5.3.5 *FWHM* — acronym for full width at half maximum, the width of the absorbance band at half its magnitude as measured from the baseline.

5.3.6 *reference spectrum*, *n* — the spectrum of the reference specimen.

5.3.6.1 *Discussion* — For true double-beam instruments, it is obtained directly with the reference specimen in the sample beam and the reference (second) beam empty. For FT-IR and other single-beam instruments, it is the result of ratioing the single-beam spectrum of the reference specimen to the background spectrum (see Section 5.3.1).

5.3.7 *sample spectrum*, *n* — the spectrum obtained when the sample specimen is placed in the infrared beam.

5.3.7.1 *Discussion* — For true double-beam instruments, it is obtained directly with the sample in the sample beam and the reference (second) beam empty. For FT-IR and other single-beam instruments, it is the result of ratioing the single-beam spectrum of the sample to the background spectrum (see Section 5.3.1).

6 Summary of Test Method

6.1 At room temperature, test slices are prepared that are polished on both sides to a nominal thickness of 2.0 mm. At cryogenic temperatures, the thickness of the test slices can range from 2.0 to 4.0 mm.

6.2 A reference slice of known low carbon content (see Section 3.2) is prepared in the same manner.

6.3 After verifying that the instrument is suitably set up, transmittance spectra of the sample and reference are obtained over the range from 700 to 500 cm^{-1} (14.3 to 20.0 μm) on a double-beam dispersive or single-beam Fourier Transform Infrared (FT-IR) spectrophotometer in accordance with manufacturer's instructions.

6.4 Absorbance spectra are derived from these transmittance spectra and a carbon-only spectrum is obtained as the difference between the two absorbance spectra.

6.5 A baseline is drawn between the regions on both sides of the carbon peak on this difference absorbance spectrum, and absorbance values of both the peak and baseline are recorded.

6.6 The absorbance peak height is taken as the difference between these two values. This peak height, corrected for sample thickness, is multiplied by a constant to calculate the substitutional carbon concentration. Two constants are used, one for measurements at room temperature (300 K), and one for measurements at cryogenic temperatures (below 80 K).

7 Apparatus

7.1 *Infrared Spectrophotometer* — Either a dispersive (computerized or non-computerized) or a Fourier transform (FT-IR) spectrophotometer may be used. The resolution of the spectrophotometer must be at least 2 cm^{-1} at room temperature, or 1 cm^{-1} at cryogenic temperatures, over the range from 500 to 700 cm^{-1} for either dispersive or Fourier transform infrared spectrophotometers. The total operating range of the spectrophotometer shall include the range from 500 to 2000 cm^{-1}.

7.2 *Micrometer Caliper* — or other instrument, capable of measuring the thickness of the specimens to an accuracy of 0.005 mm.

7.3 *Equipment and Materials for Slicing and Polishing Silicon* — to a final thickness tolerance of 0.005 mm or less, and a total thickness variation of 0.01 mm or less.

7.4 *Thermometer* — or other instrument capable of measuring the temperature of the sample chamber (for room temperature measurements) or the sample holder (for cryogenic temperature [below 80 K] measurements) to within ± 2°C.

7.5 *Calcium Fluoride Crystal (CaF$_2$)*, cut to nominal thickness of 5 mm.

8 Sampling

8.1 Unless otherwise specified, a silicon slice used for the carbon test is to be measured at the nominal slice center.

9 Test and Reference Specimens

9.1 A single crystal slice of about 2-mm thickness must be used at room temperature, and a slice of 2.4 to 3.5-mm thickness is preferable at cryogenic temperatures.

9.2 Both the test and reference specimens must be carefully shaped to the following criteria:

9.2.1 Thickness variation over the measurement area shall be 0.005 mm or less.

9.2.2 Surface preparation shall be identical,

9.2.3 Thickness equality shall meet one of the following:

9.2.3.1 When the measurement is made with double beam simple dispersive infrared spectrophotometers, the final thickness of test and reference slices shall be equal to within ± 0.01 mm (see Section 10.4).

9.2.3.2 When the measurement is made with computer assisted double-beam dispersive or single-beam FT-IR spectrophotometers, the final thickness of test and

reference slices shall be equal to within ± 0.5 mm (see Section 10.5).

9.2.4 Surface area large enough such that with respect to the holders no incident radiation can bypass either the test or reference specimen.

9.3 The reference specimen must be selected from float zone silicon with a minimal substitutional carbon concentration and a carrier concentration such that there is no measurable free carrier absorption of infrared radiation in the range from 500 to 2000 cm^{-1}.

NOTE 1: A satisfactory method of selecting usable reference specimens is to prepare polished slices of equal thickness from many different low-carbon silicon crystals produced by the float zone method, and then compare them to each other in an infrared radiation (IR) spectrophotometer. The selection must be carried out at cryogenic temperatures for cryogenic measurements. The specimen(s) showing the highest relative transmittance at 605 cm^{-1} at room temperature, or at 607.5 cm^{-1} at cryogenic temperatures (below 80 K), can be used as reference specimen(s).

9.4 Both test and reference specimens must have resistivity greater than 3 Ω·cm for *p*-type silicon and greater than 1 Ω·cm for *n*-type silicon.

10 Procedure

10.1 *Instrumental Checks*

10.1.1 With reference to the transmittance spectrum, establish the 100% transmittance line to measure the stability and the noise level by one of the following methods.

10.1.1.1 For double-beam instruments, record the transmittance spectrum with both the sample and reference beams empty.

10.1.1.2 For single-beam instruments, record the transmittance spectrum as the ratio of two spectra taken with the sample beam empty. Take the two spectra at times separated by at least the time required to load samples and take two sequential spectra.

10.1.1.3 In both cases, plot the transmittance spectrum over the wavenumber range covering 500 to 700 cm^{-1} to obtain the 100% line.

10.1.1.4 If the 100% transmittance line is 100 ± 0.5% over the entire range, continue to the next step. Otherwise, adjust or repair the instrument so as to meet this criterion.

10.1.2 Establish the 0% transmittance line, (T).

10.1.2.1 For double-beam dispersive (DIR) instruments, record the transmittance spectrum between 700 and 500 cm^{-1} with the sample beam blocked.

10.1.2.2 For FT-IR instruments, collect a background spectrum with the beam empty. Next, collect a spectrum with a CaF$_2$ crystal wafer, 5-mm thick, in the beam. Obtain the spectrum that is the ratio of the CaF$_2$ spectrum to the background spectrum, and plot the percent transmittance spectrum between 700 and 500 cm^{-1}.

10.1.2.3 In either of these cases, the 0% transmittance must not exceed ± 0.5% over the range between 700 and 500 cm^{-1}.

10.1.2.4 Alternatively, for FT-IR instruments, plot the single-beam background spectrum (empty beam only) from 1000 to 200 cm^{-1}. The recorded energy in a region where the detector is known to give a zero response (below 300 cm^{-1} for most detectors) must not be greater than 1.0% of the maximum signal in this region.[4,5]

10.1.3 To determine the mid-scale linearity of the instrument obtain a spectrum of the silicon reference specimen over the wavenumber range from 1600 to 2000 cm^{-1}. If the value of the transmittance is not 53.8 ± 2% over this wavenumber range, align the sample at a small angle to the axis of the incoming IR beam, in order to minimize undesirable reflections between the silicon surfaces and the spectrometer components.

NOTE 2: This angle may be determined by initially placing the silicon slice normal to the axis of the incoming beam, and then gradually tilting the specimen while repeatedly obtaining the transmittance spectrum above 1600 cm^{-1}. The optimum angle is typically less than 10°.
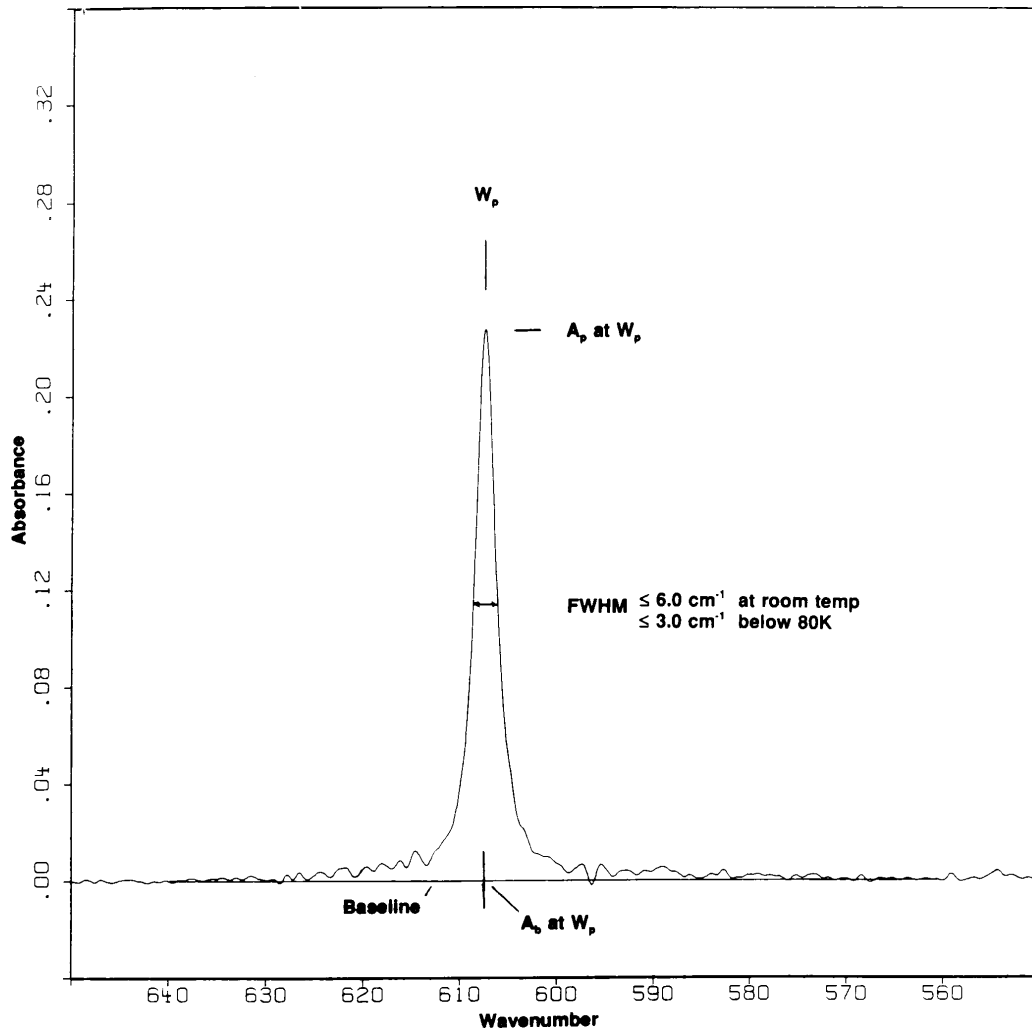
10.2 Measure and record the thicknesses of the test and reference specimens to within ± 0.005 mm, at their centers.

10.3 For room temperature measurements, measure and record the temperature of the sample chamber to ± 2°C. At cryogenic temperatures (below 80 K), measure and record the temperature of the sample holder.

10.4 Obtain the carbon-only absorbance spectrum by one of the following methods (see Figure 1):

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4 Chase, D. B., *Applied Spectroscopy* **38**(4), 491–494 (1984).

5 Hoffman, P., and Knozinger, E., *Applied Spectroscopy* **41**(8), 1303–1306, (1987).

Figure 1
Carbon-Only Absorbance Spectrum

10.4.1 For double-beam instruments, position the sample specimen in the sample beam and the reference specimen in the reference beam and collect and plot the carbon-only absorbance spectrum over the range of at least 700 to 500 cm^{-1}. Use a resolution of 2 cm^{-1} or better, at 605 cm^{-1}, at room temperature, and a resolution of 1 cm^{-1} or better, at 607.5 cm^{-1}, at cryogenic temperatures (below 80 K). Take spectra with the sample chamber in a vacuum, dry nitrogen, or dry air. Position the specimen so that the infrared beam passes through the center of the specimens.

10.4.2 For single-beam (computer-assisted) instruments, first collect a background spectrum with the beam empty, then collect sample and reference spectra sequentially under the same conditions as given in Section 10.4.5. Ratio the sample and reference spectra to the background and convert to absorbance.

Multiply the reference absorbance spectrum by the ratio of the test specimen thickness to the reference specimen thickness and subtract this normalized absorbance from the test specimen absorbance to obtain the carbon-only spectrum.

NOTE 3: If the user has interactive arithmetic capability, subtract the reference absorbance spectrum from the sample absorbance spectrum in a ratio that provides the best flatness of the non-peak baseline between 560 and 640 cm^{-1}. This eliminates the 2-phonon band due to silicon and gives a carbon-only spectrum.

10.5 With regard to the plot of the carbon-only absorbance spectrum over the range from 500 to 700 cm^{-1}, define the baseline by drawing a straight line from 560 to 640 cm^{-1}. Use the average in the regions from 550 to 570 cm^{-1}, and 630 to 650 cm^{-1}, to define the endpoints of the straight line.

10.6 For measurements at room temperature, locate the wavenumber corresponding to the maximum absorbance in the region from 603 to 607 cm^{-1}. For measurements at cryogenic temperatures (below 80 K), locate the wavenumber corresponding to the maximum absorbance in the region from 605 to 609 cm^{-1}.

10.6.1 Record the value of that wavenumber, to four significant figures, as W_p. Record the maximum absorbance as A_p, the absorbance of the absorption peak.

10.6.2 Record the baseline absorbances, A_b, as the value of the baseline defined in 10.5 at W_p.

10.6.3 Record both A_p and A_b to three significant figures.

10.7 Determine and record the full width at half maximum (FWHM) of the peak. If the FWHM of the peak is greater than 6 cm^{-1} (room temperature) or 3 cm^{-1} (cryogenic temperatures), the spectrum was not properly obtained, and the instrument needs further adjustment, or the specimen is under stress. See Section 3.4 for a list of factors that influence FWHM. Adjust the instrument in order to achieve the proper FWHM and repeat the procedure.

10.8 Record the apodization function and the number of zero-fills used (FT-IR instruments) or the spectral slit width for dispersive infrared (DIR) instruments.

11 Calculation

11.1 Calculate the absorption coefficient, α, using the expression:

$$\alpha = \frac{23.03}{X}(A_p - A_b) \qquad (1)$$

where:

X = test specimen thickness, mm,
A_p = the peak value of the carbon-only absorbance spectrum, and
A_b = the baseline value of the carbon-only absorbance spectrum.

NOTE 4: This equation does not include a correction factor for multiple reflections. Such a correction factor is not necessary, due to the very strong lattice absorption at 610 cm^{-1}.

11.2 For measurements at room temperature, calculate the substitutional carbon content in atoms/cm^3 or parts per million atomic (ppma) as follows:[6]

$$\text{Carbon Content} = 8.2 \times 10^{16}\,\alpha \text{ atoms/cm}^3 \qquad (2)$$
$$= 1.64\,\alpha \text{ ppma}$$

11.3 For measurements at cryogenic temperature, calculate the content of substitutional carbon in atoms/cm^3 or ppma as follows:

$$\text{Carbon Content} = 3.7 \times 10^{16}\,\alpha \text{ atoms/cm}^3 \qquad (3)$$
$$= 0.74\,\alpha \text{ ppma}$$

NOTE 5: The calibration factor used in this test method was determined as result of a study carried out in Japan by the Silicon Technologies Committee of the Japanese Electronic Industries Development Association (JEIDA), now JEITA,[1] in the mid-1980's. Further data analysis to eliminate recognized systematic error was carried out by the ASTM subcommittee assigned for this effort. The factor resulting from this study has an uncertainty of $\pm$ 0.4 $\times$ 10^{16} atoms/cm^2 or $\pm$0.08 ppma·cm for the room temperature measurements. The calibration factor for cryogenic measurements was calculated using the ratio given in Kolbesen and Mladcnović.[7] The uncertainty of the cryogenic temperature factor is therefore $\pm$ 0.2 $\times$ 10^{16} atoms/cm^2 or $\pm$ 0.04 ppma·cm.

12 Report

12.1 Report the following information:

12.1.1 The instrument used, the operator, and the date of the measurements,

12.1.2 Identification of test and reference specimens,

12.1.3 For room temperature measurements, the nominal temperature of the sample chamber. For cryogenic measurements, the nominal temperature of the sample holder,

12.1.4 Thickness of test and reference specimen,

12.1.5 Location and size of the illuminated area on the specimen,

12.1.6 For FT-IR instruments: (*a*) the apodization function used, (*b*) the amount of zero-filling, and (*c*) the number of data/cm^{-1}. For dispersive instruments: (*a*) the scan time/cm^{-1}, (*b*) the spectral slit width, and (*c*) the resolution at 600 cm^{-1},

12.1.7 Spectral full width at half maximum of the absorption peak,

12.1.8 W_p, wavenumber in cm^{-1} of the absorption peak,

12.1.9 The absorption coefficient due to substitutional carbon, α, in cm^{-1},

12.1.10 Carbon concentration, in ppma or in atoms/cm^3, and

12.1.11 The conversion coefficient used.

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6 Inoue, N., Stso. T. Nozaki, T., Endo, K., and Mizauma, K., "High Reliability Infrared Measurement of Oxygen and Carbon in Silicon," in *Emerging Semiconductor Technology,* ASTM STP 960, D. C. Gupta and P. H. Langer, eds., (ASTM, Philadelphia, 1987), pp. 365–377.

7 Kolbesen, B. O., and Mladcnović, T., *Kristall und Technik* **15**(1), K1–K3 (1980).

13 Precision

13.1 A round robin was carried out by JEIDA (now JEITA[1]). The results of this 21-laboratory study are shown in Table 1. Figure 2 shows that the sample standard deviation increases with concentration value.

13.2 A single-laboratory investigation of the precision of carbon measurement at cryogenic temperature with a Fourier transform spectrophotometer was conducted using a single sample analyzed once a day over a 30-day period (30 measurement values). From this single-laboratory, multiple-operator, multiple-day, single-sample investigation of precision of carbon measurement, the precision was found to be 0.02 ppma (2 standard deviations). Nominal carbon content of the sample was 0.16 ppma.

14 Bias

14.1 The bias of this test method cannot be evaluated because there are no available reference standards suitable for evaluating bias.
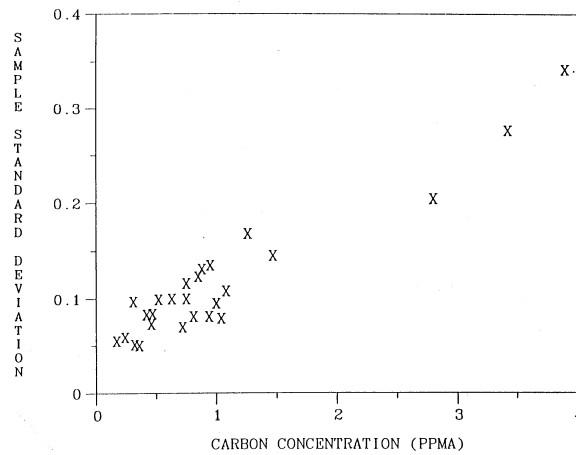
15 Keywords

15.1 carbon; infrared absorption; infrared spectroscopy; silicon; single crystal silicon

Table 1 JEIDA Round Robin Results

Sample Number	Number of Measurements	Mean Value of Concentration of Substitutional Carbon	Sample Standard Deviation
1	21	2.79	0.204
2	21	3.89	0.342
3	21	0.23	0.059
4	21	0.35	0.050
5	21	0.74	0.100
6	21	0.93	0.081
7	21	1.03	0.079
8	21	1.25	0.168
9	21	0.41	0.083
10	21	0.84	0.123
11	20	0.80	0.081
12	20	0.94	0.135
13	20	0.99	0.095
14	20	0.30	0.097
15	20	0.46	0.084
16	20	0.71	0.070
17	20	1.07	0.108
18	20	1.46	0.145
19	20	3.41	0.276
20	20	0.31	0.051
21	20	0.45	0.073
22	20	0.51	0.099
23	20	0.74	0.116
24	20	0.16	0.055
25	20	0.62	0.100
26	20	0.87	0.131

Figure 2
Round Robin Results

NOTICE: SEMI makes no warranties or representations as to the suitability of the standards set forth herein for any particular application. The determination of the suitability of the standard is solely the responsibility of the user. Users are cautioned to refer to manufacturer's instructions, product labels, product data sheets, and other relevant literature, respecting any materials or equipment mentioned herein. These standards are subject to change without notice.

