



Standard Guide for Application of Silicon Standard Reference Materials and Reference Wafers for Calibration and Control of Instruments for Measuring Resistivity of Silicon¹

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1. Scope

1.1 This guide covers the application of Standard Reference Materials (SRMs²) for resistivity measurements on silicon wafers. Specifically, this guide covers the use of these SRMs for preparing resistivity reference wafers and for ensuring the quality of the instrumentation used for preparing them.

1.2 This guide has not been evaluated for application to electronic materials other than silicon.

1.3 The guide covers the selection of materials for resistivity reference wafers, procedures for preparing and calibrating resistivity reference wafers, and use of resistivity reference wafers in qualifying, calibrating, and controlling various types of resistivity instrumentation.

1.4 The guide provides criteria for selection of instruments for determining the resistivity of silicon resistivity reference materials, procedures for maintaining such instruments in statistical quality control, and training requirements for operators engaged in making and using resistivity reference wafers.

1.5 Appendixes are included that cover (1) suggested control charting procedures for organizations that do not already have such procedures in place, and (2) errors in resistivity determination that result from uncertainties in wafer diameter, wafer thickness, and probe-tip spacing.

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 applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 5127 Guide for Ultra Pure Water Used in the Electronics and Semiconductor Industry³

F 43 Test Methods for Resistivity of Semiconductor Materials⁴

F 81 Test Method for Measuring Radial Resistivity Variation on Silicon Wafers⁴

F 84 Test Method for Measuring Resistivity of Silicon Wafers With an In-Line Four-Point Probe⁴

F 525 Test Method for Measuring Resistivity of Silicon Wafers Using a Spreading Resistance Probe⁴

F 533 Test Method for Thickness and Thickness Variation of Silicon Slices⁴

F 613 Test Method for Measuring Diameter of Semiconductor Wafers⁴

F 672 Test Method for Measuring Resistivity Profile Perpendicular to the Surface of a Silicon Wafer Using a Spreading Resistance Probe⁴

F 673 Test Method for Measuring Resistivity of Semiconductor Slices or Sheet Resistance of Semiconductor Films with a Noncontact Eddy-Current Gage⁴

F 723 Practice for Conversion Between Resistivity and Dopant Density for Boron-Doped, Phosphorus-Doped, and Arsenic-Doped Silicon⁴

F 1241 Terminology of Silicon Technology⁴

F 1392 Test Method for Determining Net Carrier Density Profiles in Silicon Wafers by Capacitance-Voltage Measurements with a Mercury Probe⁴

F 1393 Test Method for Determining Net Carrier Density in Silicon Wafers by Miller Feedback Profiler Measurements with a Mercury Probe⁴

F 1529 Test Method for Sheet Resistance Uniformity Evaluation by In-Line Four-Point Probe with the Dual-Configuration Procedure⁴

F 1530 Test Method for Measuring Flatness, Thickness, and Thickness Variation on Silicon Wafers by Automated Noncontact Scanning⁴

2.2 ISO Standards:

Guide 30:1981 Terms and Definitions Used in Connection with Reference Materials⁵

ISO 8402 Quality—Vocabulary⁵

¹ This guide is under the jurisdiction of ASTM Committee F-1 on Electronics and is the direct responsibility of Subcommittee F01.06 on Silicon Materials and Process Control.

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² SRM® is a registered trademark of the U.S. National Institute of Standards and Technology and the U.S. Government.

³ *Annual Book of ASTM Standards*, Vol 11.01.

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

⁵ ISO Central Secretariat, C. P. 56, CH-1211 Genève 20, Switzerland; available in the U.S. from American National Standards Institute, 11 West 42nd Street, 13th Floor, New York, NY 10036.

