Standard Test Methods for Measuring Resistivity of Semiconductor Slices or Sheet Resistance of Semiconductor Films with a Noncontact Eddy-Current Gage¹

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 ϵ^1 Nore—Keywords were added editorially in January 1996.

INTRODUCTION

This test method is intended to outline the principles of eddy-current measurements as they relate to semiconductor substrates and certain thin films fabricated on such substrates as well as requirements for setting up and calibrating such instruments for use particularly at a buyer-seller interface. Because such eddy-current measurements for semiconductor materials are made almost exclusively with commercial instrumentation from one of several suppliers, some details included here such as specific range limits and manner of entering slice/wafer thickness values to obtain resistivity values may not apply strictly to all instruments. In all such cases, the owner's manual for the particular instrument shall be considered to contain the correct information for that instrument. It is to be noted that an eddy-current instrument directly measures conductance of a specimen. Values of sheet resistance and resistivity are calculated from the measured conductance, with the resistivity values also requiring a measurement of specimen thickness.

1. Scope

1.1 These test methods cover the nondestructive measurement of bulk resistivity of silicon and certain gallium-arsenide slices and of the sheet resistance of thin films of silicon or gallium-arsenide fabricated on a limited range of substrates at the slice center point using a noncontact eddy-current gage.

1.1.1 The measurements are made at room temperature between 18 and 28°C.

1.2 These test methods are presently limited to singlecrystal and polycrystalline silicon and extrinsically conducting gallium-arsenide bulk specimens or to thin films of silicon or gallium-arsenide fabricated on relatively high resistivity substrates but in principle can be extended to cover other semiconductor materials.

1.2.1 The bulk silicon or gallium-arsenide specimens may be single crystal or poly crystal and of either conductivity type (*p* or *n*) in the form of slices (round or other shape) that are free of diffusions or other conducting layers that are fabricated thereon, that are free of cracks, voids or other structural discontinuities, and that have (*1*) an edge-to-edge dimension, measured through the slice centerpoint, not less than 25 mm (1.00 in.); (*2*) thickness in the range 0.1 to 1.0 mm (0.004 to

0.030 in.), inclusive, and (*3*) resistivity in the range 0.001 to 200 Ω ·cm, inclusive. Not all combinations of thickness and resistivity may be measurable. The instrument will fundamentally be limited to a fixed sheet resistance range such as given in 1.2.2; see also 9.3.

1.2.2 The thin films of silicon or gallium-arsenide may be fabricated by diffusion, epitaxial or ion implant processes. The sheet resistance of the layer should be in the nominal range from 2 to 3000 Ω per square. The substrate on which the thin film is fabricated should have a minimum edge to edge dimension of 25 mm, measured through the centerpoint and an effective sheet resistance at least $1000 \times$ that of the thin film. The effective sheet resistance of a bulk substrate is its bulk resistivity (in Ω ·cm) divided by its thickness in cm.

1.2.3 Measurements are not affected by specimen surface finish.

1.3 These test methods require the use of resistivity standards to calibrate the apparatus (see 7.1), and a set of reference specimens for qualifying the apparatus (see 7.1.2).

1.4 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.5 *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.*

¹ This test method is under the jurisdiction of ASTM Committee F-1 on Electronics and is the direct responsibility of Subcommittee F01.06 on Electrical and Optical Measurement.

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2. Referenced Documents

2.1 *ASTM Standards:*

- E 1 Specification for ASTM Thermometers²
- F 81 Test Method for Measuring Radial Resistivity Variation on Silicon Slices 3
- F 84 Test Method for Measuring Resistivity of Silicon Slices with an In-Line Four-Point Probe³
- F 374 Test Method for Sheet Resistance of Silicon Epitaxial, Diffused, and Ion-Implanted Layers Using an In-Line Four-Point Probe3
- F 533 Test Method for Thickness and Thickness Variation of Silicon Slices³

3. Summary of Test Method

3.1 There are two methods that may be used. They differ in calibration technique, sample measurement value range, data correction techniques, and suitability of instrumentation and are as follows:

3.2 Method I ascertains the conformance of the apparatus to linearity and slope limits (± 1 digit) over a broad range (2) decades) of calibration standard values. It qualifies apparatus for use over a wide range of sample values.

