Designation: F 1392 – 00

Standard Test Method for Determining Net Carrier Density Profiles in Silicon Wafers by Capacitance-Voltage Measurements With a Mercury Probe [1]

This standard is issued under the fixed designation F 1392; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method [2] 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 Ω·cm in *n*-type wafers and from about 0.24 to about 330 Ω·cm in *p*-type wafers).

1.2 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 **(1)**. [3] 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.

1.3 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.

1.4 The depth of the region that can be profiled depends on the doping level in the test specimen. Based on data reported by Severin **(1)** and Grove **(2)**, Fig. 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.

1.5 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.

NOTE 1—Test Method F 419 is an alternative method for determining net carrier density profiles in silicon wafers from capacitance-voltage measurements. This test method requires the use of one of the following structures: (*1*) a gated or ungated *p-n* junction diode fabricated using either planar or mesa technology or (*2*) an evaporated metal Schottky diode.

1.6 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 Test Method F 84 (Note 2). This test method also includes procedures for calibration of the apparatus for measuring both capacitance and voltage.

NOTE 2—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.

1.7 *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.* Specific hazard statements are given in 7.1, (7.2, 7.10.3 (Note 7), 8.2, 11.5.1, 11.6.3, and 11.6.5.

2. Referenced Documents

2.1 *ASTM Standards:*

D 5127 Guide for Ultra Pure Water Used in the Electronics and Semiconductor Industry [4]

D 4356 Practice for Establishing Consistent Test Method Tolerances [5]

E 691 Practice for Conducting an Interlaboratory Study to

[1] This test method is under the jurisdiction of ASTM Committee F-1 on Electronics and is the direct responsibility of Subcommittee F01.06 on Silicon Materials and Process Control.

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[2] 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 Committee F-1 maintains close liaison. DIN 50439 is available from Beuth Verlag GmbH, Burggrafenstraße 4-10, D-1000, Berlin 30, Germany.

[3] The boldface numbers in parentheses refer to the list of references at the end of this test method.

[4] *Annual Book of ASTM Standards*, Vol 11.01.

[5] *Annual Book of ASTM Standards*, Vol 14.02.

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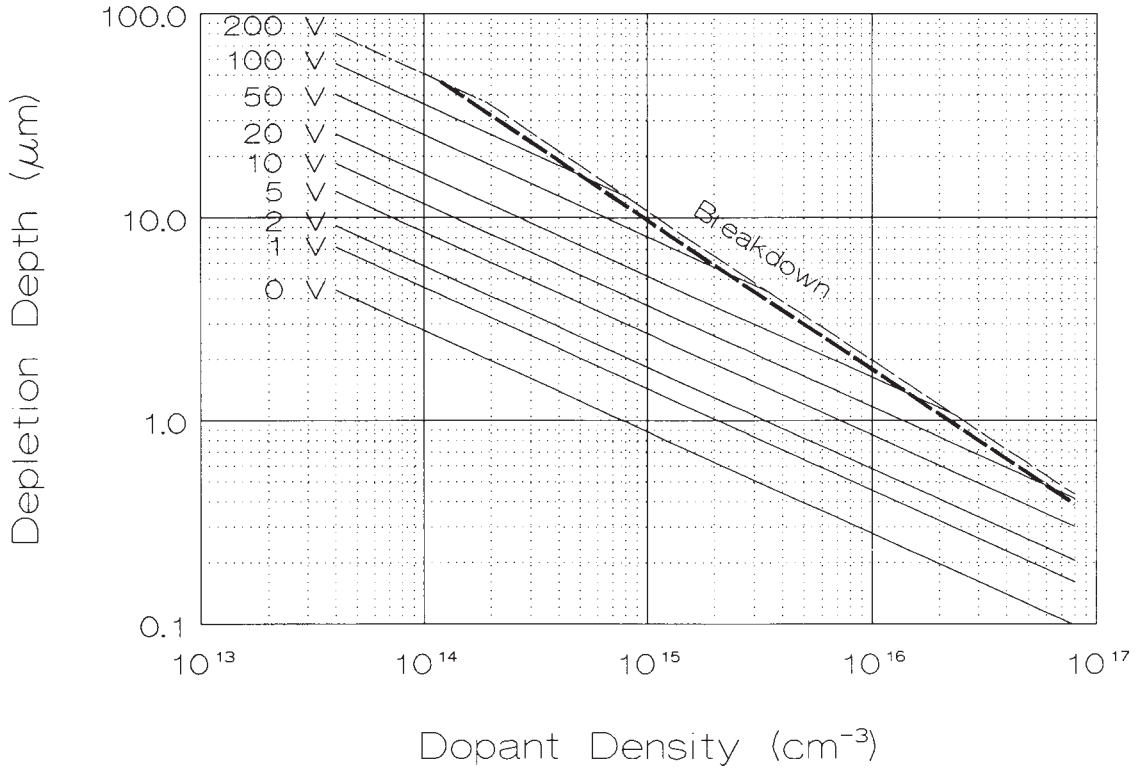
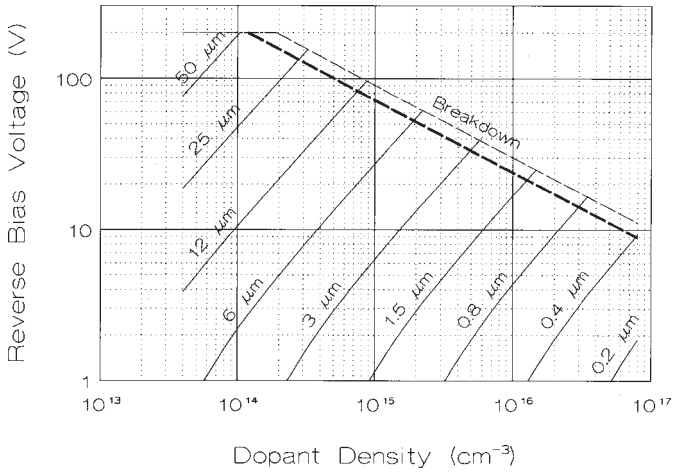


