Designation: F 397 – 93 (Reapproved 1999)

DIN 50430

Standard Test Method for Resistivity of Silicon Bars Using a Two-Point Probe[1]

This standard is issued under the fixed designation F 397; 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[2] covers the measurement of the resistivity of single-crystal bars having cross sections that are uniform in area and square, rectangular or round in shape, and having resistivity between 0.0009 and 3000 Ω·cm. The resistivity of a silicon crystal is an important acceptance requirement.

1.2 This test method is intended for use on single crystals of silicon of either *n*- or *p*-type for which the uniformity of the crystal cross section is such that the area can be accurately calculated. The specimen cross-sectional area shall be constant to within ±1 % of the average area as determined by measurements along the crystal axis (see 12.2).

1.3 The ratio of the length to the maximum dimension of the cross section of the specimen shall not be less than 3:1 (see 12.1). The largest diameter tested by round robin was 3.75 cm (1.5 in.), and this is the largest diameter that can be measured by this method. The specimen shall normally have a surface finish of 0.4 µm (16 µin.) rms or less (see ANSI B46). Other surface finishes may be used if mutually acceptable; however, the multilaboratory precision figures of this test (see 16.1) then may no longer apply.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific hazard statements are given in Section 9.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[3]

E 1 Specification for ASTM Thermometers[4]

F 42 Test Methods for Conductivity Type of Extrinsic Semiconducting Materials[5]

2.2 *Other Standard:*

ANSI B46 Surface Texture[6]

2.3 *SEMI Standard:*

C1 Specifications for Reagents[7]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *resistivity,* ρ[Ω·cm]—*of a semiconductor,* the ratio of the potential gradient (electric field) parallel with the current to the current density.

4. Summary of Test Method

4.1 A direct current is passed through ohmic contacts at the ends of a bar specimen and the potential difference is determined between two probes placed along the current direction (see 7.3.1). The resistivity is calculated from the current and potential values and factors appropriate to the geometry. This test method includes procedures for checking both the probe assembly and the electrical measuring apparatus.

4.1.1 The spacing between the two probe tips is determined from measurements of indentations made on a polished single-crystal surface.

4.1.2 The accuracy of the electrical measuring equipment is tested by means of an analog circuit containing a known resistance together with other resistors which simulate the resistance at the contacts between the probe tips and the semiconductor surface.

4.2 Procedures for preparing the specimen, for measuring its size, and for determining the temperature of the specimen during measurement are also given. A table of temperature coefficient of resistivity versus resistivity is included with the method so that appropriate calculations can be made.

5. Significance and Use

5.1 This test method is recommended for material acceptance and manufacturing control of single-crystal bulk silicon. It is also applicable to other semiconductor materials but neither the appropriate conditions of measurement nor the expected precision have been experimentally determined.

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

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[2] DIN 50430 is an equivalent method. It is the responsibility of DIN Committee NMP 221, with which Committee F-1 maintains close technical liaison. DIN 50430, Testing Inorganic Semiconductor Materials: Measurement of the Specific Electrical Resistance of Bar-Shaped Monocrystals of Silicon or Germanium by the Two-Probe Direct Current Method is available from Beuth Verlag GmbH, Burggrafenstrasse 4-10, D-1000 Berlin 30, Federal Republic of Germany.

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

[4] *Annual Book of ASTM Standards*, Vol 14.03.

[5] *Annual Book of ASTM Standards*, Vol 10.05.

[6] Available from American National Standards Institute, 11 West 42nd St., 13th Floor, New York, NY 10036.

[7] Available from the Semiconductor Equipment and Materials International, 805 E. Middlefield Rd., Mountain View, CA 94043.

5.2 This test method assumes no radial resistivity variation in the crystal. If such variations are present in the crystal, this method yields an average resistivity of some unknown cross section of the crystal. In such cases, the resistivity measured on a slice cut from the crystal may not correlate with the resistivity measured by this test method for the region about that slice.

6. Interferences

6.1 Photoconductive and photovoltaic effects can seriously influence the observed resistivity, particularly with nearly intrinsic material. Therefore, all determinations should be made in a dark chamber unless experience shows that the material is insensitive to ambient illumination.

6.2 Spurious currents can be introduced in the testing circuit when the equipment is located near high-frequency generators. If the equipment is located near such sources, adequate shielding must be provided.

