



Designation: F 84 – 02

Standard Test Method for Measuring Resistivity of Silicon Wafers With an In-Line Four-Point Probe¹

This standard is issued under the fixed designation F 84; 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 method² covers the measurement of the resistivity of silicon wafers with a in-line four-point probe. The resistivity of a silicon crystal is an important materials acceptance requirement. This test method describes a procedure that will enable interlaboratory comparisons of the room temperature resistivity of silicon wafers. The precision that can be expected depends on both the resistivity of the wafer and on the homogeneity of the wafer. Round-robin tests have been conducted to establish the expected precision for measurements on *p*-type wafers with room temperature (23°C) resistivity between 0.0008 and 2000 Ω·cm and on *n*-type wafers with room-temperature (23°C) resistivity between 0.0008 and 6000 Ω·cm.

1.2 This test method is intended for use on single crystals of silicon in the form of circular wafers with a diameter greater than 16 mm (0.625 in.) and a thickness less than 1.6 mm (0.0625 in.). Geometrical correction factors required for these measurements are available in tabulated form.³

1.3 This test method is to be used as a referee method for determining the resistivity of single crystal silicon wafers in preference to Test Methods F 43.

NOTE 1—The test method is also applicable to other semiconductor materials but neither the appropriate conditions of measurement nor the expected precision have been experimentally determined. Other geomet-

rics for which correction factors are not available can also be measured by this test method but only comparative measurements using similar geometrical conditions should be made in such situations.

NOTE 2—DIN 50431² is a similar, but not equivalent, method for determining resistivity. It is equivalent to Test Methods F 43.

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.* Specific hazard statements are given in Section 8.

2. Referenced Documents

2.1 ASTM Standards:

D 5127 Guide for Ultra Pure Water Used in the Electronics and Semiconductor Industry⁴

E 1 Specification for ASTM Thermometers⁵

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods⁶

F 42 Test Methods for Conductivity Type of Extrinsic Semiconducting Materials⁷

F 43 Test Methods for Resistivity of Semiconductor Materials⁷

F 2074 Standard Guide for Measuring Diameter of Silicon and Other Semiconductor Wafers⁷

2.2 SEMI Standard:

C19 Specification for Acetone⁸

C28 Specifications and Guidelines for Hydrofluoric Acid⁸

C31 Specification for Methanol⁸

C35 Specifications and Guidelines for Nitric Acid⁸

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

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² DIN 50431 is a similar, but not equivalent, method. It is the responsibility of DIN Committee NMP 221, with which Committee F01 maintains close liaison. DIN 50431, Testing of Inorganic Semiconductor Materials: Measurement of the Specific Electrical Resistance of Monocrystals of Silicon or Germanium by the Four-Point Direct-Current Technique with Linearly Arranged Probes, is available from Beuth Verlag GmbH Burggrafenstrasse 4-10, D-1000 Berlin 30, Federal Republic of Germany.

³ Smits, F. M., "Measurement of Sheet Resistivities with the Four-Point Probe" *Bell System Technical Journal*, BSTJA, Vol 37, 1958, p. 711; Swartzendruber, L. J., "Correction Factor Tables for Four-Point Probe Resistivity Measurements on Thin, Circular Semiconductor Samples." *Technical Note 199*, NBTNA, National Bureau of Standards, April 15, 1964.

⁴ *Annual Book of ASTM Standards*, Vol 11.01.

⁵ *Annual Book of ASTM Standards*, Vol 14.03.

⁶ *Annual Book of ASTM Standards*, Vol 14.02.

⁷ *Annual Book of ASTM Standards*, Vol 10.05.

⁸ Available from the Semiconductor Equipment and Materials International, 3081 Zanker Road, San Jose, CA 95134 (www.semi.org).

3. Terminology

3.1 Definitions of Terms Specific to This Standard:

3.1.1 *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.

3.1.2 *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.

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

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

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

3.1.6 *resistivity, ρ [$\Omega \cdot \text{cm}$]*—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 An in-line four-point probe is used in determining the resistivity in this test method. A direct current is passed through the specimen between the outer probe pins and the resulting potential difference is measured between the inner probes. The resistivity is calculated from the measured current and potential values using factors appropriate to the geometry.

4.2 This test method includes procedures for checking both the probe head and the electrical measuring apparatus.

4.2.1 The spacing between the four probe tips is determined from measurements of indentations made by the probe tips in a polished silicon surface. This test also is used to determine the condition of the probe tips.