(a) Depletion Depth as a Function of Dopant Density with Applied Reverse Bias Voltage as a Parameter.



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

NOTE 1—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.

FIG. 1 Relationships Between Depletion Depth, Applied Reverse Bias Voltage, and Dopant Density

Determine the Precision of a Test Method [5]

F 26 Test Methods for Determining the Orientation of a Semiconductive Single Crystal [6]

F 42 Test Methods for Conductivity Type of Extrinsic Semiconducting Materials [6]

F 81 Test Method for Measuring Radial Resistivity Variation on Silicon Wafers [6]

F 84 Test Method for Measuring Resistivity of Silicon Wafers with an In-Line Four-Point Probe [6]

F 419 Test Method for Determining Carrier Density in Silicon Epitaxial Layers by Capacitance-Voltage Measurements on Fabricated Junction or Schottky Diodes [6]

[6] *Annual Book of ASTM Standards*, Vol 10.05.

F 672 Test Method for Measuring Resistivity Profiles Perpendicular to the Surface of a Silicon Wafer Using a Spreading Resistance Probe [6]

F 723 Practice for Conversion Between Resistivity and Dopant Density for Boron-Doped, Phosphorus-Doped, and Arsenic-Doped Silicon [6]

F 1153 Test Method for Characterization of Metal-Oxide-Silicon (MOS) Structures by Capacitance-Voltage Measurements [6]

F 1241 Terminology of Silicon Technology [6]

2.2 *SEMI Standards:*

SEMI C28 Specifications for Hydrofluoric Acid [7]

SEMI C29 Specification for Hydrofluoric Acid, 4.9 %[7]

SEMI C30 Specification for Hydrogen Peroxide[7]

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in silicon wafer technology refer to Terminology F 1241.

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

3.2 *Definitions of Terms Specific to This Standard:*

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

3.2.2 *low-resistance contact*—an electrically and mechanically stable contact **(3)** in which the resistance across the contact does not result in excessive series resistance as determined in 11.4 (see also 6.4).

3.2.2.1 *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.

3.2.3 *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.

4. Summary of Test Method

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

4.2 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).

4.3 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.

4.4 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.

4.5 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.

4.6 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 3—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 7.2 of Practice F 723 (conversion from dopant density to resistivity). Note that in applying this conversion procedure in either direction it is assumed that the net carrier density is equal to the dopant density.