6.3 Minority carrier injection during the measurement can occur due to the electric field in the specimen. With material possessing high lifetime of the majority carriers and high resistivity, such injection can significantly influence the results. Carrier injection can be detected by repeating the measurements at lower current. In the absence of injection no change in resistivity should be observed. The current level recommended should reduce the probability of difficulty from this source to a minimum but in cases of doubt, the measurements of 13.3 to 13.7 should be repeated at a lower current. If the proper current is being used, doubling or halving its magnitude should not cause a change in observed resistance that is more than 0.5 %. Minority carrier effects should be most pronounced near current contacts.

6.4 Semiconductors have a significant temperature coefficient of resistivity. Consequently, the current used should be small to avoid resistive heating. If resistive heating is suspected, it can be detected by a change in readings as a function of time starting immediately after the current is applied. Resistive heating can also be detected by changing the current level as in 6.3.

6.5 Vibration of the voltage probes sometimes causes troublesome changes in contact resistance. If difficulty is encountered, the apparatus should be shock mounted.

6.6 Non-catastrophic cracks (not generally visible) or other mechanical damage can give erroneous readings. If visible cracks are seen, it may be necessary to saw the crystal into sections and measure separately.

7. Apparatus

7.1 *Specimen Preparation*:

7.1.1 *Grinding Facilities* capable of giving a finish of 0.4 µm rms, or less.

7.1.2 *Sandblasting Facility* that uses aluminum oxide, SiC, or equivalent abrasive commercially specified as 27 µm.

7.1.3 *Triple Beam, Dial, or Trip Balance.*

7.1.4 *Diamond Abrasive Saw* to cut proper specimen shape, if not previously done.

7.1.5 *Chemical Laboratory Apparatus* such as plastic beakers, graduates, and plastic-coated tweezers suitable for use both with acids (including hydrofluoric) and with solvents. Adequate facilities for handling and disposing of acids and their vapors are essential.

7.2 *Measurement of Cross-Sectional Specimen Geometry*—A micrometer or vernier caliper capable of measuring the dimensions of square or rectangular bars and circular rods with a resolution of ±0.05 mm (±0.002 in.) or ±0.2 %, whichever is smaller.

7.3 *Probe Assembly*:

7.3.1 *Probes*—Either of two shapes may be used:

7.3.1.1 Chisel-shaped voltage probes should preferably be made from standard tungsten carbide flat tools 0.318 by 0.635 cm (0.125 by 0.25 in.). The included angle of the chisel should be approximately 45° and the nominal tip radius should be approximately 0.04 mm (0.0015 in.).

7.3.1.2 Conical-pointed voltage probes shall have tungsten carbide or osmium tips with included angle of 45 to 150°. The nominal radius of a probe tip should be initially 25 to 50 µm.

7.3.2 *Probe Force*:

7.3.2.1 The force on the chisel-shaped probe shall be 8 ± 1 N when the probes are against the specimen in the measurement position.

7.3.2.2 The force on the conical-pointed probes shall be 1.75 ± 0.25 N when the probes are against the specimen in the measuring position.

7.3.3 *Insulation*—The electrical isolation between a probe and any other probe or part of the probe assembly shall be at least 10^8 Ω.

7.3.4 *Probe Alignment and Separation*—The spacing for the chisel-shaped two-probe shall be nominally 1 cm. The conical probe spacing shall have a nominal value of 4.7 mm (0.18 in.). Probe spacing shall be determined in accordance with the procedure of 11.1 in order to establish the suitability of the probe assembly as defined in 11.1.5. The following apparatus is required for this determination:

7.3.4.1 A chemically polished surface on material characteristic of the specimen to be measured.

7.3.4.2 A toolmaker's microscope capable of measuring increments of 2.5 µm.

NOTE 1—The nominal spacing for the conical probe configuration is arrived at by using the outer-most pins of the "62.5 mil" four-point probe. Four-point probes of other nominal spacing may be used for the purposes of 7.3.4 by mutual agreement; however, the multilaboratory precision figures of 16.1 were arrived at using only the probes specified in 7.3.4.

7.4 *Probe Supports*— Jigs must be provided for holding the specimen and bringing the probes into contact with the specimen perpendicular to the specimen surface without lateral movement. Probe positioning must be repeatable within 0.5 % of the average probe spacing.

7.5 *End Contacts*— Contact to each specimen end should be made by metallic fiber mesh ("conductive wool") or soft plates large enough to cover the ends of the specimen and made of annealed copper or a metal with an equivalent conductivity. Prior to measurement, the ends of the specimen shall be made ohmic by electroplating or painting with conducting silver paint.

