



Designation: F 1393 – 92 (Reapproved 1997)

AMERICAN SOCIETY FOR TESTING AND MATERIALS  
100 Barr Harbor Dr., West Conshohocken, PA 19428  
Reprinted from the Annual Book of ASTM Standards. Copyright ASTM

# Standard Test Method for Determining Net Carrier Density in Silicon Wafers by Miller Feedback Profiler Measurements With a Mercury Probe<sup>1</sup>

This standard is issued under the fixed designation F 1393; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This test method<sup>2</sup> covers the measurement of net carrier density and net carrier density profiles in epitaxial and polished bulk silicon wafers in the range from about  $4 \times 10^{13}$  to about  $8 \times 10^{16}$  carriers/cm (resistivity range from about 0.1 to about 100  $\Omega$ -cm in *n*-type wafers and from about 0.24 to about 330  $\Omega$ -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)<sup>3</sup> 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 relationship 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.

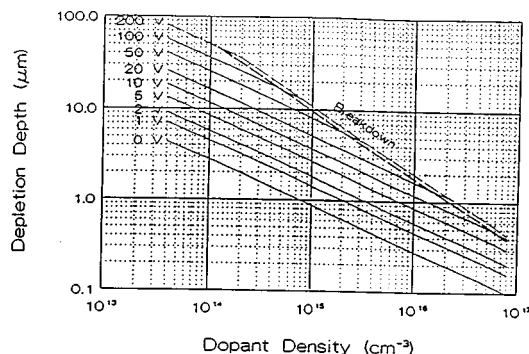
1.5 This test method is intended for rapid carrier density

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee F-1 on Electronics and its the direct responsibility of Subcommittee F01.06 on Silicon Materials and Process Control.

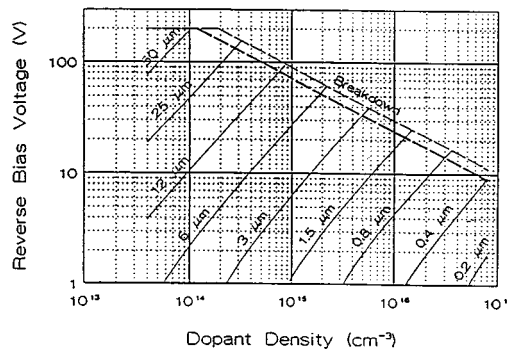
Current edition approved May 15, 1992. Published July 1992.

<sup>2</sup> 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 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.

<sup>3</sup> The boldface numbers in parenthesis refer to the list of references at the end of this test method.



(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 Depth 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

determination when extended sample preparation time or high temperature processing of the wafer is not practical.

NOTE 1—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. Although this test method was written prior to consideration of the Miller Feedback Method, the Miller Feedback Method has been satisfactorily used in measuring the round robin samples.

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 or Test Method F 673. This test method also includes procedures for calibration of the apparatus.

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 Note 4 in 7.2, 7.3, and 8.2.

## 2. Referenced Documents

### 2.1 ASTM Standards:

- D 1193 Specifications for Reagent Water<sup>4</sup>
  - F 26 Test Methods for Determining the Orientation of a Semiconductive Single Crystal<sup>5</sup>
  - F 42 Test Methods for Conductivity Type of Extrinsic Semiconducting Materials<sup>5</sup>
  - F 81 Test Method for Measuring Radial Resistivity Variation on Silicon Slices<sup>5</sup>
  - F 84 Test Method for Measuring Resistivity of Silicon Wafers with an In-Line Four-Point Probe<sup>5</sup>
  - F 419 Test Method for Determining Carrier Density in Silicon Epitaxial Layers by Capacitance Voltage of Measurements on Fabricated Junction or Schottky Diodes<sup>5</sup>
  - F 673 Test Method for Measuring Resistivity of Semiconductor Slices or Sheet Resistance of Semiconductor Films with a Noncontact Eddy-Current Gage<sup>5</sup>
  - F 723 Practice for Conversion Between Resistivity and Dopant Density for Boron-Doped and Phosphorus-Doped Silicon<sup>5</sup>
  - F 1241 Terminology of Silicon Technology<sup>5</sup>
- ### 2.2 SEMI Standard:
- SEMI C1 Specifications for Reagents<sup>6</sup>

