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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Method for Determining Net Carrier Density in Silicon Wafers by Miller Feedback Profiler Measurements With a Mercury Probe[1]

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 (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

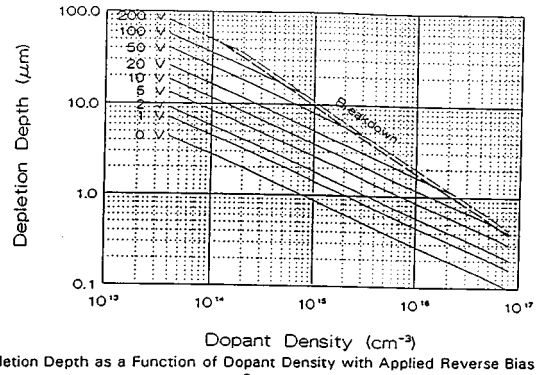
1.1 This test method[2] covers the measurement of net carrier density and net carrier density profiles in epitaxial and polished bulk silicon wafers in the range from about 4×10^{13} to about 8×10^{16} carriers/cm (resistivity range from about 0.1 to about 100 Ω-cm in *n*-type wafers and from about 0.24 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.

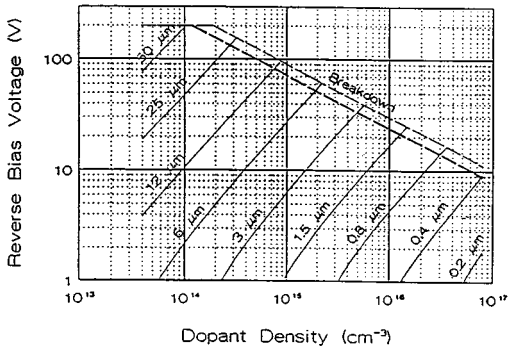
1.3 This test method may be applied to epitaxial layers on the same or opposite conductivity type substrate. This test method includes descriptions of fixtures for measuring substrates with or without an insulating backseal layer.

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



(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

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. 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

[1] 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.

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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 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 parenthesis refer to the list of references at the end of this test method.

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[4]

F 26 Test Methods for Determining the Orientation of a Semiconductive Single Crystal[5]

F 42 Test Methods for Conductivity Type of Extrinsic Semiconducting Materials[5]

F 81 Test Method for Measuring Radial Resistivity Variation on Silicon Slices[5]

F 84 Test Method for Measuring Resistivity of Silicon Wafers with an In-Line Four-Point Probe[5]

F 419 Test Method for Determining Carrier Density in Silicon Epitaxial Layers by Capacitance Voltage of Measurements on Fabricated Junction or Schottky Diodes[5]

F 673 Test Method for Measuring Resistivity of Semiconductor Slices or Sheet Resistance of Semiconductor Films with a Noncontact Eddy-Current Gage[5]

F 723 Practice for Conversion Between Resistivity and Dopant Density for Boron-Doped and Phosphorus-Doped Silicon[5]

F 1241 Terminology of Silicon Technology[5]

2.2 *SEMI Standard:*

SEMI C1 Specifications for Reagents[6]

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^2.

3.2.2 *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).

3.2.3 *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

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

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

[6] 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 μA) (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 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 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 ± 0.05 % reading + 0.05 % f.s.

7.7 *Digital Microammeter*, 0 to 200 μA, accuracy ± 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 μA/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[7] is of sufficiently high purity to permit its use without lessening the accuracy of the test.

[7] Specifications of the Committee on Analytical Reagents of the American Chemical Society, Washington, DC.