By publication of this standard, Semiconductor Equipment and Materials International (SEMI) takes no position respecting the validity of any patent rights or copyrights asserted in connection with any items mentioned in this standard. Users of this standard are expressly advised that determination of any such patent rights or copyrights, and the risk of infringement of such rights are entirely their own responsibility.

SEMI MF1392-1103
TEST METHOD FOR DETERMINING NET CARRIER DENSITY PROFILES IN SILICON WAFERS BY CAPACITANCE-VOLTAGE MEASUREMENTS WITH A MERCURY PROBE

1 Purpose

1.1 Net carrier density is a critical parameter in growth of epitaxial layers of silicon. This test method provides a means for determining net carrier density without formation of a special diode structure on the layer. It may also be used in characterizing net carrier density in polished silicon wafers.

1.2 This test method can be used for research and development, process control, and materials specification, evaluation, and acceptance purposes.

1.3 In the absence of interlaboratory test data to establish its reproducibility (see Section 14.2), this test method should be used for materials specification and acceptance only after the parties to the test have established reproducibility and correlation.

2 Scope

2.1 This test method[1] covers the measurement of net carrier density and net carrier density profiles in epitaxial and polished bulk silicon wafers in the range from about 4×10^{13} to about 8×10^{16} carriers/cm^{-3} (resistivity range from about 0.1 to about 100 $\Omega \cdot$cm in n-type wafers and from about 0.24 to about 330 $\Omega \cdot$cm in p-type wafers).

2.2 This test method requires the formation of a Schottky barrier diode with a mercury probe contact to an epitaxial or polished wafer surface. Chemical treatment of the silicon surface may be required to produce a reliable Schottky barrier diode.[2] The surface treatment chemistries are different for n- and p-type wafers. This test method is sometimes considered destructive due to the possibility of contamination from the Schottky contact formed on the wafer surface; however, repetitive measurements may be made on the same test specimen.

2.3 This test method may be applied to epitaxial layers on the same or opposite conductivity type substrate. This test method includes descriptions of fixtures for measuring substrates with or without an insulating backseal layer.

2.4 The depth of the region that can be profiled depends on the doping level in the test specimen. Based on data reported by Severin[2] and Grove,[3] Figure 1 shows the relationships between depletion depth, dopant density, and applied voltage together with the breakdown voltage of a mercury silicon contact. The test specimen can be profiled from approximately the depletion depth corresponding to an applied voltage of 1 V to the depletion depth corresponding to the maximum applied voltage (200 V or about 80% of the breakdown voltage, whichever is lower). To be measured by this test method, a layer must be thicker than the depletion depth corresponding to an applied voltage of 2 V.

2.5 This test method is intended for rapid carrier density determination when extended sample preparation time or high temperature processing of the wafer is not practical.
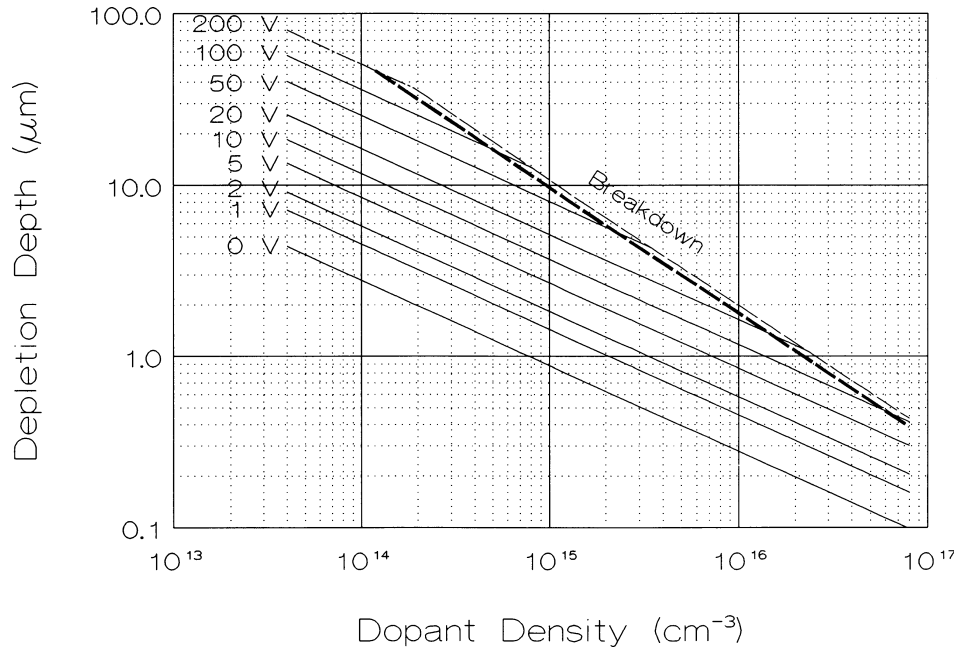
2.6 This test method provides for determining the effective area of the mercury probe contact using polished bulk reference wafers that have been measured for resistivity at 23° C in accordance with SEMI MF84 (see Note 1). This test method also includes procedures for calibration of the apparatus for measuring both capacitance and voltage.

NOTE 1: An alternative method of determining the effective area of the mercury probe contact that involves the use of reference wafers whose net carrier density has been measured using fabricated mesa or planar p-n junction diodes or evaporated Schottky diodes is not included in this test method but may be used if agreed upon by the parties to the test.
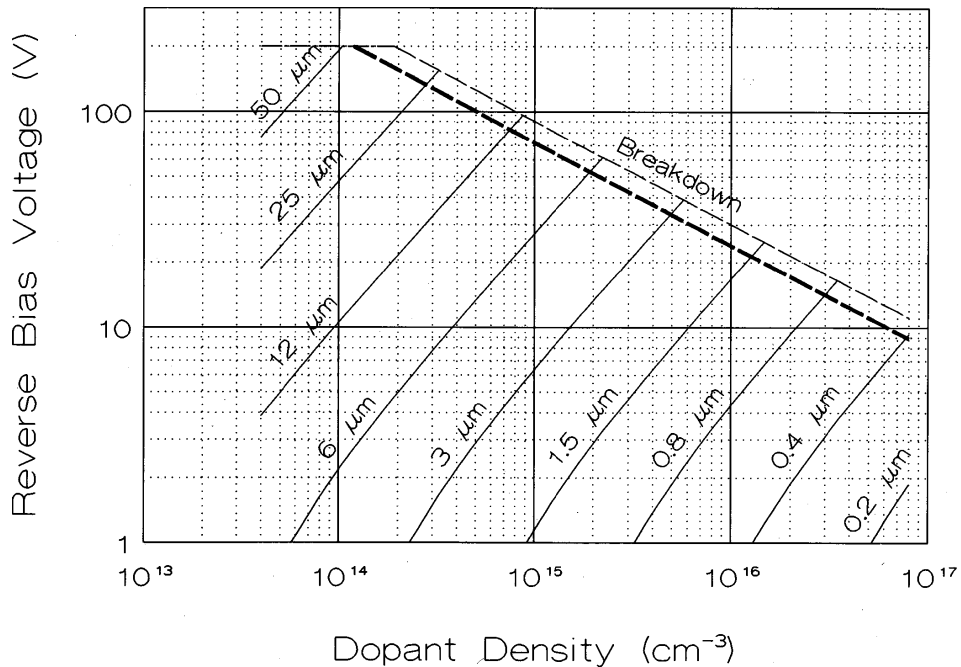
1 DIN 50439, Determination of the Dopant Concentration Profile of a Single Crystal Semiconductor Material by Means of the Capacitance-Voltage Method and Mercury Contact, is technically equivalent to this test method. DIN 50439 is the responsibility of DIN Committee NMP 221, with which SEMI maintains close liaison. DIN 50439 is available in German and English from Beuth Verlag GmbH, Burggrafenstraße 4-10, D-10772, Berlin, Germany.

2 Severin, P. J., and Poodt, G. J.,"Capacitance-Voltage Measurements with a Mercury-Silicon Diode," *J. Electrochem. Soc,* **119**, 1384–1388 (1972).

3 Grove, A. S., *Physics and Technology of Semiconductor Devices* (John Wiley and Sons, New York, 1967) Sections 6.2 and 6.7c.

(a) Depletion Depth as a Function of Dopant Density with Applied Reverse Bias Voltage as a Parameter.

(b) Applied Reverse Bias Voltage as a Function of Dopant Density with Depletion as a Parameter.

Note: The light dashed line represents the applied reverse bias voltage at which breakdown occurs in a mercury-silicon contact; the heavy dashed line represents 80 % of this voltage, it is recommended that the applied reverse bias voltage not exceed this value. The light chain-dot line represents the maximum reverse bias voltage specified in this test method.

Figure 1
Relationship Between Depletion Depth, Applied Reverse Bias Voltage, and Dopant Density

2.7 Warnings and precautionary notes regarding potential safety hazards are provided throughout the document.

NOTICE: This standard does not purport to address safety issues, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory or other limitations prior to use.

3 Limitations

3.1 A poor Schottky contact, which is generally indicated by an excessively high leakage current (see Section 11.5) is the most common problem in capacitance-voltage measurements made with mercury probe instruments. It must be emphasized that the use of a poor Schottky contact does not actually prevent a carrier density determination but produces an erroneous result.

3.2 Improper determination of the compensation capacitance, C_{comp}, (see Section 10.3) can cause significant errors in the capacitance measurement. In homogeneous material, improper zeroing or use of an improper value for C_{comp} results in an apparent monotonic increase or decrease of carrier density with distance from the Schottky barrier. In some fixtures, inherently large stray capacitances exist; in such cases, the value of C_{comp} may depend both on the diameter of the wafer and on the position of the wafer on the chuck. If these dependencies are observed, they may be reduced or eliminated by shielding the mercury probe column. If shielding is not practical, probe calibration procedures should be carried out with wafers of the same diameter as the wafers being tested and care should be taken to ensure that the geometry of wafer and probe is the same during calibration and measurement.

3.3 Alternating frequency test signals greater than 0.05 V rms may lead to errors in the measured capacitance.

3.4 Excessive series resistance in the capacitance measurement circuit can cause significant errors in the measured capacitance values. Series resistance values greater than 1 kΩ have been reported to cause measurement error in some cases.[4,5] The primary source of excessive series resistance is generally a high-resistance return contact; other possible sources are bulk resistance in the wafer and wiring defects in the

mercury probe fixture or the test cables (see Section 11.4).

3.5 When exposed to air, a scum tends to form on the exposed surface of the mercury used to form the mercury probe contact. When freed from the surface, this scum floats to the top of the mercury column. It is necessary to make certain that the mercury that contacts the wafer surface is clean by changing the mercury periodically or by otherwise removing the scum from the exposed surface.

3.6 A dirty or damaged capillary tube containing the mercury column may also result in unstable measurements (see Section 10.4.2.2).

3.7 If the reference wafer is not sufficiently uniform throughout its thickness, the value of net carrier density, N_{ref}, determined by the four-point probe measurement (see Section 8.4.3) may differ from the value of net carrier density at the surface where the mercury probe measurement is made. Use of erroneous values of N_{ref} results in incorrect values for effective probe contact area (see Section 10.3). Further, if the resistivity profile of the reference wafer is not uniform near the surface, an incorrect value of C_{comp} may be obtained (see Section 10.2). Incorrect values of probe contact area and C_{comp} result in incorrect values for the average net carrier density of the test specimen.

4 Referenced Standards

4.1 *SEMI Standards*

SEMI C28 — Specifications and Guidelines for Hydrofluoric Acid

SEMI C29 — Specifications and Guideline for 4.9% Hydrofluoric Acid (10:1 v/v)

SEMI C30 — Specifications and Guidelines for Hydrogen Peroxide

SEMI MF26 — Test Methods for Determining the Orientation of a Semiconductive Single Crystal

SEMI MF42 — Test Methods for Conductivity Type of Extrinsic Semiconducting Materials

SEMI MF81 — Test Method for Measuring Radial Resistivity Variation on Silicon Wafers

SEMI MF84 — Test Method for Measuring Resistivity of Silicon Wafers with an In-Line Four-Point Probe

SEMI MF672 — Test Method for Measuring Resistivity Profiles Perpendicular to the Surface of a Silicon Wafer Using a Spreading Resistance Probe

SEMI MF723 — Practice for Conversion Between Resistivity and Dopant Density for Boron-Doped, Phosphorus-Doped, and Arsenic-Doped Silicon

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4 Wiley, J. D., and Miller, G. L., "Series Resistance Effects in Semiconductor CV Profiling," *IEEE Trans. Electron Devices* **ED-22**, 265–272 (1975).

5 Schroder, D. K., *Semiconductor Material and Device Characterization* (Wiley-Interscience, New York, 1990) Section 2.5.2.

SEMI MF1153 — Test Method for Characterization of Metal-Oxide-Silicon (MOS) Structures by Capacitance-Voltage Measurements

SEMI MF1241— Terminology of Silicon Technology

4.2 *ASTM Standards*[6]

D 5127 — Guide for Ultra Pure Water Used in the Electronics and Semiconductor Industry

D 4356 — Practice for Establishing Consistent Test Method Tolerances

E 691 — Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

NOTICE: Unless otherwise indicated, all documents cited shall be the latest published versions.

5 Terminology

5.1 *Definitions*

5.1.1 For definitions of terms used in silicon wafer technology refer to SEMI MF1241.

5.1.2 Definitions of the statistical terms *repeatability* and *reproducibility* are given in ASTM Practice E 691.

5.2 *Definitions of Terms Specific to This Standard*:

5.2.1 *compensation capacitance, C_{comp}* — the sum of the stray capacitance of the measurement system and the peripheral capacitance of the mercury probe contact (see Section 10.3).

5.2.2 *low-resistance contact* — an electrically and mechanically stable contact[7] in which the resistance across the contact does not result in excessive series resistance as determined in Section 11.4 (see also Section 3.4).

5.2.2.1 *Discussion* — a low-resistance contact may usually be achieved by using a metal-semiconductor contact with an area much larger than that of the mercury probe contact.

5.2.3 *mercury probe contact* — a Schottky barrier diode formed by bringing a column of mercury into contact with an appropriately prepared polished or epitaxial silicon surface.

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6 Available from ASTM International, 100 Barr Harbor Drive, West Conshohoken, PA 19428-2959, USA. Telephone: 610.832.9585, Fax: 610.832-9555, Website: www.astm.org. ASTM D 5127 is published in Volume 11.01 of *Annual Book of ASTM Standards* while ASTM D 4356 and ASTM E 691 are published in Volume 14.02.

7 Rhoderick, E. H., and Williams, R. H., *Metal-Semiconductor Contacts*, 2nd Edition (Clarendon Press, Oxford, 1988).

6 Summary of Test Method

6.1 The compensation capacitance and effective mercury probe contact area are determined using a reference wafer.

6.2 The test specimen is placed in the mercury probe fixture. A column of mercury is brought into contact with the epitaxial or polished surface of the specimen by a pressure differential between the mercury and ambient to form a Schottky barrier diode (mercury probe contact).

6.3 A low-resistance return contact is also made to either the front or back surface of the wafer. This contact may be either a metal plate or a second mercury-silicon contact with an area much larger than the mercury probe contact.

6.4 The quality of the Schottky barrier diode formed by the mercury probe contact is evaluated by measuring its series resistance and its reverse current characteristics.

6.5 The small-signal, high frequency capacitance of the mercury probe contact is measured as a function of the voltage applied between the mercury probe column and the return contact. The polarity of the applied voltage is such that the mercury probe contact is reverse biased and the low-resistance return contact is forward biased.

6.6 The net carrier density profile (net carrier density as a function of depth from the surface) is calculated from the measured values of capacitance and applied voltage by one of two equivalent methods.

NOTE 2: Net carrier density values obtained by this test method are often converted to resistivity, which is generally a more familiar parameter in the industry. If this is done, the conversion should be made in accordance with the computational methods given in Section 7.2 of SEMI MF723. Note that in applying this conversion procedure in either direction it is assumed that the net carrier density is equal to the dopant density.

7 Apparatus

7.1 *Mercury Probe Fixture* — One of the following fixtures depending on the type of test specimen to be measured. **Warning:** Mercury is a toxic material. Refer to the appropriate Material Safety Data Sheet prior to use. Avoid physical contact with mercury and breathing of its vapor.

7.1.1 *Back-Surface-Return-Contact Fixture,* for use in measuring polished wafers or epitaxial layers deposited on substrates of the same conductivity type. A probe fixture that holds the treated wafer and provides a single mercury column contained in a capillary tube with nominal inside diameter of 0.4 to 2.0 mm. The fixture shall be capable of forming a mercury probe contact area on the front polished or epitaxial surface of

the wafer with a repeatability of $\pm 1\%$ or better. The fixture must also provide a low-resistance return contact to the back surface of the wafer.

7.1.2 *Front-Surface-Return-Contact Fixture,* for use in measuring epitaxial wafers deposited on substrates of the opposite conductivity type or on substrates with insulating back surface films. A probe fixture that holds the treated wafer and provides two contacts to the front polished or epitaxial surface of the wafer. One contact is the mercury probe contact as described in 7.1.1, and the other is a low-resistance return contact. The latter may be either a second mercury column or a metal plate. Its area shall be such that its capacitance is not less than 32 times the capacitance of the smaller mercury column. In addition, it is recommended that this fixture also provide a low-resistance return contact to the back surface of the wafer to permit the apparatus also to be used in the back-surface-return-contact configuration (see Section 7.1.1).

7.2 *Equipment for Handling Mercury* — Hypodermic needle or other means for transferring mercury from a storage bottle to the mercury column and equipment for neutralizing and picking up spilled mercury (**Warning**—see Section 7.1).

7.3 *Capacitance Bridge or Meter,* with ranges from 1 to 1000 pF full scale or greater, in range multiples of 10 or less. The accuracy shall be 1.0% of full scale or better for each range, and the rated reproducibility shall be 0.25% of full scale or better. The internal a-c voltage signal shall not exceed 0.05 V rms. The measurement frequency shall be in the range from 0.9 to 1.1 MHz inclusive. The instrument shall be capable of sustaining an external d-c bias of up to 200 V. Provision shall be made to compensate a compensation capacitance of up to 10 pF.

NOTE 3: Capacitance meters or bridges capable of measuring the phase angle, equivalent series resistance, conductance or total impedance in addition to the capacitance may be used.

NOTE 4: Capacitance meters with nominal frequencies from 100 kHz to 1 MHz have been used for measurements of the type covered by this test method. If an instrument with a nominal frequency other than 1 MHz is employed, the user shall demonstrate that it obtains results equivalent to the specified instrument.

7.4 *DC Power Supply,* continuously variable from 0 V to the maximum expected reverse bias or 200 V, whichever is less, capable of supplying voltages of either polarity with a peak-to-peak noise of 25 mV or less. This power supply may be integrated with the capacitance meter, if desired.

NOTE 5: The maximum reverse bias depends on the net carrier density in the wafer under test, see Figure 1.

7.5 *Digital Voltmeter,* with a minimum of four digits, ranges from ± 1 to ± 200 V full scale or greater in range multiples of 10 or less, a sensitivity of 1 mV or less, and an accuracy of 0.5% of full scale or better, a rated reproducibility of 0.25% of full scale or better, an input impedance of 100 MΩ or more, and a common-mode rejection ratio of 100 dB or greater at 60 Hz. This voltmeter may be integrated with the capacitance meter and power supply, if desired.

7.6 *Curve Tracer,* or other apparatus, capable of monitoring the reverse and forward current-voltage characteristics of the mercury probe contact. It shall be capable of applying 200 V at 0.1 mA in the reverse direction and 1.1 V at 1 mA in the forward direction and have a sensitivity of 10 μA/division or better.

7.7 *Shielded Cables,* as required, for making electrical connections between the probe fixture, power supply, capacitance bridge or meter, and digital voltmeter.

7.8 *Precision Capacitors,* with an accuracy of 0.25% or better at 1 MHz, the measurement frequency, required only for capacitance meter adjustment and verification (see Section 10.5). If used, at least two precision capacitors with values differing by at least a factor of 10 and lying within the expected capacitance ranges to be measured are required.

7.9 *Precision Voltage Source,* capable of providing output voltages from 0 to ± 200 V with an accuracy of 0.1% of the output voltage or better, required only for voltmeter verification (see Section 10.6).

7.10 *Facilities for Wafer Surface Treatment,* if required (see Sections 8.5, 10.3.5, 10.4.3.1, and 11.5.6):

7.10.1 *Fume Hood,* equipped with tanks to hold hydrofluoric acid at room temperature and, for *n*-type wafers only, hydrogen peroxide at 70° to 90° C. The tanks shall be of sufficient size to allow complete immersion of the cassettes holding the desired size wafers.

