Standard Test Method for Measuring Resistivity of Silicon Wafers With an In-Line Four-Point Probe ¹

This standard is issued under the fixed designation F 84; 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 the resistivity of silicon wafers with a in-line four-point probe. The resistivity of a silicon crystal is an important materials acceptance requirement. This test method describes a procedure that will enable interlaboratory comparisons of the room temperature resistivity of silicon wafers. The precision that can be expected depends on both the resistivity of the wafer and on the homogeneity of the wafer. Round-robin tests have been conducted to establish the expected precision for measurements on *p*-type wafers with room temperature (23°C) resistivity between 0.0008 and 2000 Ω ·cm and on *n*-type wafers with room-temperature (23°C) resistivity between 0.0008 and 6000 Ω ·cm.

1.2 This test method is intended for use on single crystals of silicon in the form of circular wafers with a diameter greater than 16 mm (0.625 in.) and a thickness less than 1.6 mm (0.0625 in.). Geometrical correction factors required for these measurements are available in tabulated form.³

1.3 This test method is to be used as a referee method for determining the resistivity of single crystal silicon wafers in preference to Test Methods F 43.

NOTE 1—The test method is also applicable to other semiconductor materials but neither the appropriate conditions of measurement nor the expected precision have been experimentally determined. Other geometrics for which correction factors are not available can also be measured by this test method but only comparative measurements using similar geometrical conditions should be made in such situations.

NOTE 2-DIN 50431 2 is a similar, but not equivalent, method for determining resistivity. It is equivalent to Test Methods F 43.

1.4 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.5 *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 Section 8.

2. Referenced Documents

- 2.1 *ASTM Standards:*
- D 1193 Specification for Reagent Water⁴
- E 1 Specification for ASTM Thermometers⁵
- E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods⁶
- F 42 Test Methods for Conductivity Type of Extrinsic Semiconducting Materials ⁷
- F 43 Test Methods for Resistivity of Semiconductor Materials ⁷
- F 613 Test Method for Measuring Diameter of Semiconductor Wafers⁷
- 2.2 *SEMI Standard:*
- C1 Specifications for Reagents⁸

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

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

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

3.1.3 *probe pin, of a four-point probe*—one of the four

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.

Current edition approved Dec. 10, 1999. Published February 2000. Originally published as F 84 – 67 T. Last previous edition F 84 – 98.

² DIN 50431 is a similar, but not equivalent, method. It is the responsibility of DIN Committee NMP 221, with which Committee F-1 maintains close liaison. DIN 50431, Testing of Inorganic Semiconductor Materials: Measurement of the Specific Electrical Resistance of Monocrystals of Silicon or Germanium by the Four-Point Direct-Current Technique with Linearly Arranged Probes, is available from Beuth Verlag GmbH Burggrafenstrasse 4-10, D-1000 Berlin 30, Federal Republic of Germany.

³ Smits, F. M., "Measurement of Sheet Resistivities with the Four-Point Probe' *Bell System Technical Journal*, BSTJA, Vol 37, 1958, p. 711: Swartzendruber, L. J., "Correction Factor Tables for Four-Point Probe Resistivity Measurements on Thin, Circular Semiconductor Samples." *Technical Note 199* , NBTNA, National Bureau of Standards, April 15, 1964.

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

⁵ *Annual Book of ASTM Standards*, Vol 14.03.

⁶ *Annual Book of ASTM Standards*, Vol 14.02.

⁷ *Annual Book of ASTM Standards*, Vol 10.05.

⁸ Available from the Semiconductor Equipment and Materials International, 805 East Middlefield Road, Mountain View, CA 94043.

needles supporting the probe tips; mounting in a bearing contained in the probe head and losded by a spring or dead weight.

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

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

3.1.6 *resistivity*, ρ [Ω ·cm]—*of a semiconductor*, the ratio of the potential gradient (electric field) parallel with the current to the current density.

4. Summary of Test Method

4.1 An in-line four-point probe is used in determining the resistivity in this test method. A direct current is passed through the specimen between the outer probe pins and the resulting potential difference is measured between the inner probes. The resistivity is calculated from the measured current and potential values using factors appropriate to the geometry.

4.2 This test method includes procedures for checking both the probe head and the electrical measuring apparatus.

4.2.1 The spacing between the four probe tips is determined from measurements of indentations made by the probe tips in a polished silicon surface. This test also is used to determine the condition of the probe tips.

4.2.2 The accuracy of the electrical measuring equipment is tested by means of an analog circuit containing a known standard resistor together with other resistors which simulate the resistance at the contacts between the probe tips and the semiconductor surface.

4.3 Procedures for preparing the specimen, for measuring its size, and for determining the temperature of the specimen during the measurements are also given. Abbreviated tables of correction factors appropriate to circular wafer geometry and a table of temperature coefficient versus resistivity are included with the test method so that appropriate calculations can be made conveniently.

5. Significance and Use

5.1 Resistivity values measured by this test method are a primary quantity for characterization and specification of silicon material used for semiconductor electronic devices.

5.2 The current level, probe force, and specimen surface preparation specified in this test method are to be preferred for all referee measurements on bulk silicon wafers. However, many changes in these conditions may be made for nonreferee applications without severe changes in measurement results. ⁹

5.3 The accuracy of the resistivity as measured by this test method has not been determined. Systematic error is introduced by characteristic radial nonuniformities in the resistivity of silicon wafers and by the finite dimensions of the wafer. The magnitude of these errors is affected by the position of the

probe head on the wafer; for referee measurements the probe head should be placed within 0.25 mm (0.01 in.) of the center of the wafer. Systematic error may also be introduced in the measurement of the separation of the probe tips. The relative error in the determination of the probe-tip spacing decreases as the nominal probe-tip spacing increases; for referee measurements a four-point probe with nominal 1.59 mm (62.5 mil) probe-tip spacing is required.