ISO 10012-1 Quality Assurance Requirements for Measuring Equipment—Part 1: Management of Measuring Equipment⁵

2.3 SEMI Standards:

SEMI C28 Specifications for Hydrofluoric Acid⁶

SEMI C35 Specification for Nitric Acid⁶

SEMI C39 Specification for Potassium Hydroxide Pellets⁶

SEMI M1 Specifications for Polished Monocrystalline Silicon Wafers⁶

3. Terminology

3.1 Definitions of Terms Related to Reference Materials:

3.1.1 *certified reference material (CRM)*—a reference material one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body (ISO Guide 30:1981).

3.1.2 *reference material (RM)*—a material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, (for) the assessment of a measurement method, or for assigning values to materials (ISO Guide 30:1981; ISO 10012-1).

3.1.3 *resistivity reference wafer*—a CRM or RM in the form of a silicon wafer or chip used for routine calibration or control of resistivity measuring equipment.

3.1.4 *Standard Reference Material (SRM)*—a certified reference material issued by the U.S. National Institute of Standards and Technology.

3.1.5 *traceability*—the ability to trace the history, application, or location of an item or activity, or similar items or activities, by means of recorded identification (ISO 8402).

DISCUSSION—ISO 8402 states that in a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, or basic physical constants or properties. In this guide, as in ISO 10012-1, the term “measuring equipment” is extended to include both measuring instruments and measurement standards (including reference wafers).

3.2 Definitions of Terms Related to Four-Point Probes:

3.2.1 *four-point probe*—an electrical probe arrangement for determining the resistivity of a material in which separate pairs of contacts are used (1) for passing current through the specimen, and (2) measuring the potential drop caused by the current.

3.2.2 *probe head, of a four-point probe*—the mounting that (1) fixes the positions of the four pins of the probe in a specific pattern such as an in-line (collinear) or square array, and (2) contains the pin bearings and springs or other means for applying a load to the probe pins.

3.2.3 *probe pin, of a four-point probe*—one of the four needles supporting the probe tips; mounted in a bearing contained in the probe head and loaded by a spring or dead weight.

3.2.4 *probe tip, of a four-point probe*—the part of the pin that contacts the wafer.

3.2.5 *probe-tip spacing, of a four-point probe*—the distance between adjacent probe tips.

3.3 Other terms related to semiconductor technology are defined in Terminology F 1241.

4. Significance and Use

4.1 Resistivity is a widely used parameter for specification and characterization of silicon wafers for use in fabricating semiconductor devices and integrated circuits. Many types of instrumentation used for making resistivity measurements including the noncontact eddy-current instruments used for measurements made in accordance with Test Method F 673 require calibration because they are relative measurements. Although measurements made with in-line four-point probes in accordance with Test Method F 84 are, in principle, absolute, control charts should be maintained for four-point probes because one cannot always be sure that the electrical resistivity of the test specimen is sufficiently homogeneous for the theoretical model of the method to apply, that the electrical thickness of the wafer is exactly equal to its measured mechanical thickness, or that the stability of the instrument is adequate.

4.2 Instruments for measuring such related parameters as spreading resistance (used in accordance with Test Method F 525 or Test Method F 672) and net carrier density (used in accordance with Test Method F 1392 or Test Method F 1393) also require calibration.

4.3 For all these purposes, wafers of known resistivity are required. Such wafers are supplied by several sources with a wide range of certified or calibrated resistivity values. Although these wafers are often used directly, the resistivity values represented by purchased standards can also be transferred to an in-house resistivity reference wafer that is then used for routine instrument calibration or control.

4.4 The accuracy with which this transfer can be effected depends not only on the procedures for using such reference wafers but also on the procedures for material selection, instrument qualification, and calibration of the reference wafer. This guide provides recommendations for procedures for these operations appropriate to obtaining the best available accuracy in use of resistivity reference wafers.

4.5 These procedures are specifically intended for use in measuring the resistivity of silicon wafers. Extension to resistivity measurements on other semiconductor materials or to resistivity values outside the range covered by the resistivity reference wafers has not been demonstrated.