## 3. Terminology

### 3.1 Definitions:

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

### 3.2 Definitions of Terms Specific to This Standard:

3.2.1 *breakdown voltage*—the reverse bias voltage at which the mercury probe contact exhibits a leakage current density of 3 mA/cm<sup>2</sup>.

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

3.2.3 *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 6.2).

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

3.2.4 *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 A calibration procedure using polished bulk wafers of known carrier density is used to determine the mercury probe contact area.

4.2 The test specimen is placed on the mercury probe fixture. A column of mercury is brought into contact with an epitaxial or polished wafer surface 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 (32 times or larger) than the mercury probe contact.

4.4 The quality of the Schottky barrier diode formed is determined by viewing the “delta X wave shape” on an oscilloscope and verifying that it is a good square wave per manufacturer’s operating instruction. It can also be evaluated by measuring its series resistance and its reverse current characteristics.

4.5 A current is driven through the diode by a radio frequency (RF) generator. The current is compared to a reference current (magnitude of which is set by the dielectric constant and area controls) at the error summation point at the input of an amplifier in a servo-controlled feedback loop that (a) keeps the RF current amplitude constant and (b) generates an output d-c signal,  $X$ , that is proportional to the depletion depth. The reverse bias ( $V$ ) on the diode is step-modulated at a low frequency and at an amplitude proportional to signal  $X$ , keeping  $dV/dX$ , the change in electric field, constant. The amplitude of the resulting modulation of the  $X$  signal ( $dX$ ) is therefore proportional to the net carrier density. A d-c signal,  $1/N$ , (net carrier density) proportional to  $dX$  is generated. The signal is used for read out information.

4.6 The net carrier density as a function of depth is determined by the profiler circuitry and computer data acquisition hardware and software.

NOTE 3—The net carrier density values obtained by this test method are frequently 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 Practice F 723, using the tabular or computational methods given in paragraph 7.2 of this practice (conversion from dopant density to resistivity) in order to eliminate the self-consistency errors in the equations given in Practice F 723. The choice of conversion direction is based on the fact that the net carrier density of the reference wafer used for determination of the area of the mercury probe contact (see 8.4 and 10.2) is traceable to National Institute of Standards and Technology using the methods of paragraph 7.2 of Practice F 723 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

<sup>4</sup> Annual Book of ASTM Standards, Vol 11.01.

<sup>5</sup> Annual Book of ASTM Standards, Vol 10.05.

<sup>6</sup> Available from Semiconductor Equipment and Materials International, 805 East Middlefield Road, Mountain View, CA 94043.

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 precision, this test method should be used for materials specifications and acceptance only after the parties to the test have established repeatability, reproducibility, and correlation.

## 6. Interferences

6.1 A poor Schottky contact, which is generally indicated by an excessively high leakage current (greater than 100  $\mu\text{A}$ ) (see 11.5) is the most common problem in measurements made with mercury probe instruments. It must be emphasized that the use of a poor Schottky contact will not actually prevent a carrier density determination but will produce an erroneous result.

6.2 Excessive series resistance in the measurement circuit can cause significant errors in the measured values. Series resistance values greater than 1  $\text{k}\Omega$  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 and excessive spacing between mercury Schottky and mercury return contact or using a backside return contact when using higher resistivity substrates (see 11.4).

6.3 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 (see **Warning** in 8.2 and Note 4).

6.4 A dirty capillary tube containing the mercury column may also result in unstable measurements. If erratic results are observed, inspect the capillary carefully. If it is dirty, clean it thoroughly. If it appears to be damaged, repair or replace the capillary and refill with clean mercury.