5.4 The recommended analog circuit (Fig. 1) is not a perfect model of a semiconductor wafer being contacted by four metallic probes, with possible rectifying effects. The most effective use of the analog circuit to test the electrical instrumentation for possible error voltage during measurement requires that readings from opposite current polarities be treated separately, and not averaged. In this manner, the calculated standard deviation of the analog measurements will have enhanced sensitivity to possible error voltages.

6. Interferences

6.1 In making resistivity measurements, spurious results can arise from a number of sources.

6.1.1 Photoconductive and photovoltaic effects can seriously influence the observed resistivity, particularly with nearly intrinsic material. Therefore, all determinations should be made in a dark chamber unless experience shows that the material is insensitive to ambient illumination.

6.1.2 Spurious currents can be introduced in the testing circuit when the equipment is located near high frequency generators. If equipment is located near such sources, adequate shielding must be provided.

6.1.3 Minority carrier injection during the measurement can occur due to the electric field in the specimen. With material possessing high lifetime of the minority carriers and high resistivity, such injection can result in a lowering of the resistivity for a distance of several centimetres. Carrier injection can be detected by repeating the measurements at lower current. In the absence of injection no increase in resistivity should be observed. For specimens thicker than 0.75 mm (0.030 in.) use of the currents recommended in Table 1 should reduce the probability of difficulty from this source to a minimum. In cases of doubt and for thinner specimens the measurements of 12.4 and 12.5 should be repeated at a lower current. If the proper current is being used, doubling or halving its magnitude should cause a change in observed resistance which is less than 0.5 %.

6.1.4 Semiconductors have a significant temperature coefficient of resistivity. Consequently, the current used should be small to avoid resistive heating. If resistive heating is suspected

NOTE 1—See Table 2 for appropriate values of *r*. **FIG. 1 Analog Test Circuit to Simulate Four-Probe Measurement**

⁹ Ehrstein, J. R.; Brewer, F. H.; Ricks, D. R.; and Bullis, W. M., "Effects of Current, Probe Force, and Wafer Surface Condition on Measurement of Resistivity of Bulk Silicon Wafers by the Four-Probe Method," Appendix E, "Methods of Measurement for Semiconductor Materials, Process Control, and Devices," *Technical Note 773* , NBTNA, National Bureau of Standards, June 1973, pp. 43–49. Available as COM 73-50534 from National Technical Information Service, Springfield, VA 22161.

TABLE 1 Recommended Nominal Measurement Current Values

NOTE 1-This table is based on achieving 10 mV of specimen voltage between Pins 2 and 3 with specimen thickness of 0.5 mm (20 mils).

it can be detected by a change in readings as a function of time starting immediately after the current is applied.

6.1.5 Vibration of the probe sometimes causes troublesome changes in contact resistance. If difficulty is encountered, the apparatus should be shock mounted.

6.1.6 The temperature corrections given in this test method are valid only if the temperature of the specimen during measurement is held constant in the range from 18 through 28°C.

6.1.7 It is not uncommon with modern digital voltmeters to find that the voltmeter itself provides a source of current of the order of 10 pA between its high and low input terminals. Currents of this magnitude will generally have no effect on measurement accuracy for specimens below about 1000 Ω ·cm. However, since such spurious currents flow through the contact resistance of the voltage sensing probes, which contact resistances may be many megohms for higher resistivity specimens, the result may be spurious voltages of several tens of microvolts. These spurious currents can often be reduced if the autozero and auto calibration functions of the voltmeter can be suppressed. They generally are of fixed sign and the effect of the resulting spurious voltages can generally be cancelled by the use of forward and reverse current measurements (see 12.4 and 12.5).

6.1.8 It is not uncommon with modern digital voltmeters to find that the measurement guard terminal is the source of electrical spikes and other electrical noise components, often due to capacitive coupling to the instrument power supply. Since such noise may be rectified by the contact of the probes to the specimen, care should be taken when choosing where to connect the input guard lead to the measurement circuit.

7. Apparatus

7.1 *Slice Preparation*:

7.1.1 *Lapping Facilities* which permit the lapping of a wafer so that the thickness varies by no more than ± 1 % from its value at the center.

7.1.2 *An Ultrasonic Cleaner* of suitable frequency (18 to 45 kHz) and adequate power.

7.1.3 *Chemical Laboratory Apparatus* such as plastic beakers, graduates, and plastic-coated tweezers suitable for use both with acids (including hydrofluoric) and with solvents. Adequate facilities for handling and disposing of acids and their vapors are essential.

7.2 *Measurement of Specimen Geometry*:

7.2.1 *Thickness*—Calibrated mechanical or electronic thickness gage capable of measuring the wafer thickness to ± 1.0 % (R3S%) at various positions on the wafer.

7.2.2 *Diameter*—A micrometer or vernier caliper.

7.3 *Probe Assembly*:

7.3.1 *Probe Pins* with conical tungsten carbide probe tips with included angle of 45 to 150°. The nominal radius of a probe tip should be initially 25 to 50 µm.

7.3.2 *Probe Force*— The force on each probe shall be 1.75 \pm 0.25 N when the probe pins are against the specimen in measurement position.

7.3.3 *Insulation*—For measurement of specimens with resistivity up to approximately 100 Ω ·cm, the electrical isolation between a probe pin (with its associated spring and external lead) and any other probe pin or part of the probe head shall be at least 100 M Ω . For measurement of specimens with higher resistivity, the electrical isolation in ohms should be at least a factor of 1 M times the specimen resistivity in ohm centimetre.

7.3.4 *Probe Alignment and Separation*—The four probe tips shall be in an equally spaced linear array. The probe-tip spacing shall have a nominal value of 1.59 mm (62.5 mils). Probe–tip spacing shall be determined in accordance with the procedure of 11.1 in order to establish the suitability of the probe head as defined in 11.1.3. The following apparatus is required for this determination:

7.3.4.1 *Silicon Surface* such as that of a wafer or block which can be conveniently placed under the probe head. The surface must be polished and have a flatness characteristic of semiconductor wafers used in transistor fabrication.