3.2.1 The apparatus is first calibrated using standards of known resistivity or sheet resistance. Then the apparatus is subjected to a test for linearity that involves measuring a set of five reference specimens. As a part of the linearity test, a plot is made of the indicated values as a function of the known values; two limiting curves are also plotted on the same graph. (If all the plotted points fall within the limit curves, the apparatus is regarded as satisfactory.)

3.2.2 For subsequent measurements of bulk samples, the thickness of each sample (wafer) is measured and entered directly, or by the operator, according to the design of the instrument. The conductance of the specimen is then measured by the apparatus and converted to a resistivity value that is displayed. These measurements are subsequently corrected to 23°C-equivalents.

3.2.3 For subsequent measurement of thin film specimens, the sheet conductance is measured, converted to sheet resistance, and displayed. See 9.1.3 for relations between these quantitites.

3.3 Method II assumes instrument linearity between calibration standards whose values are narrowly separated (typically \pm 25 % of the anticipated sample range median point).

3.3.1 The apparatus is first calibrated using standards of known resistivity or sheet resistance. The apparatus is then subjected to a test that quantifies apparatus slope at two points,

and provides a means of correcting subsequent sample measurements, for values between these two points, to a calibration line established between the two standards employed. These latter standards may be of either known bulk resistivity or sheet resistance; their material must be the same electrical type as the samples to be measured (see Note 8).

3.3.2 For subsequent measurements of bulk samples, the thickness of each sample (wafer) is measured and entered directly, or by the operator, according to the design of the instrument. These measurements are subsequently referred to the standard reference plot. The corrected values are known to a greater precision than those obtained following Method I, and in most instances are also 23°C-equivalents.

3.3.3 For subsequent measurement of thin film specimens, the sheet conductance is measured, converted to sheet resistance and displayed. See 9.1.3 for relations between these quantities. These measurements are subsequently referred to the standard reference plot.

4. Significance and Use

4.1 Resistivity is a primary quantity for characterization and specification of material used for semiconductor electronic devices. Sheet resistance is a primary quantity for characterization, specification, and monitoring of thin film fabrication processes.

4.2 This test method requires no specimen preparation.

4.3 Method II is particularly well suited to computer-based systems where all measurements can be quickly and automatically corrected for value offset and for temperature coefficient of resistivity.

4.4 Method I has been evaluated by interlab comparison (see Section 11). Until Method II has been evaluated by interlaboratory comparison, it is not recommended that the test method be used in connection with decisions between buyers and sellers.

5. Interferences

5.1 Radial resistivity variations or other resistivity nonuniformity under the transducer are averaged by this test method in a manner which may be different from that of other types of resistivity or sheet resistance techniques which are responsive to a finite lateral area. The results may therefore differ from those of four-probe measurements depending on dopant density fluctuation and the four-probe spacing used.

NOTE 1-Test Method F 81 provides a means for measuring radial resistivity variation of silicon slices.

5.2 Uncertainty of thickness values for the reference (bulk) wafers can introduce, in Method I, an uncertainty in the linearity plot; in Method II it can introduce a corresponding uncertainty in the reference line connecting the two reference wafer values, if calibration is performed in resistivity values.

5.3 Uncertainty of thickness value of sample (bulk) wafers can introduce an error in both measured and reported values for both Methods I and II. These uncertainties can be eliminated by executing the procedure using sheet resistance values for reference and sample wafers.

5.4 Spurious currents can be introduced in the test equipment when it is located near high-frequency generators. If

² *Annual Book of ASTM Standards*, Vol 14.03.

³ *Annual Book of ASTM Standards*, Vol 10.05.

FIG. 1 Schematic of Eddy-Current Sensor Assembly

equipment is located near such sources, adequate shielding must be provided. Power line filtering may also be required.

5.5 Semiconductors have a significant temperature coefficient of resistivity. Temperature-correction factors for extrinsic silicon specimens are given in Test Method F 84. Temperature differences between any of the reference or sample wafers, during calibration or measurement, or both, will introduce a measurement error in Method II.

