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DIN 50432

AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Methods for Conductivity Type of Extrinsic Semiconducting Materials [1]

This standard is issued under the fixed designation F 42; 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 These test methods [2] cover the determination of the conductivity type of extrinsic semiconductors. While explicit details are given for germanium and silicon, inclusion of other extrinsic materials such as gallium arsenide and indium antimonide should be feasible. For the latter compounds, however, applicability has not been formally verified by round-robin tests. Determinations can be made most reliably on homogeneous bulk material, but these test methods may also be used to map regions of different conductivity type on the surfaces of inhomogeneous specimens. These test methods have not been tested on layered structures such as epitaxial layers. Measurements on these structures may give erroneous indications of conductivity type.

1.2 Four test methods are described:

1.2.1 *Test Method A*—Hot-Probe Thermal EMF Conductivity-Type Test.

1.2.2 *Test Method B*—Cold-Probe Thermal EMF Conductivity-Type Test.

1.2.3 *Test Method C*—Point-Contact Rectification Conductivity-Type Test.

1.2.4 *Test Method D*—Type-All [3] system operating in two modes:

1.2.4.1 Rectification Conductivity-Type Test.

1.2.4.2 Thermal EMF Conductivity-Type Test.

1.3 Experience has shown that Test Method A (hot-probe) gives dependable results in *n*- and *p*-type silicon having a room-temperature resistivity up to 1000 Ω·cm.

NOTE 1—Resistivity of germanium specimens may be measured in accordance with Test Methods F 43 and resistivity of silicon slices may be measured in accordance with Test Methods F 43 or Test Method F 84.

1.4 Test Method B (cold-probe) gives dependable results for *n*- and *p*-type germanium having a room-temperature resistivity of 20 Ω·cm or less and for *n*- and *p*-type silicon having a resistivity up to 1000 Ω·cm (Note 1). This technique has the advantage over the hot-probe test method in that the signal amplitude can be increased by developing a greater temperature difference between the two probes.

1.5 Test Method C (rectification) is a simple convenient technique which gives dependable results for *n*- and *p*-type silicon with room-temperature resistivity between 1 and 1000 Ω·cm. This test method is not recommended for germanium (Note 1).

1.6 Test Method D (type-all rectification mode) is appropriate for use on *n*- and *p*-type silicon having a room-temperature resistivity between 0.1 and 1000 Ω·cm, inclusive (Note 1).

1.7 Test Method D (type-all thermal emf mode) is appropriate for use on *n*- and *p*-type silicon having a room-temperature resistivity between 0.002 and 0.1 Ω·cm, inclusive (Note 1).

1.8 These test methods may apply outside the limits given above, but their suitability outside these limits has not been verified experimentally.

1.9 It is recommended that if satisfactory results can not be obtained with the use of these test methods that conductivity type be determined from Hall-effect measurements as described in Test Methods F 76.

NOTE 2—DIN 50432 covers technical equivalents to Test Methods A and C of these test methods, but does not include Test Methods B and D.

1.10 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1125 Test Methods for Electrical Conductivity and Resistivity of Water [4]

F 43 Test Methods for Resistivity of Semiconductor Materials [5]

F 76 Test Methods for Measuring Resistivity and Hall Coefficient and Determining Hall Mobility in Single-Crystal Semiconductors [5]

[1] These test methods are under the jurisdiction of ASTM Committee F-1 on Electronics, and are the direct responsibility of Subcommittee F01.06 on Silicon Materials and Process Control.

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[2] DIN 50432 is an equivalent method. It is the responsibility of DIN Committee NMP 221, with which Committee F-1 maintains close technical liaison. DIN 50432, Testing of Inorganic Semiconductor Materials: Determining the Conductivity Type of Silicon or Germanium by Means of the Rectification Test or Hot Probe, is available from Beuth Verlag GmbH, Burggrafenstrasse 4-10, D-1000 Berlin 30, Federal Republic of Germany.

[3] Keenan, W. A., Schneider, C. P., and Pillus, C. A., "Type-All System for Determining Semiconductor Conductivity Type," *Solid State Technology*, Vol 14, No. 3, March 1971.