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

4.3 Procedures for preparing the specimen, for measuring its size, and for determining the temperature of the specimen during the measurements are also given. Abbreviated tables of correction factors appropriate to circular wafer geometry and a table of temperature coefficient versus resistivity are included with the test method so that appropriate calculations can be made conveniently.

5. Significance and Use

5.1 Resistivity values measured by this test method are a primary quantity for characterization and specification of silicon material used for semiconductor electronic devices.

5.2 The current level, probe force, and specimen surface preparation specified in this test method are to be preferred for all referee measurements on bulk silicon wafers. However,

many changes in these conditions may be made for nonreferee applications without severe changes in measurement results.⁹

5.3 The accuracy of the resistivity as measured by this test method has not been determined. Systematic error is introduced by characteristic radial nonuniformities in the resistivity of silicon wafers and by the finite dimensions of the wafer. The magnitude of these errors is affected by the position of the probe head on the wafer; for referee measurements the probe head should be placed within 0.25 mm (0.01 in.) of the center of the wafer. Systematic error may also be introduced in the measurement of the separation of the probe tips. The relative error in the determination of the probe-tip spacing decreases as the nominal probe-tip spacing increases; for referee measurements a four-point probe with nominal 1.59 mm (62.5 mil) probe-tip spacing is required.

5.4 The recommended analog circuit (Fig. 1) is not a perfect model of a semiconductor wafer being contacted by four metallic probes, with possible rectifying effects. The most effective use of the analog circuit to test the electrical instrumentation for possible error voltage during measurement requires that readings from opposite current polarities be treated separately, and not averaged. In this manner, the calculated standard deviation of the analog measurements will have enhanced sensitivity to possible error voltages.

6. Interferences

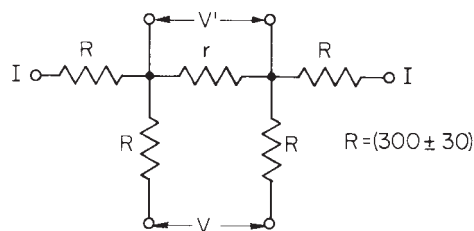
6.1 In making resistivity measurements, spurious results can arise from a number of sources.

6.1.1 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.

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

6.1.3 Minority carrier injection during the measurement can occur due to the electric field in the specimen. With material possessing high lifetime of the minority carriers and high

⁹ Ehrstein, J. R.; Brewer, F. H.; Ricks, D. R.; and Bullis, W. M., "Effects of Current, Probe Force, and Wafer Surface Condition on Measurement of Resistivity of Bulk Silicon Wafers by the Four-Probe Method," Appendix E, "Methods of Measurement for Semiconductor Materials, Process Control, and Devices," *Technical Note 773*, NBTNA, National Bureau of Standards, June 1973, pp. 43–49. Available as COM 73-50534 from National Technical Information Service, Springfield, VA 22161.



NOTE 1—See Table 2 for appropriate values of r .

FIG. 1 Analog Test Circuit to Simulate Four-Probe Measurement

resistivity, such injection can result in a lowering of the resistivity for a distance of several centimetres. Carrier injection can be detected by repeating the measurements at lower current. In the absence of injection no increase in resistivity should be observed. For specimens thicker than 0.75 mm (0.030 in.) use of the currents recommended in Table 1 should reduce the probability of difficulty from this source to a minimum. In cases of doubt and for thinner specimens the measurements of 12.4 and 12.5 should be repeated at a lower current. If the proper current is being used, doubling or halving its magnitude should cause a change in observed resistance which is less than 0.5 %.

6.1.4 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.

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

6.1.6 The temperature corrections given in this test method are valid only if the temperature of the specimen during measurement is held constant in the range from 18 through 28°C.

6.1.7 It is not uncommon with modern digital voltmeters to find that the voltmeter itself provides a source of current of the order of 10 pA between its high and low input terminals. Currents of this magnitude will generally have no effect on measurement accuracy for specimens below about 1000 $\Omega \cdot \text{cm}$. However, since such spurious currents flow through the contact resistance of the voltage sensing probes, which contact resistances may be many megohms for higher resistivity specimens, the result may be spurious voltages of several tens of microvolts. These spurious currents can often be reduced if the autozero and auto calibration functions of the voltmeter can be suppressed. They generally are of fixed sign and the effect of the resulting spurious voltages can generally be cancelled by the use of forward and reverse current measurements (see 12.4 and 12.5).

