



# Standard Test Method for Sheet Resistance of Silicon Epitaxial, Diffused, Polysilicon, and Ion-implanted Layers Using an In-Line Four-Point Probe with the Single-Configuration Procedure<sup>1</sup>

This standard is issued under the fixed designation F 374; 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 covers the direct measurement of the average sheet resistance of thin layers of silicon with diameters greater than 15.9 mm (0.625 in.) which are formed by epitaxy, diffusion, or implantation onto or below the surface of a circular silicon wafer having the opposite conductivity type from the thin layer to be measured or by the deposition of polysilicon over an insulating layer. Measurements are made at the center of the wafer using a single-configuration of the four-probe, that is, with the current being passed through the outer pins and the resulting potential difference being measured with the inner pins.

1.2 This test method is known to be applicable on films having thickness at least 0.2  $\mu\text{m}$ . It can be used to measure sheet resistance in the range 10 to 5000  $\Omega$ , inclusive.

1.2.1 The principle of the test method can be extended to cover lower or higher values of sheet resistance; however, the precision of the method has not been evaluated for sheet resistance ranges other than those given in 1.2.

NOTE 1—The minimum value of the diameter is related to tolerances on the accuracy of the measurement through the geometric correction factor. The minimum layer thickness is related to danger of penetration of the probe tips through the layer during measurement.

1.3 Procedures for preparing the specimen, for measuring its size, and for determining the temperature of the specimen during the measurement are also given. Abbreviated tables of correction factors appropriate to circular geometry are included with the method so that appropriate calculations can be made conveniently.

NOTE 2—The principles of this test method are also applicable to other semiconductor materials, but neither the appropriate conditions nor the expected precision have been determined. Other geometries can also be measured, but only comparative measurements using similar geometrical conditions should be used unless proper geometrical correction factors are known.

NOTE 3—Some relaxations of test conditions are mentioned in order to assist in applying the principles of the method to nonreferee applications,

for which a complete nonreferee method has not yet been developed. The relaxed test conditions given are consensus conditions only and their effect on measurement precision and accuracy has not been explored.

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 9.

## 2. Referenced Documents

### 2.1 ASTM Standards:

D 5127 Guide for Ultra Pure Water Used in the Electronics and Semiconductor Industry<sup>2</sup>

E 1 Specification for ASTM Thermometers<sup>3</sup>

F 42 Test Method for Conductivity Type of Extrinsic Semiconducting Materials<sup>4</sup>

F 1529 Test Method for Sheet Resistance Uniformity Evaluation by In-Line Four-Point Probe with the Dual-Configuration Procedure<sup>4</sup>

### 2.2 SEMI Standard:

C 3.15 Specifications for Nitrogen Gas<sup>5</sup>

C 28 Specification for Hydrofluoric Acid<sup>5</sup>

C 31 Specification for Methanol<sup>5</sup>

## 3. Terminology

### 3.1 Definitions:

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.

3.1.1.1 *Discussion*—It may consist of a unitized probe head holding all four probes or it may have each of the four individual probes attached to a separate cantilevered arm.

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

Current edition approved Dec. 10, 2000. Published February 2001. Originally published as F 374 – 74 T. Last previous edition F 374 – 00.

<sup>2</sup> *Annual Book of ASTM Standards*, Vol 11.01.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 14.03.

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 10.05.

<sup>5</sup> Available from the Semiconductor Equipment and Materials Institute, 625 Ellis St., Suite 212, Mountain View, CA 94043.

3.1.2 *probe head, of a four-point probe*—the mounting that (1) fixes the position of the four-point 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*—one of the four needles supporting the probe tips; mounting in a bearing contained in the probe head and loaded 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 *sheet resistance,  $R_s$*  [ $\Omega$  or  $\Omega$  per square]—of a semiconductor or thin metal film, the ratio of the potential gradient (electric field) parallel with the current to the product of the current density and thickness.

3.1.6.1 *Discussion*—The sheet resistance is formally equal to the bulk resistivity divided by the thickness of the material, taken in the limit as the thickness approaches zero.

#### 4. Summary of Method

4.1 A in-line four-point probe is used to determine the specimen sheet resistance.<sup>6</sup> A direct current is passed through the specimen between the outer probe pins, and the resulting potential difference is measured between the inner probe pins. The sheet resistance is calculated from the ratio of the measured voltage to current values using correction factors appropriate to the geometry.

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

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

#### 5. Significance and Use

5.1 The sheet resistance of silicon epitaxial, diffused, and implanted layers is an important materials acceptance and process control parameter. The sheet resistance measurement may be used by itself or may be combined with a value of layer thickness, obtained separately, to obtain an estimate of the resistivity of an epitaxial layer or of the surface concentration of dopant for diffused layers.

5.2 This test method is suitable for use in materials acceptance, manufacturing control, research, and development.

NOTE 4—An alternate method, Test Method F 1529, will generally provide superior measurement precision that may be very important for spatial uniformity mapping requirements. That test method will also avoid the need to apply a lateral geometry correction to the measurements. However, that test method will generally require the use of a fully automated four-probe measurement system.

<sup>6</sup> Smits, F. M., "Measurement of Sheet Resistivities with the Four-Point Probe," Bell System Technical Journal, BSTJA, Vol. 37, 1948, p. 711; Swartzendruber, L. J., "Correction Factor Tables for Four-Point Probe Resistivity Measurements on Thin, Circular Semiconductor Samples, NBS Technical Note 199, NBTNA, April 15, 1964.

#### 6. Interferences

6.1 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.

6.2 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.

6.3 Minority carrier injection during the measurement can occur due to the electric field in the specimen. With material possessing long lifetime of the minority carriers and high resistivity, such injection can result in a lowering of the resistivity for a distance of several centimeters from the point of injection. Carrier injection can be detected by repeating the measurements at lower current. In the absence of injection, no increase in resistivity should be observed at the lower current. The current level recommended (Table 1) should reduce the probability of difficulty from this source to a minimum, but in cases of doubt the measurements of 12.4 through 12.8 should be repeated at a lower current. If the proper current is being used, doubling or halving its magnitude should cause a total change in observed resistance which is less than 0.5 %.

6.4 Semiconductors have a significant temperature coefficient of resistivity. Consequently, the current used should be small to avoid resistive heating. The current level recommended (Table 1) should reduce the chances of this difficulty. If resistive heating is suspected, it can be detected by a change in readings as a function of time starting immediately after the current is applied. If such a change is observed, repeat the measurements of 12.4 through 12.8 at a lower current.

6.5 Vibration of the probe head may cause variations in contact resistance, which is often manifested in unstable readings. If difficulty is encountered, the apparatus should be shock mounted.

6.6 Penetration of either current or voltage probe tip through the layer to be measured to the substrate can result in erroneous readings. This can usually be checked by mounting the specimen in direct contact with a metallic support grounded to the current supply and looking for a reduction in measured specimen voltage in at least one polarity. If this condition obtains, examine the probe tips microscopically for sharp asperities and remove these by polishing, or reduce probe force, or obtain probe pins with blunter tips.

6.7 The accuracy with which the separation of the probe tips is measured affects the accuracy of the calculated sheet

**TABLE 1 Current Values Required for Measurements of Sheet Resistance**

| Sheet Resistance, $\Omega$ | $I^A$       |
|----------------------------|-------------|
| 2.0–25                     | 10 mA       |
| 20–250                     | 1 mA        |
| 200–2500                   | 100 $\mu$ A |
| 2000–25 000                | 10 $\mu$ A  |

<sup>A</sup> The proper value of current depends on layer thickness and probe spacing in addition to layer sheet resistance. The current used shall be stable to within 0.05 % during the time of measurement and shall be selected to give a measured specimen voltage between 5 and 20 mV, inclusive. The overlap in ranges in the table is intentional since the table illustrates starting points for current selection.

resistance. The relative accuracy of probe tip spacing measurement decreases as the nominal value of the probe tip spacing decreases. For referee measurement purposes, use of a four-point probe with 1.59 mm (0.0625 in.) nominal spacing is required. Four-point probes having other nominal probe tip spacings are suitable for nonreferee measurements.

6.8 The accuracy of the final calculated value of sheet resistance is degraded if the four-point probe is not placed at the center of the specimen during measurement (see 12.4). For referee measurements, the center of the tip array probe shall not be more than 1.0 mm from the center of the specimen as measured along a nonflatted diameter.

6.9 The sheet resistance value calculated from the measurements may be in error if the thin film intended for the front surface is also formed on the rear surface of the wafer, and if the wafer edges provide a conducting path between the front-surface and rear-surface films. The effect of complete coverage of the wafer front surface, edge, and rear surface by a thin conducting film is to make the appropriate value of the correction factor  $F_2$  equal to the limiting value of 4.532, regardless of wafer diameter or probe spacing. It is generally difficult or impossible to test for the conductivity type of the wafer edges. However, if a conductivity-type test of the rear surface of the wafer shows this surface to be of the same conductivity type as the front surface, the resulting sheet resistance measurements may be in error. The absolute value of the maximum error is given by  $\frac{|F_2 - 4.532|}{F_2}$ .

## 7. Apparatus

### 7.1 Specimen Preparation:

7.1.1 *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.

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

### 7.2 Measurement of Specimen Geometry:

7.2.1 *Means for Measuring Specimen Diameter*, such as a micrometer or vernier caliper.

### 7.3 Probe Head:

#### 7.3.1 Probe Pins:

7.3.1.1 *For Specimen Layers Having Thickness of 3  $\mu\text{m}$  or Less*—Probe pins shall have blunt conical tips of a durable material such as tungsten carbide, with included angle in the nominal range from 45 to 150°. The probe tips shall terminate in a hemisphere with a radius in the nominal range from 100 to 250  $\mu\text{m}$ , or in a flat circular truncation with a circle radius in the nominal range from 50 to 125  $\mu\text{m}$ .

7.3.1.2 *For Specimen Layers Having Thickness Greater Than 3  $\mu\text{m}$* —Probe pins shall have sharp conical tips of a durable material such as tungsten carbide, with included angle in the nominal range from 45 to 150°. The probe tips shall terminate in a hemisphere with a radius in the nominal range from 35 to 100  $\mu\text{m}$ .

7.3.2 *Probe Force*—For hemispherical-tipped probe pins with tip radius greater than 100  $\mu\text{m}$  or for flat-tipped probe pins with tip radius greater than 50  $\mu\text{m}$ , the force on each probe tip

shall be in the range from 0.30 to 0.80 N (31 to 81 gf), inclusive, when the four-point probe is against the specimen in measurement position. For hemispherical-tipped probe pins with tip radius less than 100  $\mu\text{m}$ , the force on each probe tip shall be  $0.30 \pm 0.03$  N ( $31 \pm 3$  gf), inclusive, when the four-point probe is against the specimen in measurement position.

NOTE 5—The combination of probe tip radius and probe pin load, which is chosen, affects not only the immunity from probe tip penetration of very thin layers but also the electrical quality of contact and hence the noise and accuracy of measurement. The presence of higher resistivity values at the top surface of the silicon layer to be measured may require an increase in the force of probe pin or use of sharper probe tips. An example of this situation is a buried peak boron implant.