7.6 *Thermometer*— ASTM precision thermometer having a range from − 8 to 32°C and conforming to the requirements for Thermometer 63C as prescribed in Specification E 1.

7.7 *Electrical Measuring Apparatus*:

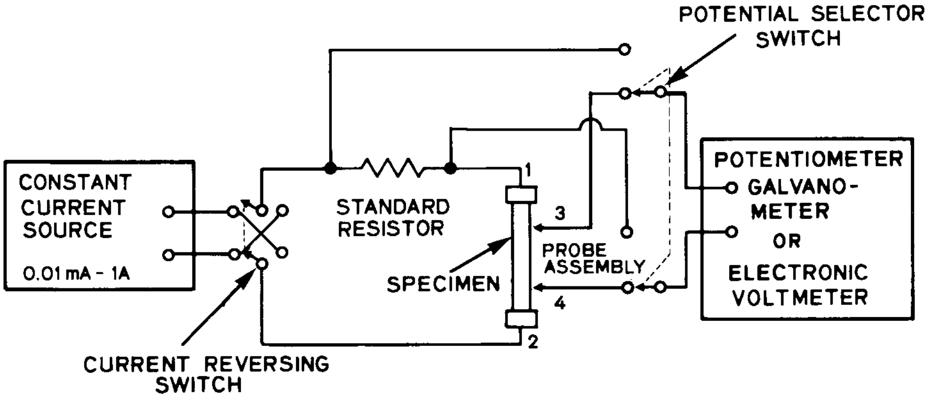

FIG. 1 Recommended Electrical Circuit

7.7.1 Any circuit that meets the requirements of 11.2 may be used to make the electrical measurements. The recommended circuit, connected as shown in Fig. 1, consists of the following:

7.7.1.1 *Constant-Current Source* capable of producing current densities shown in Table 1, constant to 0.1 %. In order to cover the full range of resistivities listed and crystal cross-section dimensions from 1.25 to 3.75 cm, full scale current values from 10^{-5} to 1 A are necessary (Note 2).

7.7.1.2 *Current Reversing Switch.*

7.7.1.3 *Double-Throw, Double-Pole Potential Selector Switch.*

7.7.1.4 *Potentiometer-Galvanometer or Electronic Voltmeter*—The instrument may be used to read the potential drop in volts or it may be calibrated in conjunction with the current ratio directly. The instrument must be capable of measuring potential differences of 10^{-4} to 1 V with two significant-figure resolution on the 10^{-4} scale and three significant digit resolution on all other scales. The input impedance should generally be greater than 10^{8} Ω (Note 3).

NOTE 2—Larger-diameter crystals may in principal be measured by this technique. However, due to the possibility of crystal heating or current injection at the end contacts (see 6.3 and 6.4) current levels cannot be scaled based on Table 1 for all resistivity ranges. Consequently a higher-resolution voltmeter than required in 7.7.1.4, or reduced accuracy and precision, or both, may be necessary for some resistivity and diameter combinations.

TABLE 1 Resistivity Range Appropriate to Analog Test Circuit Resistance and Recommended Standard Resistance Values

NOTE 1—These current densities are those which were tested by multilaboratory round robin for specimens whose diameters were 3.75 cm (1.5 in.) or less. These currents will not give the suggested 10-mV voltage drop (see 13.3) for the lower resistivity values. Some reduction from desirable signal level was necessitated by possible heating and injection effects (see 6.3 and 6.4) and current handling capabilities of available power supplies. For crystals larger than 3.75 cm in diameter further compromise may be necessary (see Note 2).

Resistivity, Ω·cm	Current Density (mA/cm ^{2})	
	1-cm Point Spacing	0.476-cm Point Spacing
0.001	100	200
0.01	20	40
0.1	10	20
1.0	2	4
10	1	2
100	0.2	0.4
1000	0.02	0.04

NOTE 3—It is generally found that for source impedances well in excess of 1000 Ω, manufacturers' specifications of digital voltmeter input impedance are not fully adequate. Hence an operational test for accuracy and precision is necessary (see 7.9 and 11.2).

7.7.2 *Ohmmeter* capable of indicating a leakage path of 10^{8} Ω.

7.8 *Conductivity-Type Determination*—Apparatus in accordance with Method A of Test Methods F 42.

7.9 *Analog Test Circuits* to test the suitability and accuracy of the electrical equipment (see Fig. 2 and Table 2).

