



Designation: F 525 – 00a

Standard Test Method for Measuring Resistivity of Silicon Wafers Using a Spreading Resistance Probe¹

This standard is issued under the fixed designation F 525; 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 measurement of the resistivity of a silicon substrate of known orientation and type, or of a uniform silicon epitaxial layer of known orientation and type that is deposited on a substrate of the same or opposite type. Resistivity of the epitaxial films can be evaluated without the necessity of thin film correction factors provided that the ratio of layer thickness to effective probe contact radius is greater than 20.

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

1.3 This test method is intended for use on silicon substrates and epitaxial layers. Within-laboratory precision has been determined through a multilaboratory experiment on substrates having resistivities from 0.01 to 200 Ω·cm.

1.3.1 The principles of this test method can be extended to lower and higher specimen resistivity values, but the precision of the test method has not been evaluated for values other than those in the range given in 1.3.

1.4 This test method is nondestructive in the sense that the specimen is not totally destroyed in making the measurements, the specimen need not be cut into a special shape, and no destructive processing need be done on the specimen. However, the probe can produce mechanical damage that may be detrimental to a device fabricated in the probed area.

1.5 The volume of semiconductor material sampled is proportional to the third power of the effective electrical contact radius of the probe. For an effective electrical contact radius of 2 μm, the volume sampled by a single probe is approximately 10⁻¹¹ cm⁻³.

1.6 *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 applica-*

bility of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

- E 1 Specification for ASTM Thermometers²
- F 26 Test Methods for Determining the Orientation of a Semiconductive Single Crystal³
- F 42 Test Method for Conductivity Type of Extrinsic Semiconducting Materials³
- F 84 Test Method for Measuring Resistivity of Silicon Slices with an In-Line Four-Point Probe³
- F 95 Test Method for Thickness of Lightly-Doped Silicon Epitaxial Layers on Heavily Doped Silicon Substrates Using an Infrared Dispersive Spectrophotometer³
- F 110 Test Method for Thickness of Epitaxial or Diffused Layers in Silicon by the Angle Lapping and Staining Technique³
- F 374 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 Procedure³
- F 419 Test Method for Determining Carrier Density in Silicon Epitaxial Layers by Capacitance-Voltage on Fabricated Junction or Schottky Diodes³
- F 1392 Test Method for Determining Net Carrier Density Profiles in Silicon Wafers by Capacitance-Voltage Measurements With a Mercury Probe³
- F 1393 Test Method for Determining Net Carrier Density in Silicon Wafers by Miller Feedback Profiler Measurements With a Mercury Probe³

3. Terminology

3.1 Definitions of Terms Specific to This Standard:

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

$$a = \sqrt{\rho/4R_s}$$

where:

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.

Current edition approved Dec. 10, 2000. Published February 2001. Originally published as F 525 – 77 T. Last previous edition F 525 – 00.

² Annual Book of ASTM Standards, Vol 14.03.

³ Annual Book of ASTM Standards, Vol 10.05.

n = number of current-carrying probes across which the potential drop is determined,
 ρ = resistivity of a homogeneous semiconductor specimen, $\Omega \cdot \text{cm}$, and
 R_s = measured spreading resistance, Ω .

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

3.1.2 *epitaxial layer—in semiconductor technology*, a layer of single crystal semiconducting material grown on a host substrate that determines its orientation.

3.1.2.1 *Discussion*—A structure may consist of several layers on a substrate: each layer is separated from a neighboring layer (or the substrate) by an interface region.

3.1.3 *spreading resistance, $R_s(\Omega)$* —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.

3.1.3.1 *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 these gradients or boundaries.

3.1.3.2 *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

$$R_s = V/I$$

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 probes. In this case, the voltage-to-current ratio, and hence the spreading resistance, is approximately twice that associated with a single probe.

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

3.1.4.1 *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 The spreading resistance of a reproducibly formed point contact is measured. The resistivity of the material in the vicinity of the probe is determined from a calibration curve derived from spreading resistance measurements made under the same conditions on reference specimens of known resistivity. The spreading resistance may be measured with the use of one, two, or three probes and (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 a resistance comparator technique.

5. Significance and Use

5.1 This test method provides means for directly determining the resistivity of a substrate or of an epitaxial layer of thickness greater than 20 times the effective electrical contact radius. Unlike Test Methods F 84, F 374, F 419, F 1392, and F 1393, it can provide lateral spatial resolution of resistivity on the order of a few micrometres.

5.2 This test method is intended primarily for use in process control, research, and development applications. In the absence of between-laboratory precision data, this test method is not recommended for use between supplier and purchaser.