5. Reagents

5.1 *Purity of Reagents*—All chemicals for which such specifications exist shall conform to Grade 1 SEMI specifications for those specific chemicals. Other grades may be used, provided it is first determined that the chemical is of sufficiently high purity to permit its use without lessening the accuracy of the test.

5.2 *Purity of Water*—Reference to water shall be understood to mean deionized water meeting the resistivity and impurity specifications of Type I Reagent Water in Specifications D 5127.

⁶ Available from Semiconductor Equipment and Materials International, 805 E. Middlefield Rd., Mountain View, CA 94043.

5.3 The recommended chemicals shall have the following nominal assays:

5.3.1 *Nitric Acid*, HNO₃, concentrated, 70 to 71 %,

5.3.2 *Hydrofluoric Acid*, HF, concentrated, 49.00 ± 0.25 %, and

5.3.3 *Potassium Hydroxide*, KOH, pellets, 85 % min.

5.4 *KOH Etching Solution*—Dissolve potassium hydroxide (KOH) in water to make a 50 weight % KOH solution in sufficient volume to allow complete immersion of the largest wafers to be prepared for test. This solution is to be used at a temperature of 65 to 75°C.

5.5 *Etching Solution (15 + 1)*—Mix 15 parts of nitric acid (HNO₃) with one part of hydrofluoric acid (HF) in sufficient volume to allow complete immersion of the largest wafers to be prepared for test.

6. Description of Resistivity SRMs

6.1 *Type, Form, and Nominal Resistivity of SRMs*—The National Institute of Standards and Technology has supplied, or does supply, two types of SRMs for resistivity measurements.

6.1.1 SRMs for spreading resistance measurements (no longer being produce) were in accordance with Test Method F 672 were supplied as sets of 16 rectangular chips, approximately 6 mm by 12 mm, covering the resistivity range from about 0.001 Ω-cm to about 100 Ω-cm. Four sets were supplied, one for each combination of conductivity type and surface orientation as listed in Table 1. Procedures for selecting materials and certifying the values of these chips have been described in detail (1).⁷ This report also provides detailed guidance on care and use of these SRM sets.

6.1.2 SRMs for bulk wafer measurements of resistivity have been supplied as sets of 2-in. diameter, 625-μm thick lapped silicon wafers (see Table 2).⁸ Wafers with nominal resistivity of 10 Ω-cm and lower were cut from boron-doped (*p*-type) (111) float zoned (FZ) crystals or (111) or (100) Czochralski (Cz) crystals. Wafers with nominal resistivity of 25 Ω-cm and higher were cut from high purity (111) FZ crystals that were phosphorus doped by neutron transmutation. These crystal types and doping methods were selected to provide the best possible radial and axial uniformity of resistivity. Nevertheless, resis-

⁷ The boldface numbers in parentheses refer to the references at the end of this guide.

⁸ Resistivity SRMs are available from the Standard Reference Materials Program, Room 204, Bldg. 202, National Institute of Standards & Technology, 100 Bureau Dr., Gaithersburg, MD 20899, (301) 975-6776. SRMs 2528, 2529, 1521, 1522, and 1523 are no longer in stock; a small quantity of SRMs 2526 and 2527 are available for resistivity by spreading resistance. SRMs 2541 through 2547 for sheet resistance and resistivity are available in the form of 100-mm diameter wafers, nominally 625-μm thick. Calibrated resistivity Reference Materials, in several sizes, and traceable to NIST, are available from VLSI Standardards Inc., 3087 N. 1st St., San Jose, CA 95134; (408) 428-1800.