6.1.8 It is not uncommon with modern digital voltmeters to find that the measurement guard terminal is the source of electrical spikes and other electrical noise components, often due to capacitive coupling to the instrument power supply. Since such noise may be rectified by the contact of the probes to the specimen, care should be taken when choosing where to connect the input guard lead to the measurement circuit.

TABLE 1 Recommended Nominal Measurement Current Values

NOTE 1—This table is based on achieving 10 mV of specimen voltage between Pins 2 and 3 with specimen thickness of 0.5 mm (20 mils).

Resistivity ($\Omega \cdot \text{cm}$)	Current
<0.03	100 mA
0.03 to 0.3	25 mA
0.3 to 3	2.5 mA
3 to 30	250 μA
30 to 300	25 μA
300 to 3000	2.5 μA
>3000	0.25 μA

7. Apparatus

7.1 Slice Preparation:

7.1.1 *Lapping Facilities* which permit the lapping of a wafer so that the thickness varies by no more than ± 1 % from its value at the center.

7.1.2 *An Ultrasonic Cleaner* of suitable frequency (18 to 45 kHz) and adequate power.

7.1.3 *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 Specimen Geometry:

7.2.1 *Thickness*—Calibrated mechanical or electronic thickness gage capable of measuring the wafer thickness to ± 1.0 % (R3S%) at various positions on the wafer.

7.2.2 *Diameter*—A micrometer or vernier caliper.

7.3 Probe Assembly:

7.3.1 *Probe Pins* with conical tungsten carbide probe 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*—The force on each probe shall be 1.75 ± 0.25 N when the probe pins are against the specimen in measurement position.

7.3.3 *Insulation*—For measurement of specimens with resistivity up to approximately 100 $\Omega \cdot \text{cm}$, the electrical isolation between a probe pin (with its associated spring and external lead) and any other probe pin or part of the probe head shall be at least 100 M Ω . For measurement of specimens with higher resistivity, the electrical isolation in ohms should be at least a factor of 1 M times the specimen resistivity in ohm centimetre.

7.3.4 *Probe Alignment and Separation*—The four probe tips shall be in an equally spaced linear array. The probe-tip spacing shall have a nominal value of 1.59 mm (62.5 mils). Probe-tip spacing shall be determined in accordance with the procedure of 11.1 in order to establish the suitability of the probe head as defined in 11.1.3. The following apparatus is required for this determination:

7.3.4.1 *Silicon Surface* such as that of a wafer or block which can be conveniently placed under the probe head. The surface must be polished and have a flatness characteristic of semiconductor wafers used in transistor fabrication.

7.3.4.2 *Micrometer Movement* capable of moving the probe head or silicon surface in increments of 0.05 to 0.10 mm (2 to 4 mils) in a direction perpendicular to a line through the probe tips and parallel to the plane of the surface.

7.3.4.3 *Toolmaker's Microscope* capable of measuring increments of 2.5 μm .

7.3.4.4 *Microscope* capable of a magnification of at least 400 $\times$.

7.4 Specimen and Probe Head Supports:

7.4.1 *Specimen Support*—A copper block at least 100 mm (4 in.) in diameter and at least 38 mm (1.5 in.) thick shall be used to support the specimen and provide a heat sink. It shall contain a hole that will accommodate a thermometer (see 7.5) in such a manner that the center of the bulk of the thermometer shall be not more than 10 mm (0.4 in.) below the central area of the heat sink where the specimen will be placed. A layer of

mica 12 to 25 μm thick shall be placed on top of the heat sink to provide electrical isolation between the specimen and heat sink (Fig. 2). Mineral oil or silicone heat sink compound shall be used between the mica layer and copper block to reduce the thermal resistance. The heat sink shall be arranged so that the center of the probe tip array can be placed within 0.25 mm (10 mils) of the center of the specimen (Note 3). The heat sink shall be connected to the ground point of the electrical measuring apparatus (see 7.6).

NOTE 3—Shallow rings, concentric with the center of the copper block, may be machined into the heat sink in order to assist in rapid centering of wafers.

7.4.2 Probe Head—The probe head shall allow the probe pins to be lowered onto the surface of the specimen with negligible lateral movement of the probe tips (see 11.1.3.4).

7.5 Thermometer—ASTM Precision Thermometer covering a range from -8 to 32°C and conforming to the requirements for Thermometer 63°C as prescribed in Specification E 1. The thermometer hole should be filled with mineral oil or silicone heat sink compound to provide good thermal contact between heat sink and thermometer.

