+0.44 ohm•cm -0.57 ohm•cm

Measurement Procedure: Each unit was individually measured by using a single in-line probe with nominal 1.59 mm probe point separation and meeting the specifications of ASTM F84 [1] for bulk silicon. The dual-configuration method [2,5] of four-point probe measurements was used, with configuration “□” being identical to that in ASTM F84. Eighteen measurements were taken on each unit: six were taken at the wafer center, separated by rotations of approximately 30° (these are averaged and reported for the center as a single location); six were taken at sites separated by rotations of 60° around a circle of radius 5 mm (0.2 in); and six were taken at sites around a circle of radius 10 mm (0.4 in), again with rotational separations of 60°. The measurements described by these two circles started on the positive X-axis (negative Y-axis is normal to the wafer flat) and proceeded counter clockwise. Due to the nature of resistivity variations in silicon, the values certified for these circles cannot be used to predict the resistivity in other regions of the wafer.

A DC current value was selected to give a measured specimen voltage between 9.95 mV and 10.05 mV in configuration “□” for the first measurement at the wafer center. This current setting was maintained on the current supply for all subsequent measurements. Values of current at each of the measurement sites were determined to five or more digits using a standard resistor.

Thickness uniformity for the region of the electrical measurements was evaluated for each wafer by using the nine-site plan of ASTM Method F84, with a radius of approximately 19 mm for the locations of the corners of this pattern. All points were required to have a thickness within 1 µm of the value at the center of the wafer to be acceptable for use as an SRM. The average of all nine thickness measurements for this wafer is given in Table 3, and should be used to convert from sheet resistance to resistivity for each of the electrical measurement sites.

Other possible sources of uncertainty were considered but were estimated to be negligible based on experience. A detailed description of the certification procedure, measurement process control, and uncertainty analysis is contained in Reference [4].

Table 3. Measurement Conditions for Certification

Date of Certification Measurements: ______________________________

Average Wafer Thickness:

__________ µm ± __________ µm

Nominal Measurement Current: 15 µA

Temperature of Heat Sink While Measuring at:

First Measurement Site: __________________________ °C

Last Measurement Site: __________________________ °C

Temperature Coefficient of Resistivity, C_T (first site) [1]: __________________________ %/°C

Relative Humidity: < 50 % RH

Correction Terms Used

1) Average factor to convert the voltage-to-current (V/I) ratio to sheet resistance, for configuration “□” at the wafer center:

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2) Correction factor related to ratio of wafer thickness to probe separation for calculating wafer sheet resistance:

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3) Correction for temperature to 23 °C (first site):

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Notes on SRM Use: A four-point probe with spacing equal to or greater than twice the wafer thickness effectively averages sheet resistance from front to back surfaces of a wafer. For this reason, either surface of the SRM wafer may be measured by the user’s four-point probe with negligible expected differences in the values measured.

When the user needs to convert sheet resistance values measured on this SRM to resistivity values, NIST recommends using the thickness value given on this Certificate to obtain best agreement with the certified resistivity value. Due to the lapped surface texture of this wafer, independent thickness measurements by the user are likely to result in a slightly different value from that reported here, particularly if a different type of thickness gauge is used. This would result in a degraded transfer of the resistivity scale using this SRM although it would have no bearing on the transfer of the sheet resistance value. Due to its lapped surface texture, **this wafer cannot be used as a reference material for wafer thickness.**

Care and Handling: The lapped surfaces of this SRM give it a general robustness and improved measurement stability. Nevertheless, reasonable care in handling, such as the use of gloves, finger cots, vacuum paddles, or tweezers should be exercised to maintain wafer cleanliness. Storage in any reasonable, noncorrosive, nontoxic environment, in conjunction with the packaging provided, should prove adequate for maintaining stability of the SRM. Cleaning with the proper surfactant or with isopropyl alcohol, together with thorough rinsing in deionized water and thorough drying, has been found acceptable for removing most surface stains. The use of hydrofluoric acid or of elevated temperature treatments is discouraged. The SRM should not be stored or measured in an ambient relative humidity above 50 %.

Cautions to User: NIST strongly recommends that this SRM not be used alone for calibration, or for testing the linearity or other performance characteristic of sheet resistance/resistivity measuring equipment. For any of these applications, use one or more of the other SRM wafers in the SRM series 2541 through 2547 in addition to this SRM.

NIST recommends the user generate secondary standards based on this SRM rather than employ it for routine use [6]. This is particularly important for users of four-point probe instruments which may gradually damage silicon wafers with repeated probing.

This SRM may not be appropriate for calibrating sheet resistance measurement instruments or for evaluating the sheet resistance/resistivity of materials other than silicon due to differences of material and instrument parameters relative to single crystal silicon wafers and to DC conduction measurements. Among the material parameters are conduction and carrier scattering mechanisms, grain boundary effects on conduction, and the relative importance of sample surface vs. bulk in responding to the measurement stimulus. Instrument parameters include electromagnetic frequency of the measurement stimulus, the associated skin depth in the materials being measured, and sampling volume of the instrumentation in relation to material grain size. Parties interested in extending the calibration use of these SRMs are advised to contact Dr. James R. Ehrstein of the NIST Semiconductor Electronics Division at (301) 975-2060.

Silicon has a noticeable temperature dependence for resistivity. Accurate values of the temperature coefficient of silicon resistivity are available for the range 18 °C to 28 °C [1]. If the user makes measurements at a temperature other than 23 °C, the resulting resistivity and sheet resistance values must be corrected to 23 °C for best agreement with the certified values. Corrections for measurements made outside of the 18 °C to 28 °C range may suffer from reduced accuracy.

