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Standard Test Method for Measuring Resistivity Profiles Perpendicular to the Surface of a Silicon Wafer Using a Spreading Resistance Probe [1]

This standard is issued under the fixed designation F 672; 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.

ε1 NOTE—Keywords were added editorially in January 1995.

INTRODUCTION

The measurement of resistivity profile by means of a spreading resistance probe is a complex procedure, with a number of commonly accepted options for carrying out the component measurements. ASTM Committee F-1 on Electronics has designed this test method to allow a range of choices, consistent with good practice, for the electronic configuration, type of specimen preparation, and method for measuring bevel angle. Items not specified by this test method are to be agreed upon by the parties to the test, usually from a specified set of choices in the context of a general restriction. The measurement of bevel angle is particularly difficult to specify, as the selection of an appropriate method depends not only on the range of angle measured but also on the quality of the instrumentation available for that method. Although ideally the beveled surface and the original surface should be two planes intersecting along a straight line, the actual geometry may differ from this ideal, further complicating the measurement. These points are recognized in the section on interferences and in Appendix X1 and associated references on the bevel-angle measurement.

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1. Scope

1.1 This test method covers measurement of the resistivity profile perpendicular to the surface of a silicon wafer of known orientation and type.

NOTE 1—This test method may also be applicable to other semiconductor materials, but feasibility and precision have been evaluated only for silicon and germanium.

1.2 This test method may be used on epitaxial films, substrates, diffused layers, or ion-implanted layers, or any combination of these.

1.3 This test method is comparative in that the resistivity profile of an unknown specimen is determined by comparing its measured spreading resistance value with those of calibration standards of known resistivity. These calibration standards must have the same surface preparation, conductivity type, and crystallographic orientation as the unknown specimen.

1.4 This test method is intended for use on silicon wafers in any resistivity range for which there exist suitable standards. Polished, lapped, or ground surfaces may be used.

1.5 This test method is destructive in that the specimen must be beveled.

1.6 Correction factors, which take into account the effects of boundaries or local resistivity variations with depth, are needed prior to using calibration data to calculate resistivity from the spreading resistance values.

NOTE 2—This test method extends Method F 525 to depth profiling.

NOTE 3—This test method provides means for directly determining the resistivity profile of a silicon specimen normal to the specimen surface. Unlike Method F 84 and Test Methods F 374 and F 419, it can provide lateral spatial resolution of resistivity on the order of a few micrometres, and an in-depth spatial resolution on the order of 10 nm (100 Å). This test method can be used to profile through *p-n* junctions.

1.7 This test method is primarily a measurement for determining the resistivity profile in a silicon wafer. However, common practice is to convert the resistivity profile information to a density profile. For such purposes, a conversion between resistivity and majority carrier density is provided in Appendix X2.

1.8 *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:*

[1] This test method is under the jurisdiction of ASTM Committee F-1 on Electronicsand is the direct responsibility of Subcommittee F01.06 on Silicon Material and Process Control.

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D 1125 Test Methods for Electrical Conductivity and Resistivity of Water [2]

E 1 Specification for ASTM Thermometers [3]

F 26 Test Methods for Determining the Orientation of a Semiconductive Single Crystal [4]

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

F 84 Test Method for Measuring Resistivity of Silicon Slices with an In-Line Four-Point Probe [4]

F 374 Test Method for Sheet Resistance of Silicon Epitaxial, Diffused, Polysilicon, and Ion-Implanted Layers Using an In-Line Four-Point Probe [4]

F 419 Test Method for Net Carrier Density in Silicon Epitaxial Layers by Capacitance Voltage Measurements on Fabricated Junction Schottky Diodes [4]

F 525 Test Method for Measuring Resistivity of Silicon Wafers Using a Spreading Resistance Probe [4]

F 674 Practice for Preparing Silicon for Spreading Resistance Measurements [4]

F 723 Practice for Conversion Between Resistivity and Dopant Density for Boron-Doped and Phosphorus-Doped Silicon [4]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *conducting boundary— for the purposes of this test method*, a boundary between two specimen layers of the same conductivity type taken to be the point at which the spreading resistance increases to twice the local minimum value it has in the layer of lower resistivity (Fig. 1*a*).