5.6 High levels of humidity may affect the indicated value.

6. Apparatus

6.1 *Electrical Measuring Apparatus*, with instructions for use and consisting of the following assemblies:

6.1.1 *Eddy-Current Sensor Assembly*, having a configuration of a fixed gap, between two opposed transducers, into which a specimen slice is inserted. The assembly shall include support(s) on which the slice rests, a device for centering the slice, and a high-frequency oscillator to excite the sensing elements. The frequency of the oscillator shall be chosen to provide a skin depth at least five times the thickness of the slice or thin film to be measured. The skin depth is a function of the resistivity of the specimen. The assembly shall provide an output signal proportional to sheet conductance. This assembly and associated apparatus are shown schematically in Fig. 1.

NOTE 2—A typical conductance apparatus is described in detail in a paper by Miller, Robinson, and Wiley.⁴ This paper also discusses skin-depth as a function of thickness and resistivity.

6.1.2 *Signal Processor*— Means for electronically converting, by analog or digital circuitry, the sheet conductance signal to a sheet resistance value, and in the case of bulk substrate measurements, means for conversion to resistivity values using the measured thickness of the substrate. The processor shall incorporate a means for displaying sheet resistance or resistivity, a means of zeroing the conductance signal in the absence of a specimen and a means for calibrating the instrument with known calibration specimens.

NOTE 3-For Method I, the linearity of the apparatus is checked in an operational qualification test (see 9.1.3).

NOTE 4—A typical apparatus operates as follows. When a specimen is inserted into the fixed gap between the two colinear sensing elements, or transducers, in a special oscillator circuit, eddy currents are induced in the specimen by the alternating field between the transducers. The current needed to maintain a constant voltage in the oscillator is determined internally; this current is a function of the specimen conductance. The specimen conductance is obtained by monitoring this current. Sheet resistance or resistivity values are obtained from the specimen conductance by analog or digital electronic means; calculation of resistivity values also requires knowledge of specimen thickness.

6.2 *Thermometer*— ASTM Precision Thermometer having a range from -8 to $+32^{\circ}$ C and conforming to the requirements for Thermometer 63C as specified in Specification E 1.

6.3 *Thickness Gage*, as specified in 7.1 of Test Method F 533 (included for completeness).

6.4 Calibration and linearity checking must be done in consistent units, whether resistivity, sheet resistance or sheet conductance, according to the requirements of the given instrument. If resistivity values are used, knowledge of the specimen thickness is also required. For bulk calibration or linearity-check specimens, the thickness is the as-measured thickness in centimetres. For thin film specimens, the total thickness of the thin film plus substrate should be measured and used; if this cannot be done, an effective thickness of 0.0508 cm (0.020 in.) should be used. (See 9.1.3.)

7. Reagents and Materials

7.1 Resistivity standards or other reference specimens to check the accuracy and linearity of the instrument. Preferably, these are bulk silicon slices but may also be fabricated by ion implantation into silicon.

7.1.1 Bulk silicon standards or other reference specimens are to be measured for resistivity in accordance with Test Method F 84. The thickness of these specimens shall be within ± 25 % of the specimens to be measured unless otherwise agreed to by the parties to the test.

7.1.2 Ion implant specimens are to be measured for sheet resistance by four point probe in accordance with Test Method F 374.

7.1.3 The standards and other reference specimens for Method I shall be at least five in number and should have a range of values that span the full range of the instrument. For Method II, where the specimens to be measured have a narrow range of resistivity or sheet resistance values, the standards and other reference specimens shall be two in number. Their values shall span a range at least as large as the specimens to be measured. Table 1 gives a list of values recommended for

⁴ Miller, G. L., Robinson, D. A. H., and Wiley, J. D., "Contactless Measurement of Semiconductor Conductivity by Radio Frequency-Free-Carrier Power Absorption," *Review of Scientific Instruments*, Vol 47, No. 7, July 1976.

checking the full range of a typical instrument for Method I application; these values should be met within ± 25 %.

NOTE 5—Resistivity standards are available from the National Bureau of Standards in the form of bulk silicon slices with nominal thicknesses of 0.025 in. at the following resistivity levels: 0.01, 0.1, 1, 10, 25, 75 and 180 Ω ·cm.

NOTE 6—Ion implanted sheet resistance reference specimens may be used subject to the requirement that the sheet resistance of the substrate is at least $1000 \times$ the sheet resistance of the implanted layer.