TABLE 1 Standard Reference Materials for Spreading Resistance Profiling

SRM	Conductivity Type	Orientation
SRM 2526	<i>p</i>	(111)
SRM 2527	<i>n</i>	(111)
SRM 2528	<i>p</i>	(100)
SRM 2529	<i>n</i>	(100)

TABLE 2 Resistivity Standard Reference Material Wafers^A

SRM	Nominal Resistivity, Ω-cm	Growth Method	Orientation	Conductivity Type	Dopant
SRM 1521	0.1, 10	Float Zone ^B	(111) ^B	<i>p</i>	boron
SRM 1522	25, 75, 180	Float Zone	(111)	<i>n</i>	phosphorus (NTD)
SRM 1523	0.01	Czochralski	(100)	<i>p</i>	boron
	1	Float Zone ^B	(111) ^B	<i>p</i>	boron

^A Nominal diameter: 2 in.; nominal thickness: 625 μm.

^B As the original supply of float zone (111) crystals was consumed, they were replaced by (111) or (100) Czochralski crystals.

tivity nonuniformity remains one of the most significant limiting factors in establishment of the resistivity value.

6.2 *Selection of Crystals for Resistivity SRM Wafers*—Crystals for use in preparation of resistivity SRM wafers are chosen on the basis of nominal center-point resistivity value, longitudinal resistivity uniformity, and radial resistivity uniformity. The resistivity at each end of a crystal section proposed for use in fabricating resistivity SRMs is measured in accordance with the Four-Probe Method of Test Methods F 43. If the range is acceptable, wafers are sliced from the crystal section. The radial resistivity variation of selected wafers is determined along two perpendicular diameters in accordance with Sampling Plan D of Test Method F 81.

6.3 *Preparation of SRM Wafers*—If the variation is acceptable, the wafers are etched in KOH etching solution to remove about 25 μm from each side, and lapped with 5- to 9-μm alumina abrasive to provide a specimen with flat, matte surfaces and a thickness variation (to half radius) of less than ±1 % of the thickness at the center of the wafer.

6.4 *Certification of Parameter Values*—The resistivity at the center point is determined in accordance with Test Method F 84 except that (1) the number of measurements is six instead of 10 and (2) the current is set to maintain a measured specimen voltage between 10 and 12 mV for all except the lowest resistivity (0.01 Ω-cm) wafers for which the developed voltage is smaller. An in-line four-point probe that has a nominal probe-tip spacing of 1.59 mm and that meets the conditions for suitability imposed by Test Method F 84 is used. Average center-point resistivity is corrected to a temperature of 23°C. Results obtained with the procedures used for the certification measurements have been related to industry consensus values through interlaboratory evaluations of Test Method F 84 (2).

6.5 *Repeatability and Reproducibility Achievable with SRM Wafers and Test Method F 84*—Several multi-instrument, multi-operator single laboratory and interlaboratory experiments have been conducted to determine the repeatability and reproducibility of average resistivity that can be obtained.

6.5.1 One six-laboratory test (3), conducted over a twelve-month period with a previously issued resistivity SRM,⁹ yielded the result that there is a 95 % probability that the averages of five replications taken in two different laboratories will not differ by more than 1.7 %.

⁹ SRM 1520, which contained wafers with characteristics of those in SRM 1521 except that the nominal thickness was 1 mm. SRM 1520 has been superceded by SRM 1521 and is no longer available.

6.5.2 A two-operator, two-instrument experiment using five wafers at each of the three resistivity levels in SRM 1522 showed that the 95 % confidence interval for the measurement averages derived from the instrument-operator combination used for normal certification were ± 0.11 %, ± 0.28 %, and ± 0.38 % for the 25, 75, and 180 $\Omega\cdot\text{cm}$ levels, respectively.¹⁰ Results of an interlaboratory experiment conducted to evaluate Test Method F 84 indicated that the reproducibility (at 95 % confidence) is ± 1.4 % of the average value for specimens with resistivity up to 120 $\Omega\cdot\text{cm}$ and ± 3.6 % of the average value for specimens with resistivity between 120 and 500 $\Omega\cdot\text{cm}$. Because the wafers used in this experiment were not as uniform as the neutron transmutation doped (NTD) wafers in SRM 1522, the latter are expected to result in better reproducibility.