3.1.2 *effective electrical contact radius, a (cm)—of a spreading resistance probe assembly*, an empirical quantity defined by

$$a = (n\rho)/4R_s \tag{1}$$

where:

n = number of current-carrying probes across which the potential drop is determined,

ρ = resistivity of a homogeneous semiconductor specimen, Ω·cm, and

R_s = measured spreading resistance, Ω.

3.1.2.1 *Discussion*—For a three-probe arrangement, $n = 1$; for a two-probe arrangement, $n = 2$.

3.1.3 *insulating boundary—for the purposes of this test method*, a boundary between two specimen layers of opposite conductivity type, taken to be the point at which the local maximum of the spreading resistance occurs (Fig. 1*b*).

3.1.4 *spreading resistance, R_s (Ω)—of a semiconductor*, the ratio of (*1*) the potential drop between a small-area conductive metal probe, and a reference point on the semiconductor, to (*2*) the current through the probe.

3.1.4.1 *Discussion*—This ratio, in fact, measures metal to semiconductor contact resistance as well as classical spreading resistance for a homogeneous specimen without electrical boundaries in the vicinity of the probes. For a specimen having resistivity gradients or electrical boundaries, this ratio also includes an effect due to those gradients or boundaries.

3.1.4.2 *Discussion*—In a three-probe arrangement, the experimental conditions approximate those of the definition (based on a single probe) and the spreading resistance is given by

$$R_s = V/I \tag{2}$$

where:

V = potential drop between one of the current-carrying probes and the reference (non-current-carrying) probe on the front surface, mV, and

I = current through the metal probe, mA.

In a two-probe arrangement, the potential drop, V, is measured between two similar current-carrying metal probes. In this case, the voltage-to-current ratio, and hence the spreading resistance, is approximately twice that associated with a single probe.

3.1.5 *substrate—in semiconductor technology*, a wafer which is the basis for subsequent processing operations in the fabrication of semiconductor devices or circuits.

3.1.5.1 *Discussion*—The devices or circuits may be fabricated directly in the substrate or in a film of the same or another material grown or deposited on the substrate.

4. Summary of Test Method

4.1 A portion of the specimen wafer is beveled at an angle. The spreading resistance of a reproducibly formed point pressure contact (or contacts) is measured at a sequence of locations on the beveled surface. The spreading resistance may be measured using two, or three, probes (*1*) by applying a known constant voltage and measuring the current, (*2*) by applying a known constant current and measuring the voltage, or (*3*) by using a resistance comparator technique. A correction factor must be used **(1, 2, 3)** [5] which takes into account the effect of local resistivity gradients and boundaries on the finite sampling volume of the probes. The resistivity of the material immediately under the probes is then determined from a calibration curve derived from spreading resistance measurements made under the same conditions on calibration standards of known resistivity.

4.2 The following quantities are not specified by this test method and shall be agreed upon by the parties to the test:

4.2.1 Probe spacing, µm (7.3.1.3),

4.2.2 Sampling plan (10.1,

4.2.3 Minimum bevel length, mm, if required (11.1.1),

4.2.4 Bevel angle, deg, appropriate to the total depth of interest and desired resolution of the test specimen data (11.3 and Table 1),

4.2.5 Beveling technique (11.6),

4.2.6 Method for obtaining calibration curve (13.4),

4.2.7 Method for measuring bevel angle (14.10),

4.2.8 Probe spacing and probe step increment, µm, appropriate to the resolution desired along the profile of interest (13.2, 14.4),

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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] The boldface numbers in parentheses refer to the list of references at the end of this test method.

4.2.9 Algorithm for sampling volume correction factor (15.4), and

4.2.10 Conversion from resistivity profile to carrier density profile (see Appendix X3).

NOTE 4—Information relating the depth resolution and bevel angle for probe step increments of 5 and 10 µm and also bevel length to the layer thickness and bevel angle is given in Table 1. The probe step increment should be larger than the diameter of the specimen area damaged by the probes.

NOTE 5—Model data, of the type used to qualify participants in the round robin is provided in Annex A1. These are idealized data, free of measurement noise and contact calibration nonlinearity. They may be used to study the effects on a calculated resistivity profile of data round-off error or input measurement noise (if random or systematic noise is added to the model data). While they may be used to compare the results from different algorithms, such comparisons may be misleading. It has been found that some algorithms do a highly satisfactory analysis of certain real structures despite their relatively poorer performance on model data as described in Annex A1, **(4)**. This is thought to be due to their relatively better ability to deal with measurement noise and with probe calibration nonlinearity.