The localized variations in resistivity typically found in silicon wafers such as those of this SRM may limit the user's ability to obtain exactly the certified values if instrumentation is employed having a sampling volume different from that of a 1.59 mm four-point probe. However, it is likely that imprecision of the user's instrumentation will be a more important limitation to transfer of the certified value than will differences in sampling volume.

REFERENCES

[1] ASTM Method F84-93 "Standard Test Method for Measuring Resistivity of Silicon Wafers With an In-Line Four-Point Probe," Annual Book of ASTM Standards **Vol. 10.05**, West Conshohocken, PA 19428.

[2] ASTM Method F1529-96, "Standard Test Method for Sheet Resistance Uniformity Evaluation by In-line Four-Point Probe with the Dual-Configuration Procedure," Annual Book of ASTM Standards **Vol. 10.05**, West Conshohocken, PA 19428.

[3] *Guide to the Expression of Uncertainty in Measurement*, ISBN 92-67-10188-9, 1st Ed. ISO, Geneva, Switzerland, (1993): see also Taylor, B.N., and Kuyatt, C.E., "Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results," NIST Technical Note 1297, U.S. Government Printing Office, Washington DC, (1994).

[4] Ehrstein, J.R. and Croarkin, M.C., Standard Reference Materials: The Certification of 100 mm Diameter Silicon Resistivity SRMs 2541 through 2547 Using Dual-Configuration Four-Point Probe Measurements, NIST Special Publication 260-131, (1997).

[5] Perloff, D.S., Gan, J.N., and Wahl, F.E., Dose Accuracy and Doping Uniformity of Ion Implantation Equipment, Solid State Technology, **Vol. 24**, No. 2, pp. 112-120, (1981).

[6] ASTM Guide F1527-94 "Standard Guide for Application of Standard Reference Materials and Reference Wafers for Calibration and Control of Instruments for Measuring Resistivity of Silicon," Annual Book of ASTM Standards, **Vol. 10.05**, West Conshohocken, PA 19428.

Certificate Revision History: 31 Mar 99 (This revision reports corrections to "Angle" listing, first column, Table 1b only; 20 Jun 97 (original certificate date)

It is the responsibility of users of this SRM to assure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: Phone (301) 975-6776 (select "Certificates"), Fax (301) 926-4751, e-mail srminfo@nist.gov, or via the Internet http://ts.nist.gov/srm.

APPENDIX

The SRM 2541 through SRM 2547 series is intended to replace the previously issued silicon resistivity sets, SRM

1521 through SRM 1523, and offer several comparative advantages: 1) larger size for better equipment compatibility; 2) better uniformity of both thickness and resistivity; 3) reduced measurement uncertainty due to an improved certification procedure; and 4) additional measurement sites for better characterization of resistivity uniformity.

Stability Tests: Silicon wafers from all previous resistivity SRMs have been monitored by NIST for as long as 15 years with no cumulative drift of the measured resistivity values. While there were some measurement values in that monitoring sequence that were outside the confidence limits determined from the certification uncertainty, those outlier values are believed to have been due to long-term measurement environment variability as well as to the use of single-configuration probe measurements which are more sensitive to changes of probes or to probe tip condition than are the dual-configuration measurements used for current SRM certification.

Tests of the stability of silicon resistivity reference materials with repeated probing have shown that it is possible to make several thousand probe measurements without significant change of value, even when the probing is confined to a very localized area. **However, the use of excessively sharp or damaged probes, or of unconditioned new probes, may accelerate the accumulation of crystalline damage** that leads to a shift in resistivity values or degraded measurement repeatability. This is particularly true for float zone or NTD silicon, both of which have low oxygen content and are more susceptible to crystalline damage from mechanical contact.

Multilaboratory Measurement Reproducibility: Test results of multilaboratory comparisons of four-probe resistivity measurements are useful for guidance on confidence intervals that might be expected when using this SRM to transfer resistivity scale values between NIST and the user's laboratory. While there have not been any reported multilaboratory tests using the dual-configuration method of measurement, a number of tests have been conducted by ASTM using the single-configuration methodology (ASTM F84). These tests yielded a multilaboratory precision of just under 0.7 % for wafers with resistivities up to 120 ohm•cm, and just under 1.7 % for wafers between 120 ohm•cm and 500 ohm•cm. These values lead to 99 % confidence intervals of ± 2 % for wafers up to 120 ohm•cm and ± 5 % for wafers between 120 ohm•cm and 500 ohm•cm [1]. Because of the relatively nonuniform, small (5 cm) diameter wafers used for those ASTM multilaboratory tests, the resulting confidence intervals are believed to be quite conservative compared to what could be expected on wafers with improved uniformity such as those from this SRM series, particularly if the dual-configuration four-point probe methodology were employed by all laboratories.

Equipment[1] Used for Wafer Qualification and for SRM Certification:

For the Determination of Thickness:

Random wafers - large area thickness uniformity prescreening:	ADE Model 6033T®
All wafers - certified thickness, 9 sites:	Haidenhain Certo 60®
Gauge blocks for calibration of Certo 60:	Webber Croblox® 0.60965 mm
	Webber Croblox 0.62500 mm
	Webber Croblox 0.66045 mm

For Electrical Measurements:

Current Supply:	Electronic Measurements Inc. Model #C 612
Standard Resistor:	1000 ohm, Leeds & Northrup, NBS Type #1592167
DVM:	Hewlett-Packard Model #3456
Probe:	Kulicke & Soffa Fell Probe #SRM 1

REFERENCE

[1] Bullis, W.M., Standard Measurements of the Resistivity of Silicon by the Four-Probe Method, Nat. Inst. Std. Tech. NBSIR 74-496, p. 75, (August 1974).

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