7.6 Electrical Measuring Apparatus:

7.6.1 Any circuit that meets the requirements of 10.2 may be used to make the electrical measurements. The recommended circuit, connected as shown in Fig. 3, consists of the following:

7.6.1.1 Constant-Current Source—The value of current to be used depends on specimen resistivity and thickness. The current supply must have a compliance of at least 10 V, have ripple and noise no more than 0.1 % of the d-c current level being used, and must be stable to at least 0.05 % during the time required for measurement of a specimen. Currents between about 10^{-7} A and 100 mA are necessary to cover the resistivity range from about 0.05 to 10 000 $\Omega\cdot\text{cm}$ with equivalent precision at the specimen voltage level recommended in 12.4, assuming a specimen thickness of 0.5 mm (20 mils). Recommended current values are given in Table 1.

NOTE 4—For specimens of lower resistivity, it is advisable not to use currents significantly above 100 mA due to the risk of joule heating at the current probe tips (see 6.1.4). Rather, to maintain equivalent measurement precision at resistivities below about 0.05 $\Omega\cdot\text{cm}$ it is advisable to use a voltmeter of higher sensitivity and resolution (see 7.6.1.5).

NOTE 5—A smaller range of current values can be used to cover the full range of specimen resistivity and thickness values in the scope of this

method without loss at measurement precision if the electronic voltmeter exceeds the minimum resolution requirements of 7.6.1.5.

7.6.1.2 Current-Reversing Switch.

7.6.1.3 Standard Resistor—The resistance of the standard resistor shall be selected so that it is within a factor of 100 of that of the specimen to be measured. Recommended values of resistance for various resistivity ranges are listed in Table 2.

NOTE 6—It is recommended that the standard resistor be chosen, where possible, to yield a potential difference that is larger than that measured on the specimen, with no upper limit other than that imposed by current carrying limits imposed to retain accuracy of certified resistor value

7.6.1.4 Double-Throw, Double-Pole-Potential-Selector Switch—This switch is needed in the recommended circuit of Fig. 2 to select between the standard resistor and the specimen for voltage measurements.

7.6.1.5 Electronic Voltmeter—This instrument may be used to measure the necessary potential differences in millivolts or it may be calibrated in conjunction with the current source to read voltage-current ratio directly. To cover the full range of specimen resistivities and thicknesses allowed in this test method, the instrument must be at least capable of measuring potential differences from 10^{-4} to 0.05 V with a resolution of 0.05 % of the measured value (at least $3\frac{1}{2}$ significant digits). The instrument must have an input impedance of at least 10^6 times the resistivity of the specimen (see also 6.1.7 and 6.1.8).

NOTE 7—A smaller range of full-scale voltages may be sufficient if only a limited range of specimen resistivity values is to be measured.

7.6.2 Analog Test Circuit—Five resistors connected as shown in Fig. 1 shall be used in testing the electrical measuring apparatus according to the procedure given in 10.2. The resistance of the central resistor, r , shall be selected according to the resistivity of the specimen to be measured as listed in Table 2.

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

7.8 Ohmmeter capable of indicating a leakage path of $10^9 \Omega$.

8. Reagents and Materials

8.1 Purity of Reagents—All chemicals for which such specifications exist shall conform to the assay and to impurity levels of Grade 1 SEMI Specifications for the appropriate chemical. 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, where such specifications are available.¹⁰ 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 Type E-3 or better water as described in Guide D 5127.

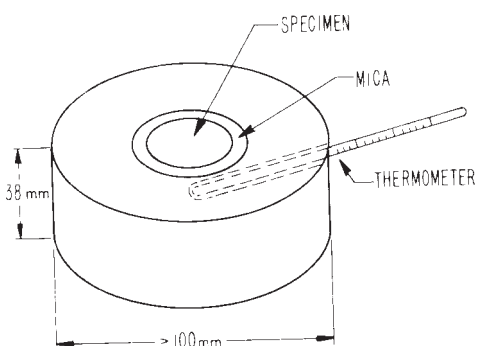


FIG. 2 Heat Sink with Specimen, Mica Insulator, and Thermometer

¹⁰ "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 "Analytical Standards for Laboratory U.K., Chemicals," BDH Ltd., Poole, Dorset, and the "United States Pharmacopoeia."