5. Significance and Use

5.1 This test method can be used for research and development, process control, and materials specification, evaluation, and acceptance purposes. However, in the absence of interlaboratory test data to establish its reproducibility, this test method should be used for materials specification and acceptance only after the parties to the test have established reproducibility and correlation.

6. Interferences

6.1 A poor Schottky contact, which is generally indicated by an excessively high leakage current (see 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.

6.2 Improper determination of the compensation capacitance, C_{comp}, (see 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.

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

6.4 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 11.4).

6.5 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

[7] Available from Semiconductor Equipment and Materials International, 805 East Middlefield Road, Mountain View, CA 94043.

the mercury that contacts the wafer surface is clean by changing the mercury periodically or by otherwise removing the scum from the exposed surface.

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

6.7 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 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 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 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.

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 ±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 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 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 4—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 5—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 (Note 6) 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 6—The maximum reverse bias depends on the net carrier density in the wafer under test, see Fig. 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 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 10.6).

7.10 *Facilities for Wafer Surface Treatment*, if required (see 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 7—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 Timer*, 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 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 6.5) (**Warning**—see 7.1).

8.3 *Purity of Water*—Reference to water shall be understood to mean deionized water (DI) meeting the resistivity and impurity specifications of Type I Reagent Water in Specifications 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 6.7, 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 Test Method F 672 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 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 Test Method F 81.

8.4.2.2 Establish axial uniformity (see 6.7) 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 Test Method F 84.

8.4.3.2 Convert the resistivity value to net carrier density using the computational methods given in 7.2 (conversion from dopant density to resistivity) of Practice F 723.

NOTE 8—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 Practice F 723. 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. Note that in applying this conversion procedure in either direction it is assumed that the net carrier density is equal to the dopant density.

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

NOTE 9—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 6.7). As an alternative, epitaxial wafers may be used as reference wafers. In this case, the epitaxial layer thickness must be large enough that the resistivity profile in the near-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 are still under development by ASTM Subcommittee F01.95 on Reference Materials, use of epitaxial wafers as reference wafers shall be permitted only if agreed upon between the parties to the test. If used, the epitaxial reference wafer shall meet the requirements of 8.4.1 for a flat spreading resistance profile. Net carrier density shall be determined using an metal oxide silicon (MOS) capacitor structure in accordance with Test Method F 1153 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 chemcials are to be preferred (see Appendix X1).

8.5.1 *Hydrofluoric Acid*, HF, concentrated, 49.00 ± 0.25 %, or dilute, 4.9± 0.1 %.

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 %.

8.5.2.1 Every eight hours, fill the hydrogen peroxide tank with a fresh 15 % H_2O_2 solution by mixing equal volumes of

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

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 two 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 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 7.2). Be certain that the series resistance of the diode circuit formed with the reference wafer meets the requirements of 11.4.1.7 or 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 (10.2.2) or from V_i as a function of C_i^{-2}(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 10—If the incremental method of calculation (see 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 12.4) is used $n \geq 5$.

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

NOTE 11—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 10.2.2.3 and 10.2.2.4 and decrease it after the first slope change as directed in 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 (Note 11). For the recalculation, correct each of the C_i to C'_i as follows:

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

NOTE 12—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 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 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 value 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 = the sum of the squares of the normalized residuals,
V_i = the measured voltage corresponding to the capacitance C_i,
$\hat{V}_i$ = the voltage corresponding to the capacitance C_i, calculated from the least-squares fit, and
n = the 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 10.2.3.4.

10.2.3.6 Repeat 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 10.2.3.8.

10.2.3.10 Repeat 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 10.2.2.6 or 10.2.3.11, determine the net carrier density profile in the central region (see 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 11.4.1.7 or 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 13—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}, Note 8). 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:

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

where:
N_{ij} = the 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 = the 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:

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

where:
A = nominal capillary area, cm^2,
$(N_{ref})_k$ = known net carrier density of reference wafer, cm^{-3}, and
$(N_{avg})_k$ = the calculated average net carrier density, 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 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 (Note 14) and repeat the calibration procedure (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 14—Recommended chemical surface treatment procedures are described in Appendix X2.

