SEMI MF672-0412
GUIDE FOR MEASURING RESISTIVITY PROFILES PERPENDICULAR TO THE SURFACE OF A SILICON WAFER USING A SPREADING RESISTANCE PROBE

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1 Purpose

1.1 The resistivity profile of a silicon wafer perpendicular to its surface is frequently a necessary or useful property of the wafer.

1.2 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. This Guide describes 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 Guide 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 limitations and in Appendix 1 and associated references on the bevel-angle measurement.

1.3 This Guide extends the procedures of SEMI MF525 to depth profiling.

1.4 The procedures in this Guide can be used for process control, research and development, and materials acceptance purposes, but see ¶ 17.5 for restrictions on use for materials acceptance.

2 Scope

2.1 This Guide covers procedures for measurement of the resistivity profile perpendicular to the surface of a silicon wafer of known orientation and type in any resistivity range for which there exist suitable standards. Polished, lapped, or ground surfaces may be used.

NOTE 1: The procedures of this Guide may also be applicable to other semiconductor materials, but feasibility and precision have been evaluated only for silicon and germanium.

NOTE 2: Unlike the methods of SEMI MF84, SEMI MF374, and SEMI MF1392, the procedures of this Guide provide lateral spatial resolution of resistivity on the order of a few micrometers and an in-depth spatial resolution on the order of 10 nm (100 Å).

2.2 The procedures of this Guide can be used to profile through *p-n* junctions.

2.3 The procedures of this Guide may be used on epitaxial films, substrates, diffused layers, or ion-implanted layers, or any combination of these.

2.4 The procedures of this Guide are 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.

2.5 The procedures of this Guide are intended for use on silicon wafers.

2.6 The procedures of this Guide are destructive in that the specimen must be beveled.

2.7 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.

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2.8 The procedures of this Guide are primarily a measurement for determining the resistivity profile in a silicon wafer. However, common practice is to convert the resistivity profile information to a carrier density profile using the conversion between resistivity and majority carrier density provided in Appendix 2.

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

2.9.1 Probe spacing, µm (see ¶ 7.3.1.3),

2.9.2 Sampling plan (see ¶ 10.1),

2.9.3 Minimum bevel length, mm, if required (see ¶ 11.2),

2.9.4 Bevel angle, degree, appropriate to the total depth of interest and desired resolution of the test specimen data (see ¶ 1.4 and Table 1),

2.9.5 Beveling technique (see ¶ 11.7),

2.9.6 Method for obtaining calibration curve (see ¶ 13.4),

2.9.7 Method for measuring bevel angle (see ¶¶ 7.2 and 14.10, and also Appendix 1),

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

2.9.9 Algorithm for sampling volume correction factor (see ¶ 15.4), and

2.9.10 Conversion from resistivity profile to carrier density profile (see Appendix 2).

NOTE 3: 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 4: Model data, of the type used to qualify participants in the round robin multi-laboratory experiment conducted to estimate the precision to be expected from the procedures in this Guide, is provided in Related Information 1. 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 Related Information 1.[1] This is thought to be due to their relatively better ability to deal with measurement noise and with probe calibration nonlinearity. The round robin multi-laboratory experiment is described in Related Information 2.

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 *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.

3.2 *Light* — Photoconductive and photovoltaic effects can seriously influence the resistance determined by the procedures of this Guide, especially on wafers having *p-n* junctions.

3.3 *Radio Frequency Fields* — If the apparatus is located near unshielded radio frequency 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.

3.4 *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).

[1] Pawlik, M., "Spreading Resistance: A Comparison of Sampling Volume Correction Factors in High Resolution Quantitative Spreading Resistance." *Emerging Semiconductor Technology, ASTM STP 960*, D. C. Gupta and P. H. Langer, eds., ASTM International, West Conshohocken, PA (1987): pp. 502–520.

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3.5 *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.

3.6 *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 non-reproducible measurements. Probes and specimens shall be protected from such exposure. Relative humidity in excess of 60% should be avoided.

3.7 *Semiconductor Surface*

3.7.1 *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 not reproducible. 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.[2,3]

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

3.7.3 *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.