## 7. Apparatus

7.1 *Facilities for Wafer Surface Treatments*—A fume hood equipped with an acid-proof sink and suitable beakers to hold wet chemicals (such as nitric acid at 70 to 80°C, hydrogen peroxide at 90°C, hydrofluoric acid at room temperature, and boiling water), water quench or cascade rinse system, and a spin dryer or other equivalent wafer drying system is required. Under some circumstances a means for baking the wafer drying system is required. Under some circumstances a means for baking the wafer at 200° in air or nitrogen may also be required.

7.2 *Mercury Probe Fixture*—One of the following fixtures depending on the type of test specimen to be measured such as:

NOTE 4—**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.

7.2.1 *Back-Side-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 (one standard deviation). The fixture must also provide a low-resistance return contact to the back surface of the wafer.

7.2.2 *Front-Surface-Return-Contact Fixture*, for use in measuring epitaxial wafers deposited on substrates of the opposite conductivity type or on substrates with high resistivity or 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 7.2.1, 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 7.2.1).

7.3 *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 Note 4).

7.4 *Miller Feedback Profiler Electronics* (6), having a frequency of 1 MHz nominal and an input capacitance range from 5 pF to 700 pF (see Appendix X3). Provision should be made for calibration of stray capacitance of up to 10 pF.

7.5 *D-C Power Supplies*, 0 to 1 vdc, 0 to -200 V.

7.6 *Digital Panel Meter*, 0 to 200 V, accuracy  $\pm 0.05\%$  reading + 0.05 % f.s.

7.7 *Digital Microammeter*, 0 to 200  $\mu\text{A}$ , accuracy  $\pm 0.05\%$  reading + 0.05 % f.s.

7.8 *Oscilloscope*, used to monitor Delta X and RF. Dual trace at least 20 MHz capability.

7.9 *Shielded Cables*, shielded coaxial cables, maximum length 36 in (0.9 m).

7.10 *Precision Capacitors*, nominal 100 pF.

7.11 *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  $\mu\text{A}$ /division or better.

## 8. Reagents and Materials

8.1 *Purity of Reagents*—All chemicals for which such specifications exist shall conform to SEMI Specifications C1. Other grades may be used, provided it is first determined that the reagent<sup>7</sup> is of sufficiently high purity to permit its use without lessening the accuracy of the test.

<sup>7</sup> Specifications of the Committee on Analytical Reagents of the American Chemical Society, Washington, DC.



8.2 Mercury shall be triple distilled and conform to reagent grade as specified in reagent chemicals. It shall be changed regularly or otherwise maintained in a clean state to avoid interference from surface scum (see 6.3) (**Warning:** see Note 4).

8.3 *Purity of Water*—Reference to water shall be understood to mean deionized water meeting the resistivity and impurity specifications of Type I Reagent Water in Specifications D 1193.

8.4 *Reference Wafers*—Polished bulk silicon wafers of the same conductivity type as the layer or wafer to be tested. Reference wafers shall have the following characteristics:

8.4.1 The net carrier density determined as follows shall lie between one-half and two times the net carrier density of the layer or wafer to be tested:

8.4.1.1 Measure the resistivity using Test Methods F 81 or F 673 at the center of the wafer and at  $\pm 3$  mm and at  $\pm 6$  mm from center on two diameters. Then calculate the average of all measurements and convert to 23°C.

8.4.1.2 Convert resistivity values to net carrier density using the tabular or computational methods given in 7.2 (conversion from dopant density to resistivity) of Practice F 723 (Note 3).

8.4.1.3 Record the net carrier density, in  $\text{cm}^{-3}$ , as  $N_{\text{ref}}$ .

8.4.2 The resistivity variation of the central region of the wafer shall be  $\leq 5\%$  as determined from 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 and analyzed in accordance with the maximum-minimum convention of Sample Plan D of Test Method F 81.