7.3.3 *Insulation*—The electrical isolation between a probe pin (with its associated spring and external lead) and any other probe pin or probe head part shall be at least  $10^9 \Omega$ .

7.3.4 *Probe Alignment and Separation*—The four-point probe tips shall be in an equally spaced linear array. The separations between adjacent probe tips shall have a nominal value of 1.59 mm (0.0625 in.). (Other nominal probe spacings such as 1.0 and 0.6 mm (0.040 and 0.025 in.) are suitable for nonreferee measurements.) The spacing between probe pins shall be determined in accordance with the procedure in 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 *Piece of Material*, such as porous silicon or germanium that is softer than single crystal silicon, for use with blunt probes, and a slice or block of silicon for use with sharp probe tips as designated for layers more than 3- $\mu\text{m}$  thick. In each case the surface of the piece of material must be polished and have a flatness characteristic of semiconductor wafers used in microelectronic device fabrication. The surface must have lateral dimensions adequate to span the outermost of the probe tips.

7.3.4.2 *Micrometer Movement*, capable of moving the probe head or silicon surface in increments in the nominal range from 0.05 to 0.10 mm in a direction perpendicular to a line through the probe tips and parallel to the plane of the surface.

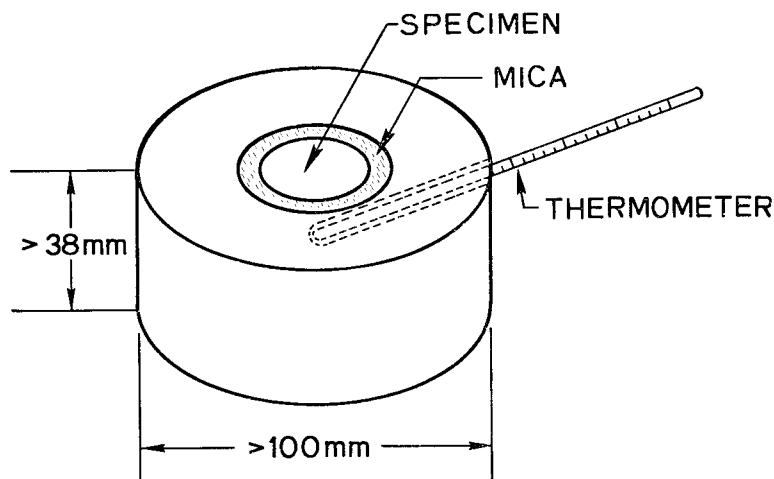
7.3.4.3 *Toolmaker's or Other Traveling Microscope*, capable of measuring increments of 2.5  $\mu\text{m}$ .

7.3.4.4 *Microscope*, with a magnification of at least 600 $\times$  with an eyepiece magnification no greater than 15 $\times$ .

### 7.4 Specimen and Probe Pin Supports:

7.4.1 *Specimen Support*—A copper block at least 100 mm (4 in.) diameter and at least 40 mm (1.6 in.) thick, or a rectangular block of equivalent mass and thickness, shall be used to support the specimen and provide a heat sink. For adequate heat transfer, vacuum clamping or other means for rigidly clamping the specimen to the heat-sink is necessary. The heat sink shall contain a hole that can accommodate a thermometer (see 7.5) in such a manner that the center of the bulb of the thermometer is not more than 10 mm below the central area of the heat-sink where the specimen will be placed (see Fig. 1). Comparable provision for the installation of a thermocouple, thermistor or resistance temperature detector (RTD) be made instead. An insulating disk, less than 0.076 mm thick and suitably perforated, shall be placed over the center

**ASTM F 374**



**FIG. 1 Heat Sink with Specimen, Mica Insulator, and Thermometer**

area of the copper to provide electrical isolation between the specimen and the heat sink. Mineral oil or silicone heat sink compound shall be used between the insulating disk and the copper block to reduce the thermal resistance. The heat sink shall be arranged so that the center of the four-probe array can be placed within 1.0 mm of the center of the specimen (see 12.4). The heat-sink shall be connected to the ground point of the electrical measuring apparatus (see 7.6). The heat-sink shall be at a temperature of  $23 \pm 1^\circ\text{C}$  during measurement.

*NOTE 6*—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 specimens.

**7.4.2 Probe Assembly Support**—The probe head support shall allow the probe pin to be lowered onto the surface of the specimen with no evidence of lateral movement of the probe tips as observed under a magnification of at least  $600\times$  using an eyepiece having a magnification no larger than  $15\times$ .

**7.5 Thermometer**—ASTM Precision Thermometer having a range from  $-8$  to  $32^\circ\text{C}$  inclusive and conforming to the requirements for Thermometer 63C as prescribed in Specification E 1. The thermometer hole shall be filled with mineral oil or silicone heat sink compound to provide good thermal contact between heat sink and thermometer.

**7.5.1** A thermocouple, thermistor or RTD known to be accurate to at least  $0.1^\circ\text{C}$  over the range of normal room temperatures may be mounted, instead of the glass bulb thermometer, in a comparable location in the heat sink.

**7.6 Electrical Measuring Apparatus:**

**7.6.1** Any circuit that meets the requirements of 11.2 may be used to make the electrical measurements. The recommended circuit, connected as shown in Fig. 2, consists of the following:

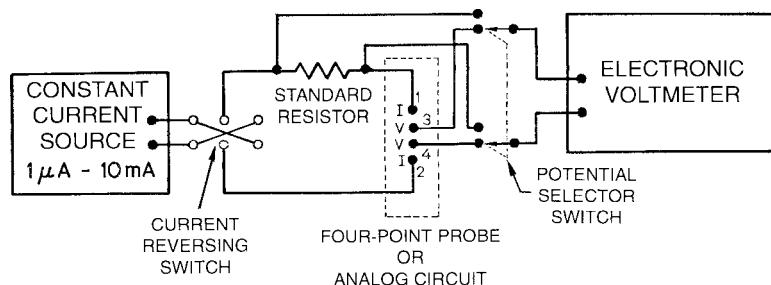
**7.6.1.1 Constant-Current Source**—The value of current to be used depends on the layer sheet resistance and shall be selected so that the potential difference across the inner probes is between 2 and 60 mV. Currents between  $10^{-6}$  and  $10^{-2}$  A are required if the sheet resistance range 1 to 25 000  $\Omega$  inclusive is to be covered (Table 1).

**7.6.1.2 Double-Pole, Double-Throw Current-Reversing Switch**.

**7.6.1.3 Standard Resistor**, selected so as to yield a potential difference which is in the range from 0.1 to 10 times the potential difference measured across the layer when using the appropriate current value for the layer. Recommended resistances for various layer sheet resistance ranges are given in Table 2. Such a standard resistor is not needed when using a precision current supply that is known to have an output accuracy of 0.1 % or better of the nominal set point value for the measurement being made.

**7.6.1.4 Electronic Voltmeter**—To read the potential drop in volts or (when calibrated in conjunction with the current source) to read the volt-current ratio directly. The instrument shall be capable of measuring potential differences between  $10^{-3}$  V and  $10^{-1}$  V full scale and be able to resolve increments as small as 0.1 % of reading for each range, and must have a d-c input resistance of  $100^9\Omega$  or greater.

**7.6.2 Analog Test Circuit**—Five resistors connected as



**FIG. 2 Recommended Electrical Circuit**

**TABLE 2 Sheet Resistance Range Appropriate to Analog Test Circuit Resistance,  $r$ , and Recommended Standard Resistance,  $R_s$ , Values**

| Sheet Resistance, $\Omega$ | Analog and Standard Resistor, $\Omega$ <sup>A</sup> |
|----------------------------|---|
| 2.0–25                     | 10  |
| 20–250                     | 100   |
| 200–2500                   | 1000  |
| 2000–25 000                | 10 000  |

<sup>A</sup> The resistance shall be within a range from one half to twice the nominal value given, inclusive, and shall be known to  $\pm 0.05\%$ . The value of the standard resistor,  $r$  (Fig. 3), should be chosen to yield a voltage drop comparable to that measured across the specimen.

shown in Fig. 3 shall be used in testing the electrical measuring apparatus in accordance with the procedure given in 11.2. The resistance of the central resistor,  $r$ , shall be selected according to the sheet resistance of the layer to be measured as listed in Table 2.

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

7.8 *Ohmmeter*, capable of indicating any leakage resistance up to  $10^9\Omega$ .

## 8. Reagents and Materials

8.1 *Purity of Reagents*—All chemicals for which specifications exist shall conform to Grade 1 SEMI specifications for those specific chemicals. 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 either Type I or Type II Reagent Water as specified in Guide D 5127.

8.3 *Dry Nitrogen*.

8.4 *Hydrofluoric Acid (HF)*,  $49.0 \pm 0.25\%$ .

8.5 *Insulator*, 0.076 mm (0.003 in.) thick or less (See 7.4.1).

8.6 *Methanol (CH<sub>3</sub>OH)*.

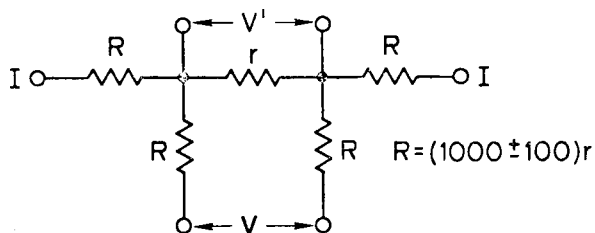
8.7 *Mineral Oil or Silicone Heat Sink Compound*.

## 9. Hazards

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

9.2 **Warning**—Hydrofluoric acid solutions are particularly hazardous. They should not be used by anyone who is not familiar with the preventive measures and first aid treatment given in the appropriate Material Safety Data Sheet.

9.3 Constant-current power supplies are capable of producing high voltages if not connected to an external circuit.



**FIG. 3 Analog Test Circuit to Simulate Four-Point Probe Measurement**

Therefore any changes of connection to a constant-current supply should be made either with the current supply turned off or with its output short-circuited.

## 10. Suitability of Test Specimen

10.1 Determine the average specimen diameter by measuring individual specimen diameters as follows. For specimens that are expected to be more than 51 mm (2 in.) in diameter, measure the length of three diameters at angular separations of 50 to 70°; for specimens that are expected to be between 32 and 51 mm (1.25 and 2.0 in.) in diameter, measure five diameters at angular separations of 30 to 45°. For specimens smaller than 32 mm in diameter, measure ten diameters at angular separations of 15 to 20°. Do not measure along any diameter that intersects an orientation notch or flat. Calculate the simple arithmetic average of these measurements,  $\bar{D}$ . For the specimen to be suitable,  $\bar{D}$  must be greater than 10 times the average probe spacing,  $\bar{S}$  (see 11.1.2.4), and the sample standard deviation of the diameter measurements shall be less than  $(\bar{D}/5\bar{S})\%$  of  $\bar{D}$ . Record the value of  $\bar{D}$ .

10.2 Determine the conductivity type of the layer to be measured and the substrate of the specimen according to Method A of Test Methods F 42 if conductivity types are unknown. Follow the procedure as given in Test Method F 42 except that the surfaces shall be cleaned in accordance with 12.2 of this test method. The layer and substrate must be of opposite conductivity type in order to make sheet resistance measurements.