5. Significance and Use

5.1 This test method can be used for process control, research and development, and materials acceptance purposes.

6. Interferences

6.1 *Temperature*—If the calibration and specimen measurements are not made at the same temperature, the accuracy of the results is likely to be adversely affected, as spreading resistance measurements are sensitive to the temperature of the specimen.

6.2 *Light*—Photoconductive and photovoltaic effects can seriously influence the resistance determined by this test method, especially on wafers having *p-n* junctions.

6.3 *Radiofrequency Fields*—If the apparatus is located near unshielded radiofrequency sources, the precision and accuracy of the results may be adversely affected, as spurious currents can be introduced in the measurement circuit in the presence of high-frequency fields.

6.4 *Mechanical Vibration*—If the apparatus is not sufficiently isolated from building-induced or other vibration sources, the precision and accuracy of the results may be adversely affected, as the probes are delicate (the entire probe assembly and the manner in which the probes contact the specimen surface are sensitive to shock and displacement).

6.5 *Minority Carrier Injection*—Caution should be taken to prevent minority carrier injection during the measurement. Experience has shown that if the potential applied between the current-carrying probes is kept to 20 mV or less, significant minority carrier injection should not occur.

6.6 *Reactive Atmosphere*—Exposure of the probe or specimen to reactive atmospheres, such as those produced in the vicinity of epitaxial reactors or by high humidity, may lead to changes in the characteristics of the instrument and to nonreproducible measurements. Probes and specimens shall be protected from such exposure. Relative humidity in excess of 60 % should be avoided.

6.7 *Semiconductor Surface*:

6.7.1 *Surface Instability*—It has been found that spreading resistance measurements made on surfaces that have been exposed to an aqueous solution may be erratic and nonreproducible. Surfaces exposed to solutions containing fluorine ions may also exhibit instability. The heat treatment included in the procedure (see 11.8) has been found to reduce these instabilities for *p*-type specimens **(5, 6)**.

6.7.2 *Surface Damage*—Spreading resistance measurements made in areas of severe or nonuniform mechanical damage may give erroneous results. Such damage may be caused by previous spreading resistance probe marks, or by improper surface preparation.

6.7.3 *High Impurity Concentration*—At impurity concentrations greater than approximately 10^{20} cm $^{-3}$ the defects caused by the impurity may have an effect on the measured spreading resistance. These defects and consequent effects may not be the same for all heavily doped specimens.

6.7.4 *Imperfect Bevel*—An ideal beveled surface is planar and intersects sharply along a straight line with a planar original surface of the specimen. Deviations from an ideal bevel can be caused by a number of factors such as nonuniform specimen thickness, specimen warp during mounting on the beveling block, rocking of the specimen mount during beveling, flexing or compression of the plate against which the beveling is done, and preferential attack of the beveling medium at the edge of the bevel. A non-ideal bevel may cause an incorrect bevel angle to be measured, present a changing depth scale along the line scanned by the probes, or both. Two simple limiting-case beveling defects can be described.

6.7.4.1 Bevel edge rounding is shown in Fig. 2. It is characterized by a gradual transition between the original and beveled surfaces of the specimen. It is found more likely to occur when a chem-mechanical beveling process is used, when a reciprocating motion is used during beveling, or when too soft a material is used for the polishing plate. Its existence is difficult to recognize by casual observation. Its presence can be seen, in general, when using bevel measurement methods 1, 3, or 4 in Appendix X1. The effect of this defect can be reduced if the specimen is covered with an oxide or nitride layer prior to beveling.

6.7.4.2 Bevel edge arcing is shown in Fig. 3. It is characterized by a curved or arced intersection of the original and beveled surfaces of the specimen, indicating that one or both surfaces are non-planar. However, a sharp transition from one surface to the other exists across the intersection. This defect is found more likely to occur when orbital motion is used during beveling, but if caused by a non-planar original surface on the specimen, it will occur regardless of motion used. This defect results in an inaccurate value of bevel angle with any measurement techniques (such as X1.1, X1.2, and X1.4 in Appendix X1) that sense an area of the specimen rather than scanning a line across the intersection.

