SEMI MF397-0812 (Reapproved 0718)
TEST METHOD FOR RESISTIVITY OF SILICON BARS USING A TWO-POINT PROBE

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1 Purpose

1.1 Control of the resistivity of semiconductor crystals is an important aspect of wafer manufacturing. Also, the resistivity of a silicon crystal is an important materials acceptance requirement.

1.2 This Test Method is recommended for material acceptance and manufacturing control of single-crystal bulk silicon. It is also applicable to other semiconductor materials but neither the appropriate conditions of measurement nor the expected precision have been experimentally determined.

2 Scope

2.1 This Test Method covers the measurement of the resistivity of single-crystal bars having cross sections that are uniform in area and square, rectangular or round in shape, and having resistivity between 0.0009 and 3000 Ω·cm.

NOTE 1: DIN 50430 is an equivalent method.

2.2 This Test Method is intended for use on single crystals of silicon of either *n*- or *p*-type for which the uniformity of the crystal cross section is such that the area can be accurately calculated. The specimen cross-sectional area shall be constant to within ±1% of the average area as determined by measurements along the crystal axis (see ¶ 12.2).

2.3 The ratio of the length to the maximum dimension of the cross section of the specimen shall not be less than 3:1 (see ¶ 12.1). The largest diameter tested by round robin was 37.5 mm (1.5 in.), and this is the largest diameter that can be measured by this method. The specimen shall normally have a surface finish of 0.4 µm (16 µin.) rms or less (see ANSI B46). Other surface finishes may be used if mutually acceptable; however, the multilaboratory precision figures of this test (see ¶ 16.1) then may no longer apply.

NOTICE: SEMI Standards and Safety Guidelines do not purport to address all safety issues associated with their use. It is the responsibility of the users of the Documents to establish appropriate safety and health practices, and determine the applicability of regulatory or other limitations prior to use.

3 Limitations

3.1 This Test Method assumes no radial resistivity variation in the crystal. If such variations are present in the crystal, this method yields an average resistivity of some unknown cross section of the crystal. In such cases, the resistivity measured on a wafer cut from the crystal may not correlate with the resistivity measured by this test method for the region about that wafer.

3.2 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.

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

3.4 Minority carrier injection during the measurement can occur due to the electric field in the specimen. With material possessing high lifetime of the majority carriers and high resistivity, such injection can significantly influence the results. Carrier injection can be detected by repeating the measurements at lower current. In the absence of injection, no change in resistivity should be observed. The current level recommended should reduce the probability of difficulty from this source to a minimum but in cases of doubt, the measurements of ¶¶ 13.3 through 13.7 should be repeated at a lower current. If the proper current is being used, doubling or halving its magnitude should not cause a change in observed resistance that is more than 0.5%. Minority carrier effects should be most pronounced near current contacts.

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3.5 Semiconductors have a significant temperature coefficient of resistivity. Consequently, the current used should be small to avoid resistive heating. 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. Resistive heating can also be detected by changing the current level as in ¶ 3.4.

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

3.7 Noncatastrophic cracks (not generally visible) or other mechanical damage can give erroneous readings. If visible cracks are seen, it may be necessary to saw the crystal into sections and measure separately.

4 Referenced Standards and Documents

4.1 *SEMI Standards and Safety Guidelines*

SEMI C19 — Specification for Acetone

SEMI C28 — Specification and Guide for Hydrofluoric Acid

SEMI C31 — Specification for Methanol

SEMI C35 — Specification and Guide for Nitric Acid

SEMI M59 — Terminology for Silicon Technology

SEMI MF42 — Test Method for Conductivity Type of Extrinsic Semiconducting Materials

4.2 *ANSI Standards*[1]

ANSI B46 — Surface Texture (Surface Roughness, Waviness, and Lay)

4.3 *ASTM Standards*[2]

ASTM D5127 — Guide for Ultra-Pure Water Used in the Electronics and Semiconductor Industries

ASTM E2251 — Specification for Liquid-in-Glass ASTM Thermometers with Low-Hazard Precision Liquids

4.4 *DIN Standards*[3]

DIN 50430 — Testing Inorganic Semiconductor Materials: Measurement of the Specific Electrical Resistance of Bar-Shaped Monocrystals of Silicon or Germanium by the Two-Probe Direct Current Method

NOTICE: Unless otherwise indicated, all documents cited shall be the latest published versions.