NOTE 7—It is possible to load the oscillator of the instrument by using a bulk specimen which has too much conductance. This may be caused by a combination of low resistivity and large thickness. Such loading of the oscillator will make it generally impossible to simultaneously meet the linearity requirements of Section 9 for this specimen and the less conductive reference specimens being used.

8. Sampling

8.1 If the test method is not used as a 100 % inspection test, sampling procedures shall be agreed upon by the parties to the test.

8.2 If sampling by lot is required, the determination of what constitutes a lot and the procedures for sampling by lot shall be agreed upon by the parties to the test.

9. Method I (Full Range)

9.1 *Calibration:*

9.1.1 With the thermometer, measure the room ambient temperature, T , to the nearest 0.1° C and record this value.

9.1.2 If bulk silicon resistivity standards or reference specimens are used, or both, calculate in accordance with the following equation, the resistivity, ρ_T , at the ambient temperature. (Proceed to 9.3 if implanted reference specimens are used.)

$$
\rho_T = \rho_{23} (1 + C_T (T - 23))
$$

where:

 ρ_{23} = is the resistivity at 23°C, and
 C_T = silicon temperature coefficien = silicon temperature coefficient of resistivity (Test Method F 84, Table 5).

9.1.3 Convert the values of the standards or reference specimens, or both, to units of sheet resistance, resistivity, conductivity; or conductance as necessary according to the calibration section of the instrument's instruction manual, using the following relation:

$$
R = \rho/t = 1/\sigma t = 1/G
$$

where:

- $R =$ sheet resistance.
- ρ = resistivity
- σ = conductivity
- $G =$ conductance, and $f =$ thickness of spec
- t = thickness of specimen in centimetres (taken to be 0.0508 cm for the thin films).
- 9.1.4 Test the apparatus linearity as follows:

9.1.4.1 Position the specimen between the transducers so that the center of the specimen is within 1 mm (0.04 in.) of the axis of the transducer assembly.

9.1.4.2 With the apparatus, measure the resistivity of each of the five reference specimens in accordance with the instructions provided by the apparatus manufacturer. Record these values.

9.1.4.3 Plot each specimen's measured values, in whatever units are chosen, against the values, in the same units, obtained from four probe measurements where the four probe values have been corrected to ambient temperature in the case of bulk specimens. (See Fig. 2 for examples of plots in units of resistivity and of conductance.)

9.1.4.4 Construct two curves corresponding to the following relations:

indicated value = known value + 5% of the known value + 1 digit indicated value = known value – 5% of the known value – 1 digit

9.1.4.5 Examine the plot. If all five points fall between the two curves, proceed to 9.2. If all five points do not fall between the two curves, refer to the manufacturer's instructions and ensure that all adjustments to the electrical measuring apparatus have been properly made, and repeat 9.1.4.1 through 9.1.4.5. If only three or four adjacent points fall within the two curves, the instrument may be used over the inclusive range bounded by the upper and lower values of these adjacent points. If fewer than three adjacent points fall within the two curves, discontinue the test, as the apparatus does not satisfy the linearity requirement.

9.2 *Procedure:*

9.2.1 With the thermometer, measure the room ambient temperature to the nearest 0.1°C and record this value.

9.2.2 If bulk specimens are being measured, first measure and record specimen centerpoint thickness in accordance with Test Method F 533. Omit this step if thin film specimens are being measured.

9.2.3 Enter the thickness value into the apparatus in accordance with the instructions provided by the apparatus manufacturer if measuring bulk specimens; if measuring thin film sheet resistance, follow the instructions provided by the instrument manufacturer for entering a normalizing thickness or using a mode switch to select an output in units of sheet resistance.

9.2.4 With the apparatus, measure the resistivity or sheet resistance of each specimen slice in accordance with the

instructions provided by the apparatus manufacturer. Record these values.

9.2.4.1 Position each slice between the transducers so that the center of the slice is within 1 mm (0.04 in.) of the axis of the transducer assembly.

9.2.4.2 If bulk specimens are being measured, correct the measured resistivity values of each specimen to 23°C equivalent in accordance with Test Method F 84. Record these values. If the specimen is a thin film or is bulk gallium arsenide, omit this step.