## 11. Suitability of Test Equipment

11.1 *Probe Head*—Establish the spacing between probe pins and probe tip condition in the following manner immediately prior to each referee test.

NOTE 7—This test procedure need be performed only once even if several specimens are to be measured during a single referee test.

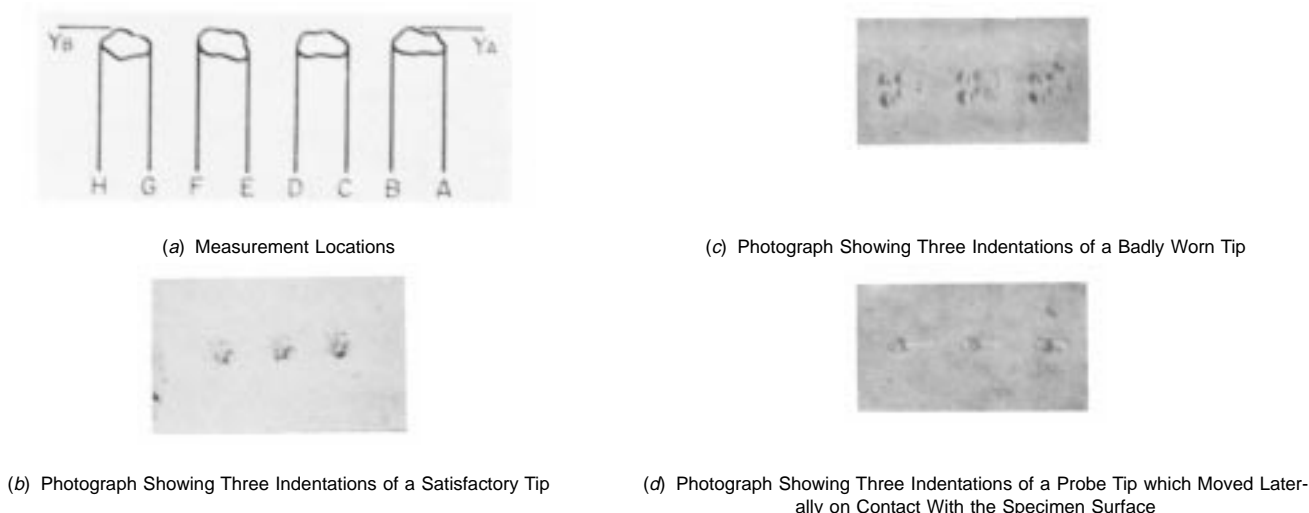
### 11.1.1 Procedure:

11.1.1.1 Make a series of indentations of the probe tips on a polished silicon surface or other polished surface (depending on the sharpness of the probe tips, see 7.3.4.1) with the four-point probe. Make these indentations by applying the probe tips to the surface using loads as specified in 7.3.2. Lift the probe pins and move either the chosen polished surface or the probe head 0.05 to 0.10 mm in a direction perpendicular to the line through the probe tips. Again apply the probe tips to the polished surface. Repeat the procedure until a series of ten indentation sets is obtained.

NOTE 8—It is recommended that the silicon specimen or the probe tips be moved twice the usual distance after every second or third indentation set in order to assist the operator in identifying the indentations belonging to each set.

11.1.1.2 Ultrasonically degrease the specimen in acetone, rinse with methanol, and let dry. Place the specimen in a pliable plastic beaker during ultrasonic agitation in order to reduce the risk of breakage.

11.1.1.3 Place the chosen polished specimen on the stage of the toolmaker's microscope so that the Y-axis readings ( $Y_A$  and  $Y_B$  in Fig. 4(a)) do not differ by more than 0.15 mm (0.006 in.). For each of the ten indentation sets record the readings  $A$



NOTE 1—The indentations are 0.05 mm apart.  
**FIG. 4 Typical Probe Tip Indentation Pattern**

through  $H$  (defined in Fig. 4(a)) on the  $X$ -axis of the toolmaker's microscope and the readings  $Y_A$  and  $Y_B$  on the  $Y$ -axis. These data should be recorded to the nearest  $2.5 \mu\text{m}$  ( $0.0001 \text{ in.}$ ). Use a data sheet similar to that shown in Fig. 5.

11.1.1.4 Examine the indentations for continuity of the contact region and evidence of horizontal motion under a microscope of at least  $600\times$  magnification.

11.1.2 *Calculations:*

11.1.2.1 For each of the ten sets of measurements calculate the separations,  $S_{1j}$ ,  $S_{2j}$ ,  $S_{3j}$ , between adjacent probe tips as follows:

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

$$S_{2j} = [(E_j + F_j)/2] - [(C_j + D_j)/2], \text{ and}$$

$$S_{3j} = [(G_j + H_j)/2] - [(E_j + F_j)/2].$$

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

11.1.2.2 Calculate the average value for each of the three probe tip spacings as follows, using the  $S_{ij}$  calculated above:

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

where the index  $i$  takes the values 1, 2, 3 and record  $S_1$ ,  $S_2$ , and  $S_3$  in the appropriate box of a table such as that shown in Fig. 5.

11.1.2.3 Calculate the sample standard deviation,  $s_i$ , for each of the three spacing between probe pins using the  $S_i$  calculated from Eq 2, and the  $S_{ij}$  calculated from Eq 1, and the equation:

$$S_{3j} = [(G_j + H_j)/2] - [(E_j + F_j)/2] \quad (3)$$

11.1.2.4 Calculate the average probe pin spacing,  $\bar{S}$ , for the four-point probe:

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

11.1.2.5 Calculate the probe pin spacing correction factor,  $F_{sp}$ :

$$F_{sp} = 1 + 1.082[1 - (S_2/S)] \quad (5)$$

11.1.2.6 For a referee test, record results of all above calculations on a data sheet such as that shown in Fig. 5.

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

11.1.3.1 The set of ten measurements for each of the  $S_i$  shall have a sample standard deviation  $s_i$  not more than 0.3 % of  $\bar{S}_i$ . (For a four-point probe with nominal 1.59-mm (0.0625-in.) spacing, this is equivalent to  $s_i \leq 4.7 \mu\text{m}$ .)

11.1.3.2 The individual average separations ( $\bar{S}_1$ ,  $\bar{S}_2$ , and  $\bar{S}_3$ ) shall not differ by more than 2 % of the overall average,  $\bar{S}$  (see 11.1.2.4).

11.1.3.3 The indentations obtained should show only a single area of contact for each probe tip when examined under a microscope having a magnification of at least  $600\times$  (Fig. 4). If the indentations obtained show disconnected areas of contact for one or more of the probe tips, the probe pin or probe pins should be replaced and the test rerun.

11.1.3.4 The probe tip indentations shall not show any evidence of horizontal sliding movements along the surface when observed at a magnification of at least  $600\times$ . An example of such evidence is shown in Fig. 4(c).

NOTE 9—In some instances, lateral movement of the probe tips 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 tip 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 immediately prior to a referee test (Note 7).

11.2.1 *Procedure:*

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

**ASTM F 374**

Probe Serial No. \_\_\_\_\_  
Date \_\_\_\_\_  
Operator \_\_\_\_\_

S \_\_\_\_\_  
F<sub>sp</sub> \_\_\_\_\_

DATA

| Set No. | A | B | C | D | E | F | G | H | Y <sub>A</sub> | Y <sub>S</sub> |
|---------|---|---|---|---|---|---|---|---|----------------|----------------|
| 1       |   |   |   |   |   |   |   |   |                |                |
| 2       |   |   |   |   |   |   |   |   |                |                |
| 3       |   |   |   |   |   |   |   |   |                |                |
| 4       |   |   |   |   |   |   |   |   |                |                |
| 5       |   |   |   |   |   |   |   |   |                |                |
| 6       |   |   |   |   |   |   |   |   |                |                |
| 7       |   |   |   |   |   |   |   |   |                |                |
| 8       |   |   |   |   |   |   |   |   |                |                |
| 9       |   |   |   |   |   |   |   |   |                |                |
| 10      |   |   |   |   |   |   |   |   |                |                |

COMPUTATIONS

| Set No. | A + B<br>2 | C + D<br>2 | E + F<br>2 | G + H<br>2 | S <sub>1</sub> | S <sub>2</sub> | S <sub>3</sub> |
|---------|------------|------------|------------|------------|----------------|----------------|----------------|
| 1       |            |            |            |            |                |                |                |
| 2       |            |            |            |            |                |                |                |
| 3       |            |            |            |            |                |                |                |
| 4       |            |            |            |            |                |                |                |
| 5       |            |            |            |            |                |                |                |
| 6       |            |            |            |            |                |                |                |
| 7       |            |            |            |            |                |                |                |
| 8       |            |            |            |            |                |                |                |
| 9       |            |            |            |            |                |                |                |
| 10      |            |            |            |            |                |                |                |

S̄ (Average) \_\_\_\_\_

s (Sample Standard Deviation) \_\_\_\_\_

Record data to nearest 2.5 μm (0.0001 in.)

**FIG. 5 Typical Data Sheet for Computing Spacing Between Probe Pins**

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

11.2.1.3 If equipment for direct measurement of resistance (voltage-to-current ratio) is to be 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 difference across the standard resistor, or  $I_{sf}$ , the forward current through the analog circuit (Fig. 3). Reverse the direction of the current. Measure  $V_{sr}$ , the potential difference across the standard resistor, or  $I_{sr}$ , the reverse current through the analog circuit. Measure  $V_{ar}$ , the potential difference across the analog circuit. Record the data taken on a sheet such as that in Fig. 6(a).

11.2.1.4 Repeat the procedure of 11.2.1.3 until five sets of data have been taken. Proceed to 11.2.2.

11.2.1.5 If using direct resistance-measuring equipment, with the equipment initially connected in either polarity (to be called “forward”) measure the resistance,  $r_f$ , of the analog

circuit. Use Table 1 to obtain appropriate current levels. Reverse the polarity of the equipment connection and measure the resistance,  $r_r$ , of the analog circuit. Record all data on a data sheet such as that in Fig. 6(b).

11.2.1.6 Repeat the procedure of 11.2.1.5 until five sets of data have been taken.

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 is followed, calculate the resistance of the analog box for the current in both the forward and reverse directions and record (Fig. 6(b)) for each measurement as follows:

$$r_f = V_{af}R_s/V_{sf} = V_{af}/I_{af}$$

$$= V_{ar}R_s/V_{sr} = V_{ar}/I_{ar} \tag{6}$$

where:

$V_{af}$ ,  $V_{sf}$ ,  $V_{ar}$ ,  $V_{sr}$ , and  $I_{ar}$  are defined in 11.2.1.3, and

$R_s$  = resistance of standard resistor,  $\Omega$ .