5 Terminology

5.1 Terms relating to silicon and other semiconductor technology are defined in SEMI M59.

6 Summary of Test Method

6.1 A direct current is passed through ohmic contacts at the ends of a bar specimen and the potential difference is determined between two probes placed along the current direction (see ¶ 7.3.1). The resistivity is calculated from the current and potential values and factors appropriate to the geometry. This test method includes procedures for checking both the probe assembly and the electrical measuring apparatus.

6.1.1 The spacing between the two probe tips is determined from measurements of indentations made on a polished single-crystal surface.

[1] American National Standards Institute, 25 West 43rd Street, New York, NY 10036, USA; Telephone: +1.212.642.4900, Fax: +1.212.398.0023, http://www.ansi.org

[2] ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, USA; Telephone: +1.610.832.9585, Fax: +1.610.832.9555, http://www.astm.org

[3] Deutsches Institut für Normung e.V., Available from Beuth Verlag GmbH, Burggrafenstrasse 4-10, D-10787 Berlin, Germany; http://www.din.de

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6.1.2 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.

6.2 Procedures for preparing the specimen, for measuring its size, and for determining the temperature of the specimen during measurement are also given. A table of temperature coefficient of resistivity versus resistivity is included with the method so that appropriate calculations can be made.

7 Apparatus

7.1 *Specimen Preparation*

7.1.1 *Grinding Facilities* — Capable of giving a finish of 0.4 µm rms, or less.

7.1.2 *Sandblasting Facility* — That uses aluminum oxide, SiC, or equivalent abrasive commercially specified as 27 µm.

7.1.3 Triple beam, dial, or trip balance.

7.1.4 *Diamond Abrasive Saw* — To cut proper specimen shape, if not previously done.

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

7.2 *Measurement of Cross-Sectional Specimen Geometry* — A micrometer or Vernier caliper capable of measuring the dimensions of square or rectangular bars and circular rods with a resolution of ±0.05 mm (±0.002 in.) or ±0.2%, whichever is smaller.

7.3 *Probe Assembly*

7.3.1 *Probes* — Either of two shapes may be used:

7.3.1.1 Chisel-shaped voltage probes should preferably be made from standard tungsten carbide flat tools 3.18 by 6.35 mm (0.125 by 0.25 in.). The included angle of the chisel should be approximately 45° and the nominal tip radius should be approximately 0.04 mm (0.0015 in.).

7.3.1.2 Conical-pointed voltage probes shall have tungsten carbide or osmium tips with included angle of 45° to 150°. The nominal radius of a probe tip should be initially 25 to 50 µm.

7.3.2 *Probe Force*

7.3.2.1 The force on the chisel-shaped probe shall be 8 ± 1 N when the probes are against the specimen in the measurement position.

7.3.2.2 The force on the conical-pointed probes shall be 1.75 ± 0.25 N when the probes are against the specimen in the measuring position.

7.3.3 *Insulation* — The electrical isolation between a probe and any other probe or part of the probe assembly shall be at least 10^8 Ω.

7.3.4 *Probe Alignment and Separation* — The spacing for the chisel-shaped two-probe shall be nominally 10 mm. The conical probe spacing shall have a nominal value of 4.76 mm (0.1875 in.). Probe spacing shall be determined in accordance with the procedure of ¶ 11.1 in order to establish the suitability of the probe assembly as defined in ¶ 11.1.5. The following apparatus is required for this determination:

7.3.4.1 A chemically polished surface on material characteristic of the specimen to be measured.

7.3.4.2 A toolmaker's microscope capable of measuring increments of 2.5 µm.

NOTE 2: The nominal spacing for the conical probe configuration is arrived at by using the outer-most pins of the '62.5 mil' four-point probe. Four-point probes of other nominal spacing may be used for the purposes of ¶ 7.3.4 by mutual agreement; however, the multilaboratory precision figures of ¶ 16.1 were arrived at using only the probes specified in ¶ 7.3.4.