8. Reagents and Materials

8.1 *Purity of Reagents*—All chemicals for which specifications exist shall conform to SEMI Specifications C 1. Reagents for which SEMI specifications have not been developed shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society[8], where such specifications are available. Other grades may be used provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 *Purity of Water*— Reference to water shall be understood to mean either distilled water or deionized water meeting

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[8] "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Analar Standards for Laboratory U.K., Chemicals," BDH Ltd., Poole, Dorset, and the "United States Pharmacopeia."

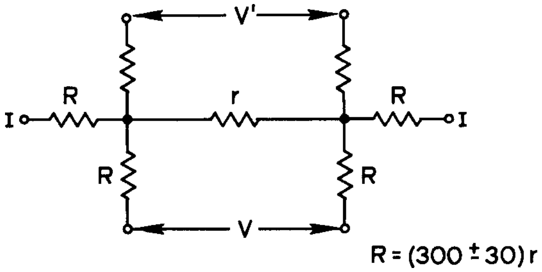

FIG. 2 Analog Circuit to Simulate Resistivity Measurement

TABLE 2 Current Values Required for Measurement of Resistivity

Resistivity, Ω·cm	Standard Resistor (R_s) and Analog Resistor (r),Ω
0.001	0.001
0.01	0.01
0.1	0.1
1.0	1.0
10	10
100	100
1000	1000

the resistivity and purity requirements of Class I reagent water according to Specification D 1193.

8.3 *Etching Solution* (15 + 1)—Mix 90 mL of nitric acid (HNO_3) and 6 mL of hydrofluoric acid (HF).

8.4 *Acetone* ($(CH_3)_2CO$).

8.5 *Methanol* (CH_3OH).

8.6 *Lapping Abrasive*— Aluminum oxide or silicon carbide commercially specified as 27-µm grade.

8.7 *End Contacts*:

8.7.1 *Copper Solution*— Dissolve 20 g of cupric sulfate ($CuSO_4 \cdot 5H_2O$) in 90 mL of water; add 15 mL of hydrofluoric acid (HF) and mix.

8.7.2 *Colloidal Graphite*—Mix by weight 60 g of water with 40 g of a 22 % colloidal graphite-in-water slurry.

8.7.3 *Silver*—Mix by volume 2 parts of acetone ($(CH_3)_2CO$) and 4 parts of methanol (CH_3OH) with 1 part of conducting silver paint.

9. Safety Hazards

9.1 The acids used in this test method are extremely hazardous. All precautions normally used with these chemicals should be strictly observed.

NOTE 4—**Warning:** Hydrofluoric acid solutions are particularly hazardous.

NOTE 5—**Precaution:** They should not be used by anyone who is not familiar with the specific preventive measures and first aid treatments given in the appropriate Material Safety Data Sheet.

9.2 Constant current power supplies are capable of producing high voltages if not connected to an external circuit. Therefore, any changes of connection to a constant current supply should be made either with the current supply turned off or with its output terminals short circuited.

10. Test Specimens

10.1 If not previously prepared using a diamond impregnated saw, saw enough material off both ends of the crystal to leave the ends flat and perpendicular to the length of the crystal. Further cut to square or rectangular cross section if necessary for referee measurement. Make certain to mark or otherwise note the seed end of the crystal.

10.2 Clean the specimen with acetone, rinse with methanol, and air dry.

10.3 Sandblast the specimen ends uniformly with 27-µm grade aluminum oxide or silicon carbide.

10.4 Determine the conductivity type at both ends of the specimen and at 1-cm intervals along the length of the specimen using Method A of Test Methods F 42. Record on a data sheet such as that of Fig. 3. If any specimen shows more than one conductivity type, repeat the conductivity type testing procedure. Resistivity measurements cannot be made by this method on any crystal showing more than one conductivity type.

10.5 Plate the specimen ends electrolytically in copper solution (see 8.7.1) or use the following alternative procedure:

10.5.1 Coat the ends uniformly with the colloidal graphite solution (see 8.7.2). Allow to dry in air for 5 min.

10.5.2 Place a uniform coat of silver paint (see 8.7.3) on the specimen ends. Allow to dry 5 min.

10.6 Allow the specimen to reach thermal equilibrium with the environment of the test station. If the specimen has recently been removed from a crystal grower or heat-treating furnace this will take a minimum of 24 h if air cooled or ½ h if water cooled in addition to ½ h in the ambient environment of the test station. For reference purposes it is required that the tests be performed at 23 ± 5°C.