7.10.2 *Cassettes,* for holding the wafers in hydrofluoric acid.

7.10.3 *Additional Cassettes,* for holding *n*-type wafers in hydrogen peroxide at 70° C. **Warning:** When using a cassette that has been previously used in hydrofluoric acid in hydrogen peroxide without prior cleaning make sure that the cassette is clean because of the likelihood of contamination of the hydrogen peroxide bath.

NOTE 6: A cassette that has been previously used in hydrofluoric acid may be cleaned for the hydrogen peroxide treatment by boiling in water for 1 h.

7.10.4 *Dump Rinser,* with air atmosphere.

7.10.5 *Spin Dryer,* for drying the wafers in an air atmosphere.

7.10.6 *Hot Plate,* for *p*-type wafers or other means for baking the wafer at 120 ± 10° C in air may also be required.

7.10.7 *Interval Time,* for controlling treatment times.

7.10.8 *Nonmetallic Tweezers or Vacuum Wand,* for holding and manipulating wafers.

8 Reagents and Materials

8.1 *Purity of Reagents* — All chemicals for which such specifications exist shall adhere to Grade 1 SEMI specifications for those specific chemicals. Other grades may be used, provided it is first determined that the chemical is of sufficiently high purity to permit its use without lessening the accuracy of the test.

8.2 *Mercury* shall be triple distilled and conform to reagent grade, as specified in *Reagent Chemicals.*[8] It shall be changed regularly or otherwise maintained in a clean state to avoid interference from surface scum (see Section 3.5) (**Warning**—see Section 7.1).

8.3 *Purity of Water* — Reference to water shall be understood to mean Type E-3 or better water as described in ASTM Guide D 5127.

8.4 *Reference Wafers* — One or more polished bulk silicon wafers of the same conductivity type as the layer or wafer to be tested. If one reference wafer is used, its net carrier density shall lie between one-half and two times the net carrier density of the layer or wafer to be tested. If several reference wafers are used, their net carrier densities shall cover the range of net carrier densities of the layers or wafers to be tested. Bulk reference wafers shall have the following characteristics:

8.4.1 Flat spreading resistance profile over a depth equal to or greater than that to be profiled in this test method. The flat profile is required to ensure correct determination of C_{comp} (see Section 3.7, Section 10.2). Determine that the spreading resistance profile is flat in the following manner:

8.4.1.1 Measure the spreading resistance profile in accordance with SEMI MF672 on a small chip from a portion of the wafer. Use a minimum of 10 spreading resistance values in constructing the spreading resistance profile.

8.4.1.2 Fit the spreading resistance data to a straight line by a least-squares method.

8.4.1.3 For the profile to be considered flat, the fitted values at the beginning and end of the profile shall be equal to within ± 2% and the maximum deviation of any measured value from the fitted line shall not exceed 5%.

8.4.2 Resistivity variation over the central region of the wafer ≤ 5%. Resistivity variation over this region should be as small as possible to obtain maximum accuracy of the determination of the net carrier density of the reference wafer; accurate determination of the net carrier density is required for accurate determination of the probe contact area (see Section 10.3).

8.4.2.1 Determine radial unformity from resistivity measurements taken at 2.0 mm intervals along two perpendicular diameters for a distance of 6 mm from the center of the wafer in each direction. Analyze the data in accordance with the maximum/minimum convention of Sample Plan D of SEMI MF81.

8.4.2.2 Establish axial uniformity (see Section 3.7) by spreading resistance measurements across a cleaved portion of the wafer or by some other method agreed upon by the parties to the test.

8.4.3 *Known Net Carrier Density —* Determine the net carrier density as follows:

8.4.3.1 Measure the resistivity at the center of the wafer and correct it to 23° C in accordance with SEMI MF84.

8.4.3.2 Convert the resistivity value to net carrier density using the computational methods given in Section 7.2 of SEMI MF723.

NOTE 7: In applying this conversion procedure in either direction it is assumed that the net carrier density is equal to the dopant density. The appropriate equation given in this section for resistivity must be solved iteratively for the net carrier density. It is necessary to use the same equation for conversion from net carrier density to resistivity and vice versa in order to eliminate the self-consistency errors in SEMI MF723. The choice of conversion direction in this test method was made so that the more laborious, iterative procedure is applied to the less frequently measured reference wafers and the direct conversion procedure is applied to material being evaluated by this test method.

8.4.3.3 Record the net carrier density just obtained, as N_{ref}, in cm^{-3}.

NOTE 8: The advantage of using bulk reference wafers is that the net carrier density can be related to that of resistivity standard reference materials issued by the National Institute of Standards and Technology. However, material inhomogeneity may make it difficult to obtain accurate values of net carrier density from bulk reference wafers (see Section 3.7). As an alternative, epitaxial wafers may be used as reference wafers. In this case, the epitaxial layer thickness should be large enough that the resistivity profile in the near-

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8 "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC.

surface region may be obtained from spreading resistance measurements through the use of a calibration curve only, without the need for using correction factors. Since the procedures for characterizing such reference wafers have not been standardized, epitaxial wafers should be used as reference wafers only if agreed upon between the parties to the test. If used, the epitaxial reference wafer should meet the requirements of Section 8.41 for a flat spreading resistance profile. Net carrier density should be determined using an metal oxide silicon (MOS) capacitor structure in accordance with SEMI MF1153 or by another mutually acceptable method.

8.5 *Reagents for Surface Treatment* — If surface treatment is required, the following chemicals may be needed. Grade 1 chemicals are preferred (see Related Information 1).

8.5.1 *Hydrofluoric Acid*, HF, concentrated, 49.00 ± 0.25%, in accordance with grade 1 of SEMI C28 or dilute, 4.9 ± 0.1%, in accordance with grade 1 of SEMI C29.

8.5.1.1 Once a week, fill the hydrofluoric acid tank with fresh HF, either concentrated or dilute.

8.5.2 *Hydrogen Peroxide*, H_2O_2, unstabilized, 30%, in accordance with grade 1 of SEMI C30.

8.5.2.1 Every 8 h, fill the hydrogen peroxide tank with a fresh 15% H_2O_2 solution by mixing equal volumes of H_2O_2 (30%) and water. Wait until the freshly poured bath reaches 70 ± 5° C before using to treat wafer surfaces.

8.5.2.2 During use, approximately every 2 h (or when the H_2O_2 level falls below the wafers being treated), replenish the tank by adding 30% H_2O_2.

9 Sampling

9.1 It is generally impractical to measure every wafer in a particular lot owing to the potential for contamination from the handling and chemical treatments involved. A wafer sampling plan shall therefore be agreed upon between the parties to the test.

9.2 Locations on the wafer where measurements are to be made shall also be agreed upon between the parties to the test.

10 System Calibration and Control

10.1 *Frequency of Calibration and Control Procedures*

10.1.1 Calibrate the capacitance meter and voltmeter in accordance with manufacturer's instructions on initial installation and following hardware or software modifications. Calibration of the capacitance meter and voltmeter may be carried out in-house or by a qualified testing laboratory.

10.1.2 Determine the compensation capacitance and effective area of the mercury probe in accordance with the procedures of this section on initial installation and after any corrective action has been carried out to bring the system back into control.

10.1.3 Conduct periodic tests and maintain control charts in accordance with the procedures in this section to demonstrate that the instruments are in control and that the variability is within the requirements of this test method.

10.2 *Determination of Compensation Capacitance*, C_{comp}

10.2.1 Measure a series of capacitance-voltage pairs on the reference wafer carrier density of 1×10^{-14} cm^{-3} or less or, if such a reference wafer is not available, on the reference wafer with the lowest value of net carrier density (see Section 8.4) in accordance with Procedure (Section 11), using either the front- or back-surface-return-contact configuration depending on the type of wafer to be tested (see Section 7.2). Be certain that the series resistance of the diode circuit formed with the reference wafer meets the requirements of Section 11.4.1.7 or Section 11.4.2.7 before proceeding with the measurement of the capacitance-voltage pairs. Record each measured capacitance, C_i, and its associated voltage, V_i. Determine the value of C_{comp} either from the net carrier density profile (Section 10.2.2) or from V_i as a function of C_i^{-2} (Section 10.2.3).

10.2.2 *Net Carrier Density Profile*

10.2.2.1 Measure a total of *n* pairs, sufficient to calculate at least five values of net carrier density, N_i, and depth, W_i.

NOTE 9: If the incremental method of calculation (see Section 12.3) is used, $n \geq k + 5$, where k is an integer chosen such that C_{i+k} is between 80 and 85% of C_i; if the curve-fitting method of calculation (see Section 12.4) is used, $n \geq 5$.

10.2.2.2 Calculate the net carrier density profile in accordance with the incremental method (see Section 12.3) or the curve-fitting method (see Section 12.4) taking $C_{comp} = 0$ (see Section 12.1).

NOTE 10: If an approximate value of C_{comp} is known, this procedure may be shortened by starting with this approximate value. In this case, if the slope of the net carrier density profile is positive, increase C_{comp} as directed in Section 10.2.2.3 and Section 10.2.2.4 and decrease it after the first slope change as directed in Section 10.2.2.5; if the slope is negative, decrease C_{comp} initially and increase it after the first slope change.

10.2.2.3 Increase C_{comp} by 0.1 pF and recalculate the net carrier density profile (see Note 10). For the recalculation, correct each of the C_i to C'_i as follows:

$$C_i' = C_i + C_{comp} \tag{1}$$

NOTE 11: On some instruments, it may be necessary to enter a new value for C_{comp} and remeasure the reference wafer each time the value of C_{comp} is changed.

10.2.2.4 Repeat Section 10.2.2.3, increasing the value of C_{comp} by 0.1 pF each time until the slope of the calculated net carrier density profile changes sign.

10.2.2.5 After the slope of the calculated net carrier density profile changes sign, repeat Section 10.2.2.3, decreasing the value of C_{comp} by 0.02 pF each time until the slope of the calculated net carrier density profile changes sign again.

10.2.2.6 Record as C_{comp} the average of the values obtained just before and just after the sign change of the slope.

10.2.3 *V_i as a Function of C_i^{-2}*

10.2.3.1 Measure at least 50 capacitance-voltage pairs over the entire range to be measured.

10.2.3.2 Taking $C_{comp} = 0$, fit a straight line to the $V_i - C_i^{-2}$ data by the least-squares method, and compute the sum of the squares of the normalized residuals as follows:

$$R_N = \sum_{i=1}^{n} \left(\frac{V_i}{\hat{V}_i} - 1 \right)^2 \tag{2}$$

where:

R_N = sum of the squares of the normalized residuals,
V_i = measured voltage corresponding to the capacitance C_i,
$\hat{V}_i$ = voltage corresponding to the capacitance C_i, calculated from the least-squares fit, and
n = number of measured capacitance-voltage pairs.

10.2.3.3 Set $C_{comp} = 0.1$ pF.

10.2.3.4 Correct each of the C_i to C_i' using Eq. 1, fit a straight line to the new data, and recompute the sum of the squares of the normalized residuals using Eq. 2.

10.2.3.5 If the sum of the squares of the normalized residuals is less than the sum obtained from the previous case, increase C_{comp} by 0.1 pF, and repeat Section 10.2.3.4.

10.2.3.6 Repeat Section 10.2.3.5, increasing the value of C_{comp} by 0.1 pF each time until the sum of the squares of the normalized residuals increases.

10.2.3.7 At this point, decrease the value of C_{comp} by 0.02 pF.

10.2.3.8 Correct each of the C_i to C_i' using Eq. 1, fit a straight line to the new data, and recompute the sum of the squares of the normalized residuals using Eq. 2.

10.2.3.9 If the sum of the squares of the normalized residuals is less than the sum obtained from the previous case, decrease C_{comp} by 0.02 pF, and repeat Section 10.2.3.8.

10.2.3.10 Repeat Section 10.2.3.9, decreasing C_{comp} by 0.02 pF each time until the sum of the squares of the normalized residuals again increases.

10.2.3.11 Record as C_{comp} the average of the values obtained just before and just after the second increase in the sum of the squares of the normalized residuals.

10.3 *Determination of Effective Mercury Probe Contact Area*

10.3.1 Using the value of C_{comp} recorded in Sections 10.2.2.6 or 10.2.3.11, determine the net carrier density profile in the central region (see Section 8.4.2) of each reference wafer five times in accordance with Procedure (Section 11) and Calculations (Section 12). Be certain that the series resistance of the diode circuit formed with the reference wafer meets the requirements of Section 11.4.1.7 or Section 11.4.2.7 before proceeding with the measurement of the capacitance-voltage pairs. For these measurements, take the area of the mercury probe contact as the nominal area of the capillary tube holding the mercury, determined as follows:

$$A = \frac{\pi d^2}{400} \tag{3}$$

where:

A = nominal capillary tube area, cm^2, and
d = nominal diameter of the capillary tube, mm.

NOTE 12: If the nominal diameter of the capillary tube is not known, it can be measured with a toolmaker's microscope or other appropriate instrument. The area need not be known precisely, an estimate within about 20% is adequate for the purpose.

10.3.2 In each of the five measurement sets (j from 1 to 5), record each measured capacitance, C_{ij}, and its associated voltage, V_{ij}, for a total of n pairs (i from 1 to n, where n is sufficient to calculate at least five values of net carrier density, N_{ij}, and depth, W_{ij}, see Note 7). Measure the same number of pairs in each of the five net carrier density profiles.

10.3.3 Calculate the average net carrier density, N_{avg}, in cm^{-3}, as the grand average of the individual net carrier densities:

$$N_{avg} = \frac{1}{5n} \sum_{j=1}^{5} \sum_{i=1}^{n} N_{ij} \qquad (4)$$

where:

N_{ij} = net carrier density, cm^{-3}, for the ith depletion depth in the jth measurement set, as calculated in accordance with Calculations (Section 12), and

n = number of capacitance-voltage pairs measured in each of the five measurement sets.

10.3.4 Calculate the effective mercury probe contact area, A_{eff}, in cm^2, for each reference wafer as follows:

$$(A_{eff})_k = A \sqrt{\frac{(N_{avg})_k}{(N_{ref})_k}} \qquad (5)$$

where:

A = nominal capillary area, cm^2,

$(N_{ref})_k$ = known net carrier density of reference wafer k, cm^{-3}, and

$(N_{avg})_k$ = the calculated average net carrier density of reference wafer k, cm^{-3}.

10.3.4.1 If one reference wafer was measured, record this value as the effective mercury probe contact area, A_{eff}.

10.3.4.2 If more than one reference wafer was measured, determine and record the effective mercury probe contact area, A_{eff}, as the average of the effective contact areas for each of the reference wafers.

10.3.5 For control charting purposes, repeat Sections 10.3.2 and 10.3.3 using A_{eff} as the area of the mercury probe contact. If the value of average net carrier density for any reference wafer differs from the known value by more than 2%, condition the surfaces of the appropriate reference wafer or wafers (see Related Information 1) and repeat the calibration procedure (Sections 10.2 and 10.3). If it is impossible to achieve deviations of 2% or less for all wafers, it may be necessary to subdivide the net carrier density range and assign effective probe contact areas to each subgroup.

NOTE 13: Recommended chemical surface treatment procedures are described in Related Information 1.

10.4 *Control Procedures* — Periodically carry out tests and maintain control charts to ensure proper operation of the electronics (Section 10.4.1), the electronics plus the mechanical functionality of the probe station (Section 10.4.2), and the entire system (Section 10.4.3).

10.4.1 Monitor the performance of the electronic components with a moving range control chart of the net carrier density of a packaged Schottky diode.

10.4.1.1 Determine the net carrier density as follows: Attach the diode to the capacitance meter terminals and carry out the procedure, beginning with Section 11.6.4, and Calculations, Section 12.

10.4.1.2 If out-of-control conditions are noted, or if the control limits are wider than is desirable for the application, verify the performance of the capacitance meter and voltmeter independently in accordance with Sections 10.5 and 10.6, respectively, to establish which requires adjustment or recalibration.

10.4.2 Monitor the performance of the electronic components together with the mechanical functionality of the probe station with a moving range control chart of the oxide capacitance, C_{ox}, of an oxidized reference wafer.

10.4.2.1 Determine C_{ox} of an oxidized reference wafer in accordance with SEMI MF1153, with the mercury probe serving as the metal capacitor electrode.

10.4.2.2 If out-of-control conditions are noted, or if the control limits are wider than is desirable for the application, and the electronics are known to be performing as expected, inspect the mercury capillary carefully for dirt or damage. If it is dirty, clean it thoroughly and refill with clean mercury. If it appears to be damaged, repair or replace the capillary and refill with clean mercury.

10.4.3 Monitor the overall system performance with moving range control charts of net carrier density of the reference wafers (see Sections 8.4 and 10.3) or test specimens used as controls.

10.4.3.1 Determine the net carrier density of these specimens in accordance with this test method. Two or more controls are required to ensure that the control wafers are stable; if one control becomes unstable, retreat the surface (see Related Information 1) and redetermine its net carrier density. If this does not bring the specimen into control, discard it and replace with a fresh control.

10.4.3.2 If system out-of-control conditions are noted, or if the control limits are wider than is desirable for the application, and they cannot be attributed to control wafer instability, mechanical malfunctions (Section 10.4.2) or electronic malfunction (Section 10.4.1), redetermine C_{comp} (see Section 10.2) and the effective area of the mercury probe contact (see Section 10.3).

10.5 *Capacitance Meter Adjustment and Verification*

10.5.1 Connect to the capacitance bridge or meter shielded cables of a length suitable for measuring the precision capacitors. Zero the capacitance bridge or meter with the cables attached only to the bridge or meter, not to a precision capacitor.

10.5.2 Connect the cables to one of the precision capacitors. Measure and record the capacitance in pF to three significant figures. Disconnect the capacitor. Repeat for the other precision capacitors.

10.5.3 If the measured capacitance values are not within 1% of the known values, make necessary adjustments consistent with the appropriate instrument instruction manuals to bring the instrument to within specifications before proceeding with measurements of test specimens.

10.6 *Voltmeter Verification*

10.6.1 Measure the precision voltage source at five or more voltages within the range from 0 to ± 200 V, inclusive to verify that the digital voltmeter is within specification over this range.

10.6.2 If any measured voltage value is not within 0.5% of the known value, make necessary adjustments consistent with the appropriate instrument instruction manuals to bring the instrument to within specifications before proceeding with measurements of test specimens.

11 Procedure

11.1 Refer to Related Information 2 for suggested data sheet formats for recording the data if the data collection and calculations are carried out manually or off-line.

NOTE 14: The following procedures are given in sufficient detail for manual data collection and calculations to be carried out. However, it is strongly recommended that both data collection and analysis be carried out using computer controlled equipment, with data storage and display capabilities. In such cases, the procedures and algorithms employed must be equivalent to those given in this test method.

11.2 If not known, determine the conductivity type and surface orientation of the test wafers in accordance with SEMI MF42 and SEMI MF26, respectively.

11.3 Estimate the reverse bias voltage range over which the measurements are to be made based on the curve in Figure 1, an estimate of the value for the dopant density of the test specimen, and the range of depth over which the profile is desired. Do not exceed 200 V or 80% of the breakdown voltage, whichever is lower.

11.4 *Determination of the Series Resistance of the Diode Circuit* — Determine the series resistance of the diode circuit in one of the following ways:

11.4.1 *Measurement of Forward Resistance* — If the capacitance bridge or meter measures capacitance only, measure the diode forward resistance, R, in ohms, at 1 V forward bias as follows:

11.4.1.1 Connect the curve tracer to the mercury probe column and to the return contact of the probe fixture.

11.4.1.2 Place the wafer to be tested onto the mercury probe fixture set up in the configuration used to determine C_{comp} (see Section 10.3) in such a way that the mercury column(s) will contact the polished or epitaxial surface of the wafer. If the back-surface-return-contact configuration is used, make a suitable return contact to the substrate or back surface of the wafer.

11.4.1.3 Bring the mercury column(s) into contact with the surface of the wafer.

11.4.1.4 Measure and record as I_1 the current through the diode at 0.9 V forward bias, in mA, to two significant figures.

11.4.1.5 Measure and record as I_2 the current through the diode at 1.1 V forward bias, in mA, to two significant figures.

11.4.1.6 Calculate the forward resistance, R, in kΩ, as follows:

$$R = \frac{0.2}{I_2 - I_1} \qquad (6)$$

where:
I_1 = current at 0.9 V forward bias, mA, and
I_2 = current at 1.1 V forward bias, mA.

11.4.1.7 If the forward resistance is 1 kΩ or less, proceed to Section 11.5. If the forward resistance exceeds 1 kΩ, improve the return contact, and repeat Section 11.4.1.