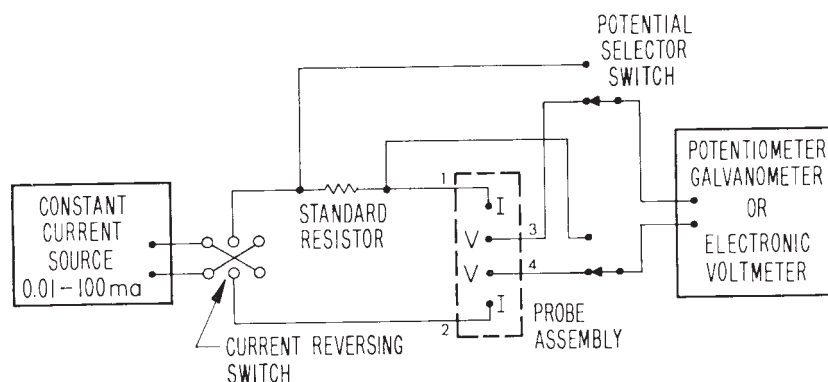


FIG. 3 Recommended Electrical Circuit

TABLE 2 Minimum Recommended Standard Resistor Values for Various Specimen Resistivities

Resistivity ($\Omega \cdot \text{cm}$)	Standard Resistor, Ω ^{A,B}
<0.0025	0.01
0.0020 to 0.025	0.1
0.020 to 0.25	1
0.20 to 2.5	10
2.0 to 25	100
20 to 250	1000
>200	10 000

^A Value must be within $\pm 20\%$ of the nominal value listed and must be known to $\pm 0.05\%$.

^B These values also apply to analog test circuit resistors.

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

8.4 *Acetone* ($(\text{CH}_3)_2\text{CO}$).

8.5 *Methanol* (CH_3OH).

8.6 *Lapping Abrasive*—Aluminum oxide commercially specified as 5- μm grade.

8.7 *Detergent Solution*—An aqueous, nonionic surfactant solution.

8.8 *Mineral Oil or Silicone Heat Sink Compound*.

9. Hazards

9.1 The chemicals used in this evaluation procedure are potentially harmful and must be handled in an acid exhaust fume hood, with utmost care at all times. **Warning**—Hydrofluoric acid solutions are particularly hazardous. 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 supplies are capable of producing high output voltages if not connected to an external circuit. Therefore any changes of circuits connected to a constant current supply should be made either with the current supply turned off or with its output short circuited.

10. Preparation of Test Specimen

10.1 Make ten measurements of diameter, D , for specimens up to 1.9 in.; make five measurements for specimens from 1.9 to 2.9 in.; make three measurements selected in accordance with Guide 2074 for specimens with diameter greater than 2.9 in. (74 mm). The specimen shall be circular; its diameter shall be greater than ten times the average probe-tip spacing S (see

11.1) and shall have a range of values not greater than $D/5S\%$ of D . Record the value of D .

10.2 If wafers are received in an as-sawed condition take at least 50 μm (2 mils) from each side to remove saw damage. This may be done conveniently by etching with the solution listed in 7.4 before lapping.

NOTE 8—Rotating the specimen during etching helps provide a more uniform etch.

10.3 Finish the surface by lapping with 5–9- μm aluminum oxide abrasive. The finished surface shall have a matte rather than a polished nature. The finished thickness w shall be less than the average probe-tip spacing $\bar{S}$. Determine the thickness at nine locations on the specimen (Fig. 4). It shall not vary more than $\pm 1\%$ from the value at the center. Record the value of w at the center of the specimen.

10.4 After lapping, clean the specimen ultrasonically in warm water and detergent, rinse with flowing deionized water, ultrasonically degrease in acetone, rinse with methanol, and air dry. Cushion the specimen with paper or place in a pliable plastic beaker during ultrasonic agitation in order to reduce the risk of breakage.

11. Suitability of Test Equipment

11.1 *Four-Point Probe*—The probe-tip spacing and probe-tip condition shall be established in the following manner. It is recommended that this be done immediately prior to a referee measurement.

11.1.1 *Procedure*:

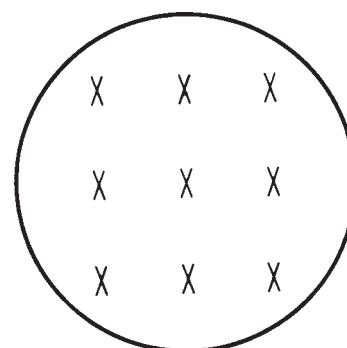


FIG. 4 Crosses Indicate Approximate Locations at Which Specimen Thickness Is to Be Measured