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

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

F 84 Test Methods for Measuring Resistivity of Silicon Slices with a Collinear Four-Probe Array [5]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *conductivity type*—Defines the nature of the majority of carriers in the specimen.

3.1.2 *n-type*—a variety of semiconductive material in which the majority current carriers are electrons, formed when donor impurities are incorporated into the crystal structure in small concentrations.

3.1.3 *p-type*—a variety of semiconductive material in which the majority current carriers are holes, formed when acceptor impurities are incorporated into the crystal structure in small concentrations.

3.1.4 *thermal emf*—the net emf set up in a thermocouple under conditions of zero current. Synonymous with *Seebeck emf.*

4. Summary of Test Methods

4.1 *Test Methods A and B*—In both of these test methods, the sign of the thermal emf generated between two metal probe contacts to the specimen held at different temperatures is used to determine conductivity type. One of the probes is maintained at room temperature while the other is heated (Test Method A) or cooled (Test Method B). The warmer probe will be positive with respect to the cooler probe when the specimen is *n*-type and negative when the specimen is *p*-type. [6] The polarity is observed on a center-zero meter which may be either voltage or current sensitive. Since most of the temperature difference occurs in the region of the probe which is not at room temperature, the sign observed is governed by the conductivity type of the portion of the specimen at this probe contact.

4.2 *Test Method C*—In this test method, the direction of the current through a point contact is used to determine the conductivity type of the specimen. A metal point contact to a *p*-type semiconductor will pass current when the semiconductor is positive while a metal point contact to an *n*-type semiconductor will pass current when the semiconductor is negative. An alternating potential is applied between the point contact and a second large area contact. The direction of current is observed on a zero-center current sensitive meter, an oscilloscope, or a curve tracer. Since rectification occurs at the point contact rather than at the large area contact, the direction of current is governed by the conductivity type of the portion of the specimen at the point contact.

4.3 *Test Method D, Rectification Mode*—In this test method, the polarity of the voltage required to reverse-bias a point contact is used to determine the conductivity type of the specimen. An alternating potential is applied between two point contacts to the specimen. During one half-cycle a given contact will be reverse-biased and will experience the major portion of the voltage drop. During the following half-cycle this junction will be forward-biased and the voltage drop will be small compared to that of the first half-cycle. This inequality of voltages results in a d-c component which is detected by a third point contact.

4.4 *Test Method D, Thermal EMF Mode*—In this test method, a thermal gradient is established in the specimen by an alternating current passing through a pair of point contacts. The thermal emf resulting from the thermal gradient is then detected by a second pair of point contacts. The point contact nearest the first pair will be the warmer and, in *n*-type material, will be positive with respect to the second point contact of the pair. With *p*-type material the warmer contact will be negative with respect to the second contact.

5. Significance and Use

5.1 The determination of conductivity type and the presence of junctions in semiconductors is important in research and development, and in processing or inspection of semiconducting materials for device fabrication.

6. Interferences

6.1 *Test Method A (Hot-Probe)*:

6.1.1 Some high-resistivity silicon and germanium specimens may be nearly intrinsic at the hot-probe temperature; since the mobility of the electrons exceeds that of holes, the thermoelectric power is always negative at these temperatures.

6.1.2 Oxide coating buildup on the hot probe can produce unreliable measurements.

6.1.3 *n*-type germanium with room-temperature resistivity greater than 40 Ω·cm can show *p*-type conductivity due to insufficient probe force (Note 1).

6.2 *Test Method B (Cold Probe)*:

6.2.1 The cold probe should be maintained free of ice. Ice formed during prolonged periods of usage in ordinary ambient air has been found to give erratic results.

6.2.2 Oxide coating buildup on the cold probe can produce unreliable measurements.

6.2.3 *n*-type germanium with room-temperature resistivity greater than 20 Ω·cm can show *p*-type conductivity due to insufficient probe force (Note 1).