NOTE 15: The diode forward resistance at 1 V, R, determined in this way is a measure of the total series resistance of the test circuit that includes the bulk, cable, and return contact resistances.

11.4.2 *Direct Determination of Equivalent Series Resistance* — For capacitance meters or bridges capable of measurement of phase angle, conductance, or total impedance, determine the equivalent series resistance directly as follows:

11.4.2.1 Place the wafer to be tested onto the mercury probe fixture set up in the configuration used to determine C_{comp} (see Section 10.3) in such a way that the mercury column(s) are just above but do not contact the polished or epitaxial surface of the wafer. If the back-surface-return-contact configuration is used, make a suitable return contact to the substrate or back surface of the wafer.

11.4.2.2 Zero the meter in accordance with the manufacturer's instructions.

11.4.2.3 Bring the mercury column(s) into contact with the surface of the wafer.

11.4.2.4 Set the bias to a nominal value of 0 V, and read the capacitance, C, and the phase angle, θ, or the conductance, G_m.

11.4.2.5 If the phase angle and capacitance were measured, calculate the series resistance, R_s, in kΩ, as follows:

$$R_s = \frac{10^3}{2\pi f C \tan\theta} \tag{7}$$

where:

θ = measured phase angle, degrees,
C = measured capacitance, pF, and
f = measurement frequency, MHz.

11.4.2.6 If the capacitance and conductance were measured, calculate the series resistance, R_s, in kΩ, and the phase angle, θ, in degrees, as follows:

$$R_s = \frac{G_m}{G_m^2 + (2\pi f C \times 10^{-3})^2} \tag{8}$$

and

$$\theta = \tan^{-1}\left(-\frac{10^3}{2\pi f R_s C}\right) \tag{9}$$

where:

G_m = measured conductance, S,
f = measurement frequency, MHz, and
C = measured capacitance, pF.

11.4.2.7 If the series resistance is 1 kΩ or less and the phase angle is between $-87°$ and $-90°$, proceed to Section 11.5. If either of these conditions is not met, improve the return contact, and repeat Section 11.4.2. If improvement of the return contact decreases the series resistance but does not bring the phase angle within the desired range, qualify the Schottky contact in accordance with Section 11.5; when the Schottky contact is satisfactory, repeat Section 11.4.2 to verify that the phase angle is within the desired range.

11.5 *Qualification of Schottky Contact* — To qualify the Schottky contact, determine the reverse current characteristics of the mercury probe contact as follows:

11.5.1 If a curve tracer was used to determine the diode forward resistance, do not disconnect it. If the series resistance was measured directly, connect a curve tracer or other apparatus for monitoring the current-voltage characteristics of the mercury probe contact (see Section 7.7). Apply a reverse bias voltage of about 1 V

to the mercury column. **Warning:** Avoid physical contact with the probe fixture when bias is applied.

11.5.2 Measure and record this voltage as V_l, and measure the current that exists at this voltage. Calculate the current density at this value of reverse bias voltage, J_{rl}, in mA·cm^{-2}, as follows:

$$J_{r1} = \frac{I_{r1}}{A_{eff}} \tag{10}$$

where:

I_{rl} = current, mA, at the reverse bias voltage V_1, and
A_{eff} = mercury probe contact area, cm^2, see Section 10.3.4.

11.5.3 Increase the magnitude of the reverse voltage at intervals until the maximum reverse bias voltage that is to be applied during the test (see Section 11.3) is reached. Measure each current and calculate the current density, J_r, at each value of voltage. In addition, calculate the rate of increase of the reverse current density with voltage, in mA·V^{-1}·cm^{-2}, as follows:

$$\frac{\Delta J_r}{\Delta V} = \frac{J_{r(i+1)} - J_{ri}}{V_{i+1} - V_i} \tag{11}$$

where:

$\Delta J_r/\Delta V$ = rate of increase of the reverse current density with voltage,
J_{ri} = current density at voltage, V_i, and
$J_{r(i+1)}$ = current density at voltage, V_{i+1}.

11.5.4 Also observe whether or not the reverse current density is stable with time.

11.5.5 If the reverse current density, J_r, equals or exceeds 3 mA/cm^2 at any voltage up to the maximum value applied, first determine if this is due to carrier density variations in the structure. In this case, reduce the maximum applied reverse bias voltage to be used in the test to the highest value for which the reverse current density is less than 3 mA·cm^{-2}.

NOTE 16: If the depletion depth extends to a region with a rapidly increasing doping density, the breakdown voltage may be significantly lower than estimated from the expected net carrier density. For example, if the test specimen consists of a lightly doped epitaxial layer on a heavily doped substrate and if the profile extends deeper into the structure than the flat region of the layer, the breakdown voltage would be lower than that estimated from the expected net carrier density in the flat region.

11.5.6 Otherwise, if $J_r > 3$ mA·cm^{-2}, or if $\Delta J_r/\Delta V > 0.3$ mA·V^{-1}·cm^{-2}, or if the reverse current is unstable in time, treat the wafer surface with an acceptable

chemical process (see Related Information 1), and repeat the procedure beginning with Section 11.4.

11.5.7 When the Schottky contact is satisfactory, disconnect the curve tracer or other apparatus for monitoring the current-voltage characteristics of the mercury probe contact.

NOTE 17: To ensure the greatest possible accuracy of the measurement, monitor the attributes of the Schottky contact during the collection of data (Section 11.6) and discontinue data collection if any of the conditions in Section 11.5.6 are violated.

11.6 *Collection of Data* — Measure a series of capacitance-voltage pairs from which the net carrier density profile can be calculated.

11.6.1 Disconnect the curve tracer from the mercury probe fixture if one was used to determine the diode current density.

11.6.2 Connect the capacitance bridge or meter to the mercury probe fixture in accordance with the manu‐facturer's instructions and zero the capacitance bridge or meter, if required.

11.6.3 Bring the mercury column(s) into contact with the test wafer surface, and apply a nominal 1 V reverse bias between the mercury probe contact and the return contact (**Warning**—see Section 11.5.1).

11.6.4 Read and record the capacitance and applied voltage, each to three or more significant figures, as C_1 and V_1, respectively. Use a data table format appropriate to the calculation method to be selected. In all cases, record the voltages as positive numbers even though reverse biases are involved.

11.6.5 Adjust the bias voltage to obtain a new value of capacitance that is up to 5% lower than the previous value. Read and record the capacitance and applied voltage, each to three or more significant figures, as C_2 and V_2, respectively (**Warning**—see Section 11.5.1).

11.6.6 Repeat Section 11.6.5, adjusting the voltage for a decrease in capacitance such that the ratio of C_{i+1} to C_i is approximately equal to C_2/C_1 at each step, until the maximum applied reverse bias voltage (see Section 11.3) is reached. Discontinue the measurement before the maximum applied reverse bias voltage is reached if the capacitance values start to increase with increased reverse bias. Obtain a minimum of n capacitance-voltage pairs, where n is sufficient to calculate at least five values of net carrier density, N_i, and depth, W_i (see Note 9).

11.7 *Shut-Down Cycle* — When the measurement sequence is complete, reduce the reverse bias voltage to 0 V, disengage the mercury column(s) from the wafer

surface, and remove the test wafer from the probe fixture.

12 Calculations

12.1 Subtract the compensation capacitance, C_{comp}, from each measured capacitance, C'_i, to obtain the corrected capacitance, C'_i:

$$C'_i = C_i - C_{comp} \tag{12}$$

Record these values of C'_i.

12.2 Calculate the net carrier density profile by the Incremental Method (see Section 12.3) or by the Curve-Fitting Method (see Section 12.4).

12.3 *Incremental Method*:[9]

12.3.1 Calculate the average depletion depth corresponding to each interval as follows:

12.3.1.1 Calculate and record the dimensionless quantity S_i for each value of i from 1 to $n-k$ as follows:

$$S_i = \frac{\ln\left(\dfrac{V_{i+k} + 0.6}{V_i + 0.6}\right)}{\ln\left(\dfrac{C'_i}{C'_{i+k}}\right)} \tag{13}$$

where:

V_i = i^{th} recorded voltage, V,
V_{i+k} = $(i+k)^{th}$ recorded voltage, V,
C'_i = i^{th} corrected capacitance, pF,
C'_{i+k} = $(i+k)^{th}$ corrected capacitance, pF,
k = whole number such that C_{i+k} is between 80 and 85% of C_i, and
n = number of measured capacitance-voltage pairs.

12.3.1.2 Calculate and record the depth, W'_i, in μm, corresponding to each corrected capacitance, C'_i, as follows:

$$W'_i = 10359 \frac{A_{eff}}{C'_i} \tag{14}$$

where:

A_{eff} = mercury probe contact area, cm^2, as determined in 10.4.4, and
C'_i = the ith corrected capacitance, pF.

9 Niehaus, W. C., van Gelder, W., Jones, T. O., and Langer, P., "Variations of a Basic Capacitance-Voltage Technique for Determination of Impurity Profiles in Semiconductors," *Semiconductor Device Processing*, NBS Spec. Publ. 337, (U.S. National Institute of Standards and Technology, Washington, DC, 1970) pp. 266–268.

12.3.1.3 Calculate and record the average depth, W_i, in μm, for each value of i from 1 to $n - k$ as follows:

$$W_i = \frac{(W'_{i+k})^{S_i} - (W'_i)^{S_i}}{S_i(W'_{i+k} - W'_i)} \cdot \frac{1}{S_i - 1} \quad (15)$$

where:

W'_i = ith calculated depth, μm,
W'_{i+k} = $(i + k)$th calculated depth, μm,
S_I = ith dimensionless value calculated in Section 12.3.1.1,
k = whole number such that C_{i+k} is between 80 and 85% of C_i, and
n = number of measured capacitance-voltage pairs.

12.3.2 Calculate and record the net carrier density, N_i, in cm^{-3}, corresponding to each average depth, W_i, as follows:

$$N_i = 6.466 \times 10^{14} \frac{V_{i+k} - V_i}{W_i(W'_{i+k} - W'_i)} \quad (16)$$

where:

V_i = ith recorded voltage, V,
V'_{i+k} = $(i + k)$th recorded voltage, V,
W_i' = ith calculated depth, μm,
W'_{i+k} = $(i + k)$th calculated depth, μm, and
k = whole number such that C_{i+k} is between 80 and 85% of C_i.

12.3.3 Proceed to Section 12.5.

12.4 *Curve-Fitting Method*

12.4.1 Fit a polynomial of the following form[10] to the capacitance-voltage pairs, $C_i - V_i$:

$$\frac{1}{C_{fi}^2} = a_0 + a_1(V_i + 0.6)$$
$$+ a_2(V_i + 0.6)^2 + \ldots + a_{k'}(V_i + 0.6)^{k'} \quad (17)$$

where:

k' = order of the polynomial, chosen so that it represents the lowest-order fit for which $|(C_i' - C_{fi})/C_i'| \leq 0.01$ for all values of i and for which $k' \leq 10$ or $n - 1$, whichever is smaller (see Note 18).
n = number of capacitance-voltage pairs determined in the test,
C_{fi} = ith value of capacitance, pF, calculated from the fit,
$a_0 \ldots a_k$ = coefficients determined such that the quantity $(C_i' - C_{fi})^2$ summed over all values of i is minimized,

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10 Daniel, C., and Wood, F. S., *Fitting Equations to Data* (Wiley-Interscience, New York, NY) 1971, p. 19.

C_i' = ith corrected capacitance, pF, and
V_i = ith recorded (positive) voltage, V.

NOTE 18: Failure to meet this condition for a low-order polynomial ($k' \leq 3$) may be caused by a single outlier; in such cases, the polynomial should be redetermined without this datum. Failure to meet this condition may also suggest that the method is inappropriate for analysis of the data set; in such cases, use of the incremental method is preferred.

12.4.2 Record the values of C_{fi} and $a_0, a_1, \ldots a_{k'}$.

12.4.3 Calculate and record D_i, the derivative of the polynomial for $1/C_{fi}^2$ with respect to diode voltage at the depth appropriate to the ith voltage-capacitance pair, as follows:

$$D_i = a_1 + 2a_2(V_i + 0.6) + \ldots + k'(V_i + 0.6)^{k'-1} \quad (18)$$

where the other symbols are defined following Eq. 15.

12.4.3.1 Calculate and record the depletion depth, W_i, in μm, and the net carrier density, N_i, in cm^{-3}, corresponding to each capacitance-voltage pair as follows:

$$W_i = 10359 \frac{A_{eff}}{C_{fi}} \quad (19a)$$

and

$$N_i = -\frac{1.2050 \times 10^7}{A_{eff}^2 D_i} \quad (19b)$$

where:

A_{eff} = mercury probe contact area, cm^2, as determined in Section 10.3.4,
C_{fi} = the ith calculated capacitance, pF, and
D_i = the derivative of the polynomial for $1/C_{fi}^2$ with respect to diode voltage at the depth W_i.

12.5 Determine the average net carrier density as follows:

12.5.1 Take the number of individual values of net carrier density to be averaged as the number of values that fall within the flat zone of the epitaxial layer or within the depth range of the specimen over which the average net carrier density is desired. Record this number as m; m may not exceed $n - k$ if the incremental method was used for the calculations or n if the curve-fitting method was used for the calculations.

12.5.2 Calculate the average net carrier density, N_{avg}, in cm^{-3}, as follows:

$$N_{avg} = \frac{1}{m} \sum_{i=1}^{m} N_i \quad (20)$$

where:

m = number of individual values of net carrier density to be averaged (see Section 12.5.1),

k = whole number such that C_{i+k} is between 80 and 85% of C_i,

n = number of capacitance-voltage pairs measured, and

N_i = net carrier density, cm^{-3}, corresponding to each average depth, W'_i.

12.5.3 Calculate the sample standard deviation of the net carrier density as follows:

$$s = \frac{1}{\sqrt{m-1}} \sqrt{\sum_{i=1}^{m} (N_i - N_{avg})^2} \qquad (21)$$

12.6 If the test wafer is not homogeneously doped or if s exceeds 10% of N_{avg}, plot the net carrier density profile as a graph of N_i as a function of W_i .

NOTE 19: Related Information 3 describes composition of the numerical constants used in the above calculations.

13 Report

13.1 Report the following information:

13.1.1 Type and model number of instrumentation used including software type and revision, if a computer controlled system is employed,

13.1.2 Probe configuration used,

13.1.3 Operator identification,

13.1.4 Date of measurement,

13.1.5 Lot number and test specimen identification including conductivity type and surface orientation,

13.1.6 Wafer and sampling plan, if applicable,

13.1.7 Compensation capacitance, pF, as determined in Section 10.2,

13.1.8 Method of calculation used, and

13.1.9 Average net carrier density, N_{avg}, and sample standard deviation, s, or net carrier density profile (plot of N_i as a function of W_i), as determined in Section 12.5.

13.2 For referee measurements, also report the following:

13.2.1 Mercury probe contact area, A_{eff}, cm^2, as determined in Section 10.3.4,

13.2.2 Forward resistance at 1 V bias or equivalent series resistance, Ω, as determined in Section 11.4,

13.2.3 Phase angle, degrees, as determined in Section 11.4.2.4, if measured,

13.2.4 Maximum applied reverse bias voltage, V, as determined in Section 11.5,

13.2.5 Maximum leakage current density, J_r, mA/cm^2, as determined in Section 11.5,

13.2.6 Surface treatment used, if applicable, and

13.2.7 Other data as tabulated in a data sheet appropriate to the calculation method used.

14 Precision and Bias

14.1 *Repeatability* — The within-laboratory precision achievable with this test method was estimated from the results of several experiments. In the first experiment, ten nominally 50 $\Omega\cdot$cm n-type wafers were measured on two days by two different operators using a single instrument. The pooled coefficient of variation was 0.183%; the largest observed coefficient of variation was 0.35%.

14.1.1 Based on the pooled value, the repeatability, r, is estimated to be about 0.51%. In the second experiment, ten nominally 1 $\Omega\cdot$cm n-type wafers were measured three times on a single instrument by a single operator. The wafer surfaces were treated prior to each measurement. The pooled coefficient of variation was 0.493%; based on this value the repeatability, *r*, is estimated to be about 1.37%.

14.2 *Reproducibility* — The reproducibility of this test method has not been evaluated because of difficulties in establishing and applying a common specimen surface preparation for use in a round robin to evaluate reproducibility. No such tests are planned.

14.3 Calculations of the errors in net carrier density, N_i, and depletion depth, W_i, expected on the basis of the precision requirements of the various parameters measured in the test procedure could, in principle, be made in accordance with the procedures of Practice D 4356. However, because the formulas include ratios of differences of both capacitance and voltage and because the voltage and capacitance are coupled, these errors depend both on the intervals chosen in the experiment and on the physical characteristics of the test specimen. The calculation procedure for the incremental method included in this test method uses large intervals in order to minimize the errors from the use of finite intervals for the determination. For some examples of the effect of both random errors and finite interval errors on the calculated value of net carrier density, N_i.[9]

14.4 *Bias* — The bias of this test method cannot be evaluated because there are no available reference standards suitable for evaluating bias.

15 Keywords

15.1 capacitance-voltage method; carrier density; carrier density profile; depth profile; epitaxial wafers; mercury probe; net carrier density; polished wafers; profiles; resistivity; silicon; single crystal silicon

RELATED INFORMATION 1
RECOMMENDED WAFER SURFACE TREATMENTS

NOTICE: This related information is not an official part of SEMI MF1392. It was developed during the original development of the document. This related information was approved for publication by full letter ballot on July 12, 2003.

R1-1.1 The following surface treatments have been found to result in satisfactory surfaces under a variety of conditions and are recommended for use in connection with this test method.   Other surface treatments may also be satisfactory and may be used if agreed upon between the parties to the test.

R1-1.2 Place the wafer to be treated in a center slot of a hydrofluoric acid cassette.

R1-1.3 Dip in HF for 30 s (concentrated HF) or 5 min (dilute HF).

R1-1.4 Rinse in water for 10 min.

R1-1.5 For *p*-type wafers, spin dry in an air atmosphere. If subsequent measurements are unstable, heat treat the wafer for 30 min at $120 \pm 10°$ C in air.

R1-1.6 For *n*-type wafers, transfer the wafer immediately from the hydrofluoric acid cassette to a center slot of a hydrogen peroxide cassette (**Caution**—see Note 1), and continue with the following:

R1-1.6.1 Immerse in 15% H_2O_2 for 10 min at 70° to 90° C.

R1-1.6.2 Rinse in water for 2 min.

R1-1.6.3 Spin dry in an air atmosphere (Note 1).

NOTE 1: Alternatively, the wafers may be dried by blow drying with a filtered nitrogen blow-off gun while holding the wafer at a 45° angle with the lower edge of the wafer resting on a clean-room wipe.

RELATED INFORMATION 2
SAMPLE DATA SHEETS

NOTICE: This related information is not an official part of SEMI MF1392. It was developed during the original development of the document. This related information was approved for publication by full letter ballot on July 12, 2003.

R2-1.1 The following are examples of data sheet formats for manual collection and manual or off-line analysis of C-V data. Figure R2-1 is for data to be analyzed by the Incremental Method and Figure R2-2 is for data to be analyzed by the Curve-Fitting Method. The data listed must be stored in a format such that it can be retrieved if the system employed performs the calculations internally.

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Type of instrument _____

Model number _____

Software type/revision _____

Probe configuration _____

Operator _____

Date _____

Lot number _____

Test specimen identification _____

Conductivity type _____

Surface orientation _____

Lot sampling plan _____

_____

Measurement locations _____

Compensation capacitance, C_{comp} _____ pF

* Series resistance, R_s _____ kΩ

   * Phase angle, θ _____ °

* Max applied reverse bias voltage _____ V

* Max leakage current density, J_r _____ mA/cm^2

* Mercury probe contact area, A_{eff} _____ cm^2

* Surface treatment, if used _____

* Data Table:

C_i, pF	C'_i, pF	V_i, V	S_i	W''_i, μm	W_i, μm	N_i, cm^{-3}
C_1	C'_1	V_1	S_1	W''_1	W_1	N_1
C_2	C'_2	V_2	S_2	W''_2	W_2	N_2
C_3	C'_3	V_3	S_3	W''_3	W_3	N_3
.	.	. .	.	.	.	.
.	.	.	.	.	.	.
.	.	.	.	.	.	.
C_{n-k-1}	C'_{n-k-1}	V_{n-k-1}	S_{n-k-1}	W''_{n-k-1}	W_{n-k-1}	N_{n-k-1}
C_{n-k}	C'_{n-k}	V_{n-k}	S_{n-k}	W''_{n-k}	W_{n-k}	N_{n-k}
C_{n-k+1}	C'_{n-k+1}	V_{n-k+1}		W''_{n-k+1}		
.	.	.		.		
.	.	.		.		
C_n	C'_n	V_n		W''_n		

NOTE: Items marked with an asterisk (*) are required only for referee measurements.