8.5 *Reagents for Surface Treatment*—If surface treatment is required, the following chemicals may be needed (see Fig. X1.1 in Appendix X1).

8.5.1 *Nitric Acid*,  $\text{HNO}_3$ , concentrated, 70 to 71 %.

8.5.2 *Hydrofluoric Acid*, HF, concentrated, 49.00  $\pm$  0.25 %.

8.5.3 *Hydrogen Peroxide*,  $\text{H}_2\text{O}_2$ , unstabilized, 30 %.

## 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. Calibration

10.1 *Miller Feedback Profiler Calibration Check*—Profile the calibration diode, which is a packaged diode provided by the manufacturer, using the manufacturer's operating instructions to ensure that the profiler is in calibration.

10.2 Choose the proper orifice diameter to ensure that the capacitance at the profiler input terminals will be between 5 and 700 pF. Refer to Fig. X3.1 in Appendix X3.

10.3 Determine the area of the orifice used to define the mercury contact area initially by measuring the diameter of the orifice used to form the mercury contact area with a toolmakers microscope. Calculate the initial orifice area, in  $\text{cm}^2$ , as follows:

$$A = \frac{\pi}{4} d^2$$

where:

$A$  = initial orifice area, and

$d$  = orifice diameter, cm.

### 10.4 Orifice Area Carrier Density Calibration:

10.4.1 Place the reference wafer on the mercury probe fixture so the polished or epitaxial surface is in contact with the mercury column.

10.4.2 Measure the carrier density, in  $\text{cm}^3$ , of the center point of the reference wafer in accordance with Section 11. Adjust the stray capacitance to obtain a constant (flat) carrier density profile; then adjust the area input to get a value that is within  $\pm 3\%$  of the reference wafer's known value. Repeat procedure as necessary to verify the 1 % repeatability.

## 11. Procedure

11.1 Refer to Fig. X1.1 in Appendix X1 for suggested data sheet forms for recording the data if the data collection and calculations are carried out manually or off-line.

NOTE 5—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 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 curves 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 *Measurement of the Diode Forward Resistance and Circuit Series Resistance:*

11.4.1 Place the test wafer onto the mercury probe fixture such that the mercury contacts the polished or epitaxial surface of the test wafer. If using single column probe configuration, make the return contact to the substrate or back surface of the wafer.

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 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  $\text{k}\Omega$ , as follows:

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

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 k $\Omega$  or less, proceed to 11.5. If the forward resistance exceeds 1 k $\Omega$ , improve the return contact, and repeat 11.4.1.

NOTE 6—The diode forward resistance,  $R$ , at 1 V 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 Determine that a rectifying contact is formed for the mercury silicon contact by viewing the delta X wave shape on an oscilloscope and verify that it is a good square wave per manufacturer's operating instruction.

11.5 *Qualification of Schottky Contact by Determination of Reverse-Current Characteristics:*

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 for the mercury probe contact (see 7.7). Apply to the mercury column a reverse bias voltage of about 1 V.

NOTE 7—**Precaution:** Avoid physical contact with the probe fixture when bias voltage 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<sup>2</sup>, as follows:

$$J_{r1} = \frac{I_{r1}}{A_{\text{eff}}}$$

where:

$I_{r1}$  = the current, mA, at the reverse bias voltage  $V_1$ , and

$A_{\text{eff}}$  = the mercury probe contact area, cm<sup>2</sup>, as determined in 10.4.

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.

11.5.4 If the current density,  $J_r$ , equals or exceeds 3 mA/cm<sup>2</sup> at any voltage up to the maximum value applied, first determine if this is due to carrier density variations in the structure (Note 8). In this case, reduce the maximum applied reverse bias voltage to be used in the test to the highest value for which the current density is less than 3 mA/cm<sup>2</sup>. Otherwise, treat the wafer surface with an acceptable chemical process (Note 9), and repeat the procedure beginning with 11.4.

NOTE 8—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.

NOTE 9—Selected chemical surface treatments that have been reported to condition the surface suitably are described in Appendix X2.