10.4 *Control Procedures*—Periodically carry out tests and maintain control charts to ensure proper operation of the electronics (10.4.1), the electronics plus the mechanical functionality of the probe station (10.4.2), and the entire system (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, Section 11, beginning with 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 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 Test Method F 1153, 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 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 (Note 14) 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 (10.4.2) or electronic malfunction (10.4.1), redetermine C_{comp} (see 10.2) and the effective area of the mercury probe contact (see 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 Appendix X2 for suggested data sheet formats for recording the data if the data collection and calculations are carried out manually or off-line.

NOTE 15—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 Test Methods F 42 and Test Methods F 26, respectively.

11.3 Estimate the reverse bias voltage range over which the measurements are to be made based on the curve in Fig. 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 (Note 16).

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 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:

$$R = \frac{0.2}{I_2 - I_1} \tag{6}$$

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 11.5. If the forward resistance exceeds 1 kΩ, improve the return contact, and repeat 11.4.1.

NOTE 16—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 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:

$$R_s = -\frac{10^3}{\tan\theta \times (2\pi fC)} \tag{7}$$

where:

θ = the measured phase angle, degrees,
C = the measured capacitance, pF, and
f = the 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:

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

and

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

where:
G_m = the measured conductance, S,
f = the measurement frequency, MHz, and
C = the 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 11.5. If either of these conditions is not met, improve the return contact, and repeat 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 11.5; when the Schottky contact is satisfactory, repeat 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 7.7). Apply to the mercury column a reverse bias voltage of about 1 V. **Warning:** Avoid physical contact with the probe fixture when bias is applied.

11.5.2 Measure and record this voltage as V_1, and measure the current that exists at this voltage. Calculate the current density at this value of reverse bias voltage, J_{r1}, in mA·cm^{-2}, as follows:

$$J_{r1} = \frac{I_{r1}}{A_{eff}} \quad (10)$$

where:
$I_{r\,1}$ = the current, mA, at the reverse bias voltage V_1, and
A_{eff} = the mercury probe contact area, cm^2, see 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 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:

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

where:
$\Delta J_r / \Delta V$ = rate of increase of the reverse current density with voltage,
J_{ri} = the current density at voltage, V_i, and
$J_{r(i+1)}$ = the 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 (Note 17). 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 17—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 (Note 14), and repeat the procedure beginning with 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 18—To ensure the greatest possible accuracy of the measurement, monitor the attributes of the Schottky contact during the collection of data (11.6) and discontinue data collection if any of the conditions in 11.5.6 are violated.

11.6 *Collection of Data*—Measure a series of capacitance-voltage pairs from which the net carrier density profile will 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 manufacturer'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 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 11.5.1).

11.6.6 Repeat 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 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 (Note 10).

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:

$$C'_i = C_i - C_{comp} \quad (12)$$

Record these values of C'_i.

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

12.3 *Incremental Method* **(6):**

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:

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

where:

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

$$W'_i = 10359\left[\frac{A_{eff}}{C'_i}\right] \quad (14)$$

where:

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

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:

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

where:

W'_i = ith calculated depth, µm,
W'_{i+k} = $(i + k)$th calculated depth, µm,
S_i = ith dimensionless value calculated in 12.3.1.1,
k = a whole number such that C_{i+k} is between 80 and 85 % of C_i, and
n = the 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:

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

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

12.3.3 Proceed to 12.5.

12.4 *Curve-Fitting Method*:

12.4.1 Fit a polynomial of the following form **(7)** to the capacitance-voltage pairs, $C_i - V_i$ (Note 19):

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

where:

k' = the order of the polynomial, chosen so that it represents the lowest-order fit for which $(C'_i - C_{fi})/C'_i| \leq 0.01$ (Note 20) for all values of i and for which $k' \leq 10$ or $n - 1$, whichever is smaller.
n = the number of capacitance-voltage pairs determined in the test,
C_{fi} = the ith value of capacitance, pF, calculated from the fit,
$a_0, a_1, \ldots a_{k'}$ = coefficients determined such that the quantity $(C'_i - C_{fi})^2$ summed over all values of i is minimized,
C'_i = the ith corrected capacitance, pF, and
V_i = the ith recorded (positive) voltage, V.