3.7.4 *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.

3.7.4.1 Bevel edge rounding is shown in Figure 1. It is characterized by a gradual transition between the original and beveled surfaces of the specimen that makes it impossible to determine the beginning of the beveled surface. In the transition region, the depth being probed is not a linear function of the probe step increment. 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 1. The effect of this defect can be reduced if the specimen is covered with an oxide or nitride layer prior to beveling.

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[2] Ehrstein, J. R. "Semiconductor Measurement Technology: Spreading Resistance Symposium." NBS Special Publication 400-10, J. R. Ehrstein, ed., December 1974, pp. 249–255.

[3] Vieweg-Gutberlet, F. G., in *Semiconductor Silicon, 1977*, Proceedings of the Third International Symposium on Silicon Materials Science and Technology, H. R. Huff and E. Sirtl, eds., The Electrochemical Society, Pennington, NJ, May 1977, p. 387.

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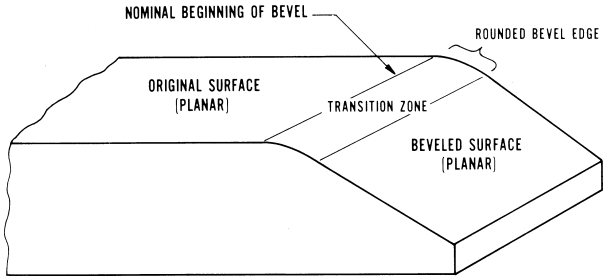
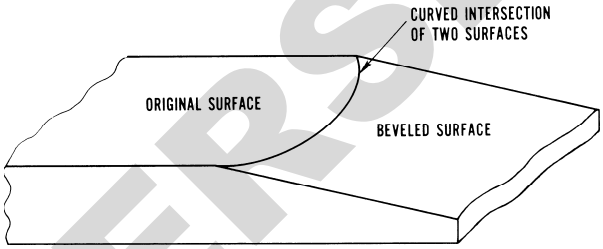


#1 Instead of a sharp intersection between two surfaces, a transition region exists that makes it impossible to determine the beginning of the beveled surface. In the transition region, the depth being probed is not a linear function of the probe step increment.

Figure 1
Sketch Illustrating Bevel Edge Rounding

3.7.4.2 Bevel edge arcing is shown in Figure 2. 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 nonplanar. 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 nonplanar original surface on the specimen, it occurs regardless of motion used. This defect results in an inaccurate value of bevel angle with any measurement techniques (such as 1, 2, and 4 in Appendix 1) that sense an area of the specimen rather than scanning a line across the intersection.



#1 Instead of a straight-line intersection between the original and beveled surfaces, an arc-shaped intersection exists indicating that one or both surfaces are nonplanar. In the presence of this defect any means for determining the bevel angle that samples a finite width of the specimen along the arc does not provide a unique angle value.

Figure 2
Sketch Illustrating Bevel Edge Arcing

3.7.5 *Flatness* — 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 (see ¶ 14.9), may adversely affect the precision of the measurement of bevel angle and therefore of the method.

3.8 *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.

4 Referenced Standards and Documents

4.1 *SEMI Standards and Safety Guidelines*

SEMI C28 — Specifications for Hydrofluoric Acid

SEMI C29 — Specifications and Guide for 4.9% Hydrofluoric Acid (10:1 v/v)

SEMI C31 — Specification for Methanol

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SEMI M59 — Terminology for Silicon Technology

SEMI MF26 — Test Methods for Determining the Orientation of a Semiconductive Single Crystal

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

SEMI MF84 —Test Method for Measuring Resistivity of Silicon Wafers with an In-Line Four-Point Probe

SEMI MF374 — 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

SEMI MF525 — Test Method for Measuring Resistivity of Silicon Wafers Using a Spreading Resistance Probe

SEMI MF674 — Practice for Preparing Silicon for Spreading Resistance Measurements

SEMI MF723 — Practice for Conversion Between Resistivity and Dopant or Carrier Density for Boron-Doped, Phosphorus-Doped, and Arsenic-Doped Silicon

SEMI MF1392 — Test Method for Determining Net Carrier Density Profiles in Silicon Wafers by Capacitance-Voltage Measurements with a Mercury Probe

4.2 *ASTM Standards*[4]

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

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

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.