9.3 *Report:*

9.3.1 Report the following information:

9.3.1.1 Date of test,

9.3.1.2 Identification of operator,

9.3.1.3 Identification of resistivity standards and reference specimens (7.1) ,

9.3.1.4 Identification of specimen,

9.3.1.5 Ambient temperature during calibration, \degree C, and, for each slice,

(a) Ambient temperature during measurement, °C,

(b) Thickness, cm (or in.),

(c) Measured resistivity, Ω ·cm (room temperature), and

(d) Corrected resistivity, Ω ·cm (23°C equivalent).

10. Method II (Partial Range)

10.1 *Calibration:*

10.1.1 With the thermometer, measure the room ambient temperature, T , to the nearest 0.1° C and record this value.

10.1.2 Select two reference specimens whose values span the range of samples to be measured.

10.1.3 Position the specimen between the transducers so that the center of the specimen is within 1 mm (0.04 in.) of the axis of the transducer assembly.

10.1.4 With the apparatus, measure the resistivity of the two specimens in accordance with the instructions provided by the manufacturer. Record these values.

10.1.5 Plot the specimen's ambient-measured values on a graph as a function of their 23°C-certified values (see Fig. 3, Fig. 4).

10.1.6 Construct a line connecting the two data points.

10.2 *Procedure:*

10.2.1 If bulk specimens are being measured, locate the samples' measured values on the graph's ordinate, and extend a horizontal line from the measured value to its intersection

with the calibration line constructed in 10.1.6. Extend a vertical line from above the calibration intersect point to the actual abscissa (see Fig. 4). Record the abscissa intersect point as the sample's actual value.

10.2.1.1 For calibration specimens and samples with resistivity above 0.1 Ω ·cm, the temperature coefficient of resistivity of silicon is monotonic and changes gradually with increasing resistivity (see Test Method F 84, Table 5). Procedure II produces for these samples actual values automatically corrected to 23°C.

10.2.1.2 For calibration specimens spanning a resistivity range that contains a non-monotonic temperature coefficient of resistivity, the measured values should be corrected to their 23°C-equivalents using Test Method F 84, Table 5 factors, prior to locating the measured values on the graph ordinate. The actual correction values in this range are extremely small.

NOTE 8—The maximum error derived from calibrating on one conductivity type and measuring the other ≤ 0.12 % / °C in the resistivity range 0.01Ω · cm to 1000 Ω · cm.

10.3 *Report:*

10.3.1 Report the following information:

10.3.1.1 Date of test,

10.3.1.2 Identification of operator,

10.3.1.3 Identification of resistivity standards and reference specimens (7.1) ,

10.3.1.4 Identification of specimen,

10.3.1.5 Ambient temperature during calibration, °C, and, for each slice,

(a) Ambient temperature during measurement, °C,

(b) Thickness, cm (or in.),

(c) Measured resistivity, Ω ·cm (room temperature), and

(d) Corrected resistivity, Ω ·cm (23°C equivalent).

11. Precision and Bias

11.1 Method I precision has been determined by roundrobin test (see Annex A1).

11.1.1 The first interlaboratory test used to estimate multilaboratory precision, or reproducibility, of this test method is detailed in Annex A1. It used three large-grain polycrystalline silicon specimens of very similar resistivity as the test specimens and ten single-crystal silicon-reference specimens to check instrument linearity. The two lowest-**FIG. 3 Reference Line Construct** resistivity value reference specimens are strictly applicable

only to low-range eddy current instruments and are not necessary to qualify instruments for use on the polycrystal test specimens. While none of the five participating laboratories successfully passed the linearity test, within ± 5 % limits, for all reference specimens, an analysis was made based on assumptions detailed in A1.4 and A1.5. Based on these assumptions, an estimate of multilaboratory reproducibility of the average of four measurements of the polycrystalline specimens is ± 12 % (3S %).