6.7.5 *Deviation from Flatness* of the original surface, including the effects of stripping an anodic oxide layer that was applied to only a portion of the specimen (14.9), may adversely affect the precision of the measurement of bevel angle and therefore of the method.

6.8 *Correction Factor Assumptions*— All formulations of the correction factor assume the measurement is being made on a surface perpendicular to the impurity gradient. Also, the

assumption of lateral impurity uniformity is employed. Since neither of these assumptions is strictly true on a beveled surface, the corrected data may not represent the actual profile.

7. Apparatus

7.1 *Apparatus to Bevel the Test Specimen*:

7.1.1 *Lapping or Polishing Methods*—A mounting plug (beveling block) having the agreed-upon angle (see 4.2.4) and plug holder as shown in Fig. 4 and a flat plate of glass prepared in accordance with 6.1.1 of Practice F 674 or a flat plate of a suitable plastic such as methyl methacrylate.

7.1.1.1 *Polishing Machine*, if required, of the shaker, oscillating-tub, or rotary-plate type.

7.1.2 *Grinding Methods*—A motor-driven plastic or other soft-matrix wheel charged with diamond grit having a particle size of 3μ m or less.

7.2 *Means for Measuring Bevel Angle*, appropriate for the agreed-upon method (4.2.7, Appendix X1, and Fig. 5).

7.3 *Mechanical Apparatus*:

7.3.1 *Probes and Probe Assembly*—Spreading resistance probe assembly with provision for supporting and lowering either two or three replaceable probe tips to the wafer surface at a reproducible descent rate and with a predetermined static load. The supporting mechanism shall provide for lateral positioning of the probes for adjustment of the contact site, and for aligning the probes parallel the bevel edge to within± 2 μm (see 14.4.1).

7.3.1.1 *Probe Tips*—A hard, durable, low-resistivity substance that wears well without flaking, such as tungsten-osmium, tungsten-carbide, or tungsten-ruthenium alloys. The mechanical radius of curvature of the probe tips in the region that will touch the specimen shall be less than or equal to 25 μm. The tip angle of the probe shall be within the range from 30 to 60°, inclusive.

7.3.1.2 *Probe Loading and Descent Rate*— The loading applied to each point shall be in the range from 5 to 50 gf (49 to 490 mN), inclusive. A dashpot, or other means, for controlling the descent rate of the probes must be available if the load is applied by dead weight, but may not be necessary if the probes are spring-loaded (see 12.4).

NOTE 6—The sampling depth of the spreading resistance probes increases with increased probe loading, as does the risk of premature penetration to underlying layers. For best profile resolution, particularly for thin layers, probe loads should be kept in the low end of the above force range.

7.3.1.3 *Probe Spacing* shall be as agreed upon by the parties to the test.

NOTE 7—Since sensitivity of the measurement to the presence of lateral specimen boundaries (and sampling volume asymmetry resulting from the use of a beveled specimen) near the probe site is reduced with decreased probe spacing, the probe spacing should generally be as small as possible for the apparatus being used. Typical probe spacings are between 10 and 100 μm.

7.3.1.4 *Probe Insulation* to provide a d-c isolation resistance of 1 GΩ or greater between any pair of probes and between each probe and any guard circuit used.

7.3.2 *Specimen Holder*—Insulated vacuum chuck or other means for holding the specimen tightly while measurements are made (the mounting plug of 7.1.1 or an equivalent piece of apparatus may be used).

7.3.3 *Translation Microscope Stage*—Means for supporting, translating, rotating, and vertical adjustment of the specimen holder to facilitate alignment of probes and bevel edge. The stage shall provide translation position resolution of at least 1 μm. Gear boxes or stepper-motor drives for stage movement shall allow step intervals in the range from 1 to 100 μm per step, inclusive.

NOTE 8—Typical step intervals are 1, 2.5, 5, and multiples of 10 times these values.

7.4 *Vibration-Free Table* for supporting apparatus as required (see 6.4).

7.5 *Apparatus Enclosure* for providing darkened environment for spreading resistance measurements, if required by specimen material (see 6.2).

7.6 *Electrical Measuring Apparatus*—For a two-probe arrangement, use the apparatus of 7.6.1, 7.6.2, or 7.6.3. For a three-probe arrangement, use the apparatus of 7.6.2.