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

Date \_\_\_\_\_  
Rs \_\_\_\_\_ Ω

| Run No. | V <sub>sr</sub> , mV | V <sub>ar</sub> , mV | V <sub>ar</sub> , mV | V <sub>sr</sub> , mV | I <sub>ar</sub> , mA | I <sub>ar</sub> , mA |
|---------|----------------------|----------------------|----------------------|----------------------|----------------------|----------------------|
| 1       |                      |                      |                      |                      |                      |                      |
| 2       |                      |                      |                      |                      |                      |                      |
| 3       |                      |                      |                      |                      |                      |                      |
| 4       |                      |                      |                      |                      |                      |                      |
| 5       |                      |                      |                      |                      |                      |                      |

(a) For standard circuit or direct measurement of current

| Run No. | r <sub>f</sub> , Ω | r <sub>r</sub> , Ω | r <sub>m</sub> , Ω |
|---------|--------------------|--------------------|--------------------|
| 1       |                    |                    |                    |
| 2       |                    |                    |                    |
| 3       |                    |                    |                    |
| 4       |                    |                    |                    |
| 5       |                    |                    |                    |

(b) For direct measurement of resistance or calculation from Fig. 6(a)

r<sub>m</sub> (Average) = \_\_\_\_\_ Ω  
s<sub>a</sub> (Sample Standard Deviation) = \_\_\_\_\_ Ω

Record at least 3 digits for all data

**FIG. 6 Typical Data Sheet for Analog Circuit Measurement**

11.2.2.2 Calculate the mean measured resistance  $r_m$  of the analog circuit for each measurement position using values  $r_f$  and  $r_r$  for resistance as calculated in 11.2.2.1 or as obtained by direct measurement. Record the values of  $r_m$  on a data sheet such as that shown in Fig. 6(b).

$$r_m = \frac{1}{2}(r_f + r_r). \quad (7)$$

11.2.2.3 Calculate the average value,  $r_m$ , of the analog circuit resistance as follows:

$$\bar{r}_m = \frac{1}{5} \sum_{i=1}^5 r_{mi} \quad (8)$$

where:

$r_{mi}$  = one of the five values of resistance determined in 11.2.2.2.

11.2.2.4 Calculate the sample standard deviation,  $s_a$ , from the equation as follows:

$$s_a = (1/2) [\sum_{j=1}^5 (r_{mj} - \bar{r}_m)^2]^{1/2} \quad (9)$$

11.2.3 *Requirements*— For the electrical equipment to be acceptable, it must meet the following requirements:

11.2.3.1 The value of  $\bar{r}_m$  must be within 0.3 % of the known value  $r$ .

11.2.3.2 The sample standard deviation,  $s_a$ , must be no greater than 0.3 % of  $\bar{r}_m$ .

NOTE 10—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  $r = V'/I$ .

11.2.3.3 The resolution of the equipment must be that differences in resistance of 0.1 % of the full-scale range in use can be detected.

11.2.4 Reconnect the probe head to the electrical circuit as

shown in Fig. 2. Allow sufficient warm-up time to meet manufacturer's specifications.

## 12. Procedure

12.1 If the specimens to be measured have been kept in a laminar-flow hood with a noncontaminating atmosphere and are to be measured within 3 h after either etching or fabrication, omit the cleaning steps and proceed to 12.3.

12.2 Clean the specimens by the following procedure:

12.2.1 Rinse in methanol for 1 min.

12.2.2 Allow to dry.

12.2.2.1 Repeat methanol rinse as necessary until the dried specimen is free from stain.

12.2.3 Place in hydrofluoric acid for 1 min (see warning notice in 9.2).

12.2.4 Thoroughly rinse in water to remove hydrofluoric acid.

12.2.5 Rinse in methanol for 1 min.

12.2.6 Allow to dry.

12.2.6.1 Repeat methanol rinse as necessary until the dried specimen is free from stain.

12.2.7 Blow free of dust with dry nitrogen.

12.3 Using clean tweezers, handle the specimen carefully to avoid contaminating or damaging the surface and place the specimen on the insulator on top of the heat-sink. Clamp the specimen to the heat sink by the method chosen. Lower the four-probe array onto the specimen. Measure the electrical resistance between any one of the four probe pins and the heat-sink with an ohmmeter in order to verify that the specimen is electrically isolated ( $>10^9 \Omega$ ) 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



**ASTM F 374**

established. At thermal equilibrium, the specimen temperature should be  $23 \pm 1^\circ\text{C}$ .

NOTE 11—For specimens that have been in the same room environment as the heat sink for 30 min or more, the time required for equilibration shall 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 measurements are made.

12.4 Lower the probe tip onto the surface of the specimen so that the center of the probe-tip array is within 1.00 mm of the center of the specimen as measured along a nonflatted diameter.

NOTE 12—For nonreferee measurements, a value for specimen centering of 5 mm is recommended for circular specimens.

12.5 With the current initially in either direction (called “forward”), adjust its magnitude to the appropriate value as given in Table 1 using an estimated value for the sheet resistance. Measure to at least three significant figures the following quantities, and record the data on an appropriate data sheet, such as one of those shown in Fig. 7, as follows:

12.5.1  $V_{sf}$ , the potential difference in millivolts across the standard resistor. (Substitute  $I_f$ , the current in milliamperes, if measuring the current directly; omit this measurement if using equipment that reads resistance directly.)

12.5.2  $V_f$ , the potential difference in millivolts between the two inner probe pins. (Substitute  $R_f$ , the resistance between the two inner probe pins, if measuring resistance directly.)

12.5.3  $T$ , the temperature in degrees Celsius of the specimen as measured by the thermometer placed in the heat sink.

NOTE 13—To obtain the precision stated in Section 14, the potential differences must be measured with a resolution of at least  $\pm 0.2\%$ . If several ranges of the measuring instrument meet this requirement, the range with the highest input impedance should be used.

12.6 Reverse the direction of the current. Measure the following quantities and record the data as follows:

12.6.1  $V_{sr}$ , the potential difference in millivolts across the standard resistor. (Substitute  $I_r$ , the current in milliamperes, if measuring the current density; omit this measurement if using equipment that reads resistance directly.)

12.6.2  $V_r$ , the potential difference in millivolts between the two inner probes. (Substitute  $R_r$ , the resistance between the two inner probes, if measuring resistance directly.)

12.7 Turn off or short-circuit the current, raise the probe assembly, and rotate the specimen  $30 \pm 5^\circ$ .

12.8 Repeat the procedure of 12.4-12.7 until five sets of data have been taken.

NOTE 14—Only a single set of data is required for a nonreferee test.

**SPECIMEN MEASUREMENT DATA**

Specimen \_\_\_\_\_  
 $R_s$  \_\_\_\_\_  
 Date \_\_\_\_\_

Slice Diameter \_\_\_\_\_ cm  
 Type:  $p/n$   $n/p$

| Run No. | $V_{sf}$ , mV | $V_f$ , mV | $V_r$ , mV | $V_{sr}$ , mV | $T$ , °C |
|---------|---------------|------------|------------|---------------|----------|
| 1       |               |            |            |               |          |
| 2       |               |            |            |               |          |
| 3       |               |            |            |               |          |
| 4       |               |            |            |               |          |
| 5       |               |            |            |               |          |

(a) For standard circuit

| Run No. | $I_f$ , mA | $V_r$ , mV | $V_r$ , mV | $I_r$ , mA | $T$ , °C |
|---------|------------|------------|------------|------------|----------|
| 1       |            |            |            |            |          |
| 2       |            |            |            |            |          |
| 3       |            |            |            |            |          |
| 4       |            |            |            |            |          |
| 5       |            |            |            |            |          |

(b) For direct measurement of current

| Run No. | $R_f$ , $\Omega$ | $R_r$ , $\Omega$ | $T$ , °C |
|---------|------------------|------------------|----------|
| 1       |                  |                  |          |
| 2       |                  |                  |          |
| 3       |                  |                  |          |
| 4       |                  |                  |          |
| 5       |                  |                  |          |

(c) For direct measurement of resistance

NOTE—Record all information identified at top of page.

Record at least 3 significant digits for all data.

**FIG. 7 Typical Data Sheet for Specimen Electrical Data**

**ASTM F 374**

**13. Calculation**

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

$$R_f = V_f R_s / V_{af} = V_f / I_f \tag{10}$$

and

$$R_r = V_r R_s / V_{sr} = V_r / I_r$$

where:

- $R_f$  = resistance with current in the forward direction,  $\Omega$ ,
- $R_r$  = resistance with current in the reverse direction,  $\Omega$ ,
- $R_s$  = resistance of standard resistor,  $\Omega$ , and  $V_f$ ,  $V_r$ ,  $V_{af}$ ,  $V_{sr}$ ,  $I_f$ , and  $I_r$  are defined in 12.5.

Use the expressions  $V_f / I_f$  and  $V_r / I_r$  when the current is measured directly; omit this calculation if equipment reading resistance directly is employed. Summarize these and subsequent calculations in a data sheet such as is shown in Fig. 8.

NOTE 15—In all cases,  $R_f$  and  $R_r$  must agree to within 5 % of the larger for the measurement to be accepted for referee purposes.

13.2 Calculate the mean value of resistance,  $R_m$ , for each measurement position as follows:

$$R_m = 1 / 2(R_f + R_r) \tag{11}$$

13.3 Calculate the ratio of the average probe separations,  $S$  (see 11.1.2.4), to the average specimen diameter,  $D$  (see 10.1). Find the correction factor  $F_2$  from Table 3 using linear interpolation.

13.4 Calculate the geometrical correction factor,  $F$ , as follows:

$$F = F_2 \times F_{sp} \tag{12}$$

where:

- $F_2$  = finite diameter specimen correction factor, <sup>6</sup> and
- $F_{sp}$  = probe correction factor (see 11.1.2.5).

NOTE 16—Using the least favorable value for ratio of the spacing between probe pins to specimen diameter, 1.59 mm and 15.9 mm, respectively, the maximum error in  $F_2$  for being 1.0 mm off center during measurement is 0.25 %.

13.5 Calculate the sheet resistance of the specimen at the temperature of measurement for each measurement position as follows:

$$R_s(T) = R_m \times F \tag{13}$$

where:

- $R_s(T)$  = sheet resistance,  $\Omega$ , of specimen at temperature  $T$ ,
- $R_m$  = mean resistance,  $\Omega$ , and
- $F$  = geometrical correction factor (see 13.4).

13.6 Calculate the grand average sheet resistance,  $\bar{R}_s T$  as follows:

$$\bar{R}_s(T) = (1/5) \sum_{i=1}^5 R_{si}(T) \tag{14}$$

13.7 Calculate the sample standard deviation,  $s$ , as follows:

$$s = (1/2) \{ \sum_i [R_{si}(T) - \bar{R}_s(T)]^2 \}^{1/2} \tag{15}$$

**14. Report**

14.1 The report shall include all information called for on data sheets shown in Fig. 7 and Fig. 8. It is recommended that data called for on data sheets shown in Fig. 5 and Fig. 6 also be provided.

