Designation: F 374 – 00a

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 [1]

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 (ϵ) 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 µm. It can be used to measure sheet resistance in the range 10 to 5000 Ω, 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 Elecrronics and Semiconductor Industry [2]

E 1 Specification for ASTM Thermometers [3]

F 42 Test Method for Conductivity Type of Extrinsic Semiconducting Materials [4]

F 1529 Test Method for Sheet Resistance Uniformity Evaluation by In-Line Four-Point Probe with the Duel-Configuration Procedure [4]

2.2 *SEMI Standard:*

C 3.15 Specifications for Nitrogen Gas [5]

C 28 Specification for Hydrofluoric Acid [5]

C 31 Specification for Methanol [5]

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.

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[1] 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.

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

[3] *Annual Book of ASTM Standards*, Vol 14.03.

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

[5] 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 [Ω or Ω 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. [6] 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.

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, Ω	I [A]
2.0–25	10 mA
20–250	1 mA
200–2500	100 µA
2000–25 000	10 µA

[A] 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.

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[6] 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.

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 µm 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 µm, or in a flat circular truncation with a circle radius in the nominal range from 50 to 125 µm.

7.3.1.2 *For Specimen Layers Having Thickness Greater Than 3 µm*—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 µm.

7.3.2 *Probe Force*— For hemispherical-tipped probe pins with tip radius greater than 100 µm or for flat-tipped probe pins with tip radius greater than 50 µm, 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 µm, the force on each probe tip shall be 0.30 ± 0.03 N (31 ± 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–µm 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 µm.

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

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 resistane temperature detector (RTD) be made instead. An insulating disk, less than 0.076 mm thick and suitably perforated, shall be placed over the center