7.6.1 *Constant-Voltage Method* (see Fig. 6):

7.6.1.1 *D-C Voltage Source*, with a constant output between 1 and 20 mV, inclusive. The output potential shall be constant to ±0.1 % into a load that varies from 1 to 10 MΩ, inclusive.

7.6.1.2 *D-C Current Detector*, accurate to ±0.1 % and capable of measuring currents in the range from 10^{-10} to 10^{-2} A, inclusive, to three significant figures.

7.6.2 *Constant-Current Method* (see Fig. 7):

7.6.2.1 *Variable D-C Current Source*, capable of providing currents from 10^{-10} to 10^{-2} A, inclusive. The current output shall be accurate to ±0.1 %, stable at any output value to ±0.1 %, and capable of providing a current of 10^{-10} A into a 100 MΩ load. The current source shall have sufficient adjustment capability so that the specimen voltages which are measured remain in the range from 1 to 20 mV, inclusive, for all measurement points.

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NOTE 9—The compliance voltage should not exceed 40 V for reasons of operator safety.

NOTE 10—For protection of the probes and specimen, capability should be provided for shorting the output when the probes are not in contact with the specimen, or else the compliance voltage should be reduced to 1 V or less.

7.6.2.2 *D-C Voltage Detector*, linear over the range from 1 to 50 mV, inclusive, capable of being read to three significant figures, and accurate to ±0.1 % of the reading. The input impedance shall be 1 GΩ or greater.

7.6.3 *Comparator Method* (see Fig. 8):

7.6.3.1 *D-C Voltage Source*, with a regulated output in the nominal range from 1 to 20 mV, inclusive, stable to ±3 % or better for a period of 1 min when connected to an external load in the range from 1 Ω to 100 MΩ.

7.6.3.2 *Log Comparator*, with an output proportional to the logarithm of the ratio of two currents (the logarithm of the ratio of the current, I_1, through the specimen to the current, I_2, through the standard resistor shall be directly measured by the circuitry). The comparator circuit shall contain a standard resistor, nominally 10 kΩ, which shall be known to an accuracy of ±0.1 %. The comparator shall be capable of measuring resistances from 1 Ω to 100 MΩ, inclusive, with a deviation from linearity of response not greater than ±1 %. If

the output is available as a voltage, at least two reference resistors, R_{r1} and R_{r2}, shall be supplied which can be selectively substituted for the specimen in the circuit (these resistors establish a fixed point and output voltage gain for use in calculating specimen spreading resistance from output voltage). The reference resistors shall be in the range from 1Ω to 100 MΩ, inclusive, and their values shall be known to an accuracy of at least ±1 %.

7.7 *Microscope*, capable of a magnification of at least 100× and a cross hair perpendicular to the direction of the microscope stage translation (7.3.3).

7.8 *Thermometer*— ASTM Precision Thermometer having a range from − 8 to + 32°C, inclusive, and conforming to the requirements for Thermometer 63C as described in Specification E 1.

7.9 *Means for Scribing and Breaking*— Customary means for scribing a silicon wafer and for breaking it into small pieces or dice.

7.10 *Etching Apparatus*, as required for removal of oxide or nitride layer (if present) from the specimen (14.4).

7.11 *Hot Plate*, capable of heating the specimen to a temperature of 150°C.

8. Reagents and Materials

8.1 *Reference Specimen Wafers for Calibration*, chosen in accordance with Section 13 from wafers in the resistivity range of the unknowns. The reference specimens shall be of the same conductivity type and nominal crystallographic orientation as the test specimens. The surface preparation technique used to produce the reference specimens shall match that of the test specimens; this includes preparation of the calibration specimens at a shallow bevel angle.

NOTE 11—It is desirable to use three or more reference specimens per decade of resistivity.

8.2 *Probe-Check Specimen*, consisting of *p*-type silicon and having a resistivity that is uniform to ± 15 %, as determined in accordance with Test Method F 525. This specimen shall have a nominal resistivity of 1 Ω·cm at 23°C. If the specimen is an epitaxial layer, the layer shall be at least 10 µm thick and fabricated on a substrate of the same conductivity type, and the surfaces shall be allowed to stabilize for at least 1 week subsequent to epitaxial growth. If the specimen is bulk silicon, the surface shall have been prepared by polish-etching followed by at least 1 week of aging, or by chem-mechanical polishing followed by thermal treatment of 150°C for 20 min in laboratory atmosphere. For use with a two-probe configuration, the chosen *p*-type specimen shall have a large-area ohmic contact fabricated into the rear surface of the specimen.