Figure R2-1
Example of Format for Capacitance-Voltage Data and Calculations Using the Incremental Method

Type of instrument _____ Lot sampling plan _____

Model number _____ _____

Software type/revision _____ Measurement locations _____

Probe configuration _____ Compensation capacitance, C_{comp} _____ pF

Operator _____ * Series resistance, R_s _____ kΩ

Date _____ * Phase angle, θ _____ °

Lot number _____ * Max applied reverse bias voltage _____ V

    Test specimen identification _____ * Max leakage current density, J_r _____ mA/cm²

    Conductivity type _____ * Mercury probe contact area, A_{eff} _____ cm²

    Surface orientation _____ * Surface treatment, if used _____

* Fitting coefficients:

a_0 _____ a_1 _____ a_2 _____ a_3 _____   · · · $a_{k'}$ _____

* Data table:

C_i, pF	C'_i, pF	V_i, V	C_{fi}	D_i, μm	W_i, μm	N_i, cm⁻³
C_1	C'_1	V_1	C_{f1}	D_1	W_1	N_1
C_2	C'_2	V_2	C_{f2}	D_2	W_2	N_2
C_3	C'_3	V_3	C_{f3}	D_3	W_3	N_3
.	.	.	.	.	.	.
.	.	.	.	.	.	.
.	.	.	.	.	.	.
C_n	C'_n	V_n	C_{fn}	D_n	W_n	N_n

NOTE: Items marked with an asterisk (*) are required only for referee measurements.

Figure R2-2
Example of Format for Capacitance-Voltage Data and Calculations Using the Curve-Fitting Method

RELATED INFORMATION 3
NUMERICAL CONSTANTS

NOTICE: This related information is not an official part of SEMI MF1392. It was developed during the original development of the document. This related information was approved for publication by full letter ballot on July 12, 2003.

R3-1.1 The numerical constants given in the equations in this test method are lumped constants. This appendix provides details as to the composition of these lumped constants and the values of the individual constants used in deriving them. Table R3-1 gives the lumped constants, their formulas, and their values. The units in this table include any necessary unit conversions. If there is no entry for a given equation, either there is no lumped constant in the equation or the constant is strictly numeric (for example, as for unit conversion). Table R3-2 gives the values of the individual constants used in this test method. The built-in potential, ϕ, may vary from about 0.5 to about 0.8 V; the constant value assumed is an approximation to this parameter.

Table R3-1 Lumped Constants

Equation	Formula	Value (Note 1)
(13), (17), (18)	ϕ	0.6 [V]
(14) (19a)	$K_{Si}\varepsilon_0$	10359 [pF·μm/cm^2]
(16)	$K_{Si}\varepsilon_0/q$	6.466×10^{14} [(F·μm^{-2})/(C·cm^3)]
(19b)	$2/K_{Si}\varepsilon_0 q$	1.2050×10^{-7} [(cm·F)/(C·pF^2)]

NOTE 1: The units are given in brackets; the symbol C indicates coulombs and the symbol F indicates farads.

Table R3-2 Values of Individual Constants

Constant	Symbol	Value (Note 1)
Built-in potential	ϕ	0.6 [V]
Permittivity of free space	ε_0	8.8542×10^{-14} [F/cm]
Dielectric constant of silicon	K_{Si}	11.7
Electronic charge	q	1.6022×10^{-19} [C]

NOTE 1: The units are given in brackets; the symbol C indicates coulombs and the symbol F indicates farads.

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SEMI MF1451-1104
TEST METHOD FOR MEASURING SORI ON SILICON WAFERS BY AUTOMATED NON-CONTACT SCANNING

This test method was technically approved by the Global Silicon Wafer Committee and is the direct responsibility of the North American Silicon Wafer Committee. Current edition approved for publication by the North American Regional Standards Committee on August 16, 2004. Initially available at www.semi.org September 2004; to be published November 2004. Original edition published by ASTM International as ASTM F 1451-92. Last previous edition SEMI MF1451-0704.

1 Purpose

1.1 Sori can significantly affect the yield of semiconductor device processing.

1.2 Knowledge of this characteristic can help the producer and consumer determine if the dimensional characteristics of a specimen wafer satisfy given geometrical requirements.

1.3 Changes in wafer sori during processing can adversely affect subsequent handling and processing steps. These changes can also provide an important process monitoring function.

1.4 This test method is suitable for measuring the sori of wafers used in semiconductor device processing in the as-sliced, lapped, etched, polished, epitaxial or other layer condition and for monitoring thermal and mechanical effects on the sori of wafers during device processing.

2 Scope

2.1 This test method covers a non-contacting, nondestructive procedure to determine the sori of clean, dry semiconductor wafers.

2.2 This test method employs a two-probe system that examines both external surfaces of the wafer simultaneously.

2.3 The test method is applicable to wafers 50 mm or larger in diameter, and approximately 100 μm and larger in thickness, independent of thickness variation and surface finish, and of gravitationally induced wafer distortion.

2.4 This test method is not intended to measure the flatness of either exposed silicon surface. Sori is a measure of the distortion of the front surface of the wafer.

2.5 This test method measures sori of a wafer corrected for mechanical forces applied during the test. Therefore, the procedure described gives the unconstrained value of sori.

NOTE 1: This sori is indicated by the acronym "GFLYFER" in Appendix 2, Shape Decision Tree, of SEMI M1.

2.6 This test method includes several methods for canceling gravity-induced deflection which could otherwise alter the shape of the wafer.[1]

NOTE 2: One of these methods, the Representative Wafer Inversion Method, is covered by a patent held by ADE Corporation, 80 Wilson Way, Westwood, MA 02090-1806.

NOTICE: This standard does not purport to address safety issues, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory or other limitations prior to use.

3 Limitations

3.1 Any relative motion along the probe measuring axis between the probes and the wafer holding device during scanning will produce error in the measurement data. Vibration of the test specimen relative to the probe-measuring axis will introduce error. Such errors are minimized by system signature analysis and correction algorithms. Internal system monitoring may also be used to correct non-repetitive and repetitive system mechanical translations. Failure to provide such corrections may cause errors.

3.2 If a measured wafer differs substantially in diameter, thickness, fiducials, or crystal orientation from that used for the gravitational compensation procedure, the results may be incorrect. Estimates of the errors in gravity induced deflection for differences in diameter and thickness are shown in Related Information 1. If the crystal orientation of the sample to be measured differs from the crystal orientation of the gravity-compensation wafer, then the measured sori value may differ from the actual sori value by up to 15%. Error tables for fiducial variation have not been generated.

3.3 Different methods for implementing gravitational compensation may give different results. Varying

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1 Poduje, N., "Eliminating Gravitational Effect in Wafer Shape Measurements," *NIST/ASTM/SEMI/SEMATECH Technology Conference, Dallas, TX*. Technology for Advanced Materials/Process Characterization, February 1, 1990.

levels of completeness of implementing a method may also give different results.

3.4 Mechanical variations in wafer holding devices between systems may introduce measurement differences. This test method allows the use of a variety of wafer holding devices (see Section 7.1.4.1); results obtained with different geometrical configurations of wafer holding device on the same test samples may differ.

3.5 Most equipment systems capable of this measurement have a definite range of wafer thickness combined with sori (dynamic range) that can be accommodated without readjustment. If the sample moves outside this dynamic range during either calibration or measurement, results may be in error. An over-range signal can be used to alert the operator and measurement data examiners to this event.

3.6 The quantity of data points and their spacing may affect the measurement results. This test method does not specify the data point spacing (see Section 7.1.4.2); results obtained with different data point spacings on the same test samples may differ.

4 Referenced Standards

4.1 *SEMI Standards*

SEMI M1 — Specifications for Polished Monocrystalline Silicon Wafers

SEMI MF1241 — Terminology of Silicon Technology

SEMI MF1530 — Test Method for Measuring Flatness, Thickness, and Thickness Variation on Silicon Wafers by Automated Non-contact Scanning

4.2 *ASTM Standard*

D 4356 — Practice for Establishing Consistent Test Method Tolerances[2]

NOTICE: Unless otherwise indicated, all documents cited shall be the latest published versions.

5 Terminology

5.1 *Definitions*

5.1.1 *mechanical signature, of an instrument* — that component of a measurement that is introduced by the instrument and that is systematic, repeatable, and quantifiable.

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2 Available from ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428, Tel: 610-832-9500, Fax: 610-832-9555, Website http://www.astm.org. Appears in Volume 14.02 of *Annual Book of ASTM Standards.*

5.1.2 *median surface, of a semiconductor wafer* — the locus of points equidistant from the front and back surfaces of the wafer.

5.1.3 *reference plane, of a semiconductor wafer* — a plane from which deviations of a specified surface of the wafer are measured.

5.1.4 *reference plane deviation (RPD)* — the distance from a point on a reference plane to the corresponding point on a wafer surface.

5.1.4.1 *Discussion* — The front surface of a dome-shaped wafer is considered to have positive RPD at its center; the front surface of a bowl-shaped wafer is considered to have negative RPD at its center.

5.1.5 *sori, of a semiconductor wafer* — the algebraic difference between the most positive and the most negative deviations of the front surface of a wafer that is not chucked from a reference plane that is a least squares fit to the front surface.

5.1.5.1 *Discussion* — The front surface may contain regions with upward or downward curvature or both; under some conditions the front surface may be flat.

5.1.6 *thickness, of a semiconductor wafer* — the distance through the wafer between corresponding points on the front and back surfaces.

5.2 Definitions of other terms related to silicon material technology can be found in SEMI MF1241.

6 Summary of Test Method

6.1 A calibration procedure is performed to set the instrument's scale factor and other constants. If the representative wafer inversion method is used for gravity correction, the calibration procedure also determines the mechanical signature of the instrument and the effect of gravity on the wafer.

6.2 The wafer is supported by a small-area chuck with front surface up.

6.3 Both external surfaces are simultaneously scanned along a prescribed pattern by an opposed pair of probes to obtain a set of values of the distances between each surface and the nearest probe. In each case, both members of the pair of distances is taken at the same value of the x and y coordinates.

6.4 The paired displacement values are used to construct the median surface.

6.5 A correction for gravity effects on the median surface is made either by subtracting a gravity correction obtained (1) from measurements on a representative wafer or (2) from theoretical considerations or by repeating the scan with the wafer inverted.

6.6 One half the thickness at each point is added to the corrected median surface to construct the corrected front surface.

6.7 A least-squares reference plane is constructed from the corrected front surface.

6.8 The reference plane deviation (RPD) is calculated at each measured pair of points.

6.9 Sori is reported as the algebraic difference between the most positive RPD and the most negative RPD.

7 Apparatus

7.1 *Measuring Equipment,* consisting of wafer holding device, multiple-axis transport mechanism, probe assembly with indicator, and system controller/computer, including data processor and suitable software.

7.1.1 The equipment shall be direct reading with all necessary calculations performed internally and automatically as outlined in Section 11.2.

7.1.2 The equipment shall be equipped with an over-range signal.

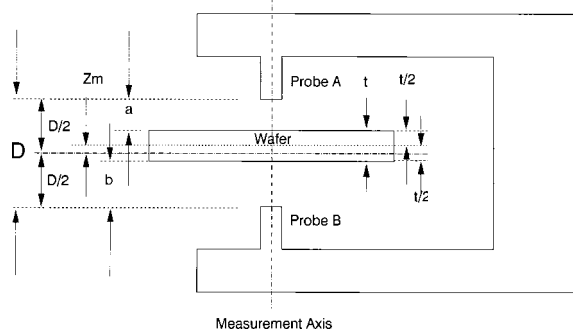7.1.3 Instrument data reporting resolution shall be 100 nm or smaller.

7.1.4 The measuring equipment contains the following subsystems:

7.1.4.1 Wafer-holding device, for example a chuck whose face is perpendicular to the measurement axis, and on which the wafer is placed for the measurement scan. The nature and size of the wafer holding device shall be agreed upon between the parties to the test.

7.1.4.2 Multiple-axis transport mechanism, which provides a means for moving the wafer-holding device, or the probe assembly, perpendicularly to the measurement axis in a controlled fashion in several directions. This motion must permit data gathering over a prescribed scan pattern covering the entire fixed quality area. Data point spacing to be used shall be agreed upon between the parties to the test.

7.1.4.3 Probe assembly with paired non-contacting displacement-sensing probes, probe supports, and indicator unit (see Figure 1).

7.1.4.3.1 The probes shall be capable of independent measurement of the distances *a* and *b* between the probed site on each surface of the sample wafer and the nearest probe surface.

Figure 1
Schematic View of Wafer, Probes, and Fixture

7.1.4.3.2 The probes shall be mounted above and below the wafer in a manner so that the probed site on one surface of the wafer is opposite the probed site on the other.

7.1.4.3.3 The common axis of these probes is the measurement axis.

7.1.4.3.4 The probe separation *D* shall be kept constant during calibration and measurement.

7.1.4.3.5 Displacement resolution shall be 100 nm or smaller.

7.1.4.3.6 The probe sensor size shall be 4 mm by 4 mm, or other value to be agreed upon between the parties to the test.

7.1.4.3.7 Measuring equipment employing either the Representative Wafer Inversion Method or the Sample Wafer Inversion Method for gravity compensation must provide precise positioning in both measurement orientations so that measurements are taken at identical locations for each orientation of the sample.

8 Materials

8.1 *Set-up Masters* — suitable to accomplish calibration and standardization as recommended by the equipment manufacturer.

8.2 *Reference Wafer* — with a reference sori value ≤20 μm that is used to determine the level of agreement between the sori value obtained by the measuring equipment under test and the reference sori value (see Section 9).

8.3 *Representative Wafer* — required only if the Representative Wafer Inversion Method is used for the gravity correction. A representative wafer shall be identical in nominal diameter, nominal thickness, fiducials, composition and crystalline orientation to those being measured. Its sori need not be known.

9 Suitability of Measuring Equipment

9.1 Determine the suitability of the measuring equipment with the use of a reference wafer and its reference sori value in accordance with the procedures of Sub-section 9.2, or by performance of a statistically-based instrument repeatability study to ascertain whether the equipment is operating within the manufacturer's stated specification for repeatability.

9.1.1 The reference sori value is the average of a number of values obtained for that wafer over a number of "passes" (repeat measurements). The reference wafer is measured on the measuring equipment under test and its reference sori value is compared against the measured sori value. The acceptable level of the agreement between the reference and measured sori values is to be agreed upon by the parties to the test.

9.2 *Procedure*

9.2.1 Select a reference wafer of appropriate criteria, together with its associated reference sori value.

9.2.2 Measure the reference wafer on the measuring equipment under test to obtain a sample sori value.

9.2.3 Subtract the two sori values to obtain the difference:

$$\Delta_{sori} = Sori_{reference} - Sori_{sample} \qquad (1)$$

9.2.4 The metric to be used to determine acceptability is difference, Δ_{sori}. Accept the measuring equipment as suitable for use if this difference is less than a value that is agreed upon between the parties to the test.

NOTE 3: If the measuring equipment is to be used to measure other parameters, such as flatness and thickness variation in addition to sori, the reference and sample sori values may be included in the reference and sample data sets specified in SEMI MF1530, but this is not necessary if only sori measurements are to be made.

10 Sampling

10.1 This test method is nondestructive and may be used on either 100% of the wafers in a lot or on a sampling basis.

10.1.1 If samples are to be taken, procedures for selecting the sample from each lot of wafers to be tested shall be agreed upon between the parties to the test, as shall the definition of what constitutes a lot.

11 Calibration and Standardization

11.1 Calibrate the measuring equipment in accordance with the manufacturer's instructions.

11.2 When using the Representative Wafer Inversion Method for correcting the gravity-induced deflection, determine $z_{gravity}$, the deflection due to gravity and machine effects on the representative wafer, in accordance with Section 12 and Section 13 through paragraph 13.7.

12 Procedure

12.1 Prepare the apparatus for measurement of wafers, including selection of diameter, peripheral fiducials, scan area and data display/output functions. Also select the gravitational correction method from one of the following:

- Reference Wafer Inversion Method (see Note 2),

- Sample Wafer Inversion Method, or

- Theoretical Modeling Method.

12.2 Select the fixed quality area (FQA) by specifying the nominal edge exclusion (*EE*).

12.3 Introduce the test specimen into the measurement mechanism with the front surface upward and initiate the measurement sequence to determine and record the distances between each probe and the nearest wafer surface in pairs, *a* and *b*, at each measurement position. Proceed directly to Section 13 unless (1) the Sample Wafer Inversion Method is being used to correct for effects of distortion due to gravity or (2) a representative wafer is being measured to obtain the gravity correction for use in the Representative Wafer Inversion Method (see Note 2).

12.4 Repeat Section 12.2 with the wafer inverted (front surface downward).

13 Calculations

13.1 The following calculations are performed automatically within the instrument. An outline of the calculation structures is provided here to indicate the nature of the procedure.

13.2 Determine the displacements (distances) between each probe and the nearest surface of the wafer (in pairs) at intervals along the scan pattern.

NOTE 4: From Figure 1, note that the distance between Probe *A* and the nearest surface of the wafer is displacement value *a* and the distance between Probe *B* and the nearest surface of the wafer is displacement value *b*.

13.3 Set the origin of the *z*-axis at the midpoint between the two probes, *A* and *B*.

13.4 Find the distance, z_m, of the median surface from the *z*-axis origin at each point. From Figure 1,

$$z_m = \frac{D}{2} - a - \frac{t}{2} \qquad (2)$$

and

$$z_m = -\frac{D}{2} + b + \frac{t}{2} \qquad (3)$$

Therefore,

$$\frac{D}{2} - a - \frac{t}{2} - \frac{D}{2} + b + \frac{t}{2} = 2z_m \qquad (4)$$

and

$$z_m = \frac{b-a}{2} \qquad (5)$$

where

D = distance between Probes A and B,
a = distance between Probe A and the nearest (top) wafer surface,
b = distance between Probe B and the nearest (bottom) wafer surface, and
t = wafer thickness.

13.5 For measurements in the normal orientation (front surface up), call the position of the median surface z_{nor}.

13.6 For measurements in the inverse orientation (back surface up), call the position of the median surface z_{inv}.

13.7 For measurements on representative wafers or on sample wafers when the Sample Wafer Inversion Method is being used, determine the gravitational correction to the median surface as follows:

$$z_{gravity} = \frac{z_{nor} + z_{inv}}{2} \qquad (6)$$

NOTE 5: This cancels the effect of the representative wafer's shape while retaining the effect of gravity.

13.8 Determine the gravity compensated median surface as follows:

13.8.1 *Representative Wafer Inversion Method* — Subtract $z_{gravity}$ from z_{nor} to produce z_{com} at each measurement point.

NOTE 6: The Representative Wafer Inversion Method deals not only with first-order gravitational effects, but also with other effects that may influence the measured value, such as wafer-periphery effects, some machine-specific signature, etc.

13.8.2 *Sample Wafer Inversion Method* — Subtract $z_{gravity}$ from z_{nor} to produce z_{com} at each measurement point. Note that this is equivalent to taking the difference between the normal and inverted measurement values at each point:

$$z_{com} = z_{nor} - z_{gravity} = z_{nor} - \frac{z_{nor} + z_{inv}}{2} = \frac{z_{nor} - z_{inv}}{2} \qquad (7)$$

13.8.3 *Theoretical Modeling Method* — Apply gravitational correction developed from a theoretical model. Although a rigorous model is not known to exist, approximate corrections have been calculated[3] (see Related Information 1).

13.9 Determine the thickness of the wafer at each point from the following equation:

$$t = D - (a + b) \qquad (8)$$

where the terms are defined after Equation (5).

13.10 Add one half the thickness to the gravity compensated median surface z value at each point to yield the gravity-compensated front surface:

$$z_{fcom} = z_{com} + \frac{t}{2} \qquad (9)$$

13.11 Construct a reference plane that is a least-squares fit to the gravity-compensated front surface data at all the points of the of the scan pattern. The reference plane is of the form:

$$z_{ref} = a_R x + b_R y + c_R \qquad (10)$$

where a_R, b_R, and c_R are constants selected so that

$$\sum_{x,y} [z_{fcom}(x, y) - (a_R x + b_R y + c_R)]^2 \qquad (11)$$

is minimized over the FQA (see Section 12.2).