11. Suitability of Test Equipment

11.1 *Probe Assembly*—Establish the probe spacing and tip condition in the following manner. It is recommended that this be done immediately prior to a referee measurement.

11.1.1 Make a series of ten sets of indentations on a chemically polished crystal slice using normal probe forces (see 7.3.2). Clean the specimen in acetone, rinse with methanol, and let dry. Place the polished silicon specimen on the toolmaker's microscope, measure, and record on the data sheet in Fig. 4 the positions of the pair of probe marks for each of the ten sets.

11.1.2 For each of the ten sets of measurements, calculate the probe separation as follows:

$$S_j = \frac{C + D}{2} - \frac{A + B}{2} \tag{1}$$

where:

j = indentation set number and takes the values 1 through 10, and
A, B, C, and D = dimensions found in Fig. 5.

11.1.3 Calculate the average of the separation as follows:

$$\bar{S} = (1/10) \sum_{j=1}^{10} S_j \tag{2}$$

11.1.4 Calculate the sample standard deviation s_p as follows:

$$s_p = (1/3) \left[\sum_{j=1}^{10} (S_j - \bar{S})^2 \right]^{½} \tag{3}$$

11.1.5 For the probe to be acceptable, the sample standard deviation shall be less than 0.25 % of $\bar{S}$.

11.2 *Electrical Equipment*—Establish the suitability and accuracy of the equipment in the following manner:

11.2.1 Disconnect the electrical leads from the two-point probe.

11.2.2 Attach the current leads (*1* and *2* of Fig. 1) to the current terminals (*I*) of the analog circuit (see Fig. 2). Attach the potential leads (*3* and *4* of Fig. 1) to the potential terminals (*V*) of the analog circuit. Follow the procedure of 11.2.3 and

Crystal No. ________ Type P N

Distance from Seed, cm	Area	T,°C	V_{af},mV	V_f,mV	V_{ar},mV	V_rmV	I_f,mA	I_r,mA	R_f,Ω	R_r,Ω
2										
3										
4										
5										
6										
7										
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9										
10										
11										
12										
13										
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15										
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18										
19										
20										

Use Identical Sheet for Measurement Made 90° Clockwise from First Set of Measurements

NOTE—Five sets of measurements are to be taken at each location.

FIG. 3 Typical Data Sheet for Two-Probe Resistivity Measurement

Probe Serial No. ____________________
Date ____________________
Operator ____________________
Nominal Probe Spacing ____________________

DATA

Run No.	A	B	C	D
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				

COMPUTATIONS

$\frac{A+B}{2}$	$\frac{C+D}{2}$	S_j

$\bar{S}$ (Average Spacing) ____________
$\bar{s}_p$ (Sample Std. Dev.) ____________

FIG. 4 Typical Data Sheet for Computing Probe Spacing

11.2.4. In the case of direct-reading equipment, follow 11.2.5 instead.

11.2.3 With the current in either direction (to be called "forward"), adjust its magnitude to the proper value given in Table 1, and measure the potential drop across the standard resistor (V_{sf}), or measure directly the current I_{af}. Record this value on a data sheet such as the one shown in Fig. 6. Measure and record the potential difference (V_{af}). Reverse the direction of the current. Measure and record the potential difference across the standard resistor (V_{sr}), or measure directly the current I_{ar}. Measure and record the potential difference across the resistor in the analog circuit (V_{ar}).

11.2.4 Repeat the procedure of 11.2.3 until five sets of data have been taken.

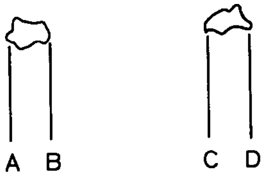

FIG. 5 Probe Indentation Measurement Locations

11.2.5 If equipment that reads resistance (voltage-current ratio) directly is being used, measure the resistance ten times. Reverse the direction of the current after each reading, and record the data using only the last two columns of the data sheet (see Fig. 6).

11.2.6 *Calculation*:

11.2.6.1 If the resistance was measured directly, proceed to 11.2.6.2. If the procedure of 11.2.3 and 11.2.4 was followed, calculate the resistance separately for forward and reverse direction of each of the five sets of data and record (see Fig. 6).

$$R = V_a R_s / V_s = V_a / I_a \tag{4}$$

where:

R = resistance of analog, Ω,
V_s = potential difference across the standard resistor, mV,
V_a = potential difference across the analog resistor, mV,
R_s = resistance of standard resistor, Ω, and
I_a = current, mA.