6.3 *Test Method C (Rectification)*:

6.3.1 Since this test method indicates primarily the surface-conductivity type, extreme care must be taken in proper surface preparation. A surface oxide can act as an insulator so that no voltage is indicated by the meter.

6.3.2 Reversed readings can sometimes occur if the large-area contact is not held firmly. In such cases, a heavy force on the point contact can cause the large-area contact to become the effective rectifying contact and give reversed meter readings.

6.3.3 Erroneous readings may arise from stray pickup caused by touching the specimen with hands or objects other than the probe.

6.3.4 An etched surface is not recommended because various etchants and etching technique may introduce uncontrolled variations in surface characteristics.

6.4 *Test Method D (Type-All)*:

6.4.1 Erroneous indications may result if the rectification mode is used with very low-resistivity material which results in a low output signal. For silicon, use of the rectification mode in

[6] Scaff, J. H. and Thearer, H. C., Edited by Scaff, J. H., Bridgers, H. E., and Shive, J. N., *Transistor Technology*, D. Van Nostrand Co., Inc., New York, Vol 1, 1958, p. 12.

cases where the output is less than 0.5 V is not recommended.

6.4.2 Erroneous indications may result if the thermal emf mode is used with high-resistivity material.

6.5 All test methods may give erroneous readings if excessive light falls on the specimen, especially with high-resistivity material.

6.6 Ambient radio-frequency energy may cause spurious rectification and erroneous indications.

7. Apparatus

NOTE 3—The instrumentation described in these test methods was in common use at the time the test methods were developed. It is now possible to perform many of the necessary functions using more modern instruments. Such instruments may be substitutes for the apparatus described in this section provided the user can show equivalence for the purposes of the measurement.

7.1 *Test Method A (Hot-Probe)*—The apparatus required consists of the following (Fig. 1):

7.1.1 *Two Probes*, preferably stainless steel or nickel, each with one end terminated in a 60° cone. One of the probes has a 10 to 25-W heater wound about its shank. The heater is electrically insulated from the probe. This probe may be conveniently fashioned from a midget-type soldering iron by attaching the connecting lead directly to a point near the tip and inserting a probe point, as described above, into the tip.

7.1.2 *Variable Power Supply*, capable of raising the temperature of the heated probe to 40 to 60°C.

7.1.3 *Center-Zero Null Indicator*, with a deflection sensitivity of at least 1×10^{-9} A/mm.

7.1.4 *Suitable Temperature Sensor*, for determining temperature of the hot probe in the range from 40 to 60°C.

7.2 *Test Method B (Cold-Probe)*—The apparatus required consists of the following (Fig. 2):

7.2.1 *Two Probes*, with tips of copper or aluminum and shanks insulated with a material such as phenolic fiber. The thermal mass of one of the probes shall be at least that of 15 g of aluminum so that it will remain at or below − 40°C for 5 min in an ambient of 25°C after immersion in liquid nitrogen. The probe tips shall be tapered to nominal 60° cones and the radius of contact shall be approximately 200 µm.

7.2.2 *Center-Zero Null Indicator*, with a deflection sensitivity of at least, 1×10^{-9} A/mm.