11.1.2 A second multilaboratory test was conducted to estimate multilaboratory reproducibility of this test method for single crystal silicon specimens over a wide range of resistivity values. This test is detailed in Annex A2. Owing to the large number of test specimens being evaluated, only three reference specimens were used for linearity check of low range instruments, and four reference specimens were used to check high range instruments. Collectively, these reference specimens covered the range 0.003 to 30Ω ·cm. For laboratories demonstrating instrument linearity of ± 5 % or better, the estimate of multilaboratory reproducibility over this resistivity range is ± 9 % (3S %). For the extrapolated test specimen range to 90 Ω ·cm, the estimate of reproducibility is ± 15 % (3S %) and from 90 to 125 Ω ·cm, it is ± 18 % (3S %).

11.1.3 *Bias*—Estimates of bias for this test method can only be established with respect to a resistivity scale determined by another technique with comparable spatial averaging over material nonuniformities, such as the four-probe technique, Test Method F 84. However, eddy current instruments are calibrated and checked for linearity of response using specimen resistivity values determined by four-probe measurements. Therefore, within the ability to establish response to \pm 5 % of reference value over a large range of resistivity values, as was demonstrated by a number of the participating laboratories, no additional bias is expected.

11.2 It is planned to determine the precision of Method II by round-robin test.

12. Keywords

12.1 contactless measurements; eddy current; nondestructive evaluation; resistivity; semiconductor; sheet resistance; silicon; thin films; wafer

ANNEXES

(Mandatory Information)

A1. RESULTS OF FIRST MULTILABORATORY TEST FOR METHOD I

A1.1 A preliminary estimate of precision was made by five laboratories taking measurements on nine single crystal and three large-grain polycrystal specimens. The single crystal specimens were measured for thickness and for resistivity at the center using a four point probe (Test Method F 84) by the laboratory which donated the specimens and also by the laboratory which coordinated the round robin. The polycrystal specimens were measured for thickness only. A summary of this data is given in Table A1.1. Thickness and resistivity values as measured by the originating laboratory were provided to all participants as were temperature coefficient of resistivity values for the single crystal specimens.

A1.2 The participating laboratories were requested to calibrate and test the linearity of the contactless resistivity instruments using the single crystal specimens and to measure the polycrystal specimens as unknown. All specimens were measured on each of 3 days. Each day, the single crystal specimens were measured four times at the center with the polished surface face up, then four times with the polished surface face down; the specimens were rotated 90° about a vertical axis between each of the four measurements. The polycrystal specimens were measured each day with one face up only; four measurements were taken on each specimen with 90° specimen rotations between the measurements. A summary of the data is given in Table A1.2.

A1.3 Differences between the measurement averages for the two face-up conditions of the single crystal specimens are generally less than 1 % and for almost all combinations of specimens and laboratories, they are smaller than the standard

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TABLE A1.2 Summary of Measurements on Linearity Test Specimens and on Unknown Test Specimens

NOTE 1—Data entries are averages of four "face-up" measurements and four "face-down" measurements on each of three days.

^A Value is outside \pm 5 % limits based on Supervisory Lab 1 data.

 B Value is outside \pm 5 % limits based on Supervisory Lab 2 data.

deviation of a set of four measurements for that specimenlaboratory combination.

A1.4 Tests of instrument linearity appear to show two kinds of results, one related to specimens and one related to instruments. The largest number of measurements which are outside of 5 % linearity limits based on four probe measurements occur for the two lowest and the very highest resistivity specimens, with a very large number of such measurements also occurring for the 20 Ω ·cm specimen. Since the specimen just below and just above 20 Ω ·cm have very few measurements reported outside of such \pm 5 % limits, the large number of poor measurements on the 20 Ω ·cm specimen are likely due to some property of the specimen, such as resistivity non-uniformity near the center, rather than malfunction of the instruments used. Laboratory 5 (see Table A1.2) shows a very high number of measurements outside the 5 % linearity limits, suggesting particular instrument problems.

A1.5 The polycrystal specimens have measured resistivity values in a region of little instrument nonlinearity as tested with the single crystal specimens. Because of the wide range of temperatures at which data was taken by the different laboratories, a temperature coefficient of resistivity with a value of 0.8 % per °C was assumed for these specimens and was applied to calculate the values shown in Table A1.2.

