SEMI MF1392-0307 (Reapproved 0718)
TEST METHOD FOR DETERMINING NET CARRIER DENSITY PROFILES IN SILICON WAFERS BY CAPACITANCE-VOLTAGE MEASUREMENTS WITH A MERCURY PROBE

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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 ¶ 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 Ω·cm in *n*-type wafers and from about 0.24 to about 330 Ω·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.

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[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 was the responsibility of DIN Committee NMP 221, with which SEMI maintained close liaison. DIN 50439 is no longer available in either German or English from Beuth Verlag GmbH, Burggrafenstraße 4-10, D-10772, Berlin, Germany. An unofficial English translation appears in the 1993 edition of volume 10.05 of the *Annual Book of ASTM Standards*.

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

[3] Grove, A. S., *Physics and Technology of Semiconductor Devices*, John Wiley and Sons, New York, 1967, §§ 6.2 and 6.7c.

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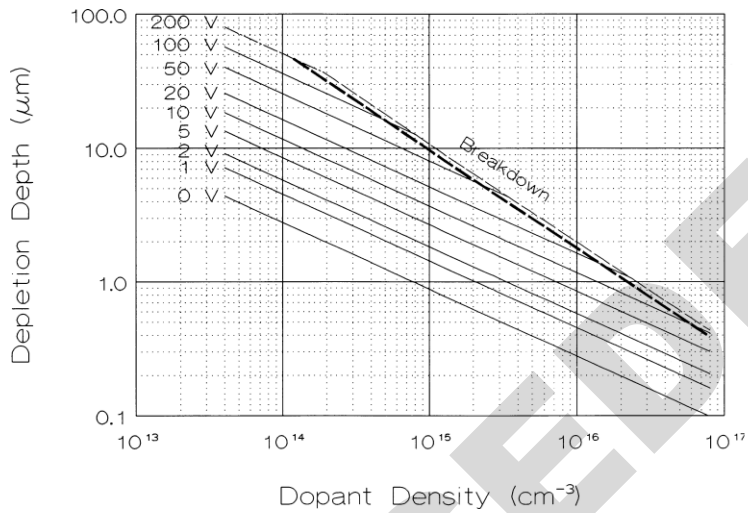
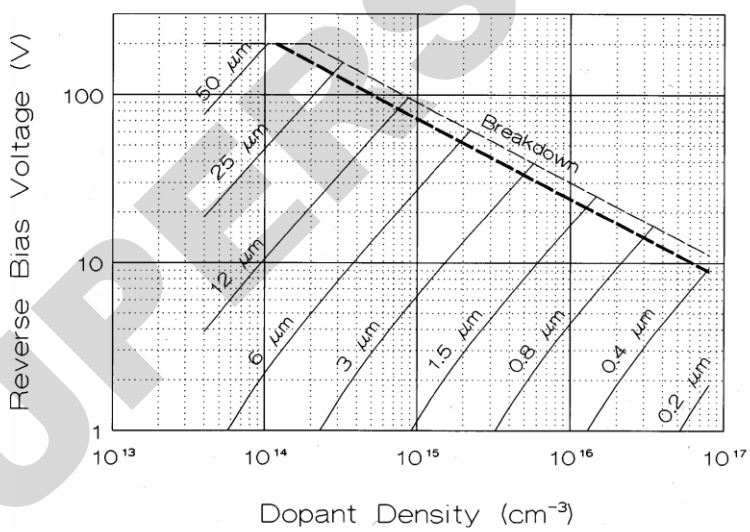
2.6 This Test Method provides for determining the effective area of the mercury probe contact using polished bulk reference wafers that have been measured for resistivity at 23°C in accordance with SEMI MF84 (see Note 1). This Test Method also includes procedures for calibration of the apparatus for 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.



(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.

#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.

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: SEMI Standards and Safety Guidelines do not purport to address all safety issues associated with their use. It is the responsibility of the users of the Documents to establish appropriate safety and health practices, and determine the applicability of regulatory or other limitations prior to use.

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3 Limitations

3.1 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.

3.2 Improper determination of the compensation capacitance, C_{comp}, (see ¶ 10.2) 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 § 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 ¶ 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 § 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.

4 Referenced Standards and Documents

4.1 *SEMI Standards and Safety Guidelines*

SEMI C28 — Specification and Guide for Hydrofluoric Acid

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

SEMI C30 — Specification for Hydrogen Peroxide

SEMI E89 — Guide for Measurement System Analysis (MSA)

SEMI M59 — Terminology for Silicon Technology

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

SEMI MF42 — Test Method 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

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

[5] Schroder, D. K. "Semiconductor Material and Device Characterization." Wiley-Interscience, New York, 1990, § 2.5.2.

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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 or Carrier Density for Boron-Doped, Phosphorus-Doped, and Arsenic-Doped Silicon

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

4.2 *ASTM Standards*[6]

ASTM D5127 — Standard Guide for Ultra Pure Water Used in the Electronics and Semiconductor Industries

ASTM D4356 — Standard Practice for Establishing Consistent Test Method Tolerances (Withdrawn 2007)

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

5 Terminology

5.1 For definitions of general terms used in silicon wafer technology refer to SEMI M59.

5.2 Definitions of the statistical terms 'repeatability' and 'reproducibility' are given in SEMI E89.

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.[7]

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 § 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 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.

[6] ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, USA; Telephone: +1.610.832.9585, Fax: +1.610.832.9555, http://www.astm.org

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

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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% of full scale or better for each range, and the rated reproducibility shall be 0.25% of full scale or better. The internal ac 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 dc 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 § 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°C 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.

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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 hour.

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°C ± 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 ¶ 3.5) (**WARNING:** see ¶ 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 D5127.

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 ¶ 3.7 and § 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 ten 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 uniformity from resistivity measurements taken at 2 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 ¶ 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 § 7.2 of SEMI MF723.

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

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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 (NIST). However, material inhomogeneity may make it difficult to obtain accurate values of net carrier density from bulk reference wafers (see ¶ 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-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 § 8.4.1 for a flat spreading resistance profile. Net carrier density should be determined using a 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 ± 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 hours, 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 hours (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 (see § 11), using either the front- or back-surface-return-contact configuration

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