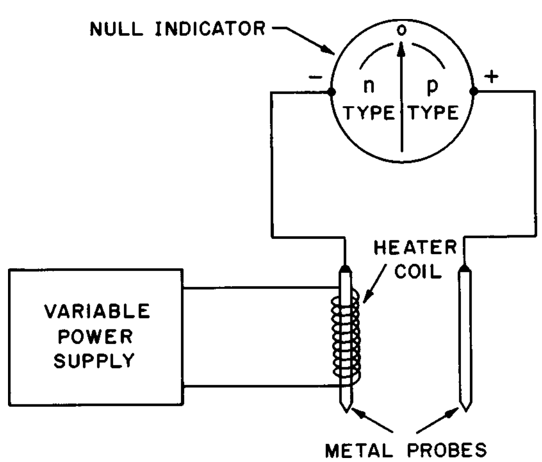


FIG. 1 Apparatus for Determination of Conductivity Type by Hot-Probe Thermal EMF

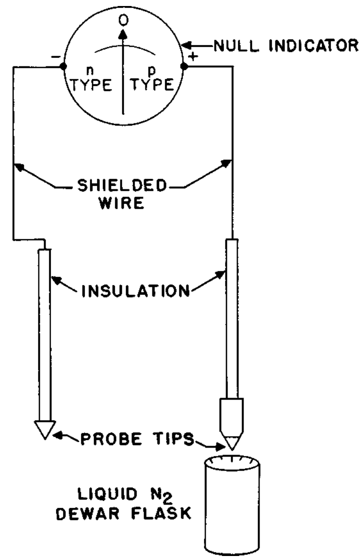


FIG. 2 Apparatus for Determination of Conductivity Type by Cold-Probe Thermal EMF

7.3 *Test Method C (Rectification)*—The apparatus required consists of the following (Fig. 3, Fig. 4, and Fig. 5):

7.3.1 *Adjustable Autotransformer*, such that a 50 to 60-Hz signal of 0 to 15-V peak to peak can be supplied to the specimen (Fig. 3 and Fig. 4).

7.3.2 *Isolation Transformer*, to avoid grounding problems and for safety (Fig. 3 and Fig. 4).

7.3.3 *Probe*, consisting of a suitable conductor such as copper, tungsten, aluminum, or silver. One end shall be tapered with a point radius not greater than 50 µm.

7.3.4 *Large-Area Ohmic Contact*, consisting of a flexible conductor such as lead or indium foil secured firmly to the specimen by a spring-loaded clamp or other equivalent means.

7.3.5 *Center-Zero Meter*, with a sensitivity of at least 200 µA full scale (Fig. 3), an oscilloscope (Fig. 4), or a curve tracer (Fig. 5).

7.4 *Test Method D (Type-All, Rectification Mode)*—The apparatus required consists of the following (Fig. 6):

7.4.1 *Three Probes*, usually part of a four-probe collinear array such as used to determine resistivity.

7.4.2 *a-c Source*, 6 to 24-V, typically 12.6 V, with means to limit the maximum current to no more than 1.0 A.

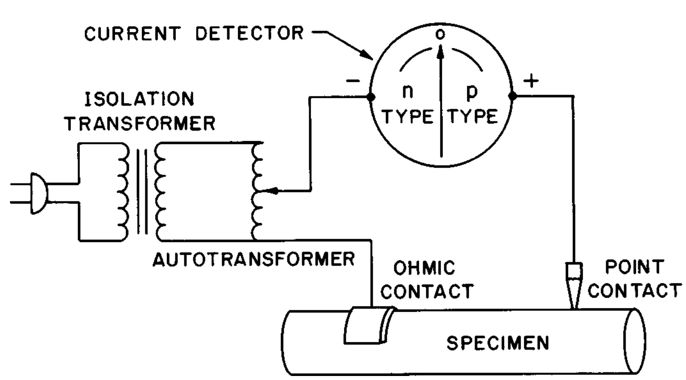


FIG. 3 Circuit for Determination of Conductivity Type by Point-Contact Rectification with the Use of a Current Detector