A1.6 The estimate of the multilaboratory precision which was made for the polycrystalline specimens was based on the assumption that each day's measurements by each laboratory was an independent set of data. However, since Laboratory number 3 differed from the other four laboratories regarding the relative values of the specimens, separate data tabulations were done with and without the data from Laboratory 3. Table A1.3 shows the calculated overall averages and relative standard deviations of the three specimens for all five laboratories (15 laboratory-day combinations for each specimen) and for the laboratories other than Laboratory 3 (12 laboratory-day combinations). If data from Laboratory 3 is omitted, a reasonably constant of relative standard deviation is calculated for all three specimens. The estimate of multilaboratory precision for the average of four individual measurements is based on the four laboratory analyses and is taken to be 4 %.

TABLE A1.3 Averages (Ω·cm) and Relative Standard Deviations of Daily Averages

Specimen	O. Ω -cm	P. Ω cm	Q, Ω -cm
	$(\%)$	(%)	(%)
Laboratories 1-5	5.448 (8.47)	4.397 (5.03)	5.771 (5.01)
Omit Laboratory 3	5.240 (3.58)	4.320 (3.83)	5.673 (3.83)

A2. RESULTS OF SECOND MULTILABORATORY TEST FOR METHOD I

A2.1 This test, conducted in 1986 and 1987, used most of single-side polished bulk silicon slices that were used as reference specimens in the first multilaboratory tests; these can be identified by comparing slice identification numbers for the two experiments. To these were added five double-side lapped slices from the NIST Standard Reference Material series, identifiable by the use of letters *U* through *Y* for slice identification in Table A2.1. Three of the single-side polished slices were replaced due to breakage following Laboratory 7; the replacements have four-digit identification numbers.

A2.1.1 The available slices were divided into two categories: three reference (linearity-check) specimens plus five test specimens to test the resistivity range 0.002 to 1 Ω ·cm, and four reference specimens plus nine test specimens to test the resistivity range 0.1 to 125 Ω ·cm.

A2.2 A summary of the data taken by the supervisory laboratory for the slices used in the low resistivity range test is given in Table A2.1. This table shows the slice identification, conductivity-type, thickness, in micrometers, measured by an electromechanical contacting gage, temperature coefficient of resistivity, and resistivity at 23°C determined by four probe, as well as the \pm 5 % and − 5 % limits on these resistivity values allowed by the linearity requirements of the test method. Table A2.2 gives corresponding values for the slices used in the high resistivity range test. Thickness values for all slices were provided to the participating laboratories. They were, however, allowed to use in situ measurement of thickness if their instruments took such data. Temperature coefficients of resistivity values were provided only for the reference specimens. No prequalification of the reference or test specimens was performed beyond the thickness and center point resistivity measurements given in Table A2.1 and Table A2.2.

A2.3 Each laboratory was required to establish the linearity of its instrument, using the reference specimens provided, on each of 3 days, and to take four measurements at the center of each test sample on each day. The averages of those four measurements are reported in the following tables: Table A2.3—low resistivity range for Laboratories 1 through 7, Table A2.4—low resistivity range for Laboratories 8 through 11, Table A2.5—high resistivity range for Laboratories 1 through 7, and Table A2.6—high resistivity range for Laboratories 8 through 11. All these values have been converted to 23°C values for comparison with four-probe values.

A2.4 Observations on reported data not contained in previous tables.

A2.4.1 Specimen thickness values provided by the coordinating laboratory were used by Laboratories 1, 10, and 11; all other laboratories reported thickness values on the data sheets that indicated they had taken their own measurements. The worst case percent offset in these thickness values compared to the reference values, was noted for each of these laboratories; these percent differences ranged from 0.3 % for Laboratories 7 and 8 to 1.5 % for Laboratories 3 and 5; Laboratory 9 has a worst case offset of 0.7 % for low resistivity range specimens but had a 4 % offset for several high range specimens. There was no meaningful difference between single-side polished and double side lapped specimens in terms of sign or magnitude of the thickness offsets.

A2.4.2 Temperature correction of resistivity value calculations were checked for low and high range reference specimens from all laboratories based on their stated ambient temperatures during measurement and their stated ambient temperature resistivity values for these specimens. All laboratories except 3, 7, and 10 clearly performed this step to within round off error amounting to less than 0.1 %. Laboratory 3 listed the ambient temperature for all measurements, but provided no record of ambient temperature resistivity values used for the reference specimens. Laboratory 7 made temperature corrections in the wrong direction, but operated close enough to 23°C that the maximum error from this source was 1.4 %. Laboratory 10 made temperature correction errors of up to 5 % but without a consistent pattern in magnitude or direction; the worst of these errors was for reference specimens above 0.8 Ω ·cm.