7.3.4.2 *Micrometer Movement* capable of moving the probe head or silicon surface in increments of 0.05 to 0.10 mm (2 to 4 mils) in a direction perpendicular to a line through the probe tips and parallel to the plane of the surface.

7.3.4.3 *Toolmaker's Microscope* capable of measuring increments of 2.5 µm.

7.3.4.4 *Microscope* capable of a magnification of at least $400\times$.

7.4 *Specimen and Probe Head Supports*:

7.4.1 *Specimen Support*— A copper block at least 100 mm (4 in.) in diameter and at least 38 mm (1.5 in.) thick shall be used to support the specimen and provide a heat sink. It shall contain a hole that will accommodate a thermometer (see 7.5) in such a manner that the center of the bulk of the thermometer shall be not more than 10 mm (0.4 in.) below the central area of the heat sink where the specimen will be placed. A layer of mica 12 to 25 µm thick shall be placed on top of the heat sink to provide electrical isolation between the specimen and heat sink (Fig. 2). Mineral oil or silicone heat sink compound shall be used between the mica layer and copper block to reduce the thermal resistance. The heat sink shall be arranged so that the center of the probe tip array can be placed within 0.25 mm (10 mils) of the center of the specimen (Note 3). The heat sink shall be connected to the ground point of the electrical measuring apparatus (see 7.6).

NOTE 3—Shallow rings, concentric with the center of the copper block, may be machined into the heat sink in order to assist in rapid centering of wafers.

7.4.2 *Probe Head*—The probe head shall allow the probe pins to be lowered onto the surface of the specimen with negligible lateral movement of the probe tips (see 11.1.3.4).

FIG. 2 Heat Sink with Specimen, Mica Insulator, and Thermometer

7.5 *Thermometer*— ASTM Precision Thermometer covering a range from − 8 to 32°C and conforming to the requirements for Thermometer 63°C as prescribed in Specification E 1. The thermometer hole should be filled with mineral oil or silicone heat sink compound to provide good thermal contact between heat sink and thermometer.

7.6 *Electrical Measuring Apparatus*:

7.6.1 Any circuit that meets the requirements of 10.2 may be used to make the electrical measurements. The recommended circuit, connected as shown in Fig. 3, consists of the following:

7.6.1.1 *Constant-Current Source*—The value of current to be used depends on specimen resistivity and thickness. The current supply must have a compliance of at least 10 V, have ripple and noise no more than 0.1 % of the d-c current level being used, and must be stable to at least 0.05 % during the time required for measurement of a specimen. Currents between about 10−7 A and 100 mA are necessary to cover the resistivity range from about 0.05 to 10 000 Ω ·cm with equivalent precision at the specimen voltage level recommended in 12.4, assuming a specimen thickness of 0.5 mm (20 mils). Recommended current values are given in Table 1.

NOTE 4—For specimens of lower resistivity, it is advisable not to use currents significantly above 100 mA due to the risk of joule heating at the current probe tips (see 6.1.4). Rather, to maintain equivalent measurement precision at resistivities below about 0.05 Ω ·cm it is advisable to use a voltmeter of higher sensitivity and resolution (see 7.6.1.5).

NOTE 5—A smaller range of current values can be used to cover the full range of specimen resistivity and thickness values in the scope of this method without loss at measurement precision if the electronic voltmeter exceeds the minimum resolution requirements of 7.6.1.5.

7.6.1.2 *Current-Reversing Switch*.

7.6.1.3 *Standard Resistor*—The resistance of the standard resistor shall be selected so that it is within a factor of 100 of that of the specimen to be measured. Recommended values of resistance for various resistivity ranges are listed in Table 2.

NOTE 6—It is recommended that the standard resistor be chosen, where possible, to yield a potential difference that is larger than that measured on the specimen, with no upper limit other than that imposed by current carrying limits imposed to retain accuracy of certified resistor value

7.6.1.4 *Double-Throw, Double-Pole-Potential-Selector Switch*—This switch is needed in the recommended circuit of Fig. 2 to select between the standard resistor and the specimen for voltage measurements.

7.6.1.5 *Electronic Voltmeter*—This instrument may be used to measure the necessary potential differences in millivolts or it may be calibrated in conjunction with the current source to read voltage-current ratio directly. To cover the full range of specimen resistivities and thicknesses allowed in this test method, the instrument must be at least capable of measuring potential differences from 10 -4 to 0.05 V with a resolution of 0.05 % of the measured value (at least 31⁄2significant digits). The instrument must have an input impedance of at least $10⁶$ times the resistivity of the specimen (see also 6.1.7 and 6.1.8).

NOTE 7—A smaller range of full-scale voltages may be sufficient if only a limited range of specimen resistivity values is to be measured.

7.6.2 *Analog Test Circuit*—Five resistors connected as shown in Fig. 1 shall be used in testing the electrical measuring apparatus according to the procedure given in 10.2. The resistance of the central resistor, *r*, shall be selected according to the resistivity of the specimen to be measured as listed in Table 2.

7.7 *Conductivity-Type Determination*—Apparatus in accordance with Test Method A of Test Methods F 42.

7.8 *Ohmmeter* capable of indicating a leakage path of $10^9 \Omega$.

8. Reagents and Materials

8.1 *Purity of Reagents*—All chemicals for which specifications exist shall conform to SEMI Specifications C 1. Reagents for which SEMI specifications have not been developed shall conform to the specifications of the Committee on Analytical

FIG. 3 Recommended Electrical Circuit

 A Value must be within ± 20 % of the nominal value listed and must be known to $+0.05 \%$

 B These values also apply to analog test circuit resistors.