NOTE 19—Appendix X2 of Test Method F 419 lists a FORTRAN subroutine that will fit data up to a 10th-order polynomial. Erroneous fluctuations in the calculated net carrier density may occur if polynomials of order greater than three are used.

NOTE 20—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, ... $a_{k'}$.

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:

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

where the other symbols are defined following Eq 15.

12.4.4 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:

$$W_i = 10359\left[\frac{A_{eff}}{C_{fi}}\right] \quad (18\,a)$$

and

$$N_i = -\frac{1.2050 \times 10^7}{A^2_{eff} D_i}$$

where:

A_{eff} = the mercury probe contact area, cm ^{2}, as determined in 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 (*1*) 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:

$$N_{avg} = \frac{1}{m} \sum_{i=1}^{m} N_i \quad (19)$$

where:

m = the number of individual values of net carrier density to be averaged (see 12.5.1),
k = a whole number such that C_{i+k} is between 80 and 85 % of C_i,
n = the 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:

$$s = \frac{1}{\sqrt{m-1}} \sqrt{\sum_{i=1}^{m} (N_i - N_{avg})^2} \quad (20)$$

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 21—Appendix X3 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 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 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 10.3.4,

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

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

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

13.2.5 Maximum leakage current density, J_r, mA/cm^2, as determined in 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 Ω·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 Ω·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, see Ref **(6)**.

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

APPENDIXES

(Nonmandatory Information)

X1. RECOMMENDED WAFER SURFACE TREATMENTS

X1.1 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.

X1.2 Place the wafer to be treated in a center slot of a hydrofluoric acid cassette.

X1.3 Dip in HF for 30 s (concentrated HF) or 5 min (dilute HF).

X1.4 Rinse in water for 10 min.

X1.5 For *p*-type wafers, spin dry in an air atmosphere. If subsequent measurements are unstable, heat treat the wafer for 30 min at 120 ± 10°C in air.

X1.6 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 7), and continue with the following:

X1.6.1 Immerse in 15 % H_2O_2 for 10 min at 70° to 90°C.

X1.6.2 Rinse in water for 2 min.

X1.6.3 Spin dry in an air atmosphere (Note X2.1).

NOTE X1.1—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.

X2. SAMPLE DATA SHEETS

X2.1 The following are examples of data sheet formats for manual collection and manual or off-line analysis of C-V data. Fig. X2.1 is for data to be analyzed by the Incremental Method and Fig. X2.2 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.

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 1—Items marked with an asterisk (*) are required only for referee measurements.

FIG. X2.1 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/cm^2

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

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 1—Items marked with an asterisk (*) are required only for referee measurements.

FIG. X2.2 Example of Format for Capacitance-Voltage Data and Calculations Using the Curve-Fitting Method

X3. NUMERICAL CONSTANTS

X3.1 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 X3.1 gives the lumped constants, their formulas, and the values in this test method and in Test Method F 419. 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 X3.2 gives the values of the individual constants used in this test method. The difference in values between this test method and Test Method F 419 is in the value of K_{Si}; this was assigned the value 11.75 in Test Method F 419, but the generally accepted value is 11.7. 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 X3.2 Values of Individual Constants

Constant	Symbol	Value [A]
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]

[A] Note that the units are given in brackets; the symbol C indicates coulombs and the symbol F indicates farads.

TABLE X3.1 Lumped Constants

Equation	Formula	Value in Test Method F 419 [A]	Value in This Test Method [A]
(10), (14), (15)	ϕ	0.6 [V]	0.6 [V]
(11), (16a)	$K_{Si}\epsilon_0$	10404 [pF·µm/cm^2]	10359 [pF·µm/cm^2]
(13)	$K_{Si}\epsilon_0/q$	6.493×10^{14}[(F·µm^2)/(C·cm^3)]	6.466×10^{14}[(F·µm^2)/(C·cm^3)]
(16b)	$2/K_{Si}\epsilon_0 q$	1.19985×10^{7}[(cm·F)/(C·pF^2)]	1.2050×10^{7}[(cm·F)/(C·pF^2)]

[A] Note that the units are given in brackets; the symbol C indicates coulombs and the symbol F indicates farads.

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