5.2 *Definitions of Terms Specific to this Standard*

5.2.1 *conducting boundary* — for the purposes of this Guide, 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.

5.2.1.1 *Discussion* — A conducting boundary is illustrated as a low-high junction in Figure 3a.

5.2.2 *insulating boundary* — for the purposes of this Guide, 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.

5.2.2.1 *Discussion* — An insulating boundary is illustrated as a *p-n* junction in Figure 3b.

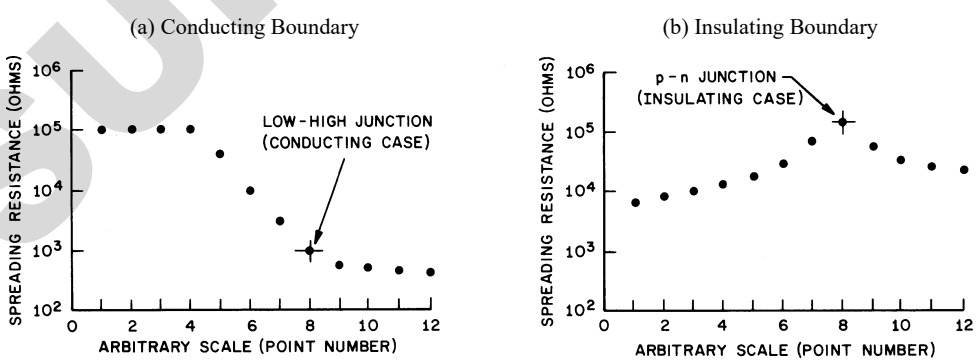

Figure 3
Identification of Boundaries from a Spreading Resistance Profile

[4] 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

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6 Summary of Measurement Procedure

6.1 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[5,6,7] must be used 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. If desired, the carrier concentration at each point on the profile is determined from the resistivity.

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 and plug holder as shown in Figure 4 and a flat plate of glass prepared in accordance with ¶ 10.1 of SEMI MF674 or a flat plate of a suitable plastic such as methyl methacrylate.

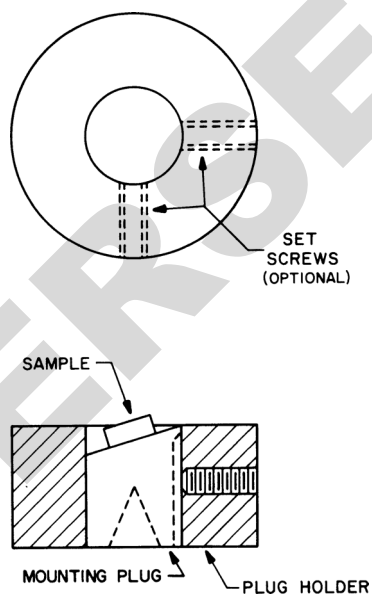

Figure 4
Typical Plug (Beveling Block) and Holder Used for Beveling

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 3 µm or less.

7.2 *Means for Measuring Bevel Angle* — Appropriate for the agreed-upon method (see Appendix 1, and Figure 5).

[5] D'Avonzo, D. L., Rung, R. D., and Dutton, R. W. "Spreading Resistance for Impurity Profiles." Stanford University Technical Report No. 5013-2, February 1977.

[6] Dickey, D. H., and Ehrstein, J. R. "Semiconductor Measurement Technology: Spreading Resistance Analysis for Silicon Layers with Nonuniform Resistivity." NBS Special Publication 400-48, May 1979.

[7] Morris, G. L., Langer, P. H., and White, J. C. "SRPROF, a Fast and Simple Program for Analyzing Spreading Resistance Profile Data." Semiconductor Measurement Technology: Spreading Resistance Symposium, op. cit., pp. 63–74.

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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 ¶ 14.1).

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 touches 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.