NOTE 12—It is desirable that the resistivity of the probe-check specimen be measured over a period of at least a month, to establish its history.

8.3 *Silicon Slice*, lapped or ground with 5-µm grit slurry, for conditioning the probe tips (see 12.5.2).

8.4 *Lapping, Polishing, or Grinding Materials*, as required for preparing the surfaces of the test specimen and calibration specimens (examples are alumina, garnet or diamond grit, and colloidal silica).

8.5 *Wax*, for mounting the test specimen to the beveling block and to the specimen holder.

8.6 *Solvent*—Methanol (CH_3OH) or other solvent recommended by the supplier of the diamond polishing medium.

8.7 *Distilled or Deionized Water*, having a resistivity greater than 2 MΩ·cm at 25°C as determined by the Nonreferee Method of Methods D 1125.

8.8 *Chemical Etch*, as required for removal of an oxide or nitride layer (if present) from the specimen (14.9).

9. Hazards

9.1 Use normal safety precautions in operating the electrical equipment.

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

10. Sampling

10.1 The sampling plan, including the definition of "lot" if sampling by lot is intended, shall be agreed upon by the parties to the test.

11. Test Specimen

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11.1 Select an area of the silicon wafer on which the profile is desired.

11.1.1 Scribe and break or saw a small piece of the wafer. The minimum width (dimension nominally parallel to intersection of original and beveled surfaces) of the test specimen shall be at least 10 times the agreed-upon probe spacing, but not less than 3 mm. The minimum length of the specimen shall be not less than 2 mm plus the bevel length given in Table 1 for the agreed-upon bevel angle and for the layer thickness values given in the table. For layer thickness and bevel angle combinations other than those given in Table 1, the minimum specimen length shall be 2 mm plus an agreed-upon minimum bevel length.

NOTE 13—The entire wafer may be used if the beveling apparatus allows. However, it may be more difficult to obtain a uniform bevel on a large specimen.

11.2 On specimens to be beveled at angles below 30 min, deposit or grow a thin layer of SiO_2 or Si_3N_4 on the specimen surface. This can help to define the bevel edge. The thin layer must be removed prior to measurement of the bevel angle (14.9).

11.3 Select a mounting plug (beveling block) with the agreed-upon angle.

11.4 If the crystallographic orientation of the specimen surface is unknown, determine and record in accordance with Test Methods F 26 the orientation of the wafer from which the beveled specimen was cut.

11.5 Mount the test specimen on the mounting plug using the wax of 8.5.

11.6 Lap, grind, or polish the specimen to form a beveled area whose length is in accordance with 11.1.1, that is, sufficient to expose the total depth of interest (see 6.7.4, Table

1, Fig. 4) using the agreed-upon procedure selected from 11.6.1 through 11.6.5. Avoid severe, nonreproducible, or non-uniform mechanical damage. Hidden severe subsurface damage may remain if the bevel is formed by a coarse lapping or grinding technique followed by a cursory fine polishing method; do not use more than one stage of polishing, that is, use only one polishing medium on a given specimen.

11.6.1 *Chem-Mechanical Polishing*—Colloidal silica in a caustic aqueous solution; used on an acrylic plate.

11.6.2 *Aqueous Mechanical Polishing*—Aluminum oxide or garnet with a particle size of 1 µm or less in an aqueous slurry; used on a glass or acrylic plate.

11.6.3 *Non-Aqueous Mechanical Polishing*— Diamond in oil suspension, with particle size of 1 µm or less; used on an acrylic plate, or on a glass plate that has been lapped with 5 to 9-µm alumina and thoroughly cleaned prior to use for beveling.

11.6.4 *Lapping*—An aqueous slurry of aluminum oxide or garnet with particle size in the nominal range from 1 to 5 µm; used on a glass plate.

11.6.5 *Grinding*—Diamond grit of particle size 3 µm or less in a plastic or other soft matrix wheel. A non-aqueous liquid such as machinist's cutting oil shall be used with this test method.

11.7 Remove the residue of the polishing medium from the specimen using water for water-based polishing media and using methanol (or other solvent recommended by the supplier of the polishing medium) for non-aqueous-based polishing media.