6.5.3 In a two-operator, two-instrument experiment¹¹ using four wafers at each of the two resistivity levels in SRM 1523, no effect due to operator or instrument was found that was larger than 0.25 %, the average wafer measurement repeatability from any one instrument-operator combination. The earlier interlaboratory experiment conducted to evaluate Test Method F 84 indicates that resistivity scales in this range can be transferred with a reproducibility of average resistivity of ± 1.4 % at a confidence level of 95 %.

7. Control of Primary Resistivity Measuring Instruments

7.1 “Primary” resistivity measuring instruments are four-point probes used for calibrating resistivity reference wafers. Such instruments must (1) meet the requirements of Test Method F 84, (2) be maintained in a state of control through the use of \bar{X} ,s instrument control charts (see 7.1.1), (3) return measured resistivity values of appropriate CRMs to within desired limits through measurement of resistivity CRMs at regular intervals (see 7.1.2), and (4) be operated within the temperature range specified in Test Method F 84 (see X2.4 of this guide).

7.1.1 *Instrument Control Charts*— \bar{X} ,s control charts should be maintained to establish the stability of the instrument over the range of resistivity that the instrument is expected to measure. A separate control chart should be maintained for each reference wafer used. Initially, resistivity CRMs or other wafers with adequate radial resistivity uniformity (see 8.5.1) should be used for this purpose. As resistivity reference wafers are prepared, these should be used for maintaining the control charts. After in-house resistivity reference wafers become available, it is recommended that three wafers at each resistivity level be set aside for control charting. Two of these should be used for maintaining the \bar{X} ,s charts on a regular basis. The third is retained as a reference to ensure that any apparently out-of-control conditions are due to the instrument and not to changes in sample surface conditions, as might occur after prolonged use of a particular sample. Each resistivity determination should consist of six to ten measurements made in

accordance with Test Method F 84. Established procedures for generating and maintaining the control charts and for determining the existence of out-of-control conditions and the need for corrective action should be used. A suggested procedure for these determinations is given in Appendix X1 for use in organizations without previously established procedures.

7.1.2 *Comparison with CRMs*—To provide traceability, measurements of the resistivity of available CRMs that encompass the range of resistivity expected to be encountered should be made on a periodic, but less frequent, basis. In addition to being in control, the instruments should return measured values of the resistivity of the CRMs that do not deviate from their certified value by more than the multilaboratory reproducibility (see 6.5). A suggested procedure for these determinations is given in Appendix X1 for use in organizations without previously established procedures.

7.1.3 *Probe Assembly and Electrical Equipment Tests*—Should an out-of-control or out-of-specification condition be encountered, the probe assembly and electrical equipment can be tested in accordance with the section on Suitability of Test Equipment in Test Method F 84. These tests may serve to isolate the cause of the problem encountered. Either or both of these components should be repaired or replaced if they fail to meet the requirements specified in this section of Test Method F 84.

NOTE 1—The stringent requirements on control of probe-tip spacing as well as the need for off-center diameter correction factors required for determination of radial resistivity uniformity (see 8.5) are a consequence of the single-configuration method of using the four-point probe in accordance with Test Method F 84. The probe-tip spacing requirements can be relaxed significantly if the dual-configuration method (4) of measuring resistivity with a four-point probe is used. Use of this method is recommended for determination of the radial resistivity uniformity (see 8.5). However, the dual-configuration method has not yet been standardized for bulk resistivity measurements; when standard procedures are developed, this method will be incorporated into this guide. Errors resulting from uncertainty in probe-tip spacing when using the single-configuration method are considered in Appendix X2.