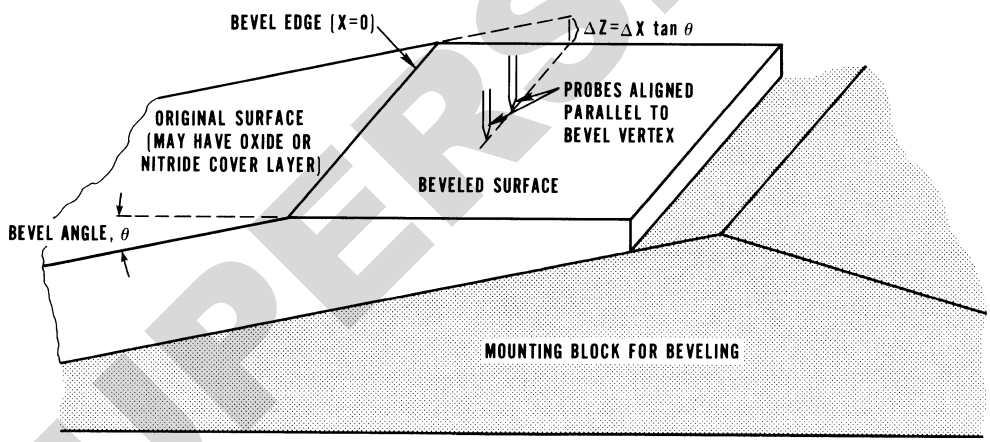
7.3.1.2 *Probe Loading and Descent Rate* — The loading applied to each point shall be in the range from 5 to 50 gf (49 to 490 mN), inclusive. A dashpot, or other means, for controlling the descent rate of the probes must be available if the load is applied by dead weight, but may not be necessary if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

NOTE 5: 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 6: 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 dc isolation resistance of 1 GΩ or greater between any pair of probes and between each probe and any guard circuit used.



#1 Ideal beveling consists of two planar surfaces intersecting along a straight line with no transition region between the planar surfaces.

Figure 5
Sketch of Beveled Silicon Wafer on Mounting Block Showing Ideal Beveling with Two Probes Aligned Parallel with the Bevel Edge

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 7: Typical step intervals are 1, 2.5, 5, and multiples of 10 times these values.

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7.4 *Vibration-Free Table* — For supporting apparatus as required (see ¶ 3.4).

7.5 *Apparatus Enclosure* — For providing darkened environment for spreading resistance measurements, if required by specimen material (see ¶ 3.2).

7.6 *Electrical Measuring Apparatus* — For a two-probe arrangement, use any of the following apparatus. For a three-probe arrangement, use the apparatus of ¶ 7.6.2.

7.6.1 *Constant-Voltage Method Usable only with the Two-Probe Arrangement (see Figure 6)*

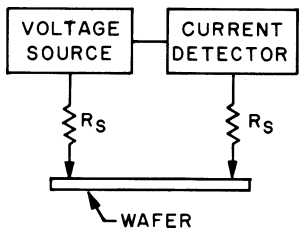
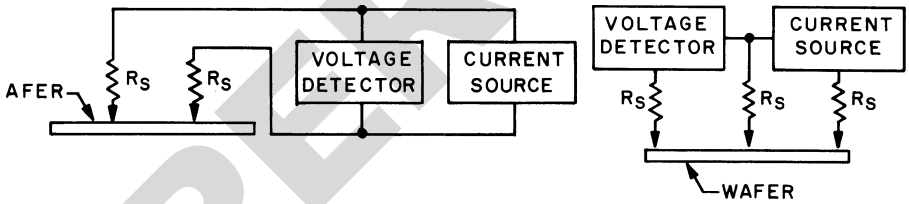


Figure 6
Electrical Circuit for the Constant-Voltage Method

7.6.1.1 *DC 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 *DC 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 Usable with Either the Two-Probe or Three-Probe Arrangement (see Figure 7)*



(a) Two-Probe Arrangement (b) Three-Probe Arrangement

Figure 7
Electrical Circuit for the Constant-Current Method

7.6.2.1 *Variable DC 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 specimen voltages that are measured remain in the range from 1 to 20 mV, inclusive, for all measurement points.

NOTE 8: The compliance voltage should not exceed 40 V for reasons of operator safety.

NOTE 9: 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 *DC 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.

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