**15. Precision and Bias**

15.1 *Specimen Layers Having Thickness Greater Than 3  $\mu\text{m}$* —Measured with sharp probe tips and nominal 0.3-N (30-gf) force on probe pins:

15.1.1 The precision of this test method as applied to epitaxial layers greater than 3  $\mu\text{m}$  thick was determined by round-robin measurements on three  $n$ -type and three  $p$ -type epitaxial specimens with epitaxial resistivity in the range 0.1 to 10  $\Omega\text{-cm}$  (sheet resistance range 10 to 5000  $\Omega$ ) and that were deposited on opposite conductivity-type substrates (see Annex A1). Due to breakage of, and substitution for, two of the  $p$ -type specimens during the experiment, not all laboratories measured all the same  $p$ -type specimens. Specimen diameter measurements were made by each laboratory, and these individual values were used for computation. Despite a change in procedure with regard to determining probe force during the latter part of the round robin, data taken by both procedures on a given specimen were pooled since the change had no effect on quality of data already taken.

15.1.2 The precision of this test method for layers greater than 3  $\mu\text{m}$  thick is given in terms of the 95 % confidence estimate (R2S) for the reproducibility of measurement averages from two laboratories. Based on analysis of the multilayer round robin, a conservative estimate of this reproducibility for six of the specimens (specimens 1A, 2, 3A, 4, 5, 6—see Annex

| COMPUTATION SHEET FOR SHEET RESISTANCE MEASUREMENT |                    |               |               |       |                 |
|--|--------------------|---------------|---------------|-------|-----------------|
| SPECIMEN   |                    |               | TYPE:         |       |                 |
| Run No.  | $R_f, \Omega$      | $R_r, \Omega$ | $R_m, \Omega$ | $p/n$ | $n/p$           |
| 1  |                    |               |               |       | $\bar{S}$<br>cm |
| 2  |                    |               |               |       | $D$<br>cm       |
| 3  |                    |               |               |       | $S/D$<br>cm     |
| 4  |                    |               |               |       | $F_{sp}$        |
| 5  |                    |               |               |       | $F_2$           |
|  |                    |               |               |       | $F$             |
|  | $(\bar{R}_s(T)) =$ |               |               |       |                 |
|  | $S =$              |               |               |       |                 |

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

**ASTM F 374**

**TABLE 3 Correction Factor  $F_2$  as a Function of the Ratio of Average Spacing Between Probe Pins,  $S$ , to Average Specimen Diameter,  $D$**

| $\bar{S}/\bar{D}$ | $F_2$ | $\bar{S}/\bar{D}$ | $F_2$ | $\bar{S}/\bar{D}$ | $F_2$ |
|-------------------|-------|-------------------|-------|-------------------|-------|
| 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 |

A1) was  $\pm 12\%$ . For the thinnest specimen in the round robin (3), which was slightly thinner than covered by the scope of this method, the estimate of this reproducibility was  $\pm 30\%$ . The remaining specimen in the round robin (1) was only measured by three laboratories before breakage, and a final estimate of reproducibility was not made for this specimen.

NOTE 17—The test conditions for layers thicker than  $3\ \mu\text{m}$  were developed, and the related round robin was run, approximately 4 years before the corresponding work for layers thinner than  $3\ \mu\text{m}$ . Despite the greater technical difficulty in measuring the thinner layers, somewhat better multilaboratory reproducibility was found for them than for the thicker layers. It is expected, but has not been tested by a round robin, that the use of the blunter probes required for measuring layers thinner than  $3\ \mu\text{m}$  would improve measurement reproducibility for measurements thicker than  $3\ \mu\text{m}$ .

15.2 *Specimen Layers Having Thickness  $3\ \mu\text{m}$  or Less*—Measured with blunt probe tips and 0.3 to 0.8-N (30 to 80-gf), inclusive, probe force:

15.2.1 The precision of this test method as applied to layers less than  $3\ \mu\text{m}$  thick was determined by round-robin measurements on three replicate sets of specimens, each set consisting of three *p*-type ion-implanted layers, three *n*-type ion-implanted layers and one each *p*-type and *n*-type epitaxial layer. In each case, the thin films were fabricated on silicon

substrates of opposite conductivity type. The sheet resistances of the specimens in each set ranged between  $200\ \Omega/\square$  and  $5000\ \Omega/\square$ , and layer thicknesses were between 0.2 and  $1.5\ \mu\text{m}$ . Laboratories were divided into three groups; one of the replicate sets was assigned to each group. Details of the specimens, number of laboratories in each group, and analysis of data are given in Annex A2.

15.2.2 The precision of this test method for layers less than  $3\ \mu\text{m}$  thick is given in terms of the 95 % confidence estimate (R2S) for the reproducibility of measurement averages from two laboratories. Based on analysis of the multilaboratory round robin, a conservative estimate of this reproducibility is  $\pm 10\%$ .

NOTE 18—A noticeably smaller value ( $\pm 3\%$ ) for this estimate was obtained from the three specimens with lowest sheet resistance ( $<600\ \Omega/\square$ ). However, layers with combinations of layer thickness and resistivity depth profile different from those tested may also have sheet resistance values less than  $600\ \Omega$  and yet not be measurable with this same low (3 %) reproducibility interval.

15.3 *Bias*—The bias of this test method cannot be evaluated because there are no available reference standards suitable for evaluating bias.

**ANNEXES**

**(Mandatory Information)**

**A1. RESULTS OF ROUND ROBIN—SPECIMEN LAYERS HAVING THICKNESS GREATER THAN  $3\ \mu\text{m}$**

A1.1 This round robin comprised two parts. The second part was necessitated by experimental difficulties in the first part of the round robin.

A1.2 *Part 1:*

A1.2.1 The first part of this round robin included three *p*-type epitaxial layers and three *n*-type epitaxial layers each on opposite conductivity-type substrates. Analog test circuits with 2, 100, and  $1\ 000\ \Omega$  resistance for the principal resistor were included with the specimens as were a set of gram loading springs adjusted approximately to the load required by the method of test. Each participant was requested to mount these springs in his four-probe head and to make a final adjustment to obtain the exact gram loading required. Nine laboratories were to have participated in this part of the round robin.

A1.2.2 The original three *n*-type epitaxial specimens which were measured by Laboratory 1 were found not to be satisfac-

tory for continued use in the round robin; they were therefore replaced before measurement by subsequent laboratories; the data for *n/p* specimens are therefore not listed for Laboratory 1 in Table A1.1. The round robin then proceeded; Specimen 1 was found to be broken when received by Laboratory 4 but it was not replaced at that time. When the specimens were received by Laboratory 8, Specimen 3 was also found to be broken and the included set of probe springs was found to be the wrong set for use in this round robin. The round robin was halted at this time.

A1.3 *Part 2:*

A1.3.1 Reasonably similar specimens called 1A and 3A were substituted for both the broken Specimens 1 and 3, and the round robin was resumed. Because of possible errors when using interchangeable probe loading springs, a complete four-probe head preadjusted to the proper gram loading was

**ASTM F 374**

**TABLE A1.1 Round Robin Part 1: Mean Sheet Resistance ( $\Omega$ ) and Sample Standard Deviation (%)<sup>A</sup> for Epitaxial Specimens**

| Specimen Type            | 1                                     | 2                    | 3                     | 4                    | 5                    | 6                   | 2 $\Omega$ analog                             | 100 $\Omega$ analog                          | 1000 $\Omega$ analog                          |
|--------------------------|---------------------------------------|----------------------|-----------------------|----------------------|----------------------|---------------------|---|--|---|
| Thickness, $\mu\text{m}$ | 10.7                                  | 13.2                 | 2.3                   | 12.1                 | 7.8                  | 16.8                |   |  |   |
| Laboratory               |                                       |                      |                       |                      |                      |                     |   |  |   |
| 1                        | 15.131<br><i>0.61</i>                 | 640<br><i>1.95</i>   | 386.9<br><i>20.54</i> | did not measure same |                      |                     | 2.0370 <sup>B</sup><br><i>0.002</i>           | 100.26<br><i>0.00</i>                        | 1002.51 <sup>B</sup><br><i>0.01</i>           |
| 2                        | 14.963<br><i>0.44</i>                 | 635<br><i>0.31</i>   | 404.8<br><i>0.47</i>  | 1051<br><i>0.15</i>  | 188<br><i>0.05</i>   | 4424<br><i>0.19</i> | 2.010<br><i>0.01</i>                          | 100.0<br><i>0.07</i>                         | 1000.0<br><i>0.03</i>                         |
| 3                        | 15.109<br><i>3.49</i><br><sub>c</sub> | 620<br><i>2.14</i>   | 374.8<br><i>2.31</i>  | 1093<br><i>0.03</i>  | 950<br><i>0.00</i>   | 4620<br><i>0.34</i> | 2.002<br><i>0.00</i>                          | 100.1<br><i>0.00</i>                         | 1013.4 <sup>B</sup><br><i>0.05</i>            |
| 4                        |                                       | 645<br><i>3.11</i>   | 350.9<br><i>10.38</i> | 1095<br><i>0.75</i>  | 190.4<br><i>0.24</i> | 4543<br><i>0.23</i> | 2.037 <sup>B</sup><br><i>0.71<sup>B</sup></i> | 99.2 <sup>B</sup><br><i>1.50<sup>B</sup></i> | 993.0 <sup>B</sup><br><i>1.84<sup>B</sup></i> |
| 5                        |                                       | 687<br><i>0.70</i>   | 500.4<br><i>1.45</i>  | 1110<br><i>0.10</i>  | 192.9<br><i>0.11</i> | 4739<br><i>0.03</i> | 2.012 <sup>B</sup><br><i>0.04</i>             | 99.9<br><i>0.20</i>                          | 1000.0<br><i>0.01</i>                         |
| 6                        |                                       | 694.1<br><i>1.38</i> | 458.3<br><i>6.08</i>  | 1114<br><i>0.74</i>  | 189.5<br><i>0.16</i> | 4636<br><i>0.15</i> | 1.9996 <sup>B</sup><br><i>0.01</i>            | 100.04<br><i>0.05</i>                        | 1000.08<br><i>0.01</i>                        |
| 7                        |                                       | 693.2<br><i>1.73</i> | 586.5<br><i>14.29</i> | 1107<br><i>0.10</i>  | 185.8<br><i>0.09</i> | 4670<br><i>0.28</i> | 1.9999 <sup>B</sup><br><i>0.004</i>           | 100.01<br><i>0.01</i>                        | 1000.07<br><i>0.003</i>                       |

<sup>A</sup> Sample standard deviation is shown in italics.

<sup>B</sup> Fails to meet criteria of this method when measuring analog resistor circuits.

<sup>C</sup> Specimen visibly cracked at this point.

included with the specimens, and each participant was requested to use the complete four-probe head. Laboratory numbering was consecutive to that of the first part. Only Laboratories 1, 2, 4, and 5 participated in both parts of the round robin; Laboratories 8 and 9 which had not yet participated in Part 1, and two additional laboratories, 10 and 11, constituted the rest of Part 2.

A1.3.2 All specimens, analog boxes, and probe head were returned to the supervising laboratory after measurement by each of the participants. At that time each specimen was visually inspected for abnormal surface damage; probe impression quality was checked and proper probe loading was verified. Specimen 2 was broken when returned from Laboratory 8. Specimen 1A, which exhibited erratic behavior for Laboratories 9 and 10, could not even be measured by Laboratory 11 and was found to be cracked after remeasure-

ment by the supervising laboratory at the end of the round robin. The possibility then exists that Wafer 1A was cracked at the same time as Wafer 2, that is, upon receipt by Laboratory 9.