Reagents of the American Chemical Society, where such specifications are available. ¹⁰ Other grades may be used provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 *Purity of Water*— Reference to water shall be understood to mean deionized (DI) water meeting the resistivity and impurity specifications of Type I reagent water in Specification D 1193.

8.3 The recommended chemicals shall have the following nominal assays:

8.4 *Etching Solution* (*15 + 1*)—Mix 90 mL of nitric acid $(HNO₃)$ and 6 mL of hydrofluoric acid (HF).

8.5 *Acetone* ((CH₃)₂CO).

8.6 *Methanol* (CH ₃OH).

8.7 *Lapping Abrasive*— Aluminum oxide commercially specified as 5-um grade.

8.8 *Detergent Solution*—An aqueous, nonionic surfactant solution.

8.9 *Mineral Oil or Silicone Heat Sink Compound*.

9. Hazards

9.1 The chemicals used in this evaluation procedure are potentially harmful and must be handled in an acid exhaust fume hood, with utmost care at all times.

NOTE 8—**Warning:** Hydrofluoric acid solutions are particularly hazardous.

NOTE 9—**Precaution:** They should not be used by anyone who is not familiar with the specific preventive measures and first aid treatments given in the appropriate Material Safety Data Sheet.

9.2 Constant current supplies are capable of producing high output voltages if not connected to an external circuit. Therefore any changes of circuits connected to a constant current supply should be made either with the current supply turned off or with its output short circuited.

10. Preparation of Test Specimen

10.1 Make ten measurements of diameter, *D*, for specimens up to 1.9 in.; make five measurements for specimens from 1.9 to 2.9 in.; make three measurements for all larger diameters selected according to Test Method F 613 for specimens with diameter greater than 2.9 in. (74 mm). The specimen shall be circular; its diameter shall be greater than ten times the average probe-tip spacing *S* (see 11.1) and shall have a range of values not greater than *D/5S* % of *D*. Record the value of *D*.

10.2 If wafers are received in an as-sawed condition take at least 50 μ m (2 mils) from each side to remove saw damage. This may be done conveniently by etching with the solution listed in 7.4 before lapping.

NOTE 10—Rotating the specimen during etching helps provide a more uniform etch.

10.3 Finish the surface by lapping with 5–9-µm aluminum oxide abrasive. The finished surface shall have a matte rather than a polished nature. The finished thickness w shall be less than the average probe-tip spacing \bar{S} . Determine the thickness at nine locations on the specimen (Fig. 4). It shall not vary more than ± 1 % from the value at the center. Record the value of w at the center of the specimen.

10.4 After lapping, clean the specimen ultrasonically in warm water and detergent, rinse with flowing deionized water, ultrasonically degrease in acetone, rinse with methanol, and air dry. Cushion the specimen with paper or place in a pliable plastic beaker during ultrasonic agitation in order to reduce the risk of breakage.

11. Suitability of Test Equipment

11.1 *Four-Point Probe*— The probe-tip spacing and probetip condition shall be established in the following manner. It is recommended that this be done immediately prior to a referee measurement.

11.1.1 *Procedure*:

11.1.1.1 Make a series of indentations on a polished silicon surface with the four-point probe. Make these inden-tations by applying the probe to the surface using normal point pressures. Lift the probes and move either the silicon surface or the probes 0.05 to 0.10 mm (2 to 4 mils) in a direction perpendicular to a line through the probe tips. Again apply the probes to the silicon surface. Repeat the procedure until a series of ten indentation sets is obtained.

NOTE 11—It is recommended that the surface or the probes be moved twice the usual distance after every second or every third indentation set in order to assist the operator in identifying the indentations belonging to each set.

FIG. 4 Crosses Indicate Approximate Locations at Which Specimen Thickness Is to Be Measured

¹⁰ "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Analar Standards for Laboratory U.K., Chemicals," BDH Ltd., Poole, Dorset, and the "United States Pharmacopeia."

(b) Photograph Showing Three Indentations of a Satisfactory Tip.

(c) Photograph Showing Three Indentations of a Badly Worn Tip.

(d) Photograph Showing Three Indentations of a Probe Tip which Moved Laterally on Contact With the Specimen Surface.

NOTE 1—The indentations are 0.05 mm apart. **FIG. 5 Typical Probe Tip Indentation Pattern**

f F 84

11.1.1.2 Ultrasonically degrease the specimen in acetone, rinse with methanol, and let dry (see 10.4).

11.1.1.3 Place the polished silicon specimen on the stage of the toolmaker's microscope so that the *Y*-axis readings (*Y*_Aand Y_B in Fig. 5*a*) do not differ by more than 0.150 mm (0.006 in.). For each of the ten indentation sets record the readings *A* through *H* (defined in Fig. 5*a*) on the *X*-axis of the toolmaker's microscope and the readings Y_A and Y_B on the *Y*-axis. Use a data sheet similar to that shown in Fig. 6.

11.1.1.4 Examine the indentations under a microscope with a magnification of at least $400\times$.

11.1.2 *Calculations*:

11.1.2.1 For each of the ten sets of measurements calculate the probe separations S_{1j} , S_{2j} , and S_{3j} from the equations:

$$
S_{1j} = [(C_j + D_j)/2] - [(A_j + B_j)/2],
$$

\n
$$
S_{2j} = [(E_j + F_j)/2] - [(C_j + D_j)/2],
$$
 and
\n
$$
S_{3j} = [(G_j + H_j)/2] - [(E_j + F_j)/2]
$$
 (1)

In Eq 1, the index j is the set number and takes values 1 through 10.