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

11.6 Apply a d-c voltage of at least 1.0 V of reverse bias voltage to the sample and increase continuously up to the expected maximum applied reverse voltage. The carrier density profile, as function of depletion depth, is displayed on the system. Monitor and record the leakage current.

11.7 The measurement system exhibits the automatically recorded carrier density on a logarithmic scale versus the depletion layer depth.

## 12. Report

12.1 Report the following information:

12.1.1 Operator identification,

12.1.2 Date of measurement,

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

12.1.4 Probe configuration used,

12.1.5 Lot number and test specimen identification including conductivity type and surface orientation, concentration range, volts per micron and test temperature,

12.1.6 Wafer and sampling plan, if applicable, and

12.1.7 Net carrier density ( $N$ ) versus depth ( $X$ ) (plot of  $N$  as a function of  $X$ ), as determined in 11.4.

12.2 For referee measurements, also report the following:

12.2.1 Lot sampling plan,

12.2.2 Measurement locations,

12.2.3 Stray capacitance in pF, as determined in 10.4.2,

12.2.4 Series resistance, as determined in 11.4,

12.2.5 Bias voltage ramping rate (slow, medium, fast, and user defined),

12.2.6 Bias voltage start and stopping points,

12.2.7 Maximum applied reverse bias voltage,  $V$ , as determined in 11.5,

12.2.7.1 Parabolic ramp generator (on/off),

12.2.7.2 Parabolic ramp time (seconds),

12.2.8 Depth processing mode (on/off),

12.2.8.1 Desired depth in micrometres,

12.2.9 Maximum leakage current density,  $J_r$ , mA/cm<sup>2</sup>, as determined in 11.5,

12.2.10 Mercury probe contact area,  $A_{\text{eff}}$  cm<sup>2</sup>, as determined in 10.2,

12.2.11 Surface treatment used, if applicable, and

12.2.12 Other data as tabulated in a data sheet appropriate to the Miller Feedback Method.

## 13. Precision and Bias

13.1 The mercury probe single laboratory repeatability tests were done using two silicon  $n$ -type epi wafers with approximate carrier concentrations of  $10^{13}$  and  $10^{15}$  cm<sup>-3</sup>. The lower doped sample showed single standard deviations that ranged from 1.1 to 4.0 % and the higher doped wafers single standard deviations ranged from 0.0 to 2.1 %. The tests were done over a ten day period. On each day ten readings were taken on each wafer; this was done by breaking contact and repositioning the wafer for each of the ten measurements. The wafer was then profiled to a specified depth. The depth was selected as a point

**F 1393**

at the center of the profile between zero bias and breakdown. The depth for the lower doped wafer was 12  $\mu\text{m}$  and for the higher doped sample was 3.0  $\mu\text{m}$ . At these depths the carrier concentration was recorded and used to calculate the standard deviation of values for each day.

13.2 The tests were done without chemically treating the wafers in any way.

**14. Keywords**

14.1 depth profile; epitaxial wafers; mercury probe; miller feedback method; net carrier density; silicon

**APPENDIXES**

**(Nonmandatory Information)**

**X1. SAMPLE DATA SHEETS**

X1.1 Fig. X1.1 is an example of a data sheet format for manual collection and manual or off-line analysis of Miller

Feedback Profiler data. If a computer is used, the data listed must be stored in a format such that it can be retrieved.