11.8 If an aqueous polish was used, heat the specimen in air at 150 + 10°C for 15 ± 5 min. If the specimen is mounted in wax, be sure that the wax does not get on the bevel surface during this heat treatment. (Ground surfaces or surfaces prepared in the absence of aqueous solutions do not need heat treatment.)

NOTE 14—Freshly prepared surfaces should have a spreading resistance that is stable and repeatable for a long enough time so that the calibration standards need not be prepared freshly for each test run. The best long-term stability is achieved by a surface preparation that excludes aqueous or fluorine-containing solutions **(6).**

12. Preparation of Apparatus

12.1 Adjust the probe spacing to the agreed-upon value (see section 3.2.1), not to exceed 100 µm.

12.2 Choose a loading in the range from 5 to 50 gf (49 to 490 mN), inclusive, to be applied to the probes. In a multiple-probe arrangement, use the same loading for each probe.

NOTE 15—Reasonable loading for most profiling is 20 gf (195 mN).

12.3 Connect the appropriate electrical circuit (see Fig. 5, Fig. 6, and Fig. 7). If a voltage source is used (constant-voltage or comparator methods), adjust the potential to 20 mV or less. If a current source is used that has a compliance voltage greater than 1 V (constant-current method), short-circuit the output before measurement begins, and at all times when the probes are lifted from the specimen surface.

12.4 Adjust the descent rate of the probes onto the specimen to an appropriate value to minimize damage, and to ensure maximum measurement reproducibility.

NOTE 16—A nominal descent rate of 1 mm/s is generally adequate for a load of 20 gf (195 mN).

NOTE 17—It is generally possible to obtain 20 measurements with a scatter in the range from 1 to 5 % for most silicon specimens with a polished surface.

12.5 Make 20 measurements of the spreading resistance of the silicon probe-check specimen (8.2) in accordance with 14.6 through 14.11.

12.5.1 If the measured resistance is within ±20 % of the value assigned to the specimen, proceed to the tests in 12.6 and 12.7.

12.5.2 If the measured spreading resistance deviates by more than ±20 % from the value assigned to the probe-check specimen, either (*1*) replace the probes and repeat the test or (*2*) condition the probes by stepping them at least 500 times on a silicon substrate that has been ground with 5-µm grit slurry (see 8.3). Step the probes at least 20 times on a polished wafer such as the probe-check wafer and repeat the test.

NOTE 18—It is permissible to leave the slurry on the substrate during conditioning, but it must be removed from probe tips before measuring test specimens.

12.6 Using the microscope at a nominal magnification of 100×, examine the probe marks for reproducibility. If the probe marks from a given probe (*1*) do not appear similar, (*2*) do not have simple, near circular shape, or (*3*) show chipping or radial crack lines, decrease the descent rate, recondition the probe in accordance with 12.5.2, or replace the probe. Repeat 12.5 and 12.6.

NOTE 19—Experience suggests that the first approach should be decreasing the descent rate.

NOTE 20—Probe imprints from different probes need not be similar.

12.7 If the two-probe arrangement is being employed, verify that the spreading resistances of each of the two probes are equal to within 10 % when measured on the probe-check specimen (8.2).

12.7.1 Accomplish this measurement by using the ohmic rear-surface contact to replace each of the probes, in turn, and by measuring between the remaining probe and rear-surface contact in the normal manner (7.4). If the single-probe measurements do not agree within 10 %, recheck or adjust the loading (12.2) and descent rate (12.4) to be nominally equal on both probes. If satisfactory results are not achieved with equal probe loading and descent rate, recondition (12.5.1) or replace one or both probes. Repeat the tests given in 12.5, 12.6, and 12.7.

13. Calibration

13.1 Measure the resistivity of each of the specimens to be used as reference specimens (8.1), in accordance with Test Method F 84. Record the results.

13.2 Prepare each reference specimen in a manner identical to that intended for the test specimen, as described in Section 11. In accordance with 14.6 to 14.9, make a minimum of 20 spreading resistance measurements on the beveled surface of each of the proposed reference specimens. Make the measurements as close as possible to the region where the four-probe measurements were made. Use the agreed-upon probe-step increment that is to be used on the test specimen. Record the results. Using the thermometer (7.8), measure and

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