A2.4.3 The most common problems with linearity performance, that is, staying within ± 5 % of the resistivity value obtained by four-probe measurement values was noted at the high end of the low resistivity range and at the low end of the high resistivity range.

A2.5 Two analyses of these data were performed, the first of which provides the basis for the precision statement in 11.1.1. Each is based on the average of four readings taken for each day's instrument setup but with each day's average taken as a separate entry since the setups for each day are assumed to be independent.

A2.5.1 *Laboratories* Passing the \pm 5 % Linearity *Requirement for All Reference Specimens On the Appropriate Instrument Range:*

A2.5.1.1 Table A2.7 gives the grand average, percent standard deviation and number of contributing measurements for the low range specimens; a pooled standard deviation based

TABLE A2.1 Low Resistivity Range Specimens: Thickness and Four-Probe Resistivity Information Taken by the Coordinating Laboratory

	Reference Slices			Test Slices						
Slice Identification		1928		58				43	2051	
Conductivity Type	Ν	N				D		N	Ν	
Thickness (µm)	524.6	587.0	501.2	504.2	522.2	633.4	636.0	372.6	601.0	506.9
ρ_{23}	0.0027	0.00302	0.1106	0.830	0.00835	0.0144	0.09816	0.4286	0.407	0.8186
ρ_{23} + 5 %	0.00284	0.00317	0.1161	0.872	0.00877	0.0151	0.1030	0.4500	0.427	0.8595
ρ_{23} – 5 %	0.00257	0.00286	0.1051	0.789	0.00793	0.0136	0.0933	0.4072	0.386	0.7777

TABLE A2.2 High Resistivity Range Specimens: Thickness and Four Probe Resistivity Information Taken by the Coordinating Laboratory

TABLE A2.3 Average Resistivity, Corrected to 23°C, for Each Day's Measurement of Low Range Specimens, Laboratories 1 through 7

on degrees of freedom is also reported for Specimens 43 and 2051. Table A2.8 gives the same parameters for the high resistivity range specimens with pooled values of standard deviation being reported for Specimen Pairs 43 and 2051 and for 56 and 1055. Each day's data from a laboratory passing the linearity requirement was taken as a separate contributing measurement under the assumption that each day's setup and qualification of the instrument was independent of the rest. No significant trend of multilaboratory reproducibility of average value with resistivity is seen on either instrument range. Based on this analysis, multilaboratory reproducibility is stated to be better than 9 % (3S %) for specimens up to 30 Ω ·cm, to be \langle 15 % (3S %) for specimens between 30 and 90 Ω ·cm and to be about 18 % (3S %) for specimens between 90 and 125

 Ω ·cm. The latter two values are derived from specimens well beyond the highest resistivity reference specimen available.

A2.5.2 *Laboratories* Passing the \pm 5% *Linearity Requirement for Reference Specimens on Either Side of a Test Specimen But Not Necessarily for All Reference Specimens On That Instrument Range:*

A2.5.2.1 This analysis generally gave more contributing measurements to the determination of multilaboratory reproducibility values (24 values each on Specimens 17, *T* , *U*, and *X*, and 27 measurements each on Specimens 6 and *W*). However, estimates of multilaboratory reproducibility values (1S %) were generally several tenths of a percent larger using this procedure rather than that preceding.

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TABLE A2.4 Average Resistivity, Corrected to 23°C, for Each Day's Measurement of Low Range Specimens, Laboratories 8 through 11

TABLE A2.5 Average Resistivity, Corrected to 23°C, for Each Day's Measurement of High Range Specimens, Laboratories 1 through 7

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TABLE A2.7 Analysis of Results, Low Resistivity Range Specimens, from Second Multilaboratory Test; Laboratories 2, 4, 8, 9, 10, 11

TABLE A2.8 Analysis of Results, High Resistivity Range Specimens, from Second Multilaboratory Test Laboratories 1, 2, 4, 9, 11

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