11.1.2.2 Calculate the average value for each of the three separations using the S_{ij} calculated above and the equation:

$$
\bar{S}_i = (1/10) \sum_{j=1}^{10} S_{ij}
$$
 (2)

11.1.2.3 Calculate the sample standard deviation s_i for each

fill F 84

COMPUTATIONS

Run No.	$\frac{A + B}{2}$	$\frac{C\,+\,D}{2}$	$\frac{\mathbf{E}+\mathbf{F}}{2}$	$\frac{G + H}{2}$	\rm{S}_1	S ₂	S_{1}
$\mathbf 1$							
$\,2\,$							
$\bf{3}$							
$\overline{\mathbf{4}}$							
5							
$\bf 6$							
$\overline{7}$							
8							
9							
10							
\bar{S} (AVERAGE)							
s (SAMPLE STD. DEV.)							

FIG. 6 Typical Data Sheet for Computing Probe–Tip Spacing

of the three separations using the \bar{S}_i calculated from Eq 2, the S_{ij} calculated from Eq 1, and the equation:

$$
s_i = \left(1/3\right) \left[\sum_{j=1}^{10} \left(S_{ij} - \bar{S}_i\right) \right]^{1/2} \tag{3}
$$

11.1.2.4 Calculate the average probe–tip spacing \bar{S} :

$$
\bar{S} = (1/3)(\bar{S}_1 + \bar{S}_2 + \bar{S}_3) \tag{4}
$$

11.1.2.5 Calculate the probe–tip spacing correction factor $F_{\rm sp}$:

$$
F_{sp} = 1 + 1.082[1 - (\bar{S}_2/\bar{S})]
$$
 (5)

11.1.3 *Requirements*— For the four-point probe to be acceptable, it must meet the following requirements:

11.1.3.1 Each of the three sets of ten measurements for \bar{S}_i shall have a sample standard deviation s_i of less than 0.30 % of \bar{S}_i .

11.1.3.2 The average values of the separations \bar{S}_1 , \bar{S}_2 , and \bar{S}_3 shall not differ by more than 2 %.

11.1.3.3 The indentations obtained should show only a single area of contact for each probe-tip (Fig. 5 *b*). If the indentations obtained show disconnected areas of contact for one or more of the probe, the probe tips or pins (Fig. 5*c*) should be replaced and the test rerun.

11.1.3.4 Probe tips that show evidence of lateral movement on contact with the specimen (see Fig. 5*d*) are not acceptable. The probe head must be modified to prevent such movement.

NOTE 12—In some instances, lateral movement of the probe pins will result in motion of the specimen and a corresponding reduction in the extent of the skid mark. In such cases, the probe head should be checked by examining indentations made by lowering the probe pins onto a polished surface that is held rigidly in place.

11.2 *Electrical Equipment*—The suitability and accuracy of the electrical equipment shall be established in the following manner. It is recommended that this be done immediately prior to a referee measurement.

11.2.1 *Procedure*:

11.2.1.1 With the current supply short circuited or turned off, disconnect the probe assembly from the electrical circuit.

11.2.1.2 Attach the current leads (*1* and *2* of Fig. 3) to the current terminals (*I*) of the analog circuit appropriate to the resistivity of the specimen to be measured (Fig. 1 and Table 2). Attach the potential leads (*3* and *4* of Fig. 3) to the potential terminals (*V*) of the analog circuit.

11.2.1.3 If equipment for the direct measurement of resistance (voltage to current ratio) is being used proceed to 11.2.1.5; if not, proceed as follows: with the current initially in either direction (to be called "forward") adjust its magnitude to the appropriate value as given in Table 1. Measure V_{sf} , the potential differences across the standard resistor, or measure directly $I_{\rm sf}$, the current through the analog circuit. Measure $V_{\rm af}$, the potential difference across the analog circuit (Fig. 3). Reverse the direction of the current. Measure $V_{\rm sr}$, the potential difference across the standard resistor, or measure directly $I_{\rm a}$ _r, the current through the analog circuit. Measure V_{ar} , the potential differences across the analog circuit. Record the data taken on a sheet such as that shown in Fig. 7*(a)*.

11.2.1.4 Repeat the procedure of 11.2.1.3 until five measurements have been taken for each polarity. Proceed to 11.2.2.

11.2.1.5 Using direct resistance-measuring equipment, with the equipment initially connected in either polarity (to be called "forward") measure r_f , the resistance of the analog circuit in the forward direction. Reverse the polarity of the analog circuit connection; measure r_r , the resistance of the analog circuit in

the "reverse" direction. Continue to measure r_f and r_r , reversing the polarity of the equipment between successive readings until five measurements have been taken for each polarity. Record the results on a data sheet such as that in Fig. 7*(b)*.

11.2.2 *Calculations*:

11.2.2.1 If the resistance is measured directly, begin the calculations with 11.2.2.2. If the procedure of 11.2.1.3 and 11.2.1.4 was followed, calculate and record, on a data sheet such as that in Fig. 7(*b*), r_f and r_r , the resistance of the analog box for the current in the forward and reverse directions, respectively, using the following for each measurement position:

$$
r_{\rm f} = V_{\rm a\,f} R_{\rm s} / V_{\rm s\,f} = V_{\rm a\,f} / I_{\rm af} \tag{6}
$$

$$
r_{\rm r} = V_{\rm af} R_{\rm s}/V_{\rm sr} = V_{\rm ar}/I_{\rm ar} \tag{7}
$$

where:

- R_s = resistance of standard resistor, Ω ,
 V_{af} = potential difference across the analog potential difference across the analog circuit, current in the forward direction, mV,
- V_{ar} = potential difference across the analog circuit, current in the reverse direction, mV,
- $V_{\rm s~f}$ = potential difference across the standard resistor, current in the forward direction, mV,
- V_{sr} = potential difference across the standard resistor, current in the reverse direction, mV,
- I_{af} = current through the analog circuit in the forward direction, mA, and
- I_{ar} = current through the analog circuit in the reverse direction, mA.

(a) For Standard Circuit or Direct Measurement of Current

(b) For Direct Measurement of Resistance or Calculation from Fig. 7(a) $T = \Omega$ $s = \Omega$

Use the right-hand most form of Eq 6 when the current is measured directly.