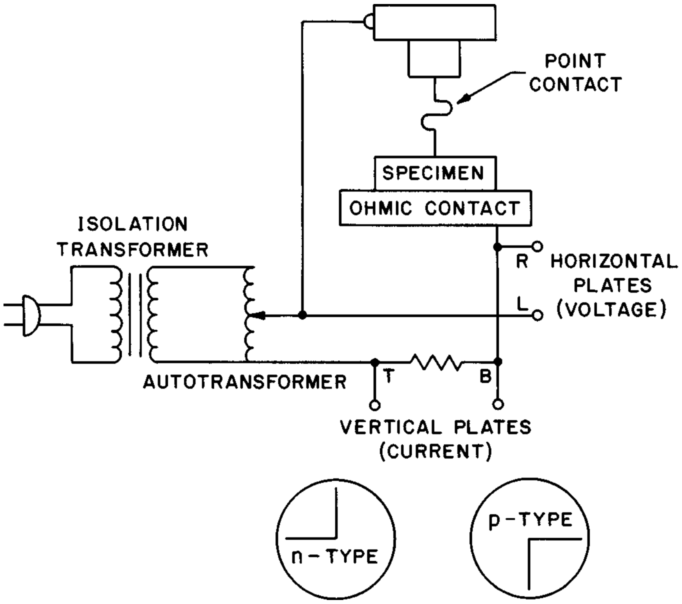
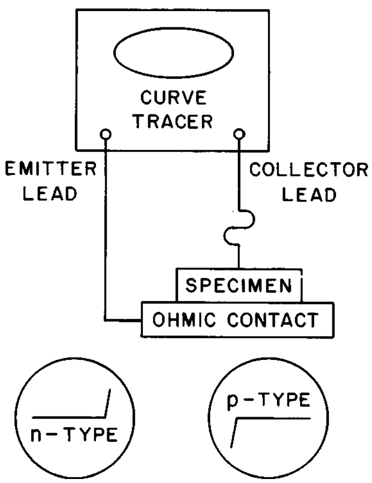
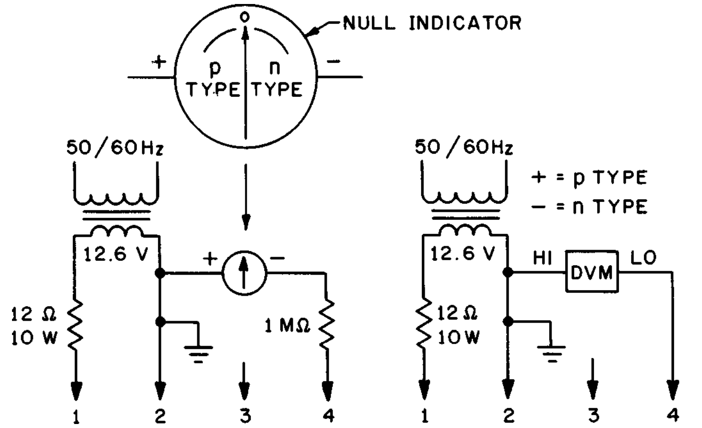


FIG. 4 Circuit and Typical Oscilloscope Displays for Determination of Conductivity Type by Point-Contact Rectification with the Use of an Oscilloscope Display



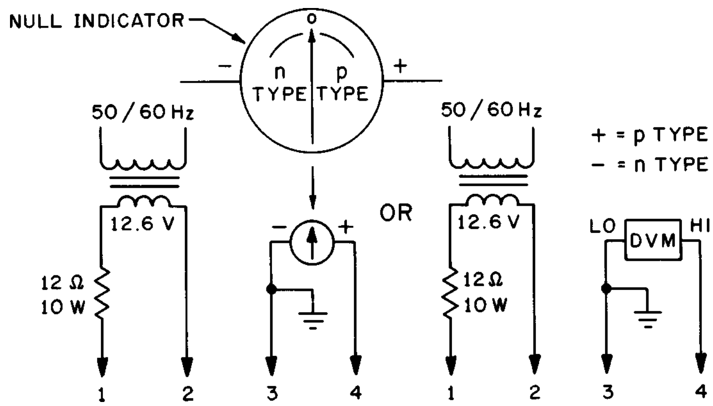
NOTE 1—To obtain complete rectification characteristics as shown it is necessary to switch manually the polarity of the curve tracer.

FIG. 5 Circuit and Typical Displays for Determination of Conductivity Type by Point-Contact Rectification with the Use of a Curve Tracer



(*a*) With Null Indicator (*b*) With Digital Voltmeter

FIG. 6 Circuits for Determination of Conductivity Type by Type-All, Rectification Mode



(*a*) With Null Indicator (*b*) With Digital Voltmeter

FIG. 7 Circuits for Determination of Conductivity Type by Type-All, Thermal EMF Mode

7.4.3 *Center-Zero Null Indicator*, with a sensitivity of at least 10^{-7} A/mm with a 1-MΩ series resistor to give a resolution of at least 0.1 V/mm, or a polarity-indicating digital voltmeter (DVM) with resolution of at least 0.1 V/digit.