Type of instrument _____	Measurement locations _____
Model Number _____	Stray capacitance _____ pF
Software type/revision _____	No. of Test Points _____
Probe configuration _____	Series resistance, R _____ $\Omega$
Operator _____	Ramp Time Selection _____
Date _____	Ramp Time (sec/volt): ramp voltage _____
Lot number _____	start stop _____
Test specimen identification _____	Parabolic ramp generator (on/off) _____
Conductivity type _____	Parabolic Ramp Time (sec) _____
Concentration Range Calib volts vits/mic _____	Max applied reverse bias voltage _____
Surface orientation _____	Depth processing on/off _____
Test temperature _____	Desired Depth (microns) _____
Lot sampling plan _____	Max leakage current density, $J_r$ _____ mA/cm <sup>2</sup>
	Mercury probe contact area, $A_{\text{eff}}$ _____ cm <sup>2</sup>
	Surface treatment, if used _____

Data Point	X (microns)	N (cm <sup>-3</sup> )	Bias (volts)	Leakage (mic-amp)	Resistivity
0	-----	-----	-----	-----	-----
1	-----	-----	-----	-----	-----
2	-----	-----	-----	-----	-----
3	-----	-----	-----	-----	-----
4	-----	-----	-----	-----	-----
5	-----	-----	-----	-----	-----
6	-----	-----	-----	-----	-----
7	-----	-----	-----	-----	-----
8	-----	-----	-----	-----	-----
Etc.	-----	-----	-----	-----	-----

**FIG. X1.1 Example of Format for Miller Feedback Profiler Measurement Data**

**X2. WAFER SURFACE TREATMENTS**

X2.1 For *p*-type wafers, two acceptable treatments are (a) 10 min in hot nitric acid at 70 to 80°C followed by a DI water quench, a DI water cascade rinse for 10 min, and spin dry (7), or (b) a dip in hydrofluoric acid for 10 s or until the wafer surface is hydrophobic, followed by a DI water quench, a DI water cascade rinse for 10 min, and spin dry. Under some circumstances, especially for wafers with resistivity of 1  $\Omega\text{-cm}$  or higher, it may be necessary to bake the wafer at 200°C for

10 min in air or nitrogen in order to stabilize the surface.

X2.2 For *n*-type wafers, two acceptable treatments are (a) 10 min in boiling DI water, followed by a DI water quench for 10 min, a DI water cascade rinse for 10 min, and spin dry, or (b) 10 min in hydrogen peroxide heated to 90°C followed by a DI water quench for 10 min, a DI water cascade rinse for 3 min, and spin dry (8).

### X3. PRACTICAL LIMITS

X3.1 The Feedback Profiler has been designed to produce accurate profiles for all diode capacitances in the range  $5\text{pF} < C < 700\text{pF}$ . (These limits are conservative, but should be observed when possible.) During profiling, the diode capacitance will vary from some maximum value at zero bias, to a minimum value at breakdown. It is therefore important to ensure that this range of values lies within the 5 to 700 pF limits. This, in turn, means that some care must be taken in the selection of diode area for a given doping range. Fig. X3.1 has been prepared as an aid in this selection process. The lines labeled "zero bias" and "breakdown" give the capacitance of a 10-mil diameter circular diode at these two bias extremes as

functions of  $N$ . Similar lines can be drawn for diodes of other diameters by using the diode diameter scales and drawing lines parallel to the given 10-mil lines. As an example of the use of Fig. X3.1, suppose we wish to profile material having  $N \approx 10^{16}\text{cm}^3$ . Fig. X3.1 tells us that, if we use 10-mil (250- $\mu\text{m}$ ) diodes, the capacitance will be 15 pF at zero bias and 2 pF at breakdown. Since 2 pF is below the low limit, the diode diameter must be increased to provide a capacitance  $\geq 5\text{pF}$  (approximately 25 mil) in order to profile out to breakdown. Using 20-mil diodes would give capacitance values four times larger, or 8 to 60 pF. Thus, 20-mil (500- $\mu\text{m}$ ) diodes would be a good choice.