8. Selection and Qualification of Materials for Resistivity Reference Wafers

8.1 Factors that must be considered in selecting materials for resistivity reference wafers are nominal resistivity (see 8.2), wafer diameter (see 8.3), radial and axial uniformity of the resistivity (see 8.4 and 8.5), wafer thickness (see 8.6), wafer conductivity type and surface orientation (see 8.7), and surface finish (see 9.1.2). It is generally desirable to qualify a crystal section for the desired parameters and then verify that individual wafers meet the requirements during the calibration procedure. The uniformity requirements, in particular, depend on the specific application of the reference wafers because different resistivity measuring instruments are sensitive to different volumes of material.

8.1.1 *Four-Point Probe*—The most straightforward application of resistivity reference wafers is for control of four-point probes used for measurement of resistivity and resistivity variation. If the wafer under test has the same geometry and surface characteristics as the reference wafer and if the same probe geometry is used for the measurement of both the reference wafer and the wafer under test, the transfer is direct

¹⁰ Certificate, SRM 1522, available from the Standard Reference Materials Program. Because this SRM is out of stock and is no longer available, the information is provided for the use of the installed base of SRM 1522 users.

¹¹ Certificate, SRM 1523, available from the Standard Reference Materials Program. Because this SRM is no longer available, the information is provided for the installed base of SRM 1523 users.

and no special precautions need be observed. If the wafer under test has different thickness or diameter from the resistivity reference wafer or if four-point probes with different probe-tip spacings are used to measure the test and reference wafers, attention must be paid to the use of the appropriate correction factors; in particular, if the ratio of thickness to probe-tip spacing becomes too large while the ratio of diameter to probe-tip spacing becomes too small, second order errors due to the use of two two-dimensional corrections for a three-dimensional geometry may become significant. Also, if different probe-tip spacings are used, differences in sampling volume must be recognized; in this case, resistivity uniformity of both the reference wafer and the wafer under test limits the transfer accuracy. If the wafer under test has a different surface condition from that of the reference wafer, it is necessary to demonstrate the equivalence of the resulting measurement value with that which would have been obtained had the same surface condition been employed for both.

8.1.2 Eddy-Current Gages—The eddy-current detector is sensitive to the integrated sheet resistance across the thickness of the wafer; in this respect it is similar to the four-point probe. However, the eddy-current probe may weight various elements of the volume sampled differently than that of a four-point probe. Therefore, the weighted average resistivity seen in nonuniform wafers by the eddy current gage may not be the same as the center-point average resistivity value determined by the four-point probe. As a result, the accuracy of transfer between these types of instruments depends strongly on the macro-scale uniformity of the resistivity over the central area of the reference wafer. Because the eddy-current gage is a bulk measurement device, the thickness uniformity over the area sampled by the eddy-current probe is also important. The upper limit of the expected reduction of transfer accuracy is given as follows:

$$\text{maximum percent reduction} = \left(\left[\left(1 + \frac{\Delta\rho}{\rho} \right) \times \left(1 + \frac{TTV}{w} \right) \right] - 1 \right) \times 100 \quad (1)$$

where:

ρ = average center-point resistivity of resistivity reference wafer, corrected to 23°C, in Ω -cm,

$\Delta\rho$ = radial resistivity variation of resistivity reference wafer, corrected to 23°C, in Ω -cm, over the area sampled by the eddy-current probe,

TTV = total thickness variation of resistivity reference wafer, in μm , over the area sampled by the eddy-current probe, and

w = average thickness of resistivity reference wafer, in μm .

8.1.3 Spreading Resistance Probes—Because the spreading resistance probe samples a volume that is exceedingly small compared with the volume sampled by the four-point probe, the most critical reference wafer parameter for accurate transfer of resistivity value is the microvariation of the resistivity both axially and radially. Very complex and time consuming procedures are required to determine the microscale depth and lateral variations of resistivity in a reference wafer (see 8.4.3 and 8.5.2, respectively). Further, the procedures for determining microscale depth profiles are destructive and cannot be

carried out on the reference wafer itself. Consequently, for this application crystals should be grown by procedures that result in the best possible uniformity. These include neutron transmutation doping of very high purity FZ crystals to obtain high resistivity (neutron transmutation doped, or NTD) n -type wafers and magnetic Czochralski (MCz) crystals for both n - and p -type wafers.