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7.4 *Probe Supports* — Jigs must be provided for holding the specimen and bringing the probes into contact with the specimen perpendicular to the specimen surface without lateral movement. Probe positioning must be repeatable within 0.5% of the average probe spacing.

7.5 *End Contacts* — Contact to each specimen end should be made by metallic fiber mesh ('conductive wool') or soft plates large enough to cover the ends of the specimen and made of annealed copper or a metal with an equivalent conductivity. Prior to measurement, the ends of the specimen shall be made ohmic by electroplating or painting with conducting silver paint.

7.6 *Thermometer* — ASTM precision thermometer having a range from −8°C to +32°C and conforming to the requirements for Thermometer S63C as prescribed in ASTM E2251.

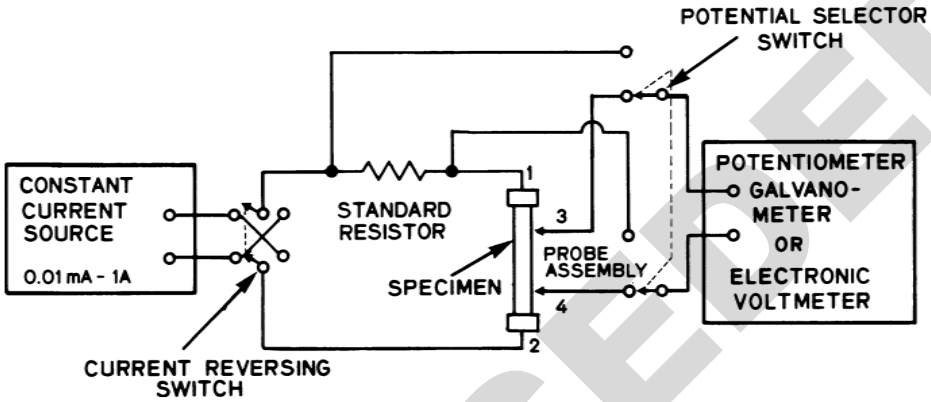

Figure 1
Recommended Electrical Circuit

7.7 *Electrical Measuring Apparatus*

7.7.1 Any circuit that meets the requirements of ¶ 11.2 may be used to make the electrical measurements. The recommended circuit, connected as shown in Figure 1, consists of the following:

7.7.1.1 *Constant-Current Source* — Capable of producing current densities shown in Table 1, constant to 0.1%. In order to cover the full range of resistivities listed and crystal cross-section dimensions from 12.5 to 37.5 mm, full scale current values from 10^{-5} to 1 A are necessary (see Note 3).

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Table 1 Resistivity Range Appropriate to Analog Test Circuit Resistance and Recommended Standard Resistance Values

Resistivity, Ω·cm	*Current Density (mA/cm²)*[#1]	
	10 mm Point Spacing	*4.76 mm Point Spacing*
0.001	100	200
0.01	20	40
0.1	10	20
1.0	2	4
10	1	2
100	0.2	0.4
1000	0.02	0.04

#1 These current densities are those which were tested by multilaboratory round robin for specimens whose diameters were 37.5 mm (1.5 in.) or less. These currents do not give the suggested 10-mV voltage drop (see ¶ 13.3) for the lower resistivity values. Some reduction from desirable signal level was necessitated by possible heating and injection effects (see ¶¶ 3.4 and 3.5) and current handling capabilities of available power supplies. For crystals larger than 37.5 mm in diameter further compromise may be necessary (see Note 3).

7.7.1.2 *Current Reversing Switch*

7.7.1.3 *Double-Throw, Double-Pole Potential Selector Switch*

7.7.1.4 *Potentiometer-Galvanometer or Electronic Voltmeter* — The instrument may be used to read the potential drop in volts or it may be calibrated in conjunction with the current ratio directly. The instrument must be capable of measuring potential differences of 10^{-4} to 1 V with two significant figure resolution on the 10^{-4} scale and three significant figure resolution on all other scales. The input impedance should generally be greater than 10^8 Ω (see Note 4).