11.2.2.2 Using the forward and reverse resistance values as separate values (whether obtained by calculation or direct measurement) calculate the average resistance \bar{r} from the equation,

$$
\bar{r} = (1/10) \sum_{i=1}^{10} r_i \tag{8}
$$

where r_i is one of the ten values for r_f and r_f already determined.

11.2.2.3 Calculate the sample standard deviation s_r from the equation:

$$
s_{\rm r} = (1/3) \left[\sum_{i=1}^{10} (r_i - \bar{r})^2 \right]^{1/2}
$$
 (9)

11.2.3 *Requirements*— For the electrical measuring equipment to be suitable, it must meet the following requirements.

11.2.3.1 The value of \bar{r} must be within 0.1 % of the known value of *r* for resistors up to 100 Ω and must be within 0.3 % of the known value of *r* for resistors above 100 Ω .

11.2.3.2 The sample standard deviation s_r must be less than 0.3 % of \bar{r} .

NOTE 13—The value of the analog circuit test resistor *r* if unknown, may be determined with the use of ordinary standards laboratory procedures by measuring current I and the potential difference V' with the potential terminals *V* open circuited (Fig. 3), and by calculating $r_a = V'/I$.

11.2.3.3 The resolution of the equipment must be such that differences in resistance of 0.05 % can be detected.

12. Procedure

12.1 Immediately before measuring the specimen, clean ultrasonically in warm water and detergent solution, rinse in flowing deionized water, ultrasonically degrease in acetone, rinse with methanol, and air dry (see 10.4).

12.2 Using clean nonmetallic tweezers place the specimen on the mica insulator on top of the heat sink. Measure the resistance between specimen and heat sink with an ohmmeter in order to verify that the specimen is electrically isolated $(>10^8 \Omega)$ from the heat sink. With the thermometer in place, allow sufficient time after placing the specimen on the heat sink for thermal equilibrium to be established.

NOTE 14—For specimens that have been in the same room as the heat sink for 30 min or more, the time required for equilibration will not exceed 30 s. The heat sink itself should have been allowed to come to equilibrium with the room (the temperature of which should not vary by more than a few degrees) for 48 h before referee measurements are made.

12.3 Lower the probe pins onto the surface of the specimen so that the center of the probe tip array is within 0.25 mm (10 mils) of the center of the specimen.

12.4 With the current initially in either direction (called forward), adjust its value to give a potential difference measured across the specimen having a recommended value of 10 to 20 mV, but no more than 50 mV. Values of this potential difference lower than 10 mV are necessary for specimens with resistivities below about 0.05 Ω ·cm to keep the maximum measurement current at approximately 100 mA. Nominal currents to achieve these potential difference values are given in Table 1 (see also 6.1.3, 6.1.4, and Note 4.) Measure to at least 31⁄2 significant figures (resolution to 0.05 % of reading) the following quantities and record.

NOTE 15—It will generally not be possible, even with the best available electronic voltmeters, to measure voltages to 31⁄2 significant figures for specimens below 0.001 Ω ·cm at the recommended maximum current levels.

12.4.1 V_{sf} , the potential difference across the standard resistor. (Substitute I_f , the current, if measuring the current directly; omit this measurement if using equipment which reads resistance directly.)

12.4.2 V_f , the potential difference between the two inner probe tips. (Substitute R_f , the resistance, between the two inner probe tips, if measuring resistance directly.)

12.4.3 *T*, the temperature of the specimen as measured by the thermometer placed in the heat sink.

NOTE 16—To obtain the precision stated in Section 15, the temperature must be measured to the nearest 0.1°C and the potential differences with a combined instrumental uncertainty no greater than ± 0.1 %.

12.5 Reverse the direction of the current. Measure the following quantities and record the data:

12.5.1 $V_{\rm sr}$, the potential difference across the standard resistor. (Substitute I_r , the current, if measuring the current directly; omit this measurement if using equipment which reads resistance directly.)

12.5.2 V_r , the potential difference between the two inner probe tips. (Substitute R_r , the resistance, between the two inner probe tips, if measuring resistance directly.)

12.6 Short circuit or turn off the current supply, raise the probe head, and rotate the specimen 15 to 20°.

12.7 Repeat the procedure of 12.3, 12.4, 12.5, and 12.6 until ten sets of data have been taken.

12.8 Record on the data sheet the specimen thickness in centimetres as measured at its center (see 10.3) and the average specimen diameter in centimetres (see 10.1).

12.9 Determine the conductivity type of the specimen in accordance with Method A of Test Methods F 42. Follow the procedure as given with the exception that the surface treatment of this method (see 10.3) shall be used.

13. Calculation

13.1 Calculate the resistance for the current in both forward and reverse directions as follows:

$$
R_{\rm f} = V_{\rm f} R_{\rm s} / V_{\rm s f} = V_{\rm f} / I_{\rm f}
$$
, and

F 84

Run No.	$R_i(\Omega)$	$R_r(\Omega)$	$R_m(\Omega)$	$\rho(T)(\Omega \cdots cm)$	\mathcal{F}_T	$\rho(23)(\Omega)$	cm)		
\mathbf{I}								\boldsymbol{D}	cm
$\sqrt{2}$								\mathbf{W}	cm
3								$\cal S$	cm
$\overline{4}$								$F_{\rm sp}$	
5								$\overline{S}/\overline{D}$	
$\sqrt{6}$								F ₂	
$\overline{7}$								$F(w/\overline{S})$	
$\bar{8}$								\boldsymbol{F}	
$\overline{9}$								C_{T}	
$\rm 10$									
				$\rho(23)$ (AVERAGE)					

FIG. 8 Typical Computation Sheet for Four-Point Probe Resistivity Measurement

$$
R_{\rm r} = V_{\rm r} R_{\rm s} / V_{\rm sr} = V_{\rm r} / I_{\rm r}, \tag{10}
$$

where:
 R_f

- = specimen resistance with current in the forward direction, Ω ,
- R_r = specimen resistance with current in the reverse direction, Ω ,
- I_f = current through the specimen in the forward direction, mA,
- I_r = current through the specimen in the reverse direction, mA,
- V_f = potential difference across the specimen, current in the forward direction, mV,
- V_r = potential difference across the specimen, current in the reverse direction, mV,
- V_{sf} = potential difference across the standard resistor, current in the forward direction, mV, and
- $V_{\rm sr}$ = potential difference across the standard resistor, current in the reverse direction, mV.