Use the right-hand form of Eq 4 if the current is measured directly.

11.2.6.2 Calculate the average resistance $\bar{R}_a$ as follows:

$$R_a = (1/10) \sum_{i=1}^{10} R_i \tag{5}$$

where:

R_i = one of the ten values of resistance determined above (see Eq 4).

11.2.6.3 Calculate the sample standard deviation, s_a, as follows:

$$s_a = (1/3) \left[\sum_{i=1}^{10} (R_i - \bar{R}_a)^2 \right]^{1/2} \tag{6}$$

$$s_a = (1/3) \left[\sum_{i=1}^{10} (R_i^{\ 2} - 0.1(\Sigma R_i)^2) \right]^{1/2} \tag{7}$$

11.2.7 *Requirements*— For the electrical measuring equipment to be suitable, it must meet the following requirements:

11.2.7.1 The value of $\bar{R}_a$ must be within 0.3 % of the known value of R.

11.2.7.2 The sample standard deviation, s_a, must be no greater than 0.3 % of $\bar{R}$.

12. Suitability of the Specimen

12.1 Determine the average cross-sectional area of the specimen.

12.1.1 *Cylindrical Specimens*—At intervals of 1.0 ± 0.1 cm along the length of the specimen starting at the seed end, measure and record the diameter of two perpendicular cross sections. Use the average of these two diameters to calculate the cross-sectional area, A, at the point of measurement. Using all values for A thus calculated, calculate the average cross-sectional area, $\bar{A}$, for the entire specimen.

12.1.2 *Bar-Shaped Specimens*—At intervals of 1.0 ± 0.1 cm along the length of the specimen starting 1.0 cm from the seed end, measure and record the width and thickness of the cross section, and calculate the cross-sectional area at the point of measurement. Using all values for A thus obtained, calculate the average cross-sectional area, $\bar{A}$, for the entire specimen.

12.2 For either specimen, if any cross-sectional area is not within ± 1 % of the average measured cross-sectional area for that entire specimen, the specimen is unsuitable for use in this test method.

13. Procedure

13.1 Sandblast two strips approximately 0.5 cm wide from end to end along the longitudinal axis of the specimen and separated by 90° around the circumference; clean with acetone.

13.2 Place the specimen between the conductive plates and lower the probes onto a sandblasted strip with the probes parallel to the length of the crystal. The first measurement shall be made 2 cm from the seed end as measured from the center of the two probes to the crystal end.

NOTE 6—If the specimen is of limited size, such as a section cut from a crystal ingot where the direction of the seed end is no longer known, and if this test method is to be used as a referee method, measurement positions shall be defined that are mutually satisfactory to those performing them.

13.3 With the current initially in either direction, to be called "forward," adjust its magnitude to the value indicated in Table 1. If the resistivity is unknown, start at a low-current value and increase the current until a potential difference of about 10 mV is observed between the two voltage probes.

NOTE 7—For a limited range of crystal rod diameters it is possible within the instrumentation accuracy limitations already listed to use an alternative method of selecting current value. See Annex A1 and Table A1.1 and Table A1.2 for explanation.

13.4 Measure the following and record the data on the data sheet of Fig. 3:

13.4.1 T, the ambient temperature, in °C to the nearest 0.2°C, and

13.4.2 V_{sf}, the potential drop, in mV, across the standard resistor, or measure the specimen current I_f, in mA, directly. Measure V_f, the potential drop, in mV, between the two probes. (Substitute for all the above R_f, the resistance, in Ω, between the two probes, if using direct reading equipment.)

13.5 Reverse the direction of the current, measure, and record the following:

13.5.1 V_{sr}, the potential drop, in mV, across the standard resistor, or measure the specimen current I_r, in mA, directly. Measure V_r, the potential drop, in mV, between the two probes. (Substitute for all the above R_r, the resistance, in Ω, between the two probes, if using direct reading equipment, and use the appropriate columns of a data sheet such as that in Fig. 3.)

13.6 Raise the probe and move the probes 1.0 ± 0.1 cm toward the tang end and repeat the procedure of 13.4 and 13.5, until the probe is within 2 cm of the tang end.

13.7 Rotate the crystal 90° and repeat the procedure of 13.4 through 13.6.

13.8 Repeat the procedure of 13.3 through 13.7 until five sets of data have been taken.