NOTE 3: Larger-diameter crystals may in principal be measured by this technique. However, due to the possibility of crystal heating or current injection at the end contacts (see ¶¶ 3.4 and 3.5) current levels cannot be scaled based on Table 1 for all resistivity ranges. Consequently, a higher-resolution voltmeter than that required in ¶ 7.7.1.4, or acceptance of reduced accuracy and precision, or both, may be necessary for some resistivity and diameter combinations.

NOTE 4: It is generally found that for source impedances well in excess of 1000 Ω, manufacturers' specifications of digital voltmeter input impedance are not fully adequate. Hence an operational test for accuracy and precision is necessary (see ¶¶ 7.9 and 11.2).

7.7.2 *Ohmmeter* — Capable of indicating a leakage path of 10^8 Ω.

7.8 *Conductivity-Type Determination* — Apparatus in accordance with Method A of SEMI MF42.

7.9 *Analog Test Circuits* — To test the suitability and accuracy of the electrical equipment (see Figure 2 and Table 2).

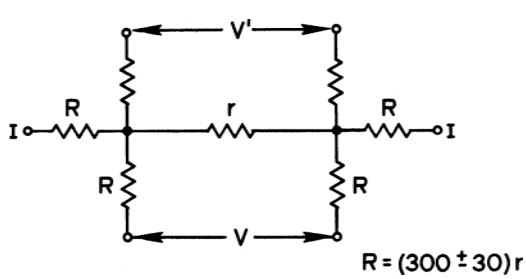

Figure 2
Analog Circuit to Simulate Resistivity Measurement

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Table 2 Current Values Required for Measurement of Resistivity

Resistivity, Ω·cm	*Standard Resistor (R_s) and Analog Resistor (r), Ω*
0.001	0.001
0.01	0.01
0.1	0.1
1.0	1.0
10	10
100	100
1000	1000

8 Reagents

8.1 *Purity of Reagents* — All chemicals for which such specifications exist shall conform to the assay and to impurity levels of Grade 1 SEMI Specifications for the appropriate chemical. Other grades may be used provided it is first ascertained that the reagent is of sufficiently high purity to permit use without lessening the accuracy of the determination.

8.2 *Purity of Water* — Reference to water shall be understood to mean Type E-3 or better water as described in ASTM D5127.

8.3 *Etching Solution* (15 + 1) — Mix 90 mL of concentrated nitric acid (HNO_3), in accordance with SEMI C35, and 6 mL of concentrated hydrofluoric acid (HF), in accordance with SEMI C28.

8.4 *Acetone ($(CH_3)_2CO$)* — In accordance with SEMI C19.

8.5 *Methanol (CH_3OH)* — In accordance with SEMI C31.

8.6 *Lapping Abrasive* — Aluminum oxide or silicon carbide commercially specified as 27 µm grade.

8.7 *End Contacts*

8.7.1 *Copper Solution* — Dissolve 20 g of cupric sulfate ($CuSO_4 \cdot 5H_2O$) in 90 mL of water; add 15 mL of concentrated hydrofluoric acid (HF), in accordance with SEMI C28, and mix.

8.7.2 *Colloidal Graphite* — Mix by weight 60 g of water with 40 g of a 22% colloidal graphite-in-water slurry.

8.7.3 *Silver* — Mix by volume 2 parts of acetone ($(CH_3)_2CO$), in accordance with SEMI C19, and 4 parts of methanol (CH_3OH), in accordance with SEMI C31, with 1 part of conducting silver paint.

9 Hazards

9.1 The acids in the etchants used for chemically preparing or treating the specimen surfaces (when such preparation or treatment is required) are extremely hazardous. All precautions normally used when handling these chemicals should be strictly observed.

9.1.1 **WARNING:** Hydrofluoric acid solutions are particularly hazardous. Make sure that the user is familiar with the specific preventive measures and first aid treatments given in the appropriate Material Safety Data Sheet.

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

10 Test Specimens

10.1 If not previously prepared using a diamond impregnated saw, saw enough material off both ends of the crystal to leave the ends flat and perpendicular to the length of the crystal. Further cut to square or rectangular cross section if necessary for referee measurement. Make certain to mark or otherwise note the seed end of the crystal.

10.2 Clean the specimen with acetone, rinse with methanol, and air dry.

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