13.12 Subtract the z-value of the reference plane (z_{ref}) from the compensated z-value, z_{com}, at all the points of the scan pattern to yield the reference plane deviation (RPD) at each point:

$$RPD = z_{fcom} - z_{ref} \qquad (12)$$

13.13 Calculate the sori of the wafer as the difference between the maximum (most positive) and minimum (most negative) RPD:

$$sori = RPD_{max} - RPD_{min} \qquad (13)$$

13.14 Record the calculated sori value.

13.15 For referee or other measurements where the wafer is measured more than once, calculate the maximum, minimum, sample standard deviation, average, and range of all measurements on the sample.

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3 Application Note: "Gravitational Sag in Silicon Wafers," ADE Corporation, 80 Wilson Way, Westwood, MA 02090-1806, Nov. 20, 1991.

13.15.1 Record sample standard deviation and other statistical parameters as agreed upon between the parties to the test.

14 Report

14.1 Report the following information:

14.1.1 Date, time, and temperature of test,

14.1.2 Identification of operator,

14.1.3 Location (laboratory) of test,

14.1.4 Identification of measuring instruments, including wafer-holding device diameter, data point spacing, sensor size, and gravitation-correction method,

14.1.5 Lot identification, including nominal diameter, nominal center-point thickness, and nominal edge exclusion (*EE*) specified.

14.1.6 Description of sampling plan, if any, and

14.1.7 Sori of each wafer measured.

14.2 For referee tests also include in the report the standard deviation of each set of wafer measurements and such other statistical parameters as have been agreed to by the parties to the test.

15 Precision

15.1 An inter-laboratory experiment to establish the precision of this test method has not been conducted.

15.2 In order to estimate the precision, an error analysis was performed in accordance with ASTM Practice D 4356. This analysis indicates the best 1s precision obtainable by this method is ±0.35 μm.

15.3 This value is obtained as follows:

15.3.1 The instrument resolution is specified to be 0.1 μm. Therefore, the values *a* and *b* cannot be known closer than this during each of the six measurements made during the determination of sori:

- two measurements on the reference wafer in the normal orientation,

- two measurements on the reference wafer in the inverted orientation, and

- two measurements on the sample wafer in the normal orientation.

15.4 Equation A1.1 in ASTM Practice D 4356 holds that:

$$\text{Precision (1s)} = \pm\sqrt{2e^2 N} \\ = \pm\sqrt{2 \cdot (0.1)^2 \cdot 6} \qquad (14) \\ = \pm 0.35$$

where:

e = single measurement error (0.1 μm), and

N = number of measurements made during the determination of sori (6).

15.5 In practice, this precision is degraded by the factors listed in Section 3.

16 Bias

16.1 *Bias* — No standards exist against which the bias of this test method can be evaluated.

17 Keywords

non-contact measurement; semiconductor; shape; silicon; sori; wafers

RELATED INFORMATION 1
MEASUREMENT ERRORS DUE TO DIFFERENCES IN DIAMETER AND THICKNESS BETWEEN A REPRESENTATIVE WAFER AND A WAFER UNDER TEST

NOTICE: This related information is not an official part of SEMI MF1451. This related information was approved for publication by full letter ballot on August 16, 2004.

R1-1 The sag, or deflection induced by gravity at the edge of a wafer supported at its center, in μm, has been estimated[3] as:

$$S = \frac{(3 \times 10^8) k g d D^4}{32 E t^2} = \frac{K D^4}{t^2} \qquad \text{(R1-1)}$$

where:

S = deflection , in μm,
k = geometrical constant (=0.5854),
g = gravitational constant (980 cm/s^2),
d = density of silicon (2.329 g/cm^3),
E = Young's modulus (~1.6×10^{12} dyne/cm^2,
D = nominal wafer diameter, in mm, and
t = nominal wafer thickness, in μm.

K, the constant of proportionality is therefore equal to 7.83 × 10^{-3} μm^3/mm^4. Table R1-1 gives estimated values of sag in micrometers for 100 mm through 300 mm diameter wafers with thickness and diameter as specified in SEMI M1.

Table R1-1. Estimated Sag, in μm, of Wafers of Nominal Diameter and Thickness

Diameter, mm	Thickness, μm	SEMI M1 Reference	Estimated Sag, μm
300	775	SEMI M1.15	105.6
200	725	SEMI M1.9	23.8
150	675	SEMI M1.8	8.7
150	625	SEMI M1.13	10.1
125	625	SEMI M1.7	4.9
100	525	SEMI M1.5	2.8

R1-2 For small variations about the nominal values of diameter and thickness, the relative change of the gravity effect is 4 times the relative change of the diameter and −2 times the relative change of thickness:

$$\frac{\partial S}{\partial d} = 4 \frac{K d^3}{t^2} \qquad \text{(R1-2)}$$

and

$$\frac{\partial S}{\partial t} = -2 \frac{K d^4}{t^3} \qquad \text{(R1-3)}$$

Therefore the relative changes are as follows:

$$\frac{\partial S}{S} = 4 \frac{\partial d}{d} \qquad \text{(R1-4)}$$

and

$$\frac{\partial S}{S} = -2 \frac{\partial t}{t} \qquad \text{(R1-5)}$$

R1-3 Table R1-2 gives examples of worst-case gravity effect errors (in micrometers), for 100 mm through 300 mm diameter wafers with thickness and diameter tolerances as specified in SEMI M1.

Table R1-2 Examples of Gravity Effect Errors

300 mm Diameter Wafers				
		Actual Diameter, mm		
		299.8	300.0	300.2
		Gravity Effect Errors, μm		
Actual Thickness, μm	755	3.90	5.67	5.96
	775	−1.68	0.00	0.28
	795	−6.84	−5.25	−4.98

200 mm Diameter Wafers				
		Actual Diameter, mm		
		199.8	300.0	200.2
		Gravity Effect Errors, μm		
Actual Thickness, μm	705	0.77	1.37	1.47
	725	−0.57	0.00	0.10
	745	−0.52	−1.26	−1.17

150 mm Diameter Wafers				
		Actual Diameter, mm		
		149.8	150.0	150.2
		Gravity Effect Errors, μm		
Actual Thickness, μm	655	0.49	0.54	0.59
	675	−0.05	0.00	0.05
	695	−0.54	−0.49	−0.45

150 mm Diameter Wafers				
		Actual Diameter, mm		
		149.8	150.0	150.2
		Gravity Effect Errors, μm		
Actual Thickness, μm	610	−0.45	0.51	0.56
	625	−0.05	0.00	0.05
	640	−0.52	−0.47	−0.42

125 mm Diameter Wafers				
		Actual Diameter, mm		
		124.5	125.0	125.5
		Gravity Effect Errors, μm		
Actual Thickness, μm	605	0.25	0.33	0.41
	625	−0.08	0.00	0.08
	645	−0.37	−0.30	−0.22

100 mm Diameter Wafers				
		Actual Diameter, mm		
		99.5	100.0	100.5
		Gravity Effect Errors, μm		
Actual Thickness, μm	505	0.17	0.23	0.29
	525	−0.06	0.00	0.06
	545	−0.26	−0.20	−0.15

SEMI MF1526-95 (Withdrawn 1104)
Test Method for Measuring Surface Metal Contamination on Silicon Wafers by Total Reflection X-Ray Fluorescence Spectroscopy

This standard was originally published by ASTM International as ASTM F 1526-94. It was formally approved by ASTM balloting procedures and adhered to ASTM patent requirements. Though ownership of this standard has been transferred to SEMI, it has not been formally approved by SEMI balloting procedures and does not adhere either to SEMI Regulations dealing with patents or to SEMI Editorial Guidelines. Available at www.semi.org September 2004, to be published November 2004. Last published by ASTM International as ASTM F 1526-95 (Reapproved 2000).

NOTICE: This document was balloted and approved for withdrawal in 2004.

1. Scope

1.1 This test method covers the quantitative determination of elemental areal density on the surface of polished single crystal silicon substrates using total reflection X-ray fluorescence spectroscopy (TXRF[1]) with a monochromatic X-ray source. [2]

1.2 This test method can be used for both *n*-type and *p*-type silicon.

1.3 This test method can be used to detect surface elemental contamination that is within the analyte depth of approximately 5 nm for highly mirror-polished silicon wafers. The analytic depth increases with surface roughness. [3]

1.4 This test method is especially useful for determining the surface elemental areal densities in the native oxide or in chemically grown oxide of polished silicon wafers after cleaning.

1.5 This test method is useful for elemental areal densities between 10^9 and 10^{15} atoms/cm^2 within the measurement area. See Annex A1 for a discussion of the relationship between repeatability and detection limit.

1.6 This test method is useful for detecting elements with atomic number between 16 (S) and 92 (U), depending upon the X-ray source provided in the instrument. This test is especially useful for detecting the following metals or elements: potassium, calcium, titanium, vanadium, chromium, manganese, iron, cobalt, nickel, copper, zinc, arsenic, molybdenum, palladium, silver, tin, tantalum, tungsten, platinum, gold, mercury, and lead.

1.7 The detection limit depends upon atomic number, excitation energy, photon flux of excitation X-rays, instrumental background, integration time, and blank value. For constant instrumental parameters, the interference-free detection limits vary over two orders of magnitude as a function of atomic number of the element.

1.8 This test method is nondestructive.

1.9 This test method is complementary to a variety of other test methods:

1.9.1 Electron spectroscopy for chemical analysis that can detect elemental surface areal densities down to the order of 10^{13} atoms/cm^2.

1.9.2 Auger electron spectroscopy that can detect elemental surface areal densities down to the order of 10^2 atoms/cm^2.

1.9.3 Nitrogen-beam Rutherford backscattering spectrometry that can detect down to 10^{10} atoms/cm^2 for some elements but cannot mass resolve heavy elements of nearby atomic number.

1.9.4 Secondary ion mass spectrometry that can detect low-atomic-number elemental areal densities in the range of 10^8 to 10^{12} atoms/cm^2 but cannot provide adequate detection limits for transition metals with atomic number between 22 titanium and 30 zinc. This method is destructive.

1.9.5 Vapor phase decomposition (VPD) of surface metals followed by atomic absorption spectroscopy (AAS), where the metal detection limits are from 10^8 to 10^{11} atoms/cm^2, but there is no spatial information available and the analysis time is longer than TXRF. This method is destructive.

1.10 This test method uses X-radiation; it is absolutely necessary to avoid personal exposure to X-rays. It is especially important to keep hands or fingers out of the path of the X rays and to protect the eyes from scattered secondary radiation. The use of commercial film badge or dosimeter service is recommended, together with periodic checks of the radiation level at the hand and body positions with a Geiger-Muller counter calibrated with a standard nuclear source. The present maximum permissible dose for total body exposure of an individual to external X-radiation of quantum energy less than 3 MeV over an indefinite period is 1.25 R (3.22×10^{-4} C/kg) per calendar quarter (equivalent to 0.6 mR/h (1.5×10^{-7} C/kg-h) as established in the Code of Federal Regulations, Title 10, Part 20. The present maximum permissible dose of hand and forearm exposure

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1 There are several acronyms in use: TXRF, TRFA, and TRXRF; however, TXRF is the most common in the technical literature.

2 There are some non-monochromatic TXRF instruments that are no longer commercially available and that do not provide the detection limits described herein.

3 The extreme case of roughness on the backside of wafers is addressed by Hockett, R. S., "TXRF Measurement of Substrate Backside Contamination," Cleaning Technology in Semiconductor Device Manufacturing, ECS Proceedings, Vol 92-12, The Electrochemical Society, Inc., Pennington, NJ, 1992, p. 350.

under the same conditions is 18.75 R (4.85×10^{-3} C/kg) per calendar quarter (equivalent to 9.3 mR/h (2.4×10^{-6} C/kg-h)). Besides the above stated regulations, various other government and regulatory organizations have their own safety requirements. It is the responsibility of the user to make sure that the equipment and the conditions under which it is used meet these regulations (see 1.11).

1.11 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*
E 122 Practice for Choice of Sample Size to Estimate a Measure of Quality of a Lot or Process [4]
E 135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials[5]
2.2 *Federal Standard:*
CFR Title 10, Part 20 [6]

3. Terminology

3.1 Most terms used in this test method are defined in Terminology E 135.
3.2 *Definitions of Terms Specific to This Standard:*
3.2.1 *anglescan*—a measurement of the emitted fluorescence signal as a function of glancing angle.
3.2.2 *critical angle*—the incident X-ray glancing angle below which total external reflection of the incident X-ray occurs.

4. Summary of Test Method

4.1 Fig. 1 shows a block diagram of the technique. Monochromatic X rays from an X-ray source impinge the surface of a polished silicon substrate at a glancing angle that is below the angle for total external reflection of the X rays. The evanescent wave of the X rays penetrates the polished silicon surface with an exponential decay of intensity versus depth dependent upon the total electron density of the native oxide and the silicon substrate. One exponential decay length is approximately 5 nm for silicon of all resistivity.

4.2 The evanescent wave excites the fluorescence energy levels of the surface atoms which then emit fluorescence X-rays characteristic of their atomic number. Emitted fluorescence X-rays are detected by a lithium-drifted silicon detector, or other solid state detector, which is an energy dispersive spectrometer. Experience indicates that for measurement of samples with high levels ($>10^{11}$ atoms/cm^2) of specific elements that have been measured with other methods, such as those listed in 1.9, the integrated counts per second under the fluorescence peaks are linearly proportional to the elemental areal density.

4.3 Reproducible, rapid analysis can be accomplished using a calibration specimen, supplied by the TXRF instrument manufacturer or developed by another company, with at least one known elemental areal density in the measurement area. This calibration specimen is analyzed by the TXRF instrument to provide a measured number of integrated fluorescence counts per second corresponding to the known elemental areal density. Then one or more test specimens are analyzed under the same instrumental conditions. The   integrated fluorescence counts per second for the elements detected on the   test specimen are quantified using relative sensitivity factors (RSFs) with respect to the calibration element count rate per known areal density, where   the RSFs are contained within the instrument software. The lack of true standards   precludes determination of the accuracy of this test method.

5. Significance and Use

5.1 TXRF can measure the elemental, particularly metal, areal densities on polished silicon wafer product.
5.2 The TXRF measurement facilitates the production of silicon wafers with controlled upper limits on metal areal densities.
5.3 This test method can be used for monitoring a mirror-polished wafer cleaning process, research and development, and materials acceptance purposes.

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4 Annual Book of ASTM Standards, Vol 14.02, ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428. Telephone: 610-832-9500, Fax: 610-832-9555, Website: www.astm.org
5 Annual Book of ASTM Standards, Vol 03.05.
6 Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

6. Interferences

6.1 The interferences in conventional X-ray fluorescence spectroscopy are common to TXRF also. These include, but are not limited to: overlap of fluorescence lines, escape peak and sum peak overlap, energy gain calibration drift, X-ray source stability, and instrumental background peaks. However, no X-ray fluorescence corrections for secondary fluorescence or for matrix absorption are required for TXRF. Interferences common to software procedures and calculations can be evaluated by comparing data sets; see Annex A1.

6.2 In addition to conventional interferences, there are interferences that are unique to TXRF as follows:

6.2.1 If the glancing angle calibration is not reproducible, variability is introduced to the measurement,

6.2.2 If the glancing angle calibration is inaccurate, bias is introduced to the measurement,

6.2.3 If the anglescan of the known elemental impurity on the calibration specimen is different from the elemental impurity anglescan on the test specimen, this may introduce a bias to the quantification. An example can be the measurement of particulate metal contamination on a test specimen while using a calibration specimen which has the calibration metal localized within the native oxide,

6.2.4 Mechanical vibration may degrade the detector energy resolution, and this may degrade detection limits,

6.2.5 If the specimen is not chem-mechanically polished, there will be a loss in detection capability, a bias in quantification, and an increase in variability of the measurement. There is some possibility that the surface roughness and waviness differences from different cleaning processes may cause these interferences also. The semiquantitative effect of surface roughness upon the TXRF measurement is under study.

6.2.6 Bias in the assigned elemental areal density of the calibration specimen can introduce bias into the TXRF measured areal densities,

6.2.7 Surface contamination introduced during handling of the test specimens or during the measurement itself will introduce a bias to the measurement, if this surface contamination contains the element(s) to be measured,

6.2.8 Bias in the RSF of a fluorescence line can introduce bias to the measurement,

6.2.9 Nonlinearity of detected fluorescence signal versus impurity areal density may occur due to high deadtime of some detectors under the condition of high-total signal count rates, and

6.2.10 Fluorescence curve smoothing may affect the quantification accuracy.

6.2.11 Instrumental peaks may be generated by the incident X-ray beam diffracting from the silicon crystal and the diffracted beam entering the detector to excite metals in the detector window or detector housing. This effect can be tested for by appropriate experiments. [7]

7. Apparatus

7.1 *TXRF Instrument*,   equipped with a monochromatic X-ray source, test specimen handling equipment, a method for glancing angle calibration, an energy-dispersive spectrometer X-ray detector, software for background subtraction, peak integration, analysis and RSFs, and an analysis ambient without argon (for example, vacuum of 10^{-2} torr, or helium gas). The methods for glancing angle calibration are presently proprietary for each TXRF instrument manufacturer. A TXRF manufacturer may include a subtraction routine for escape peaks and these signals may already be removed.

7.2 *Reference Wafer*— The suitability of the apparatus shall be determined with the use of a reference wafer and its associated data set in accordance with the procedures of Annex A2, or by performance of a statistically-based instrument repeatability study to ascertain whether the equipment is operating within the manufacturer's stated specification for repeatability.

7.3 *Class 100 Air Environment*—The area for sample transfer to the instrument measurement stage must be enclosed in this.

8. Sampling

8.1 A sampling procedure must be used to evaluate the characteristics of a group of silicon wafers. No general sampling procedure is included as part of this test method, because the most suitable sampling plan will vary considerably depending upon individual conditions. For referee   purposes, a sampling plan shall be agreed upon before conducting the test. See Practice E 122 for suggested choices of sampling plans.

9. Specimen Requirements

9.1 Test specimens must be chem-mechanical polished on the side used for analysis.

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7 Yakushiji, K., Ohkawa, S., Yoshinga, A., and Harada, J.,“ Origins of Spurious Peaks of Total Reflection X-Ray Fluorescence Analysis of Si Wafers Excited by Monochromatic X-Ray Beam W1-beta,” Japanese Journal of Applied Physics, Vol 33, 1994, pp. 1130—1135.

10. Test Specimen Measurement

10.1 Load the test specimen into the TXRF instrument in a contamination free environment of Class 100 or better.

10.2 Measure the TXRF spectrum for the test specimen.

10.3 Calculate the net integrated counts/second for detected elemental peaks.

10.4 Using the calibration wafer data for the calibration element and the RSFs for the other elements, calculate the elemental areal densities for each detected element on the test specimen according to Eq. 1.

$$D_{u,m} = \left(1/F_{s,m}\right) \times \left(CPS_{u,m}\right) \times \left(CPS_s\right)^{-1} \times \left(A_s\right) \qquad (1)$$

where:

$D_{su,m}$	=	the areal density in atoms/cm^2 of element "m" on the test specimen "u",
$F_{s,m}$	=	the RSF of element "m" with respect to the calibration element "s",
$CPS_{u,m}$	=	the integrated counts/second of element "m" detected on test specimen "u",
CPS_s	=	the integrated counts/second of the calibration element on the calibration standard, and
A_s	=	the assigned areal density in atoms/cm^2 of the calibration element.

11. Calibration Standard

11.1 The calibration standard and the method of its preparation must be agreed upon between the parties.

NOTE 1—Several methods for preparing calibration standards are in practice. [8]

The present state-of-the-art calibration standard is a chem-mechanical polished silicon substrate with a surface metal areal density of between 1 by 10^{12} and 1 by 10^{14} atoms/cm^2 located in the analysis area. The k-alpha fluorescence signal from the calibration standard metal is free of interferences, that is, escape peaks, sum peaks, and fluorescence peaks from other contamination, and free from external contamination sources. The preferred element should not be easily added as a contaminated element (for example, iron) or diffuse away from the analyte depth over time (for example, gold or copper). The preferred element is nickel or vanadium. Calibration can also be done using a set of wafers with different known areal densities of a particular element.

The calibration standard should have been measured by an appropriate analytical method to determine the metal areal density of the calibration metal. Some analytical methods which have been used for assigning quantitative values to calibration standards include:

(a) Nitrogen-Beam Rutherford Backscattering Spectrometry (N-RBS)—If this method is used, there should not be any other metals present at levels greater than 1 % of the calibration metal areal density within a mass range of ±5 atomic mass unit. The N-RBS measurement is absolute and must be made within the analysis area of the TXRF. Other backscattering methods are also used to calibrate elemental areal densities. These include forward scattering Rutherford backscattering (F-RBS) and heavy ion backscattering spectrometry (HIBS).