6. Interferences

6.1 *Temperature*—Spreading resistance measurements are sensitive to the temperature of the specimen. Therefore, it is important that the calibration and actual measurements be made at the same temperature.

6.2 *Light*—Photoconductive and photovoltaic effects can seriously influence the resistance determined by this test method, especially on slices with p - n junctions. All determinations should be made in a dark chamber, or in diffuse room light if experience shows that the material is insensitive to illumination of this type.

6.3 *Radio-Frequency Fields*—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.

6.4 *Mechanical Vibrations*—The apparatus may be sensitive to building or other vibrations. The probe should be located in an area free from significant vibrations. It may be desirable to mount the probe assembly on a vibration-free table.

6.5 *Minority Carrier Injection*—Caution should be taken to prevent minority carrier injection during the measurement. Experience has shown that if the potential on the probe is kept to 20 mV or less, minority carrier injection will 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, leads to changes in the characteristics of the instrument and to nonreproducible measurements. Probes and specimens should be protected from such exposure. Relative humidity in excess of 60 % should be avoided.

6.7 Semiconductor Surfaces:

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. Surfaces exposed to solutions containing fluorine ions may also exhibit instability. The heat treatment included in the procedure (see 13.4) may reduce these instabilities.

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.

6.8 *Correction Factors*—When the ratio of epitaxial layer thickness, t , to the effective electrical contact radius of the probe, a , is less than 20, it is necessary to employ correction factors to determine the correct resistivity value from the

spreading resistance measurement.⁴

7. Apparatus

7.1 Mechanical Apparatus:

7.1.1 Probes and Probe Assembly—A spreading resistance probe assembly with provision for supporting and lowering either one, two, or three replaceable probe tips to the wafer surface at a reproducible rate and with a predetermined load. The supporting mechanism shall provide for spatial displacement of the probes for adjustment of the point of contact.

7.1.1.1 Probe Tip Material—A hard, durable, low-resistance substance that wears well without flaking, such as osmium, tungsten carbide, or tungsten-ruthenium alloys.

7.1.1.2 Probe Tip Radius—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.

7.1.1.3 Probe Loading and Descent Rate—The loading applied to each point shall be less than 50 gf (L 490 mN). A 1-mm/s descent rate has been found to be adequate for a 40-gf (390-mN) load (see 11.3).

7.1.1.4 Probe Spacing, as used for calibration (see Section 12).

NOTE 1—Typical probe spacings are between 15 and 1000 μm .

7.1.1.5 Probe Insulation—A d-c isolation resistance of 1 G Ω or greater between any pair of probes and between each probe and any guard circuit used.

7.1.2 Sample Holder—An insulated vacuum chuck or other means for clamping the substrate tightly while measurements are made.

NOTE 2—The vacuum chuck may be inserted on a high-resolution translatable microscope stage with drum calibrated preferably in metric units. Gear boxes for stage movement should allow step-size movements in the range from 5 to 500 μm per step, inclusive. Usual increments are 5.0, 10, 25, and multiples of 10 thereof.

7.2 Electrical Measuring Apparatus:

7.2.1 Constant-Voltage Method, suitable for use with the one- or two-probe arrangement. See Fig. 1.

7.2.1.1 D-C Voltage Source, with a constant output between 1 and 20 mV, inclusive. The output potential shall be constant to within $\pm 0.1\%$ into a load that varies from 1 Ω to 10 M Ω , inclusive.

⁴ Ehrstein, J. R., Ed., *Semiconductor Measurement Technology: Spreading Resistance Symposium*, NBS Special Publication 400-10, December 1974. ASTM STP 572, ASTM, 1974.

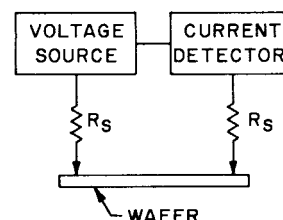
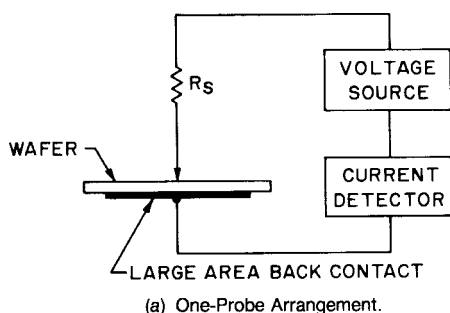


FIG. 1 Electrical Circuits for the Constant-Voltage Method

7.2.1.2 D-C Current Detector, accurate to within $\pm 0.1\%$ and capable of covering the range from 10 mA to 1 pA, inclusive.