The right-hand most form of Eq 10 is most convenient for use when the current is measured directly. This calculation is not required if direct reading equipment is employed. In all cases, R_f and R_r must agree to within 10 % of the larger for the measurement to be accepted for referee purposes. These and subsequent calculations may be summarized conveniently in the data sheet of Fig. 8.

13.2 Calculate the mean value of the resistance R_m for each measurement position:

$$
R_{\rm m} = 1 / 2 (R_{\rm f} + R_{\rm r})
$$
 (11)

13.3 Calculate the ratio of the average probe-tip spacing *S* to the wafer diameter *D*. Find the correction factor F_2 from Table 3 using linear interpolation.

13.4 Calculate the ratio of the wafer thickness w to the average probe–tip spacing \bar{S} . Find the correction factor $F(w/\bar{S})$ from Table 4 using linear interpolation or from the expression given in Appendix X1.

13.5 Calculate the geometrical correction factor *F* as follows:

$$
F = F_2 \times w \times F(w/S) \times F_{\text{sp}},\tag{12}
$$

TABLE 3 Correction Factor ^F ² as a Function of the Ratio of Probe-Tip Spacing S to Slice Diameter D

		. -			
Ŝ/D	F,	S/D	F_{2}	S/D	F_{2}
0	4.532	0.035	4.485	0.070	4.348
0.005	4.531	0.040	4.470	0.075	4.322
0.010	4.528	0.045	4.454	0.080	4.294
0.015	4.524	0.050	4.436	0.085	4.265
0.020	4.517	0.055	4.417	0.090	4.235
0.025	4.508	0.060	4.395	0.095	4.204
0.030	4.497	0.065	4.372	0.100	4.171

where:

 F_{sp} = probe-tip spacing correction factor (see 11.1.2.5) and

w = specimen thickness, cm.

13.6 Calculate the resistivity of the sample at the temperature of measurement:

$$
\rho(T) = R_{\rm m} \times F \tag{13}
$$

where:

 $\rho(T)$ = resistivity of specimen at temperature T, Ω ·cm,

 R_{m} = average resistance (see 13.2), Ω , and
F = geometrical correction factor, cm (see $=$ geometrical correction factor, cm (see 13.5).

13.7 Find the appropriate temperature coefficient 11 , 12 from Table 5. Calculate the temperature correction factor F_T by the equation:

¹¹ Bullis, W. M., Brewer, F. H., Kolstad, C. D., and Swartzendruber, L. J., "Temperature Coefficient of Resistivity of Silicon and Germanium Near Room Temperature," *Solid-State Electronics*, Vol II, 1968, pp. 639–646.

¹² Bullis, W. Murray, ed., "Methods of Measurement for Semiconductor Materials, Process Control, and Devices," NBS Technical Note 754 , pp. 8–9.

TABLE 5 Temperature Coefficient of Resistivity of Silicon in the Range from 18 to 28°C

Note 1—Values for *p*-type silicon are valid for boron dopant only. Numbers in italics are smoothed values of the results from curve fitting.¹²

$$
F_T = 1 - C_T (T - 23)
$$
 (14)

where:

T = temperature, ^oC, and

 C_T = coefficient read from Table 5.

Only one value of C_T generally need be read for a series of measurements on a given specimen, and should be based on the average value of uncorrected resistivity as calculated in Eq 13.

13.8 Calculate the resistivity corrected to 23°C as follows:

$$
\rho(23) = \rho(T) \times F_T \tag{15}
$$

where:

 $p(23)$ = resistivity corrected to 23^oC, Ω ·cm.

13.9 Calculate the value of the grand average of the corrected resistivity as follows:

$$
\bar{\rho}(23) = (1 \, / \, 10) \sum_{i=1}^{10} \rho_i(23) \tag{16}
$$

where ρ_i (23) are corrected resistivities found from Eq 15. Omit this step if only a single measurement was made.

13.10 Calculate the sample standard deviation, *s*:

$$
s = (1/3) \left[\sum_{i=1}^{10} \left[\rho_i(23) - \bar{\rho}(23) \right]^2 \right]^{1/2}
$$

14. Report

14.1 Report the following information:

14.1.1 *Referee tests*— include all information called for on data sheets (Fig. 6, Fig. 7, Fig. 8, and Fig. 9). In addition, the electrical instruments employed in the test shall be identified.

14.1.2 *Nonreferee tests*— only the value of resistivity and the number of sets of data taken need be reported. Supporting calibration data for four-point probe, electrical measuring equipment, thermometer, and standard resistors should be taken at regular intervals and an appropriate file maintained.

15. Precision

15.1 For measurements on homogeneous silicon wafers with room-temperature (23°C) resistivity between 0.0008 and

F 84

SPECIMEN MEASUREMENT DATA

(b) For Direct Measurement of Current

Nore 1—Record all information at top of page. Record four digits for all data except temperature. **FIG. 9 Typical Data Sheet for Specimen Electrical Data**

120 Ω ·cm, the interlaboratory precision, as defined by Practice E 177, is ± 2 % (3S %) when the measurement is performed by competent operators. This precision is based on results obtained during 3 round-robin experiments involving 6 laboratories and 13 wafers.