7.5 *Test Method D (Type-All, Thermal EMF Mode)*—The apparatus required consists of the following (Fig. 7):

7.5.1 *Collinear Four-Probe Array*, such as a resistivity-test probe.

7.5.2 *Center-Zero Null Indicator*, with a sensitivity of at least 10^{-9} A/mm, or a polarity-indicating DVM with a resolution of at least 100 µV/digit.

7.5.3 *a-c Source*, 6 to 24-V, typically 12.6 V, with means to limit the maximum current to no more than 1.0 A.

7.6 *Compartment*, used to shield optically the specimens under test when needed.

7.7 *Radio-Frequency Shielding* for specimen under test and connecting leads when needed.

7.8 Adequate facilities for lapping or sandblasting.

8. Materials

8.1 *Water*—Reference to water shall be understood to mean either distilled or deionized water having a resistivity greater than 2 MΩ·cm at 25°C as determined by the non-referee method of Test Methods D 1125.

8.2 *Coolant*—Liquid nitrogen, dry ice-acetone mixture, or any other coolant that will reduce the temperature of the cold probe below room temperature. Liquid nitrogen is recommended.

8.3 *Nitrogen* (N_2) *or Air*—Oil free.

8.4 *Stainless Steel Wool or Equivalent.*

9. Procedure

9.1 *Test Method A* (*Hot-Probe*):

9.1.1 Verify that the heated probe is connected to the negative terminal of the center-zero null indicator.

9.1.2 Remove oxides from the tip of the heated probe by burnishing with stainless steel wool or its equivalent.

9.1.3 Raise the temperature of the heated probe to approximately 40 to 60°C. Confirm temperature level with a suitable temperature-measuring sensor.

9.1.4 Space the two probes to within a few millimetres of each other and press both down firmly on the test specimen. Support thin slices in such a manner that they will not break.

9.1.5 Observe the deflection on the center-zero null indicator. Record the specimen as *p*-type if the deflection is to the positive side and as *n*-type if the deflection is to the negative side.

9.1.6 Move the probes about on the specimen surface and sample the area for conductivity type.

9.2 *Test Method B (Cold-Probe)*:

9.2.1 Verify that the cooled probe is connected to the positive terminal of the center-zero null indicator.

9.2.2 Immerse the cold probe in the coolant for several minutes.

9.2.3 Follow the steps in 9.1.4-9.1.6.

9.3 *Test Method C (Rectification)*:

9.3.1 Verify that the circuit is connected according to Fig. 3, Fig. 4, or Fig. 5.

9.3.2 Place the large area contact on the clean surface and clamp or hold firmly.

9.3.3 If a center-zero null indicator is being used, be sure that the point contact is connected to the positive terminal of the meter. Note the direction of deflection. Record the specimen as *p*-type if the deflection is to the positive side and as *n*-type if the deflection is to the negative side. If the meter deflection is not stable do not use this method. If an oscilloscope display is being used, record the specimen as *p*-type or *n*-type in accordance with the rectification characteristics illustrated in Fig. 4. If a curve tracer is being used, connect the point contact to the collector terminal and the area contact to the emitter terminal. Adjust the scale settings on the curve tracer until a rectification characteristic can be observed. Record the specimen as *p*-type or *n*-type in accordance with the rectification characteristics illustrated in Fig. 5.

9.3.4 Move the point probe about the specimen surface and sample the area for conductivity type.

9.4 *Test Method D (Type-All)*:

9.4.1 Verify that the probe is connected for the rectification mode (see Fig. 6) with the second terminal of the probe common to the a-c source and the positive terminal of the null detector or the high-impedance terminal of the DVM.

9.4.2 Place the probe in contact with the specimen. Support thin slices in such a manner that they will not break.

9.4.3 Observe the deflection of the center-zero null indicator or the reading of the DVM. Record the specimen as *p*-type if the indication is positive and as *n*-type if the indication is negative unless the magnitude of the indication is less than 0.5 μA with the null indicator or less than 500 mV with the DVM.