A1.4 This round robin was originally intended to measure epitaxial layer resistivity. During the course of the experiment it was decided that due to uncertainties in measuring epitaxial thickness due to unknown impurity profiles the preferred referee measurement for epitaxial layers is sheet resistance, not resistivity. Current levels for measurement were chosen by use of the original specimen supplier's value for its thickness and resistivity.

A1.5 The data from each of the laboratories are summarized in Table A1.1 and Table A1.2. Table A1.3 is a tabulation of multilaboratory precision and the reproducibility interval for

**TABLE A1.2 Round Robin Part 2: Mean Sheet Resistance ( $\Omega$ ) and Sample Standard Deviation (%)<sup>A</sup> for Epitaxial Specimens**

| Specimen Type            | 1A                      | 2                                    | 3A                   | 4                     | 5                     | 6                   | 2 $\Omega$ analog                  | 100 $\Omega$ analog              | 1000 $\Omega$ analog                          | Probe Spacing Data        |
|--------------------------|-------------------------|--------------------------------------|----------------------|-----------------------|-----------------------|---------------------|------------------------------------|----------------------------------|---|---------------------------|
| Thickness, $\mu\text{m}$ | 12.45                   | 13.2                                 | 3.3                  | 12.1                  | 7.8                   | 16.8                |                                    |                                  |   | s FSp                     |
| Laboratory               |                         |                                      |                      |                       |                       |                     |                                    |                                  |   |                           |
| 2                        | 10.646<br><i>0.28</i>   | 674.1<br><i>0.28</i>                 | 1356<br><i>0.15</i>  | 1109.5<br><i>0.15</i> | 191.86<br><i>0.21</i> | 4658<br><i>0.30</i> | 2.001<br><i>0.03</i>               | 100.02<br><i>0.09</i>            | 999.8<br><i>0.04</i>                          | 0.15894<br><i>1.00034</i> |
| 1                        | 10.440<br><i>5.53</i>   | 674.6<br><i>0.74</i>                 | 1389<br><i>0.23</i>  | 1111.2<br><i>0.29</i> | 191.73<br><i>0.05</i> | 4564<br><i>1.98</i> | 2.02 <sup>B</sup><br><i>0.00</i>   | 100.00<br><i>0.01</i>            | 1000.4<br><i>0.01</i>                         | 0.15876<br><i>0.9989</i>  |
| 4                        | 10.233<br><i>1.57</i>   | 685.1<br><i>2.89</i>                 | 1392<br><i>2.77</i>  | 1114.4<br><i>0.49</i> | 192.3<br><i>0.34</i>  | 4679<br><i>1.37</i> | 2.011<br><i>0.03</i>               | 99.99<br><i>0.57<sup>B</sup></i> | 993.2 <sup>B</sup><br><i>3.73<sup>B</sup></i> | 0.15898<br><i>0.9995</i>  |
| 5                        | 11.186<br><i>0.79</i>   | 637.8<br><i>2.00</i>                 | 1364<br><i>0.96</i>  | 1148.8<br><i>0.85</i> | 193.11<br><i>0.13</i> | 4673<br><i>0.25</i> | 2.011<br><i>0.10</i>               | 100.00<br><i>0.02</i>            | 1000.01<br><i>0.01</i>                        |                           |
| 8                        | 10.921<br><i>0.82</i>   | 688.9<br><i>1.23</i><br><sub>c</sub> | 1283<br><i>2.56</i>  | 1046.3<br><i>0.49</i> | 190.4<br><i>0.22</i>  | 4578<br><i>0.22</i> | 1.9992 <sup>B</sup><br><i>0.04</i> | 100.01<br><i>0.03</i>            | 1000.36<br><i>0.02</i>                        | 0.15895<br><i>0.9987</i>  |
| 9                        | 103.680<br><i>43.14</i> |                                      | 723.9<br><i>4.52</i> | 1110.7<br><i>0.51</i> | 183.2<br><i>1.17</i>  | 4579<br><i>0.65</i> | 2.0002<br><i>0.02</i>              | 100.00<br><i>0.01</i>            | 999.80<br><i>0.005</i>                        | 0.15914<br><i>1.0016</i>  |
| 10                       | 8.704<br><i>5.61</i>    |                                      | 1212<br><i>3.8</i>   | 1058<br><i>1.66</i>   | 168<br><i>1.15</i>    | 4130<br><i>1.97</i> | did not measure                    |                                  |   | 0.15908<br><i>1.0024</i>  |
| 11                       | nodata<br><sub>c</sub>  |                                      | 1397<br><i>0.12</i>  | 1109<br><i>0.07</i>   | 192<br><i>0.00</i>    | 4680<br><i>0.29</i> | 2.011<br><i>0.26<sup>B</sup></i>   | 100.0<br><i>0.05</i>             | 1000.2<br><i>0.04</i>                         |                           |

<sup>A</sup> Sample standard deviation is shown in italics.

<sup>B</sup> Fails to meet criteria of this method when measuring analog resistor circuits.

<sup>C</sup> Specimen visibly cracked at this point.

**ASTM F 374**

**TABLE A1.3 Summary of Results—Epitaxial Specimens Thicker Than 3  $\mu\text{m}$**

| Specimen                               | 1              | 1A       | 2        | 3        | 3A       | 4        | 5        | 6        |
|--|----------------|----------|----------|----------|----------|----------|----------|----------|
| Layer type                             | <i>p</i>       | <i>p</i> | <i>p</i> | <i>p</i> | <i>p</i> | <i>n</i> | <i>n</i> | <i>n</i> |
| Substrate type                         | <i>n</i>       | <i>n</i> | <i>n</i> | <i>n</i> | <i>n</i> | <i>p</i> | <i>p</i> | <i>p</i> |
| Nominal layer thickness, $\mu\text{m}$ | 10.7           | 12.5     | 13.2     | 2.3      | 3.3      | 12.1     | 7.8      | 16.8     |
| Nominal sheet resistance, $\Omega$     | 15             | 10       | 680      | 400      | 1400     | 1100     | 190      | 4600     |
| $n^A$                                  | 1              | 5        | 8        | 3        | 5        | 10       | 10       | 10       |
| $S, \%^B$                              | not applicable | 3.2      | 3.6      | 10.5     | 3.8      | 2.7      | 1.5      | 1.9      |
| $\bar{s}, \%^C$                        | 0.44           | 1.6      | 1.0      | 2.7      | 0.9      | 0.4      | 0.2      | 0.4      |
| Reproducibility interval, $\%^D$       | not applicable | 8.9      | 10       | 29       | 10.5     | 7.5      | 4.2      | 5.3      |

<sup>A</sup>Number of laboratories that passed analog circuit test requirements (see 11.2.3) and were within  $\pm 3 \sigma$  of multilaboratory mean (untrimmed).

<sup>B</sup>Multilaboratory relative standard deviation after trimming to meet condition above.

<sup>C</sup>Average single laboratory relative standard deviation.

<sup>D</sup>95 % confidence interval for difference in measurement averages, divided by average value.

each of the specimens after rejecting data from laboratories that failed the analog circuit test or that were significant outliers. Where laboratories participated in both parts of the round robin, their data were treated as though from different laboratories (see Table A1.4).

NOTE A1.1—The use of the reproducibility interval to characterize multilaboratory results was developed after the round robin for layers thicker than 3  $\mu\text{m}$  was originally analyzed and reported. The results are restated here in terms of the reproducibility interval because of its practical interpretation for referee measurement usage. For a discussion of the meaning of the reproducibility interval see A3.4 in Annex A2.

**TABLE A1.4 Epitaxial Resistance Round Robin Analog Circuit Measurements**

|                        | Analog #1 | Analog #2 | Analog #3 |
|------------------------|-----------|-----------|-----------|
| $n_1^A$                | 15        | 15        | 15        |
| Resistance, $\Omega^B$ | 2.011     | 99.97     | 1000.2    |
| Resistance $^C$        | 1.89      | 1.06      | 2.04      |
| Range (%)              |           |           |           |
| $n_2^D$                | 7         | 13        | 11        |
| Resistance, $\Omega^E$ | 2.007     | 100.04    | 1000.07   |
| Resistance $^F$        | 0.45      | 0.36      | 0.06      |
| Range (%)              |           |           |           |

<sup>A</sup>Total number of measurements in both parts of round robin.

<sup>B</sup>Average of all measurements on each analog circuit.

<sup>C</sup>Range of values for all measurements on each analog divided by its average value.

<sup>D</sup>Number of laboratories whose analog circuit measurements passed requirements of 10.2.3.

<sup>E</sup>Averages after omitting laboratories which failed 10.2.3.

<sup>F</sup>Range of values (%) resulting from omitting these laboratories.

**A2. RESULTS OF ROUND ROBIN—SPECIMEN LAYERS HAVING THICKNESS LESS THAN 3  $\mu\text{m}$**

A2.1 This round robin used three sets of nearly replicate specimens. Each set consisted of three *p*-type ion implanted layers, three *n*-type ion-implanted layers, and one each *p*-type and *n*-type epitaxial layer. All layers were fabricated on silicon substrates of opposite conductivity type. The sheet resistances of the layers were in the range 200  $\Omega/\square$  to 5000  $\Omega/\square$ , and the thicknesses of the layers were in the range 0.2 to 1.5  $\mu\text{m}$ . Specific description of the layers is given in the heading of

Table A2.1 which summarizes all data taken. One set of specimens was assigned to each group of participating laboratories. Each laboratory group consisted of four laboratories. The coordinating laboratory, which was designated a formal participant of laboratory group I, also took data on the specimen sets for laboratory groups II and III. These data were also used in the round-robin analysis, as described in A2.2. No analog boxes were used in the round robin. Each laboratory