7.2.2 Constant-Current Method, suitable for use with the one-, two-, or three-probe arrangement. See Fig. 2.

7.2.2.1 Variable D-C Current Source, capable of providing currents from 10 nA to 10 mA, inclusive. The current source shall be accurate to within $\pm 0.1\%$ and capable of providing a current of 10 nA into a 10-M Ω load. **Caution:** For safety reasons the compliance voltage should not exceed 40 V.

7.2.2.2 D-C Voltage Detector, linear over the range from 1 to 100 mV, inclusive, and accurate to within $\pm 0.1\%$ of the reading. The input impedance shall be 10 M Ω or greater.

7.2.3 Comparator Method, suitable for use with the one- or two-probe arrangement. See Fig. 3.

7.2.3.1 D-C Voltage Source, with a constant output between 1 and 20 mV, inclusive, but no special requirements on the precision.

7.2.3.2 Log Comparator, with an output voltage proportional to the logarithm of the ratio of two currents. Together with its standard resistor, R_0 , the comparator shall be capable of measuring resistances from 1 Ω to 10 M Ω inclusive, with a deviation from linearity not greater than $\pm 1\%$.

7.2.3.3 Standard Resistor, nominally 10 k Ω , known to an accuracy of $\pm 0.1\%$.

7.3 Microscope, capable of a magnification of at least 400 $\times$.

7.4 Thermometer—An 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.

8. Reagents and Materials

8.1 Resistivity Standards for Calibration:

8.1.1 Resistivity standards shall be chosen from substrates in the resistivity range of the unknowns in accordance with Section 12.

NOTE 3—It is desirable to use at least three resistivity values per decade of resistivity. For applications where spreading resistance measurements are being used to sort material within a narrow resistivity range (one decade or less) it may be necessary to have 8 or 10 calibration specimens spanning the decade of interest to compensate for the resistivity averaging effect of a four probe on nonhomogeneous calibration specimens. This is particularly important for resistivity ranges where calibration material is found to have significant fine-scale resistivity variations (striations).

8.1.2 The standards shall match the conductivity type and orientation of the test specimens. The surface finish of the standards shall match the surface finish of the test specimens as

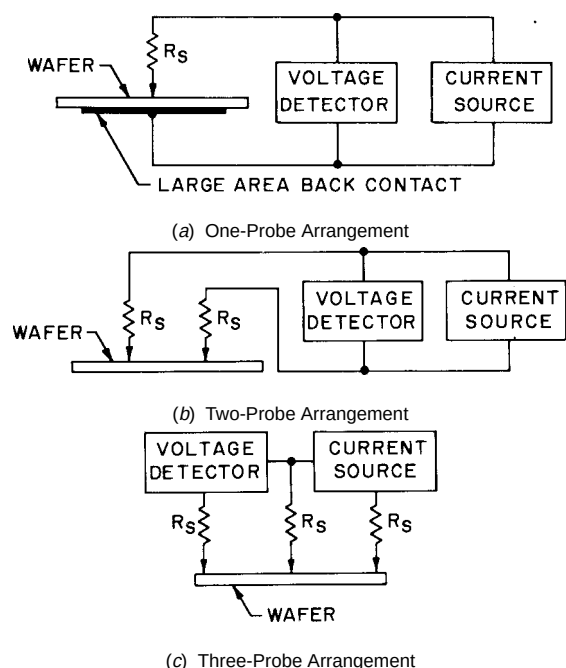


FIG. 2 Electrical Circuits for the Constant-Current Method

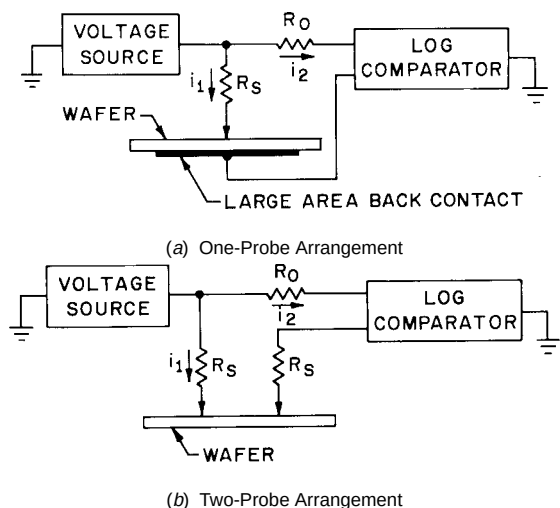


FIG. 3 Electrical Circuits for the Log Comparator Method

closely as possible. For use in connection with epitaxial layer test specimens, standards shall have a polished surface.