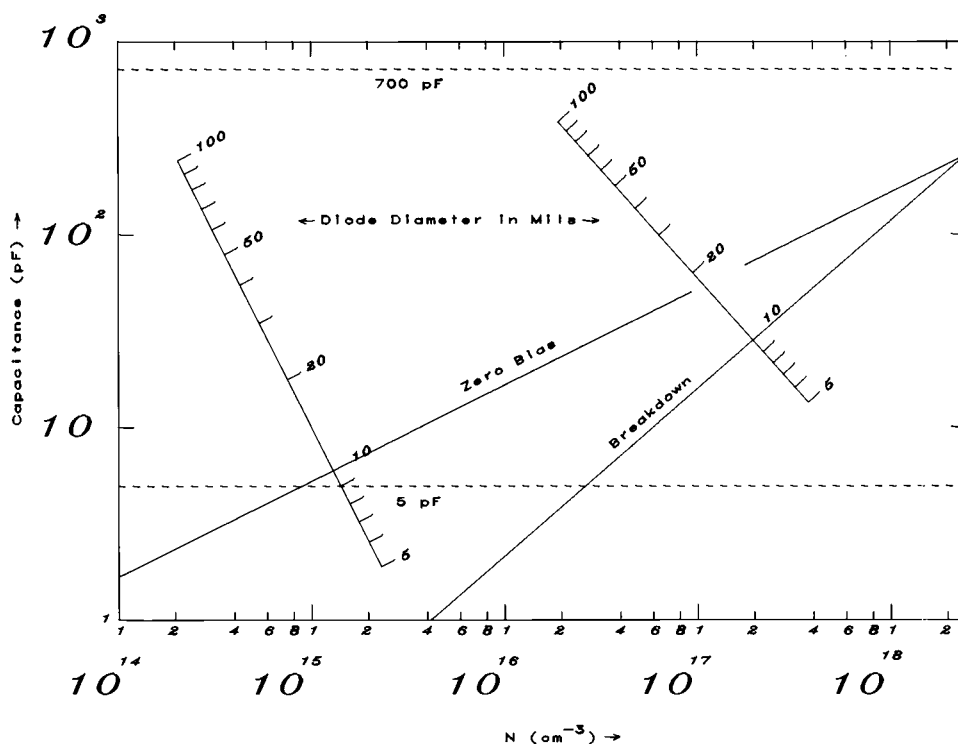


FIG. X3.1 Nomograph for Practical Limit Concern

### REFERENCES

- (1) Severin, P. J., and Poodt, G. J., "Capacitance-Voltage Measurements with a Mercury-Silicon Diode," *Journal of the Electrochemical Society*, Vol 119, No. 10, 1972, pp. 1384-1388.
- (2) Grove, A. S., *Physics and Technology of Semiconductor Devices*, John Wiley and Sons, New York, 1967, Sections 6.2 and 6.7c.
- (3) Rhoderick, E. H., and Williams, R. H., *Metal-Semiconductor Contacts*, 2nd Edition, Clarendon Press, Oxford, 1988.
- (4) Wiley, J. D., and Miller, G. L., "Series Resistance Effects in Semiconductor CV Profiling," *IEEE Transactions on Electron Devices*, Vol ED-22, 1975, pp. 265-272.
- (5) Schroder, D. K., *Semiconductor Material and Device Characterization*, Wiley-Interscience, New York, 1990, Section 2.5.2.
- (6) Miller, G. L., "A Feedback Method for Investigating Carrier Distributions in Semiconductors," *IEEE Transactions for Electron Devices*, Vol ED-19, No. 10, 1972, pp. 1103-1108.
- (7) Rehrig, D. L., and Pearce, C. W., "Production Mercury Probe Capacitance-Voltage Testing," *Semiconductor International*, Vol 3, No. 5, May 1980, pp. 151-162.
- (8) Schaffer, P. S., and Lally, T. R., "Silicon Epitaxial Wafer Profiling Using the Mercury-Silicon Schottky Diode Differential Capacitance Method," *Solid State Technology*, Vol 26, No. 4, April 1983, pp. 229-233.

**NOTICE: This standard has either been superseded and replaced by a new version or discontinued. Contact ASTM International (www.astm.org) for the latest information.**

 **F 1393**

*The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.*

*This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 100 Barr Harbor Drive, West Conshohocken, PA 19428.*