**TABLE A2.1 Summary of Round-Robin Data—Average Measured Sheet Resistance,  $\Omega$ , and Standard Deviation as Percent of Average Value**

| Laboratory Number and Probe Description                                   | Specimen   |  |  |  |  |  |  |  |
|---|--|--|--|--|--|--|--|--|
|   | 1<br><i>n</i> -implant<br>$X_j = 1$<br>$\mu\text{m}$ | 2<br><i>n</i> -implant<br>$X_j = 0.2$<br>$\mu\text{m}$ | 3<br><i>n</i> -implant<br>$X_j = 0.2$<br>$\mu\text{m}$ | 4<br><i>p</i> -implant<br>$X_j = 1$<br>$\mu\text{m}$ | 5<br><i>p</i> -implant<br>$X_j = 0.2$<br>$\mu\text{m}$ | 6<br><i>p</i> -implant<br>$X_j = 0.2$<br>$\mu\text{m}$ | 7<br><i>p</i> -epitaxy<br>$X_j = 1.5$<br>$\mu\text{m}$ | 8<br><i>n</i> -epitaxy<br>$X_j = 1.5$<br>$\mu\text{m}$ |
| (a) Laboratory Group 1  |  |  |  |  |  |  |  |  |
| Laboratory 1<br>$\bar{S} = 1.59$ mm<br>100- $\mu\text{m}$ flat tip, 30 g  | 551.7 $\Omega$<br>$\pm 0.2$ %                        | 1022 $\Omega$<br>$\pm 0.6$ %                           | 343.8 $\Omega$<br>$\pm 0.1$ %                          | 694.9 $\Omega$<br>$\pm 0.2$ %                        | 1060 $\Omega$<br>$\pm 0.2$ %                           | 275.0 $\Omega$<br>$\pm 0.1$ %                          | 3371 $\Omega$<br>$\pm 0.2$ %                           | 4135 $\Omega$<br>$\pm 0.1$ %                           |
| Laboratory 2<br>$\bar{S} = 1.59$ mm<br>100- $\mu\text{m}$ round tip, 30 g | 558.3 $\Omega$<br>$\pm 0.7$ %                        | 1042 $\Omega$<br>$\pm 0.5$ %                           | 347.5 $\Omega$<br>$\pm 0.4$ %                          | 705.3 $\Omega$<br>$\pm 0.3$ %                        | 1004 $\Omega$<br>$\pm 1.1$ %                           | 277.9 $\Omega$<br>$\pm 0.7$ %                          | 3258 $\Omega$<br>$\pm 0.3$ %                           | 4209 $\Omega$<br>$\pm 2.4$ %                           |
| Laboratory 3<br>$\bar{S} = 1.02$ mm<br>100- $\mu\text{m}$ round tip, 45 g | 554.3 $\Omega$<br>$\pm 0.2$ %                        | 1101 $\Omega$<br>$\pm 5.1$ %                           | 343.8 $\Omega$<br>$\pm 0.5$ %                          | 705.1 $\Omega$<br>$\pm 0.2$ %                        | 1088 $\Omega$<br>$\pm 0.4$ %                           | 280.4 $\Omega$<br>$\pm 0.4$ %                          | 3283 $\Omega$<br>$\pm 0.9$ %                           | 4397 $\Omega$<br>$\pm 1.6$ %                           |
| Laboratory 3<br>repeat  | 558.0 $\Omega$<br>$\pm 0.3$ %                        | 1097 $\Omega$<br>$\pm 3.2$ %                           | 343.8 $\Omega$<br>$\pm 1.2$ %                          | 713.2 $\Omega$<br>$\pm 0.3$ %                        | 1090 $\Omega$<br>$\pm 0.2$ %                           | 280.8 $\Omega$<br>$\pm 0.3$ %                          | 3362 $\Omega$<br>$\pm 0.8$ %                           | 4360 $\Omega$<br>$\pm 0.6$ %                           |
| Laboratory 4<br>$\bar{S} = 1.02$ mm<br>100- $\mu\text{m}$ round tip, 50 g | 560.3 $\Omega$<br>$\pm 0.5$ %                        | 989.2 $\Omega$<br>$\pm 10.7$ %                         | 343.4 $\Omega$<br>$\pm 0.3$ %                          | 714.7 $\Omega$<br>$\pm 0.5$ %                        | 1101 $\Omega$<br>$\pm 0.4$ %                           | 280.4 $\Omega$<br>$\pm 0.2$ %                          | 3361 $\Omega$<br>$\pm 0.6$ %                           | 4291 $\Omega$<br>$\pm 1.6$ %                           |
| Laboratory 4<br>repeat  | 558.7 $\Omega$<br>$\pm 0.2$ %                        | 998.2 $\Omega$<br>$\pm 6.2$ %                          | 342.8 $\Omega$<br>$\pm 0.3$ %                          | 713.8 $\Omega$<br>$\pm 0.2$ %                        | 1098 $\Omega$<br>$\pm 0.5$ %                           | 280.7 $\Omega$<br>$\pm 0.2$ %                          | 3371 $\Omega$<br>$\pm 0.5$ %                           | 4259 $\Omega$<br>$\pm 1.6$ %                           |
| (b) Laboratory Group II   |  |  |  |  |  |  |  |  |
| Laboratory 1<br>$\bar{S} = 1.59$ mm<br>100- $\mu\text{m}$ flat tip, 30 g  | 531.7 $\Omega$<br>$\pm 0.1$ %                        | 1084 $\Omega$<br>$\pm 0.2$ %                           | 349.5 $\Omega$<br>$\pm 0.1$ %                          | 714.0 $\Omega$<br>$\pm 0.2$ %                        | 1070 $\Omega$<br>$\pm 0.4$ %                           | 274.5 $\Omega$<br>$\pm 0.1$ %                          | 3368 $\Omega$<br>$\pm 0.2$ %                           | 3821 $\Omega$<br>$\pm 0.2$ %                           |
| Laboratory 2<br>$\bar{S} = 1.59$ mm<br>125- $\mu\text{m}$ round tip, 70 g | 529.5 $\Omega$<br>$\pm 0.1$ %                        | 1092 $\Omega$<br>$\pm 0.7$ %                           | 347.2 $\Omega$<br>$\pm 0.4$ %                          | 703.3 $\Omega$<br>$\pm 0.2$ %                        | 1053 $\Omega$<br>$\pm 0.6$ %                           | 271.5 $\Omega$<br>$\pm 0.1$ %                          | 3272 $\Omega$<br>$\pm 0.3$ %                           | 3906 $\Omega$<br>$\pm 1.3$ %                           |
| Laboratory 2<br>repeat  | 529.9 $\Omega$<br>$\pm 0.2$ %                        | 1098 $\Omega$<br>$\pm 0.3$ %                           | 347.2 $\Omega$<br>$\pm 0.3$ %                          | 705.4 $\Omega$<br>$\pm 0.2$ %                        | 1056 $\Omega$<br>$\pm 1.1$ %                           | 272.0 $\Omega$<br>$\pm 0.4$ %                          | 3267 $\Omega$<br>$\pm 0.4$ %                           | 3886 $\Omega$<br>$\pm 0.8$ %                           |
| Laboratory 2<br>repeat  | 529.7 $\Omega$<br>$\pm 0.1$ %                        | 1101 $\Omega$<br>$\pm 0.5$ %                           | 347.8 $\Omega$<br>$\pm 0.4$ %                          | 710.7 $\Omega$<br>$\pm 0.3$ %                        | 1081 $\Omega$<br>$\pm 0.5$ %                           | 274.5 $\Omega$<br>$\pm 0.2$ %                          | 3301 $\Omega$<br>$\pm 0.5$ %                           | 3895 $\Omega$<br>$\pm 1.1$ %                           |
| Laboratory 3<br>$\bar{S} = 1.27$ mm<br>100- $\mu\text{m}$ round tip, 40 g | 536.8 $\Omega$<br>$\pm 0.2$ %                        | 1121 $\Omega$<br>$\pm 0.5$ %                           | 351.8 $\Omega$<br>$\pm 0.5$ %                          | 718.6 $\Omega$<br>$\pm 0.4$ %                        | 1115 $\Omega$<br>$\pm 0.5$ %                           | 278.5 $\Omega$<br>$\pm 0.3$ %                          | 3321 $\Omega$<br>$\pm 0.9$ %                           | 3934 $\Omega$<br>$\pm 0.5$ %                           |
| Laboratory 3<br>repeat  | 531.6 $\Omega$<br>$\pm 0.2$ %                        | 1116 $\Omega$<br>$\pm 1.0$ %                           | 353.7 $\Omega$<br>$\pm 1.7$ %                          | 716.2 $\Omega$<br>$\pm 0.5$ %                        | 1126 $\Omega$<br>$\pm 0.3$ %                           | 279.5 $\Omega$<br>$\pm 0.4$ %                          | 3297 $\Omega$<br>$\pm 0.6$ %                           | 3863 $\Omega$<br>$\pm 0.8$ %                           |
| Laboratory 4<br>$\bar{S} = 76$ mm<br>250- $\mu\text{m}$ round tip, 80 g   | 544.5 $\Omega$<br>$\pm 0.4$ %                        | 1139 $\Omega$<br>$\pm 0.5$ %                           | 356.9 $\Omega$<br>$\pm 0.7$ %                          | 734.4 $\Omega$<br>$\pm 0.6$ %                        | 1125 $\Omega$<br>$\pm 0.6$ %                           | 282.4 $\Omega$<br>$\pm 0.5$ %                          | 3520 $\Omega$<br>$\pm 1.8$ %                           | 4072 $\Omega$<br>$\pm 1.7$ %                           |
| (c) Laboratory Group III  |  |  |  |  |  |  |  |  |
| Laboratory 1<br>$\bar{S} = 1.59$ mm<br>100- $\mu\text{m}$ flat tip, 30 g  | 528.2 $\Omega$<br>$\pm 0.1$ %                        | 1052 $\Omega$<br>$\pm 0.2$ %                           | 337.1 $\Omega$<br>$\pm 0.1$ %                          | 700.6 $\Omega$<br>$\pm 0.1$ %                        | 1053 $\Omega$<br>$\pm 0.2$ %                           | 275.5 $\Omega$<br>$\pm 0.1$ %                          | 3117 $\Omega$<br>$\pm 0.2$ %                           | 5195 $\Omega$<br>$\pm 0.2$ %                           |
| Laboratory 2<br>$\bar{S} = 1.59$ mm<br>100- $\mu\text{m}$ round tip, 50 g | 541.8 $\Omega$<br>$\pm 0.3$ %                        | 1079 $\Omega$<br>$\pm 0.2$ %                           | 341.0 $\Omega$<br>$\pm 0.5$ %                          | 721.6 $\Omega$<br>$\pm 0.4$ %                        | 1082 $\Omega$<br>$\pm 0.5$ %                           | 280.8 $\Omega$<br>$\pm 0.1$ %                          | 2982 $\Omega$<br>$\pm 0.8$ %                           | 5275 $\Omega$<br>$\pm 0.9$ %                           |
| Laboratory 3<br>$\bar{S} = 0.64$ mm<br>100- $\mu\text{m}$ round tip, 50 g | 541.4 $\Omega$<br>$\pm 0.8$ %                        | 1076 $\Omega$<br>$\pm 5.0$ %                           | 336.8 $\Omega$<br>$\pm 0.8$ %                          | 772.1 $\Omega$<br>$\pm 2.7$ %                        | 1152 $\Omega$<br>$\pm 2.7$ %                           | 278.7 $\Omega$<br>$\pm 0.7$ %                          | 2957 $\Omega$<br>$\pm 1.5$ %                           | 4905 $\Omega$<br>$\pm 4.4$ %                           |
| Laboratory 3<br>repeat  | 539.5 $\Omega$<br>$\pm 0.9$ %                        | 1062 $\Omega$<br>$\pm 2.1$ %                           | 340.4 $\Omega$<br>$\pm 1.3$ %                          | 747.1 $\Omega$<br>$\pm 2.2$ %                        | 1127 $\Omega$<br>$\pm 1.5$ %                           | 280.9 $\Omega$<br>$\pm 1.7$ %                          | 2926 $\Omega$<br>$\pm 5.6$ %                           | 4834 $\Omega$<br>$\pm 5.2$ %                           |
| Laboratory 4<br>$\bar{S} = 1.59$ mm<br>100- $\mu\text{m}$ round tip, 60 g | 543.1 $\Omega$<br>$\pm 0.7$ %                        | 974 $\Omega$<br>$\pm 3.5$ %                            | 339.2 $\Omega$<br>$\pm 1.0$ %                          | 714.1 $\Omega$<br>$\pm 1.2$ %                        | 938 $\Omega$<br>$\pm 2.5$ %                            | 277.5 $\Omega$<br>$\pm 0.4$ %                          | 2900 $\Omega$<br>$\pm 3.9$ %                           | 2992 $\Omega$<br>$\pm 18.2$ %                          |
| Laboratory 5<br>$\bar{S} = 1.59$ mm<br>250- $\mu\text{m}$ round tip, 80 g | 536.5 $\Omega$<br>$\pm 2.0$ %                        | 1024 $\Omega$<br>$\pm 4.7$ %                           | 337.1 $\Omega$<br>$\pm 1.7$ %                          | 713.0 $\Omega$<br>$\pm 1.5$ %                        | 903 $\Omega$<br>$\pm 1.4$ %                            | 274.9 $\Omega$<br>$\pm 0.6$ %                          | 2749 $\Omega$<br>$\pm 5.9$ %                           | 3912 $\Omega$<br>$\pm 10.8$ %                          |
| Laboratory 5<br>repeat  | 539.1 $\Omega$<br>$\pm 1.2$ %                        | 963 $\Omega$<br>$\pm 10.0$ %                           | 338.9 $\Omega$<br>$\pm 1.1$ %                          | 722.6 $\Omega$<br>$\pm 1.3$ %                        | 877 $\Omega$<br>$\pm 1.1$ %                            | 274.0 $\Omega$<br>$\pm 0.4$ %                          | 2774 $\Omega$<br>$\pm 6.6$ %                           | 4087 $\Omega$<br>$\pm 7.8$ %                           |