(b) Vapor Phase Decomposition—This followed by atomic absorption spectrometry (VPD/AAS) is used to calibrate a spin coating contamination process for making calibration standards. The VPD/AAS measurement is destructive. The contamination process should also be shown to be uniform across the wafer by using TXRF or SIMS mapping. The VPD/AAS absolute assignment is based upon Atomic Absorption Spectroscopy standards. An error in the elemental recovery rate of VPD is a source of error in accuracy.

(c) Implant—Ion implant the reference element into a pre-amorphized silicon surface and using solid phase epitaxy to regrow the amorphous silicon into crystal silicon. [9] This procedure sweeps the ion implanted reference element to the sample surface if the reference element is much more soluble in the amorphous silicon than in the crystal silicon. The assignment of the quantitative level in atoms/cm^2 is done using the ion implant current to determine the implant dose.

(d) Diluted Atomic Absorption Standard Solution—Deposit onto a polished silicon substrate a diluted atomic absorption standard solution of a metal onto a localized spot much less than the size of the TXRF analysis area. This approach assumes none of the calibration metal is lost

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8 Hockett, R. S., "TXRF Reference Standards: A Discussion," Contamination Control and Defect Reduction in Semiconductor Manufacturing III, Vol 94-9, edited by Dennis N. Schmidt, The Electrochemical Society, Pennington, NJ, 1994, pp. 323–334.

9 Hockett, R. S., and Jacobson, D. C., "A New Approach to TXRF Standards," Extended Abstracts, Vol 93-1, The Electrochemical Society, Inc., Pennington, NJ, 1993, p. 1289; Jacobson, D. C., Poate, J. M., Higashi, G. S., Boone, T., Eaglesham, D. J., and Hockett, R. S., "Ion Implanted Calibration Standards for Si Surface Contamination Detection by TXRF," Materials Research Society, Proceedings of the Spring '93 MRS Meeting, San Francisco, April 26–29, 1993.

during the drying of the solution. The deposition spot must be easily found by the TXRF instrument in order to analyze the entire deposited dried solution. The absolute assignment is based upon the AAS standards. The deposited solution must produce one of two TXRF anglescan forms:

(1) A metal fluorescence count rate versus angle that is independent of glancing angle for angles 80 % below the critical angle (see Curve *(a)* in Fig. 2 [10]).

(2) A metal fluorescence count rate versus angle that is characteristic of metal contamination localized within a 3-nm thickness of the surface (see Curve *(b)* in Fig. 2).

11.2 An anglescan of the calibration standard must be made to verify if the anglescan of the calibration element is similar to Curve *(a)* (residue) or Curve *(b)* (in the film) in Fig. 2.

11.3 An appropriate non-TXRF measurement of the calibration standard must be agreed upon between parties in order to assign the elemental areal density to the calibration standard element.

11.4 The calibration of the other elements detectable by the TXRF instrument must be completed using relative sensitivity factor (RSFs) developed by the instrument manufacturer and stored in the instrument computer program. These RSFs are a function of the X-ray source energy, the atomic number of the fluorescing element, and the fluorescence energy level, so if the X-ray source energy is changed, a different set of RSFs must be used.

12. Procedure

12.1 Turn on the instrument in accordance with the manufacturer's instructions.

12.2 *Analytical Conditions*:

12.2.1 Choose and record the analytical conditions for the test specimen measurement. This includes:

12.2.1.1 Voltage to the X-ray source,

12.2.1.2 Current to the X-ray source,

12.2.1.3 Ambient in the analysis chamber,

12.2.1.4 Glancing angle,

12.2.1.5 Integration time,

12.2.1.6 X-ray source energy, and

12.2.1.7 Analysis location on the test specimen.

13. Calibration

13.1 Load the calibration specimen into the TXRF instrument in a contamination free environment defined by Class 100 or better.

13.2 Measure the TXRF spectrum of the calibration specimen under the same operating conditions of current and voltage applied to the X-ray source and of glancing angle to be used for the measurement of the test specimen. The measurement time and the ambient (for example, vacuum, air, nitrogen, helium, etc) in the analysis chamber may be different between the calibration and test specimen measurements. If the calibration anglescan is similar to Curve *(b)* of Fig. 2, use a glancing angle which is 70 to 80 % of the critical angle. If the calibration anglescan is similar to Curve *(a)* of Fig. 2, use any glancing angle that is below 85 % of the critical angle.

13.3 A polished silicon substrate with no detectable elemental contamination (that is, a BLANK), or a series of polished silicon substrates each of which shows no detectable metal contamination for a selected element of interest (that is, a set of element-specific BLANKs) and which collectively represent a blank for all elements of interest, must be measured by the TXRF measurement under the condition of the test specimen to verify three are no instrumental background signals. An example BLANK for all elements except sulfur is shown in Fig. 3.

13.4 Have the TXRF instrument integrate the calibration elemental fluorescence signal and subtract the background to obtain a net integrated counts/second that corresponds to the assigned elemental areal density. The background may be subtracted by a deconvolution routine or by a linear fit routine.

14. Report

14.1 Record the following information (see also 12.2.1):

14.1.1 Test specimen identification,

14.1.2 Calibration specimen identification,

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10 Eichinger, P., Rath, H. J., and Schwenke, H., "Application of Total Reflection X-Ray Fluorescence Analysis for Metallic Trace Impurities on Silicon Wafer Surfaces," Semiconductor Fabrication: Technology and Metrology, ASTM STP 990, ASTM, 1989, pp. 305–313.

14.1.3 Anode material,

14.1.4 Monochromator,

14.1.5 Operator,

14.1.6 Date,

14.1.7 Type of instrument including model and manufacturer,

14.1.8 Software version,

14.1.9 Analysis time, and

14.1.10 Crystal orientation of the test specimen with respect to the incoming X-ray beam.

15. Precision and Bias[11],[12]

15.1 *Precision*—The precision was estimated through two round robins using monochromatic TXRF instruments.

15.1.1 The within-laboratory precision was estimated from a sequential round robin[12] that used two reference samples and six unknowns with surface contamination of iron, nickel, copper and zinc, between 10^{11} and 10^{12} atoms/cm^2. Thirteen laboratories participated. At each laboratory the samples were measured once per day for 4 days. The measurement conditions were X-ray rotating anode with a tungsten target, LiF (200) monochromator (9.67 keV line selected), 30 kV, 200 mA, glancing angle of 0.05°, 10^{-2} torr ambient, 10-mm diameter analysis area, and 1000-s integration time. The data were reported by each laboratory as the average of the four readings and the one standard deviation of the four readings. The 95 % confidence, within-laboratory relative precision was 28 %, that is, 2.8 times the within-laboratory relative one standard deviation of 10 %.
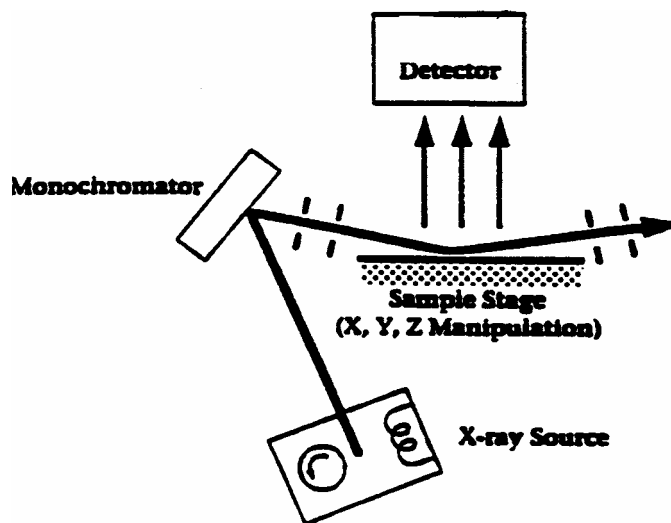
15.1.2 The between-laboratory precision was estimated from a spoke wheel round robin [13] that used sets of one reference sample and three unknowns, one of which was a BLANK. The reference sample and the unknowns were contaminated with surface nickel between 10^{11} and 10^{12} atoms/cm^2. Seventeen organizations participated. At each laboratory the samples were measured on multiple days. The measurement conditions were X-ray rotating anode with a tungsten or gold target, monochromator (9.67 keV or higher line selected), 30 kV, 200 mA or higher, glancing angle of 0.1°, 10-mm diameter analysis area, and 1000-s integration time. The 95 % confidence between-laboratory precision was: $\pm 8 \times 10^{10}$ atoms/cm^2 at an average 15×10^{10} atoms/cm^2, and $\pm 20 \times 10^{10}$ atoms/cm^2 at an average 45×10^{10} atoms/cm^2.

15.1.3 Analysis conditions other than those used for these round robins listed in 15.1.1 and 15.1.2 can be used, but the precision has not been estimated for other analysis conditions.

15.2 *Bias*—The bias of this test method cannot be estimated, because there are no absolute standards for this test method.

16. Keywords

16.1 contamination; metals; silicone; surface; TXRF; X-ray fluorescence

11 Hockett, R. S., Ikeda, S., and Taniguchi, T., "TXRF Round Robin Results," Cleaning Technology in Semiconductor Device Manufacturing, ECS Proceedings, edited by J. Ruzyllo and R. E. Novak, Vol 92-12, The Electrochemical Society, Pennington, NJ, 1992, pp. 324–337.

12 Supporting data are available from ASTM Headquarters. Request RR: F01-1009 and F01-1012.
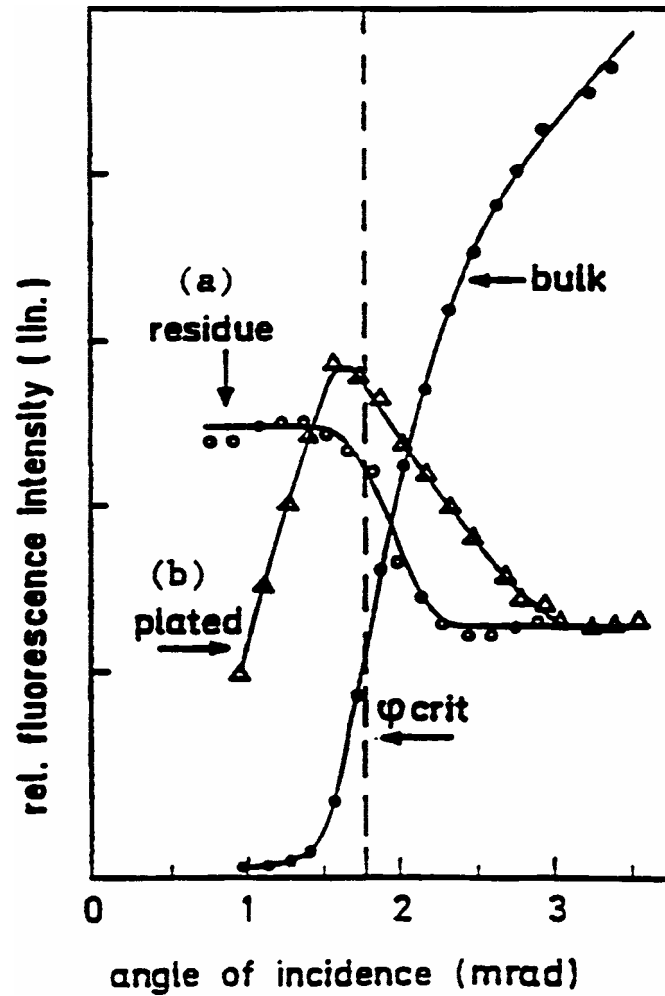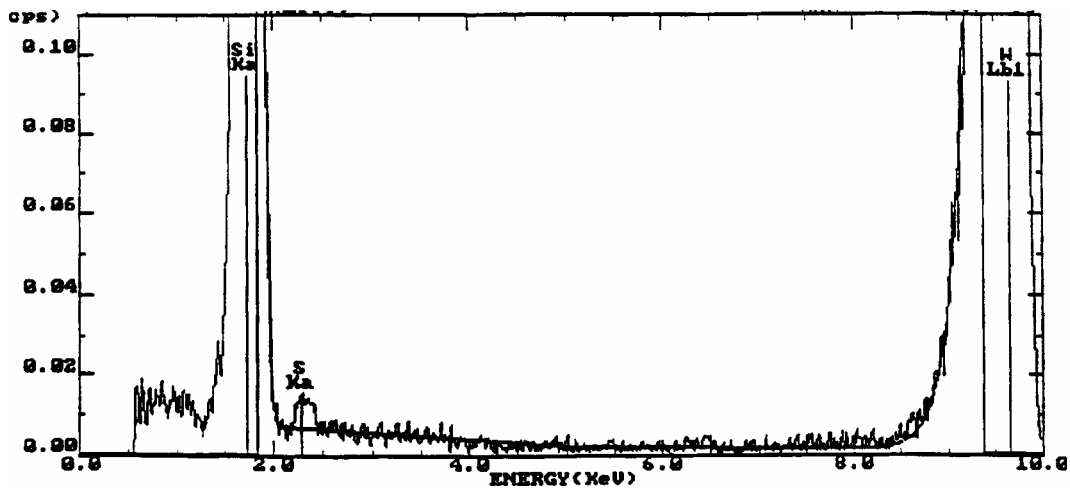
FIG. 1 Block Diagram of the TXRF Technique

FIG. 2 Experimental Curves for the Angular Dependence of the Fluorescence Intensity from Plated or Sputtered Submonoatomic Nickel-layers (Δ), Layers Produced by Evaporation of a Nickel Salt Solution (O) and the Silicon Substrate (·)

FIG. 3 Example BLANK (Except for Sulfur) Spectrum of Silicon Wafer Using TREX 610 Operated at 30 kV, 200 mA, 0.11°, 1000 s, Tungsten Target, LiF (200) Monochromator, W L-beta Line (9.67 keV)

ANNEXES

(Mandatory Information)

A1. RELATIONSHIP BETWEEN REPEATABILITY AND DETECTION LIMIT

A1.1 The consensus method to determine the limit of detection, c_L, in a photon spectroscopy, when there is no instrumental peak to subtract, is given as follows [13]:

$$c_L = 3s_b/S \qquad (A1.1)$$

where:

s_b	=	the standard deviation of the BLANK measures, and
S	=	the sensitivity (net signal divided by atoms/cm^2).

The numeral 3 in Eq. A1.1 is chosen so that a 99.6 % confidence level applies for a strictly one-sided Gaussian distribution; but it is recognized that at low concentrations, non-Gaussian distributions are more likely.

A1.1.1 It is commonly assumed that the standard deviation of the BLANK measures, for short term measurements, is given by Poisson statistics of the photons, and this leads to Eq A2.2 for the limit of detection which is commonly reported in the technical literature.

$$c_L = 3(areal\ density\ of\ ref)(background\ counts)^{1/2}/(net\ signal) \qquad (A1.2)$$

A1.1.2 This limit of detection includes the key assumption that the standard deviation of the BLANK measures is given only by Poisson statistics of the X-ray photons, and that no other variability contribution is significant for this term. This assumption may be valid only for short term estimation of the limit of detection.

A1.2 For long term estimation of the limit of detection, the standard deviation of the BLANK measures is expected to have contributions from other variabilities than just the Poisson statistics, and therefore the long term estimation of the limit of detection is expected to be larger than the short term limit of detection. Examples of other contributions to variability in the BLANK measures may include, but not be limited to, the glancing angle calibration and X-ray beam divergence.

A2. COMPARING DATA SETS

A2.1 Introduction

A2.1.1 In qualifying a measurement system for operation, it can be useful to compare values ascribed to an artifact such as a reference sample against those obtained for that artifact on a machine under test. This annex outlines a way in which the multiple element measurement data can be used to monitor the effects of interferences that may arise from software procedures and calculations in the instrument.

A2.1.2 A data set is that set of data used in computation of surface elemental contamination by TXRF.

A2.1.3 A referee wafer (artifact) is accompanied by its own data set (referee data set (RDS)), in which each data point is the average of a number of repeated measurements. The artifact is measured on a machine under test and its RDS is compared against the resultant measured sample data set. Differences in the data sets are computed. The parameter used to determine agreement between the artifact and the system under test and the acceptable level of this agreement is to be agreed upon between the using parties.

A2.2 Summary of Test Method

A2.2.1 Select a referee wafer of appropriate criteria, for which an RDS has been obtained.

A2.2.2 Measure the referee wafer on the machine under test to obtain a sample data set (SDS).

A2.2.3 Subtract the two to obtain a difference data set (DDS) as follows:

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13 International Union of Pure and Applied Chemistry, Analytical Chemistry Division, "Nomenclature, symbols, units and their usage in spectrochemical analysis—II, Data Interpretation," Spectrochemica Acta, Vol 33, 1978, pp. 242–245.

$$RDS - SDS = DDS \qquad (A2.1)$$

where:

DDS = the differences between the measurements made on the machine under test and the referee data set.

The DDS contains many values. The simplest metric that can be used to determine acceptability is the maximum difference, the largest absolute value in the DDS. This represents the worst-case disagreement between the machine under test and the referee data.

A2.2.4 Accept the machine as suitable for measurement if the maximum difference is less than a value that is agreed upon between the parties to the test.

A2.2.5 More complex calculations may also be used, for example, a histogram of the (element-by-element) values of the DDS along with statistical measures (mean, sigma, etc) may be compared. These measures can be compared to application-specific limits or used to provide insight into the nature and source of the difference, or both.

SEMI MF1527-1104
GUIDE FOR APPLICATION OF CERTIFIED REFERENCE MATERIALS AND REFERENCE WAFERS FOR CALIBRATION AND CONTROL OF INSTRUMENTS FOR MEASURING RESISTIVITY OF SILICON

1 Purpose

1.1 Resistivity is a widely used parameter for specification and characterization of silicon wafers for use in fabricating semiconductor devices and integrated circuits. Many types of instrumentation used for making resistivity measurements including the non-contact eddy-current instruments used for measurements made in accordance with SEMI MF673, DIN 50445, or DIN 50447 require calibration because they are relative measurements. Although measurements made with in-line four-point probes in accordance with SEMI MF84 or DIN 50441 are, in principle, absolute, control charts should be maintained for four-point probes because one cannot always be sure that the electrical resistivity of the test specimen is sufficiently homogeneous for the theoretical model of the method to apply, that the electrical thickness of the wafer is exactly equal to its measured mechanical thickness, or that the stability of the instrument is adequate.

1.2 Instruments for measuring such related parameters as spreading resistance (used in accordance with SEMI MF525 or SEMI MF672), net carrier density (used in accordance with SEMI MF1392, SEMI MF1393, or DIN 50439), and sheet resistance (used in accordance with SEMI MF1529) also require calibration.

1.3 For all these purposes, wafers of known resistivity are required. Such wafers are supplied by several sources with a wide range of certified or calibrated resistivity values. Although these wafers are often used directly, the resistivity values represented by purchased standards can also be transferred to an in-house resistivity reference wafer that is then used for routine instrument calibration or control.

1.4 The accuracy with which this transfer can be affected depends not only on the procedures for using such reference wafers but also on the procedures for material selection, instrument qualification, and calibration of the reference wafer. This guide provides recommendations for procedures for these operations appropriate to obtaining the best available accuracy in use of resistivity reference wafers.

1.5 These procedures are specifically intended for use in measuring the resistivity of silicon wafers. Extension to resistivity measurements on other semiconductor materials or to resistivity values outside the range covered by the resistivity reference wafers has not been demonstrated.

2 Scope

2.1 This guide covers the application of Certified Reference Materials (CRMs) for resistivity measurements on silicon wafers. Specifically, this guide covers the use of these CRMs for preparing resistivity reference wafers and for ensuring the quality of the instrumentation used for preparing them.

2.2 The guide covers the selection of materials for resistivity reference wafers, procedures for preparing and calibrating resistivity reference wafers, and use of resistivity reference wafers in qualifying, calibrating, and controlling various types of resistivity instrumentation.

2.3 The guide provides criteria for selection of instruments for determining the resistivity of silicon resistivity reference materials, procedures for maintaining such instruments in statistical quality control, and training requirements for operators engaged in making and using resistivity reference wafers.

2.4 Related Information is included that covers (1) suggested control charting procedures for organizations that do not already have such procedures in place, and (2) errors in resistivity determination that result from uncertainties in wafer diameter, wafer thickness, and probe-tip spacing.

NOTICE: This standard does not purport to address safety issues, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory or other limitations prior to use.