15.2 For measurements on silicon wafers with roomtemperature (23°C) resistivity between 120 and 500 Ω ·cm, the interlaboratory precision is expected to be better than \pm 5 % (3S %) and for silicon wafers with room-temperature (23°C) resistivity between 500 and 2000 Ω · cm, the interlaboratory precision is expected to be better than ± 15 % (3S %). These data are based on 2 round-robin experiments involving 6 laboratories and 5 wafers. These wafers all had radial resistivity gradients which were large enough to affect the results of the experiment; the precision to be expected on homogeneous wafers in these resistivity ranges has not yet been determined.

15.3 For measurements on n-type silicon wafers with room-

temperature (23 $^{\circ}$ C) resistivity from 2000 to 6000 Ω ·cm, the interlaboratory precision is expected to be better than ± 15 % (3S %) based on the results of a round-robin experiment involving 7 laboratories and 1 wafer. Measurements by the same laboratories on two p-type wafers in this resistivity range resulted in considerably more scatter. The reasons for the greater scatter have not yet been determined.

15.4 If a single pair of measurements is made instead of the series of ten specified in 12.7, the precision is expected to be somewhat degraded. For homogeneous specimens, however, the initial reading of each series of ten sets taken during the three round-robin experiments fell within the 3-sigma limits specified above.

16. Keywords

16.1 four-point probe; four-probe; resistivity; semiconductor; silicon

APPENDIXES

(Nonmandatory Information)

X1. COMPUTATION OF $F(w/\bar{S})$

X1.1 Rather than using the values in Table 4, $F(w/\bar{S})$ can be computed directly as follows:

$$
F(w/\bar{S}) = 1.3863 \bar{S/(wD)} \tag{X1.1}
$$

where:

where:

 $M =$ integer $(2 \bar{S}/w) + 1$, and

$$
D = 1 + 2\sum_{n=1}^{M} \{ [1/4 + (nw/\bar{S})^2]^{-1/2} - [1 + (nw/\bar{S})^2]^{-1/2} \} + \sum_{n=M+1}^{N} [3/4(\bar{S}/nw)^3 - 45/64(\bar{S}/nw)^5 + 315/512(\bar{S}/nw)^7]
$$

 $N =$ smallest value of *n* for which the increment in the

second summation is less than 10^{-5} .

X1.2 With two exceptions, values of $F(w/\bar{S})$ calculated from Eq X1.1 agree with those of Smits, 2 to 1 part in 10⁴ and with those of Table 4, to 6 parts in 10 $⁴$ or better. (The</sup> calculated value for $w/\bar{S} = 0.9$ is 0.9460, 0.002 higher than the value in Table 4, and the calculated value for $w / \bar{S} = 1.0$ is 0.9216, 0.0002 higher than the value quoted by Smits.) The error in Table 4 occurs because the values in the table are based on a linear interpolation between values quoted by Smits; the interval used to obtain the 0.9 value was larger than any other.

X1.3 For w/\bar{S} < 0.4, $F(w/\bar{S}) = 1.000$, and no computation is necessary.

X2. POLYNOMIAL COEFFICIENTS TO GENERATE THE TEMPERATURE COEFFICIENT OF RESISTIVITY

 $X2.1$ Polynomial coefficients were used¹³ to fit the empirical data base developed by NIST for the temperature coefficient of resistivity of *n*-type and boron-doped *p*-type silicon over the resistivity range of 0.0006 ohm cm to greater than 500 ohm cm, and the temperature interval 18°C to 28°C. The coefficients were used to calculate the values gtiven in Table X2.1 and enable the resisitivity of silicon specimens measured anywhere in the given temperature interval to be corrected to the standard temperature of 23°C.

X2.2 The polynomial coefficients are used to fit the specific temperature coefficient, C_T ($\Omega \times \text{cm}/\Omega \times \text{cm} \times {}^{\circ}C$), to a polynomial function of the natural logarithm, (*ln*), of the measured resistivity:

$$
C_T = A_0 + A_1 (\ln \rho) + A_2 (\ln \rho)^2 + A_3 (\ln p)^3 + \dots
$$
 (X2.1)

TABLE X2.1 Polynomial Coefficients for Temperature Coefficient of Silicon Resistivity

Coefficient	n-Type Silicon	p-Type Silicon
A ₀	7.364×10^{-3}	7.068×10^{-3}
A_1	6.560×10^{-4}	8.544×10^{-4}
A ₂	-3.075×10^{-4}	-1.478×10^{-4}
A_3	-2.427×10^{-5}	1.635×10^{-5}
A_4	7.5883×10^{-5}	-2.003×10^{-5}
A ₅	7.5541×10^{-6}	3.415×10^{-6}
A ₆	-1.39760×10^{-5}	2.0915×10^{-6}
A ₇	-1.159×10^{-8}	-4.3237×10^{-7}
A_8	1.106882 \times 10 ⁻⁶	-7.0532×10^{-8}
A_{9}	-4.56719×10^{-8}	1.60868×10^{-8}
A_{10}	-4.407686×10^{-8}	1.0346×10^{-9}
A_{11}	2.601512×10^{-9}	-2.5201×10^{-10}
A_{12}	9.408560×10^{-10}	-5.6419×10^{-12}
A_{13}	$-6.190700 \times 10^{-11}$	1.4445×10^{-12}
A_{14}	$-1.032377 \times 10^{-11}$	
A_{15}	6.890181 \times 10 ⁻¹³	
A_{16}	4.58514×10^{-14}	
A_{17}	-2.9432×10^{-15}	

This standard is copyrighted by ASTM, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org).

¹³ Bullis, W. Murray, ed. "Methods of Measurement for Semiconductor Materials, Process Control and Devices", NBS Technical Note 560, pp. 6–7. Available from NIST, Div. 812, Gaithersburg, MD 20899.

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, at the address shown below.