8.2 A *p*-type silicon specimen of uniform resistivity with a {111} orientation, for which the typical spreading resistance per probe tip is between 300 and 1000 Ω , shall be used to ensure that the probe tips are properly conditioned (see 11.4.1).

NOTE 4—An epitaxial layer of 0.1 to 1 $\Omega \cdot \text{cm}$, 10 μm or thicker is acceptable.

8.3 A substrate, lapped or ground with 5- μm grit slurry or a tape or cloth coated with an abrasive grit of 8000 to 12 000 grade, shall be used for conditioning the probe tips (see 11.4).

8.4 *Specimen Preparation Materials*, consisting of polishing materials and pads or other backing plates, as follows.

8.4.1 *Silica Powder*, colloidal or non-colloidal in aqueous suspension with pH from 7.0 to 11.5 and either hard plastic

such as methyl methacrylate (for small area specimens) or a poromeric polishing pad of urethane or polyurethane foam with a napped or sueded surface and attached to a rigid backing plate (for large area specimens).

8.4.2 *Diamond Suspension* in fluid or paste carrier with diamond in the range from 0.1 to 1 μm , together with (1) a glass plate lapped with aluminum oxide in the range from 5 to 12 μm (and thoroughly removed subsequent to lapping), for use with small area specimens, or (2) a nonwoven chemotextile polishing pad attached to a rigid backing plate, for use with large or small area specimens.

8.4.3 *Aluminum Oxide*, 0.3- μm particle size in water together with a non-woven chemotextile polishing pad bonded to a rigid backing plate.

8.4.4 *Aluminum Oxide*, 5- μm particle size, together with a frosted glass or cast iron lapping plate.

9. Safety Hazards

9.1 Use normal safety precautions in operating the electrical equipment.

10. Sampling

10.1 Since the most suitable sampling plan will depend upon individual conditions, no general sampling procedure is included here. For referee purposes a sampling plan shall be agreed upon by the testers before the tests are started.

11. Preparation of Apparatus

11.1 Adjust the probe separation to the desired value if a multiple-probe arrangement is employed. Record the probe separation.

11.2 Choose a loading in the range from 10 to 100 gf (98 to 980 mN), inclusive, to be applied to the probes. In a multiple-probe arrangement, use the same loading for each probe.

11.3 Once the loading on the probes has been chosen, determine an appropriate descent rate of the probes on the specimen to minimize damage to the points and avoid fracturing the specimen.

NOTE 5—The descent rate specified in 7.1.1.3 is adequate for a load of 40 gf (390 mN).

11.4 Condition for probe tips by stepping them at least 500 times on a silicon substrate that has been ground with 5- μm grit slurry (see 8.3) or very lightly abrade the individual probe tips using the abrasive cloth or tape.

NOTE 6—It is permissible to leave the slurry on the substrate during conditioning, but it must be removed from probe tips before measuring test specimens.

11.4.1 Measure the spreading resistance of the uniform *p*-type silicon specimen (see 8.2) in accordance with 13.4 through 14.2. Consider the probes to be satisfactory if the measured spreading resistance is within $\pm 10\%$ of the usual value that has been found to be consistent over a long period of time.

11.4.2 If the probes are not satisfactory, repeat the conditioning step or use new probe tips.

11.5 Using the microscope at 400 $\times$, examine the probe marks for reproducibility (Note 7). If the probe marks from a given probe do not appear similar, recondition (see 11.4) or replace the probe.

 F 525

NOTE 7—All the marks from a specific probe should appear to be similar. Probe imprints from different probes need not be identical.

11.6 If the two-probe arrangement is being employed, verify that the spreading resistances of the two probes are equal to within 10 % when measured on the uniform *p*-type specimen (see 8.2) using the one-probe configuration. If they are not, recheck or adjust the loading (see 11.2) and descent rate (see 11.3) to be equal on both probes. If satisfactory results are not achieved with equal probe loading and descent rate, recondition (see 11.4) or replace the probes.

11.7 Connect the appropriate electrical circuit (see Fig. 1, Fig. 2, and Fig. 3). 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 (constant-current method), short circuit the output.

11.8 Note and record the voltage to be applied in millivolts as *V* (constant-voltage method), the current to be applied in milliamperes as *I* (constant-current method), or the value of the standard resistor in ohms as *R*₀ (comparator method).

12. Calibration

12.1 Measure the resistivity of the specimens to be used as standards (see 8.1) at $23 \pm 3^\circ\text{C}$ in accordance with Test Method F 84, if not previously measured. Record the results.