9.4.4 If the indication is below either of these limits, disregard it and reconnect the probe for the thermal mode (see Fig. 7) with the probe tip (No. 3) nearest the source of heat (generated by current between tips 1 and 2) connected to the negative terminal of the null indicator or the low-impedance terminal of the DVM. Place the probe in contact with the specimen and observe the deflection of the center-zero null indicator or the reading of the DVM. Record the specimen as *p*-type if the indication is positive and as *n*-type if the indication is negative.

9.4.5 Using the thermal mode if readings obtained in 9.4.3 are less than 0.5 μA or 500 mV, and the rectification mode if they are greater, move the probes about on the specimen surface and sample the area for conductivity type.

9.5 *Specimen Preparation*—If stable readings and good sensitivity are obtained using the above methods, the surface is free from contamination. If erratic readings or poor sensitivity is noted, the surfaces are contaminated and should be treated in the following manner:

9.5.1 Abrade the surface by lapping or sandblasting, whenever appropriate.

9.5.2 After the surface preparation, wash the specimen in water.

9.5.3 Dry with air or nitrogen.

9.5.4 Take care that the surface remains clean.

10. Report

10.1 Report the following information:

10.1.1 Report the conductivity type as either *n* or *p* if the specimen is homogeneous,

10.1.2 When both *n*- and *p*-type regions are observed, map the surface qualitatively, delineating the regions where the respective types are found, and

10.1.3 Specify the technique used.

11. Precision

11.1 The recommendations in 1.3-1.7 through are based on interlaboratory tests of the four methods. The results of these tests are summarized in Table 1.

12. Keywords

12.1 conductivity type; germanium; rectification test; semiconductor; silicon; thermal EMF test

TABLE 1 Results of Interlaboratory Conductivity-Type Test

Resistivity, Ω-cm	Method A, Hot-Probe [A]			Method B, Cold-Probe [A]			Method C, Rectification [A]			Method D, Type-All Rectification [A]			Method D, Type-All Thermal EMF [A]		
	1	2	3	1	2	3	1	2	3	1	2	3	1	2	3
							Germanium								
0.005	*n*	6	6	*n*	7	7	*n*	3	4	...	...	...	...	...	...
0.005	*p*	6	6	*p*	7	7	*p*	1	4	...	...	...	...	...	...
1.0	*n*	6	6	*n*	7	7	*n*	3	4	...	...	...	...	...	...
1.0	*p*	6	6	*p*	7	7	*p*	3	4	...	...	...	...	...	...
20	*n*	6	6	*n*	7 [B]	7	*n*	4	4	...	...	...	...	...	...
10	*p*	6	6	*p*	7	7	*p*	3	4	...	...	...	...	...	...
50	*n*	6 [B]	6	*n*	6 [C]	7	*n*	4	4	...	...	...	...	...	...
50	*p*	6	6	*p*	7	7	*p*	1	4	...	...	...	...	...	...
							Silicon								
0.002													*n*	7	7
0.005	*n*	6	6	*n*	7	7	*n*	2	4						
0.006	*p*	6	6	*p*	7	7	*p*	1	4						
0.01													*n*	7	7
0.05										*p*	2	2	*p*	5	5
0.5										*p*	7	7			
0.7										*p*	6	6	*p*	1	1
0.9										*n*	7	7			
1.0	*n*	6	6	*n*	7	7	*n*	5	5						
1.0										*p*	7	7			
3.0	*p*	6	6	*p*	7	7	*p*	5	5						
10										*p*	7	7			
50										*n*	7	7			
100	*n*	6	6	*n*	7	7	*n*	5	5						
100	*p*	6	6	*p*	6	6	*p*	5	5						
900	*n*	6	6	*n*	7	7	*n*	5	5						
1000										*n*	7	7			
14000	*p*	6	6	*p*	6	6	*p*	5	5						

[A] Column 1: Conductivity type.
Column 2: Number of laboratories reporting correct type.
Column 3: Number of laboratories that made determination.
[B] One laboratory noted heavy pressure required to read *n*-type.
[C] Two laboratories noted heavy pressure required to read *n*-type.