8.1.4 Mercury Probes—Similar conditions exist for reference wafers intended for calibration and control of mercury probe systems that are primarily used for determining the net carrier density of epitaxial layers from capacitance measurements. Such probes sample a lateral area much smaller than the four-point probe and a depth that is only a very tiny fraction of the wafer thickness. To ensure that the resistivity in the region sampled by the mercury probe can be taken as the resistivity measured by the four-point probe, it is necessary to establish that both the macro- and micro-scale variations in resistivity of the reference wafer are within tolerable limits. Thus, it is desirable to use highly uniform crystal such as neutron transmutation doped FZ or MCz crystal for resistivity reference wafers for this application (see 8.1.3). It is further required in this case to establish the net carrier density of the reference wafer from the resistivity measurement (see 9.2.4).

8.2 Resistivity—The center-point resistivity of the reference wafer should be chosen to meet the requirements of the measuring instrument to be calibrated or controlled. Most of the test methods cited in Section 4 specify the resistivity range of the required resistivity reference wafers.

8.2.1 Four-Point Probes—For control of instrumentation for making four-point probe resistivity measurements in accordance with Test Method F 84, it is recommended that a minimum of three resistivity reference wafers, with resistivity values in the upper, middle, and lower portions of the resistivity range of interest be used. It is preferable to use more closely spaced resistivity reference wafers to ensure instrument linearity over the entire measurement range. If only a narrow resistivity range (less than $\pm 25\%$ of the nominal value) needs to be measured, it is sufficient to use two resistivity reference wafers at the extremes of this range.

8.2.2 Eddy Current Gages—For calibration of eddy current resistivity measuring instruments, Test Method F 673 specifies that for calibration by Method I, five reference wafers that span the full resistivity range of the instrument are required. However, many organizations prefer to use many more reference wafers for the Method I calibration. Two resistivity reference wafers at the extremes of a narrow resistivity range (typically less than about $\pm 25\%$ of the nominal value) are required for Method II calibration.

8.2.3 Spreading Resistance Probes—For calibration of spreading resistance equipment, Test Method F 672 recommends use of at least three resistivity reference wafers per decade over the range of resistivity to be measured. Regular spacing is desirable, but it may not be possible to secure samples with adequate uniformity with arbitrary nominal resistivity values. Selection of samples for microscale uniformity should take precedence over regular spacing.

8.2.4 Mercury Probes—The resistivity of a resistivity reference wafer intended for use in calibrating mercury probe

systems for making net carrier density measurements in accordance with Test Method F 1392 or Test Method F 1393 is specified to be between one-half and two times the resistivity of the specimens to be measured. However, there is some benefit to employ several resistivity reference wafers with a range of resistivity from very high (net carrier density less than about $1 \times 10^{14} \text{ cm}^{-3}$) to the lowest value to be measured.

8.3 Wafer Diameter—Resistivity reference wafers do not need to be the same diameter as the wafers to be measured by the system; any diameter that fits the instrumentation and its associated wafer handling is, in principle, acceptable. However, advances in crystal growing design and control together with certain aspect ratio consideration make it likely that superior radial resistivity uniformity in the central region of the wafer can be obtained with larger diameter ($\geq 100 \text{ mm}$) wafers as compared to that of smaller diameter wafers. In addition, the diameter correction factor for wafers 100 mm in diameter and larger measured with a four-point probe with probe-tip spacing of 1.59 mm is within 0.2 % of its limiting value of $\pi/\ln 2$ ($= 4.5324$). Consequently, errors due to small variations in diameter or probe placement at the wafer center are negligibly small (see Appendix X2), and the second order errors associated with the combination of the thickness and diameter correction factors can also be neglected. The latter point is particularly significant when four-point probes with small probe-tip spacings are employed on standard thickness wafers because the thickness correction factor for such geometries varies rapidly with thickness (see 8.6).