3 Referenced Standards

3.1 *SEMI Standards*

SEMI C28 — Specifications and Guidelines for Hydrofluoric Acid

SEMI C35 — Specifications and Guideline for Nitric Acid

SEMI C39 — Specification for Potassium Hydroxide Pellets

SEMI M1 — Specifications for Polished Monocrystalline Silicon Wafers

SEMI MF43 — Test Methods for Resistivity of Semiconductor Materials

SEMI MF81 — Test Method for Measuring Radial Resistivity Variation on Silicon Wafers

SEMI MF84 — Test Method for Measuring Resistivity of Silicon Wafers with an In-line Four-point Probe

SEMI MF525 — Test Method for Measuring Resistivity of Silicon Wafers Using a Spreading Resistance Probe

SEMI MF533 — Test Method for Thickness and Thickness Variation of Silicon Slices

SEMI MF672 — Test Method for Measuring Resistivity Profile Perpendicular to the Surface of a Silicon Wafer Using a Spreading Resistance Probe

SEMI MF673 — Test Method for Measuring Resistivity of Semiconductor Slices or Sheet Resistance of Semiconductor Films with a Non-contact Eddy-current Gage

SEMI MF723 — Practice for Conversion Between Resistivity and Dopant Density for Boron-doped, Phosphorus-doped, and Arsenic-doped Silicon

SEMI MF1241 — Terminology of Silicon Technology

SEMI MF1392 — Test Method for Determining Net Carrier Density Profiles in Silicon Wafers by Capacitance-voltage Measurements with a Mercury Probe

SEMI MF1393 — Test Method for Determining Net Carrier Density in Silicon Wafers by Miller Feedback Profiler Measurements with a Mercury Probe

SEMI MF1529 — Test Method for Sheet Resistance Uniformity Evaluation by in-line Four-point Probe with the Dual-configuration Procedure

SEMI MF1530 — Test Method for Measuring Flatness, Thickness, and Thickness Variation on Silicon Wafers by Automated Non-contact Scanning

SEMI MF1618 — Practice for Determining Uniformity of Thin Films on Silicon Wafers

SEMI MF2074 — Guide for Measuring Diameter of Silicon and Other Semiconductor Wafers

3.2 *ASTM Standard* [1]

D 5127 — Guide for Ultra Pure Water Used in the Electronics and Semiconductor Industry

3.3 *DIN Standards* [2]

50431 — Measurement of the Electrical Resistivity of Silicon or Germanium Single Crystals by Means of the Four-Point-Probe Direct Current Method with Collinear Four-probe Array

50439 — Determination of the Dopant Concentration Profile of Single Crystalline Semiconductor Material by Means of the Capacitance-voltage Method and Mercury Contact

50445 — Contactless Determination of the Electrical Resistivity of Semiconductor Wafers with the Eddy Current Method

50447 — Contactless Measurement of Electrical Surface Resistivity of Semiconductor Layers by Eddy-current Method

3.4 *ISO Standards* [3]

Guide 30:1981 Terms and Definitions Used in Connection with Reference Materials

ISO 8402 — Quality—Vocabulary

ISO 10012-1 — Quality Assurance Requirements for Measuring Equipment—Part 1: Management of Measuring Equipment

NOTICE: Unless otherwise indicated, all documents cited shall be the latest published versions.

4 Terminology

4.1 *Definition*s *Terms Related to Reference Materials*

4.1.1 *certified reference material (CRM)* — a reference material one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation

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1 *Annual Book of ASTM Standards,* Vol 11.01, ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428. Telephone: 610-832-9500, Fax: 610-832-9555, Website: www.astm.org.

2 Available in both German and English editions from Deutches Institut für Normung e.V., Beuth Verlag GmbH, Burggrafenstrasse 4-10, D 10787 Berlin, Germany, website: www.din.de.

3 ISO Central Secretariat, C. P. 56, CH-1211 Genève 20, Switzerland, Website: www.iso.ch, available in the U.S. from American National Standards Institute, 11 West 42nd Street, 13th Floor, New York, NY 10036, Website: www.ansi.org.

which is issued by a certifying body (ISO Guide 30:1981).

4.1.2 *Discussion* — ISO 8402 states that in a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, or basic physical constants or properties. In the present guide, as in ISO 10012-1, the term "measuring equipment" is extended to include both measuring instruments and measurement standards (including reference wafers).

4.1.3 *reference material (RM)* — a material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, (for) the assessment of a measurement method, or for assigning values to materials (ISO Guide 30:1981; ISO 10012-1).

4.1.4 *resistivity reference wafer* — a CRM or RM in the form of a silicon wafer or chip used for routine calibration or control of resistivity measuring equipment.

4.1.5 *Standard Reference Material (SRM[4]®)* — a certified reference material issued by the U.S. National Institute of Standards and Technology.

4.1.6 *traceability* — the ability to trace the history, application, or location of an item or activity, or similar items or activities, by means of recorded identification (ISO 8402).

4.2 *Definitions of Terms Related to Four-point Probes*

4.2.1 *four-point probe* — an electrical probe arrangement for determining the resistivity of a material in which separate pairs of contacts are used (1) for passing current through the specimen, and (2) measuring the potential drop caused by the current.

4.2.2 *probe head, of a four-point probe* — the mounting that (1) fixes the positions of the four pins of the probe in a specific pattern such as an in-line (collinear) or square array, and (2) contains the pin bearings and springs or other means for applying a load to the probe pins.

4.2.3 *probe pin, of a four-point probe* — one of the four needles supporting the probe tips; mounted in a bearing contained in the probe head and loaded by a spring or dead weight.

4.2.4 *probe tip, of a four-point probe* — the part of the pin that contacts the wafer.

4.2.5 *probe-tip spacing, of a four-point probe* — the distance between adjacent probe tips.

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[4] SRM® is a registered trademark of the U.S. National Institute of Standards and Technology and the U.S. Government.

4.3 For definitions of other terms used in silicon wafer technology refer to SEMI M1 and SEMI MF1241.

5 Reagents

5.1 *Purity of Reagents* — All chemicals for which such specifications exist shall conform to Grade 1 SEMI specifications for those specific chemicals. Other grades may be used, provided it is first determined that the chemical is of sufficiently high purity to permit its use without lessening the accuracy of the test.

5.2 *Purity of Water* — Reference to water shall be understood to mean water meeting the requirements of Type E-3 water or better as described in ASTM Guide D 5127.

5.3 The recommended chemicals shall have the following nominal assays:

5.3.1 *Nitric Acid*, HNO_3, concentrated, 70 to 71%, Grade 1 in accordance with SEMI C35,

5.3.2 *Hydrofluoric Acid*, HF, concentrated, 49.00 ± 0.25%, Grade 1 in accordance with SEMI C28, and

5.3.3 *Potassium Hydroxide*, KOH, pellets, 85% min, Grade 1 in accordance with SEMI C39.

5.4 *KOH Etching Solution* — Dissolve potassium hydroxide (KOH) in water to make a 50 weight % KOH solution in sufficient volume to allow complete immersion of the largest wafers to be prepared for test. This solution is to be used at a temperature of 65 to 75°C.

5.5 *Etching Solution (15 + 1)* — Mix 15 parts of nitric acid (HNO_3) with one part of hydrofluoric acid (HF) in sufficient volume to allow complete immersion of the largest wafers to be prepared for test.

6 Resistivity CRMs

6.1 Resistivity CRMs are available from several sources in a variety of configurations, orientations, conductivity types, and resistivity. Choose the configuration to match the desired application as closely as possible. For example, choose whole wafers for applications involving whole wafer measurements and chip sets with relatively closely spaced resistivity values and appropriate orientation and conductivity type for applications involving spreading resistance.

7 Control of Primary Resistivity Measuring Instruments

7.1 "Primary" resistivity measuring instruments are four-point probes used for calibrating resistivity reference wafers. Such instruments must (1) meet the requirements of SEMI MF84, (2) be maintained in a state of control through the use of $\overline{X}$ and *s* instrument

control charts (see Section 7.1.1), (3) return measured resistivity values of appropriate CRMs to within desired limits through measurement of resistivity CRMs at regular intervals (see Section 7.1.2), and (4) be operated within the temperature range specified in SEMI MF84 (see Section R2-4 of this guide).

7.1.1 *Instrument Control Charts* — $\overline{X}$ and *s* control charts should be maintained to establish the stability of the instrument over the range of resistivity that the instrument is expected to measure. A separate control chart should be maintained for each reference wafer used. Initially, resistivity CRMs or other wafers with adequate radial resistivity uniformity (see Section 8.5.1) should be used for this purpose. As resistivity reference wafers are prepared, these should be used for maintaining the control charts. After in-house resistivity reference wafers become available, it is recommended that three wafers at each resistivity level be set aside for control charting. Two of these should be used for maintaining the $\overline{X}$ and s charts on a regular basis. The third is retained as a reference to ensure that any apparently out-of-control conditions are due to the instrument and not to changes in sample surface conditions, as might occur after prolonged use of a particular sample. Each resistivity determination should consist of six to ten measurements made in accordance with SEMI MF84. Established procedures for generating and maintaining the control charts and for determining the existence of out-of-control conditions and the need for corrective action should be used. A suggested procedure for these determinations is given in Section R1-2 for use in organizations without previously established procedures.

7.1.2 *Comparison with CRMs* — To provide traceability, measurements of the resistivity of available CRMs that encompass the range of resistivity expected to be encountered should be made on a periodic, but less frequent, basis. In addition to being in control, the instruments should return measured values of the resistivity of the CRMs that do not deviate from their certified value by more than the root mean square of the two-sigma (95 % confidence level estimate) uncertainty of the CRM (Note 1) and the two-sigma instrument variability determined by control charting procedures (Note 2). A suggested procedure for control charting of CRMs is given in Section R1-3 for use in organizations without previously established procedures.

NOTE 1: A laboratory issuing a CRM should provide a comprehensive statement of its uncertainty associated with the measurement and reporting of the CRM value. This should include evaluations, or estimates, of both random and systematic "errors" following ISO procedures for Type A and Type B evaluations of components of uncertainty. The result would be expressed as a Combined Standard Uncertainty (standard deviation, or square-root of a sum of variances of uncertainty components), or as an "Expanded Uncertainty" (2 times the Combined Standard Uncertainty). If based on sufficient statistical degrees of freedom, the Combined Standard Uncertainty is a one-sigma estimate, and the Expanded Uncertainty is a two-sigma, or 95% confidence-level, estimate. If there are insufficient statistical degrees of freedom, the effective number of degrees of freedom should be reported by the CRM-issuing laboratory. It is then necessary to multiply the uncertainty value provided by the student-*t* factor appropriate to that number of degrees of freedom in order to obtain values for the one-sigma or two-sigma (95% confidence-level) measurement uncertainty.

NOTE 2: A laboratory following this procedure to establish traceability to resistivity CRMs incorporates a value of its measurement uncertainty based on its control chart measurements, which evaluate random components of uncertainty only. If the laboratory then measures a value for the CRM within the total uncertainty interval, calculated following the root-mean-square procedure, above, about the certified value, its instrument is validated for use. If it measures a value outside the prescribed interval, this is an indication that its measurement bias with respect to true value is statistically significant at the 95% level, and instrument repair, or "calibration" is necessary before proceeding.

7.1.3 *Probe Assembly and Electrical Equipment Tests* — Should an out-of-control or out-of-specification condition be encountered, the probe assembly and electrical equipment can be tested in accordance with the section on Suitability of Test Equipment in SEMI MF84. These tests may serve to isolate the cause of the problem encountered. Either or both of these components should be repaired or replaced if they fail to meet the requirements specified in this section of SEMI MF84.

NOTE 3: The stringent requirements on control of probe-tip spacing as well as the need for off-center diameter correction factors required for determination of radial resistivity uniformity (see Section 8.5) are a consequence of the single-configuration method of using the four-point probe in accordance with SEMI MF84. Errors resulting from uncertainty in probe-tip spacing when using the single-configuration method are considered in Related Information 2. The probe-tip spacing requirements can be relaxed significantly if the dual-configuration method[5] of measuring resistivity with a four-point probe is used. Use of this method is recommended for determination of the radial resistivity uniformity (see Section 8.5). However, the dual-configuration method has not yet been standardized for bulk resistivity measurements because appropriate thickness correction factors have not yet been published. Such corrections are required for thickness to probe spacing ratios greater than 0.36. Nevertheless, it is noted that the most

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5 Perloff, D. S., "Four-point Probe Correction Factors for Use in Measuring Large Diameter Doped Semiconductor Wafers," *J. Electrochem. Soc.* **123**, 1745–1750 (1976).

recently issued CRMs are calibrated and certified by this technique.[6]

8 Selection and Qualification of Materials for Resistivity Reference Wafers

8.1 Factors that must be considered in selecting materials for resistivity reference wafers are nominal resistivity (see Section 8.2), wafer diameter (see Section 8.3), axial and radial uniformity of the resistivity (see Sections 8.4 and 8.5, respectively), wafer thickness (see 8.6), wafer conductivity type and surface orientation (see Section 8.7), and surface finish (see Section 9.1.2). It is generally desirable to qualify a crystal section for the desired parameters and then verify that individual wafers meet the requirements during the calibration procedure. The uniformity requirements, in particular, depend on the specific application of the reference wafers because different resistivity measuring instruments are sensitive to different volumes of material.

8.1.1 *Four-point Probes* — The most straightforward application of resistivity reference wafers is for control of four-point probes used for measurement of resistivity and resistivity variation. If the wafer under test has the same geometry and surface characteristics as the reference wafer and if the same probe geometry is used for the measurement of both the reference wafer and the wafer under test, the transfer is direct and no special precautions need be observed. If the wafer under test has different thickness or diameter from the resistivity reference wafer or if four-point probes with different probe-tip spacings are used to measure the test and reference wafers, attention must be paid to the use of the appropriate correction factors. In particular, if the ratio of thickness to probe-tip spacing becomes too large, (Note 3) while the ratio of diameter to probe-tip spacing becomes too small, second order errors due to the use of two two-dimensional corrections for a three-dimensional geometry may become significant. Also, if different probe-tip spacings are used, differences in sampling volume must be recognized; in this case, resistivity uniformity of both the reference wafer and the wafer under test limits the transfer accuracy. If the wafer under test has a different surface condition from that of the reference wafer, it is necessary to demonstrate the equivalence of the resulting measurement value with that which would have been obtained had the same surface condition been employed for both. These considerations apply to single-configuration four-point probes. Because the standard test method for use of dual-configuration four-point probes is intended for sheet resistance, rather than bulk-resistivity measurement, these concerns with thickness and diameter issues can be ignored. However, if the thickness is too great, an unknown thickness correction factor must be used if bulk resistivity values are to be compared.

8.1.2 *Eddy-current Gages* — The eddy-current detector is sensitive to the integrated sheet resistance across the thickness of the wafer; in this respect it is similar to the four-point probe. However, the eddy-current probe may weight various elements of the volume sampled differently than that of a four-point probe. Therefore, the weighted average resistivity seen in nonuniform wafers by the eddy current gage may not be the same as the center-point average resistivity value determined by the four-point probe. As a result, the accuracy of transfer between these types of instruments depends strongly on the macro-scale uniformity of the resistivity over the central area of the reference wafer. Because the eddy-current gage is a bulk measurement device, the thickness uniformity over the area sampled by the eddy-current probe is also important. The upper limit of the expected reduction of transfer accuracy is given as follows:

$$\text{maximum percent reduction}$$
$$= \left\{ \left[\left(1 + \frac{\Delta\rho}{\rho} \right) \times \left(1 + \frac{\text{TTV}}{w} \right) \right] - 1 \right\} \times 100 \qquad (1)$$

where:

ρ = average center-point resistivity of resistivity reference wafer, corrected to 23°C, in Ω·cm,

$\Delta\rho$ = radial resistivity variation of resistivity reference wafer, corrected to 23°C, in Ω·cm, over the area sampled by the eddy-current probe,

TTV = total thickness variation of resistivity reference wafer, in μm, over the area sampled by the eddy-current probe, and

w = average thickness of resistivity reference wafer, in μm.

8.1.3 *Spreading Resistance Probes* — Because the spreading resistance probe samples a volume that is exceedingly small compared with the volume sampled by the four-point probe, the most critical reference wafer parameter for accurate transfer of resistivity value is the micro-variation of the resistivity both axially and radially. Very complex and time-consuming procedures are required to determine the micro-scale depth and lateral variations of resistivity in a reference wafer (see Sections 8.4.3 and 8.5.2,

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6 See, for example, 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, 1999 edition, 106 pp. Available from the National Technical Information Service, Springfield, VA 22161, as PB 98-113731.

respectively). Further, the procedures for determining micro-scale depth profiles are destructive and cannot be carried out on the reference wafer itself. Consequently, for this application crystals should be grown by procedures that result in the best possible uniformity. These include neutron transmutation doping of very high purity FZ crystals to obtain high resistivity (neutron transmutation doped (NTD)) *n*-type wafers and magnetic Czochralski (MCz) growth of crystals for both *n*- and *p*-type wafers.

8.1.4 *Mercury Probes* — Similar conditions exist for reference wafers intended for calibration and control of mercury probe systems that are primarily used for determining the net carrier density of epitaxial layers from capacitance measurements. Such probes sample a lateral area much smaller than the four-point probe and a depth that is only a very tiny fraction of the wafer thickness. To ensure that the resistivity in the region sampled by the mercury probe can be taken as the resistivity measured by the four-point probe, it is necessary to establish that both the macro- and micro-scale variations in resistivity of the reference wafer are within tolerable limits. Thus, it is desirable to use highly uniform crystal such as NTD FZ or MCz crystal for resistivity reference wafers for this application (see Section 8.1.3). It is further required in this case to establish the net carrier density of the reference wafer from the resistivity measurement (see Section 9.2.4).

8.2 *Resistivity* — The center-point resistivity of the reference wafer should be chosen to meet the requirements of the measuring instrument to be calibrated or controlled. Most of the test methods cited in Section 1.2 specify the resistivity range of the required resistivity reference wafers.

8.2.1 *Four-point Probes* — For control of instrumentation for making four-point probe resistivity measurements in accordance with SEMI MF84, it is recommended that a minimum of three resistivity reference wafers, with resistivity values in the upper, middle, and lower portions of the resistivity range of interest be used (Note 4). It is preferable to use more closely spaced resistivity reference wafers to ensure instrument linearity over the entire measurement range. If only a narrow resistivity range (less than ±25% of the nominal value) needs to be measured, it is sufficient to use two resistivity reference wafers at the extremes of this range. Similar considerations apply for control of instrumentation for making sheet resistance measurements in accordance with SEMI MF1529, except that, in this case, of course, the parameter of interest is the sheet resistance rather than the resistivity.

NOTE 4: For control of instrumentation for making four-point probe measurements over a narrow resistivity range, it

may be appropriate to use only one or two resistivity reference wafers.

8.2.2 *Eddy–Current Gages* — For calibration of eddy current resistivity measuring instruments, SEMI MF673 specifies that for calibration by Method I, five reference wafers that span the full resistivity range of the instrument are required. However, many organizations prefer to use many more reference wafers for the Method I calibration. Two resistivity reference wafers at the extremes of a narrow resistivity range (typically less than about ±25 % of the nominal value) are required for Method II calibration.

8.2.3 *Spreading Resistance Probes* — For calibration of spreading resistance equipment, SEMI MF672 recommends use of at least three resistivity reference wafers per decade over the range of resistivity to be measured. Regular spacing is desirable, but it may not be possible to secure samples with adequate uniformity with arbitrary nominal resistivity values. Selection of samples for micro-scale uniformity should take precedence over regular spacing.

8.2.4 *Mercury Probes* — The resistivity of a resistivity reference wafer intended for use in calibrating mercury probe systems for making net carrier density measurements in accordance with SEMI MF1392 or SEMI MF1393 is specified to be between one-half and two times the resistivity of the specimens to be measured. However, there is some benefit to employ several resistivity reference wafers with a range of resistivity from very high (net carrier density less than about 1×10^{14} cm^{-3}) to the lowest value to be measured.

8.3 *Wafer Diameter* — Resistivity reference wafers do not need to be the same diameter as the wafers to be measured by the system; any diameter that fits the instrumentation and its associated wafer handling is, in principle, acceptable. However, advances in crystal growing design and control together with certain aspect ratio considerations make it likely that superior radial resistivity uniformity in the central region of the wafer can be obtained with larger diameter (≥100 mm) wafers as compared to that of smaller diameter wafers. In addition, the diameter correction factor for wafers 100 mm in diameter and larger measured with a four-point probe with probe-tip spacing of 1.59 mm is within 0.2% of its limiting value of π/ln2 (= 4.5324). Consequently, errors due to small variations in diameter or probe placement at the wafer center are negligibly small (see Section R2-3.1), and the second order errors associated with the combination of the thickness and diameter correction factors can also be neglected. The latter point is particularly significant when four-point probes with small probe-tip spacings are employed on standard thickness wafers because the thickness