was required to take six measurements on each specimen in accordance with 2.4 to 12.8. Some laboratories remeasured all specimens in their set as noted in Table A2.1. Such repeat measurements are entered separately in the analysis.

A2.2 Each laboratory provided its own probe for this round robin. However, not all laboratories used probes with the

spacing (1.59 mm) specified in 7.3.4. Since no consistent bias could be found for any of these laboratories that might be related to errors in the probe-spacing correction factor (see Note 5 and 11.1.2.5), no laboratory's data were excluded from analysis on account of probe spacing. However, certain data were omitted from the final analysis. Those data and the

**ASTM F 374**

reasons for the omission are as follows: (1) Data for specimen 2 as obtained from Laboratories 3 and 4 in Group I were omitted because they were obtained after a large scratch or hairline crack across the central portion of this specimen had been identified by the coordinating laboratory. This specimen damage was believed to be the cause of the degraded measurement results from Laboratories 3 and 4; (2) all data from Laboratory Group III were omitted because of clear operational failures on the parts of three of the laboratories in this group, as follows: Laboratory 3 reported measurements for four of the specimens that had differences between forward and reverse-current readings in excess of 10 %; Laboratory 4 was found to have used a probe that was too sharp or too heavily loaded to meet the probe specifications; Laboratory 5 was found to have used a probe stand that caused extensive probe skidding.

**A2.3 Analysis of Data for Components of Variance:**

A2.3.1 The model used for analysis of data from each specimen in each group is:

$$Y_{ijk} = \mu + \beta_i + \gamma_{ij} + \epsilon_{ijk}$$

where:

- $Y_{ijk}$  = the  $k$ th measurement in run  $j$  at laboratory  $i$ ,
- $\mu$  = the overall mean resistance of the specimen as measured by the group of laboratories,
- $\beta_i$  = the random effect associated with laboratory  $i$ ,
- $\gamma_{ij}$  = the random effect associated with run  $j$  at laboratory  $i$ , and
- $\epsilon_{ijk}$  = the random effect associated with a given measurement of the set of six in any run.

A2.3.2 The variance,  $\sigma_{tot}^2$ , of a single measurement for a given specimen, as estimated from this model, is:

$$\sigma_{tot}^2 = \text{variance of } Y_{ijk} = \sigma_L^2 + \sigma_R^2 + \sigma_W^2$$

where:

- $\sigma_L^2$  = the variance between laboratories,
- $\sigma_R^2$  = the variance between runs within a given laboratory, and
- $\sigma_W^2$  = the variance within measurements of a given run.

A2.3.3 The precision of this method for referee purposes is based on the variance of measurement averages. From the sets of six measurements taken on each specimen during the round robin, an estimate of the variance  $\sigma^2$  of measurement averages is:

$$\sigma^2 = \text{variance of } \frac{1}{6} \left( \sum_{k=1}^6 Y_{ijk} \right) = \sigma_L^2 + \sigma_R^2 + \frac{\sigma_W^2}{6}$$

where:

$$\frac{1}{6} \left( \sum_{k=1}^6 Y_{ijk} \right) = \text{the average of 6 measurements at laboratory } i \text{ during run } j.$$

A2.3.4 Values of  $\sigma_L$ ,  $\sigma_R$ ,  $\sigma_W$ ,  $\sigma_{tot}$ , and  $\sigma$  are given for each specimen and each laboratory group in Table A2.2. These results are expressed as a percent of the mean resistance  $\mu$  for each specimen in each group.

**A2.4 Reproducibility Interval for Measurement Averages:**

A2.4.1 The variance of the difference in measurement average from two laboratories is found from propagation of variance to be  $2\sigma^2$ , where  $\sigma$  is given in A2.3.3; the standard deviation for this difference in measurement averages is  $\sqrt{2}\sigma$ .

A2.4.2 The 95 % confidence interval for the difference in measurement averages from two different laboratories is called the reproducibility interval,  $I(R)$ , and is given by:

$$I(R) = 1.96\sqrt{2}\sigma = 2.77 \sqrt{\sigma_L^2 + \sigma_R^2 + (\sigma_W^2)/6}$$

A2.4.3 Values for the reproducibility interval of measurement averages based on data sets that are trimmed as noted in A2.2 are given for each specimen in Table A2.3.

**A2.5 Pooling Estimates from the Two Laboratory Groups:**

A2.5.1 Sufficient degrees of freedom result from the total number of measurements available in each specimen category that normal distribution statistics are assumed; numerical factors related to t-distribution statistics are ignored.

A2.5.2 A pooled estimate of the reproducibility interval for measurement averages is obtained from a weighted pooling of the values of  $\sigma^2$  from each laboratory group for that specimen. The weighting factor is the number of degrees of freedom in the calculation of between-laboratory variance for the particular group. For all specimens except No. 2:

$$I(R)_{pooled} = 2.77\sigma_{pooled} = 2.77 \left( \frac{3\sigma_I^2 + 3\sigma_{II}^2}{6} \right)^{1/2}$$

where there are 3 degrees of freedom for laboratory groups I and II. For specimen 2, only two laboratories were available in group I to estimate the between-laboratory variance; hence there was only one degree of freedom for this group, and

**TABLE A2.2 Summary of Components of Variance for Each Specimen and Laboratory Group, Listed in Order of Increasing Specimen Resistance on Trimmed Data Set<sup>A,B</sup>**

| Error Component | Laboratory Group | Specimen Number |      |      |      |      |      |      |      |
|-----------------|------------------|-----------------|------|------|------|------|------|------|------|
|                 |                  | 6               | 3    | 1    | 4    | 5    | 2    | 7    | 8    |
| $\sigma_W$      | I                | 0.30            | 0.45 | 0.34 | 0.27 | 0.45 | 0.53 | 0.56 | 1.31 |
|                 | II               | 0.28            | 0.60 | 0.18 | 0.31 | 0.57 | 0.53 | 0.65 | 0.92 |
| $\sigma_R$      | I                | —               | —    | 0.32 | 0.56 | —    | —    | 1.16 | —    |
|                 | II               | 0.48            | —    | 0.39 | 0.43 | 1.18 | 0.29 | 0.40 | 0.64 |
| $\sigma_L$      | I                | 0.89            | 0.52 | 0.48 | 0.98 | 3.72 | 1.33 | 0.99 | 2.34 |
|                 | II               | 1.55            | 1.15 | 1.06 | 1.52 | 2.97 | 1.87 | 2.88 | 2.09 |
| $\sigma_{tot}$  | I                | 0.96            | 0.77 | 0.70 | 1.17 | 3.76 | 1.43 | 1.65 | 2.79 |
|                 | II               | 1.65            | 1.39 | 1.15 | 1.62 | 3.25 | 1.98 | 3.03 | 2.42 |
| $\sigma$        | I                | 0.90            | 0.55 | 0.59 | 1.13 | 3.72 | 1.35 | 1.54 | 2.40 |
|                 | II               | 1.63            | 1.18 | 1.13 | 1.58 | 3.20 | 1.90 | 2.92 | 2.22 |

<sup>A</sup> All components are expressed as a percent of specimen mean value within each group.

<sup>B</sup> Entries are missing when the value calculated is insignificant.

**ASTM F 374**

**TABLE A2.3 Reproducibility Intervals (95 % Confidence) for Measurement Averages Based on Trimmed Sets of Round-Robin Data**

|                          | Laboratory Group | Specimen Number |     |     |     |      |     |     |     |
|--------------------------|------------------|-----------------|-----|-----|-----|------|-----|-----|-----|
|                          |                  | 6               | 3   | 1   | 4   | 5    | 2   | 7   | 8   |
| Reproducibility Interval | I                | 2.5             | 1.5 | 1.6 | 3.1 | 10.3 | 3.7 | 4.3 | 6.6 |
|                          | II               | 4.5             | 3.3 | 3.1 | 4.4 | 8.9  | 5.3 | 8.1 | 6.1 |

**TABLE A2.4 Pooled Estimates of 95 % Reproducibility Interval for Measurement Averages, I(R)**

|                 | Specimen Number |       |       |       |       |       |       |       |
|-----------------|-----------------|-------|-------|-------|-------|-------|-------|-------|
|                 | 6               | 3     | 1     | 4     | 5     | 2     | 7     | 8     |
| Pooled Estimate | 3.6 %           | 2.6 % | 2.5 % | 3.8 % | 9.6 % | 5.9 % | 6.5 % | 6.4 % |

$$I(R)_{\text{pooled}} = 2.77\sigma_{\text{pooled}} = 2.77 \left( \frac{\sigma_I^2 + 3\sigma_{II}^2}{4} \right)^{1/2}$$

Table A2.4 summarizes the values of I(R) pooled for the individual specimens.

A2.6 The pooled estimates of the 95 % reproducibility interval shown in Table A2.4 are found to be noticeably larger for the specimens with sheet resistance over 1000 Ω than for the other specimens. No one value for the pooled estimate

represents all the specimens well. However, in order to allow for specimens that also fit within the scope of this method but have combinations of layer thickness and resistivity depth profile other than those tested, the final precision figure will be based on the worst case of those tested. A conservative estimate of the 95 % reproducibility interval for measurement averages is taken to be ±10 %.

*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.*

*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).*