12.2 Prepare the surface of the calibration specimens using the materials of 8.4.1, 8.4.2, 8.4.3, or 8.4.4 using manual procedure or machine, the choice of materials and procedure being governed only by the requirement that the calibration specimens must be prepared in the same manner as the specimens to be tested. If the specimen to be tested is epitaxial, the use of a lapped surface is not to be used for the calibration specimens.

12.2.1 Clean the polishing material from the calibration specimens using water, or solvent in the case of solvent-soluble vehicles used for certain diamond polishes. Place the specimen on a hot plate at $150 \pm 20^\circ\text{C}$ for 10 to 15 min if calibration specimens were prepared with silica or aluminum oxide polishing or lapping compounds.

NOTE 8—If the chosen calibration specimen preparation is anything other than lapping with 5 μm aluminum oxide, it is advisable to remove a minimum of 25 μm of specimen surface the first time the specimens are prepared, subsequent to resistivity measurement by Test Method F 84 in order to remove any subsurface damage left by the 5 μm lapping process required in Test Method F 84.

NOTE 9—The frequency with which calibration specimen surfaces should be reprepared is a function both of the inherent stability of the surface for the chosen preparation procedure, and of possible exposure of the calibration specimens to environments such as acid fumes. The stability of the specimen surfaces for the chosen procedure should be established by each user of this method.

12.3 Make a minimum of 20 spreading resistance measurements on each of the proposed standards. Make the measurements as closely as possible to the region where the four-probe measurements (see 12.1) were made. Make these measurements over a length approximately equal to the separation of the outer probe tips of the four-probe array used for the resistivity measurements. Record the results.

12.4 Compute the mean of the measurements made on each of the proposed standards and calculate the standard deviation for each set of measurements. If the standard deviation of the

spreading resistance measurements is greater than 10 % of the mean, do not use the specimen as a calibration standard.

12.5 Using the resistivity value and the corresponding spreading resistance mean for each suitable calibration standard, fit an appropriate polynomial, piecewise linear, or spline curve to the calibration data for the groups of specimens of each conductivity type and orientation. Plot the data and calibration curve on graph paper.

NOTE 10—See Appendix X1 of Test Method F 419 for a computer program to generate a polynomial fit.

13. Procedure

13.1 Handle the specimen carefully to avoid contamination or damage to the surface.

13.2 Make all measurements with an ambient temperature of $23 \pm 3^\circ\text{C}$. Record the ambient temperature.

13.3 Determine the orientation of the specimen surface in accordance with Test Methods F 26, determine the conductivity type of the substrate and, if present, the epitaxial layer in accordance with Test Method F 42. For epitaxial layers, determine the thickness in accordance with either Test Method F 95 or Test Method 110. Record the results.

13.4 Prepare all bulk specimens to be tested using the same procedure as was used for the calibration specimens in 12.2. Do not prepare the surface of any epitaxial specimens. Thoroughly clean polishing material residues from the specimen using water or solvent as necessary. If the materials of 8.4.1 or 8.4.3 were used for specimen preparation, proceed to 13.5, otherwise proceed to 13.6.

13.5 Place the specimen on a hot plate with the surface to be measured upwards, not in contact with the hot plate. Heat the specimen in air at $140 \pm 20^\circ\text{C}$ for 10 to 15 min. Remove the specimen from the hot plate. Allow the specimen to return to $23 \pm 3^\circ\text{C}$ before continuing with the measurements.

13.6 Rigidly mount specimen on stage under probes. Use of wax mounting to base block used to hold specimen during surface preparation or use of vacuum clamping is acceptable. Position the specimen on the specimen holder so that the probe or probes will be applied at the desired measurement location.

13.7 Lower the points to make contact with the specimen surface, and adjust to within 0.1 % (unless the comparator method is being used) the voltage or current source to the desired value (see 11.8).

13.8 After a stabilization period of 1 s or longer, measure and record the current in milliamperes as *I* (constant-voltage method), the voltage in millivolts as *V* (constant-current method), or the output of the log comparator as $\log(i_1/i_2)$ (log comparator method).

13.9 Adjust the voltage source to a value of 20 mV or less or short circuit the current source and lift the probes.

13.10 Move the specimen to the next position, making sure that the step spacing is larger than the diameter of the specimen area damaged by the probes.

13.11 Note and record the step spacing.

13.12 Repeat 13.6 through 13.9 until the desired number of measurements has been made.

14. Calculation

14.1 Calculate the spreading resistance, *R*_s, in ohms, for