8.3.1 Because of the relative insensitivity of the calculated center-point resistivity to the exact value of diameter for large diameter wafers, it is usually adequate to assume the nominal diameter of standard commercially available wafers specified in accordance with SEMI Specifications M 1. However, diameter correction factors are usually required for off-center resistivity measurements made in accordance with Test Method F 81. When required, diameter measurements may be made in accordance with Test Method F 613.

8.4 Axial Resistivity Uniformity—The uniformity of the resistivity along the crystal axis is important for all applications in which the sampling volume of the measuring instrument in the direction perpendicular to the wafer surface is different from that of the certifying or calibrating instrument. In addition, gross axial uniformity along a crystal section may be ascertained in order to qualify the section as a source of resistivity reference wafers of a particular resistivity prior to slicing the section.

8.4.1 Very crude indications of the range of center-point resistivity within a crystal section may be obtained from measurements made on the ends of the section in accordance with the Four-Probe Method of Test Methods F 43. While these measurements provide a useful preliminary screen for the crystal section, they cannot be relied upon to give accurate values of resistivity.

8.4.2 Somewhat more accurate but still gross indications of axial resistivity uniformity may be obtained from measurements of the resistivity at the center of wafers in accordance with Test Method F 84 as a function of position along the crystal section. However, because of local fluctuations in

dopant incorporation as the crystal was grown, this procedure does not yield reliable information about the center-point resistivity of any wafer that is not measured. In addition, it does not yield information about the local variation in resistivity as a function of depth from the wafer surface, even if center-point resistivity measurements are made on each wafer in the section.

8.4.3 For use in connection with measurements in which the sampling volume differs from the sampling volume of an in-line four-point probe with 0.159 ($1/2\pi$) cm spacing as specified in Test Method F 84, transfer accuracy is affected by local resistivity uniformity. In particular, the resistivity in the near-surface region sampled by spreading resistance or mercury probes may differ significantly from the average resistivity of the resistivity reference wafer. Depth profiles of resistivity near the wafer surface may be obtained from spreading resistance measurements on beveled sections in accordance with Test Method F 672. To obtain depth profiles across the entire wafer, spreading resistance measurements can be made on a cleaved or polished perpendicular section; special fixtures are required for these measurements, which do not provide the depth resolution of an angle beveled section. Since relative measurements only are required, accurate calibration of the spreading resistance probe is not necessary; however, linearity is important. Two difficulties with this approach must be recognized. First, if beveled specimens are employed, the possibility of mixing the vertical and horizontal resistivity variations must be considered. Second, because the method is destructive, the resistivity variation of the actual material being used for the resistivity reference wafer cannot be determined directly at the center of a resistivity reference wafer; it can only be inferred from measurements on nearby sections or adjacent wafers.

8.5 Radial Resistivity Uniformity—The uniformity of the resistivity along the surface of the resistivity reference wafer is important for all applications in which the measuring instrument samples a different area than the certifying or calibrating instrument. Since all resistivity measurements other than those conducted in accordance with Test Method F 84, including four-point probe measurements made with probes with different probe-tip spacing, sample different areas, this factor is one of the most significant limitations in transfer of resistivity values from one instrument to another.

8.5.1 Large scale (macro) variations in resistivity across the surface of a wafer can be measured with a four-point probe.

8.5.1.1 Coarse indications can be obtained using an in-line four-point probe in accordance with Test Method F 81 modified to yield a nine-site map of resistivity variation over the central 38-mm diameter region of the wafer. In addition to four-point probes, eddy current instruments are frequently used for these measurements. Although the procedures for using eddy current instruments for measuring resistivity variation are not standardized, they provide an adequate measure of radial resistivity variation, especially in the central region of the wafer where edge effects are not significant. Although more dense patterns may be measured, they are very inconvenient to obtain manually using either a four-point probe or an eddy current instrument.