Designation: F 42 – 02

DIN 50432

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 5127 Guide for Ultra Pure Water Used in the Electronics and Semiconductor Industry[4]

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

Current edition approved Dec. 10, 2002. Published February 2003. Originally approved in 1964 as F 42–64T. Last previous edition approved in 1993 as F 42 – 93 (1997).

[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.

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]
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.

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

[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.

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 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.

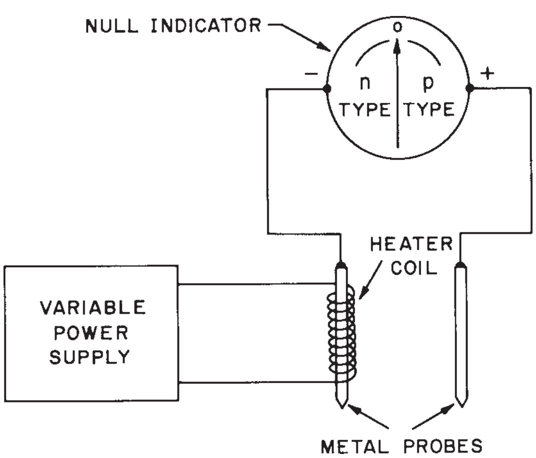

FIG. 1 Apparatus for Determination of Conductivity Type by Hot-Probe Thermal EMF

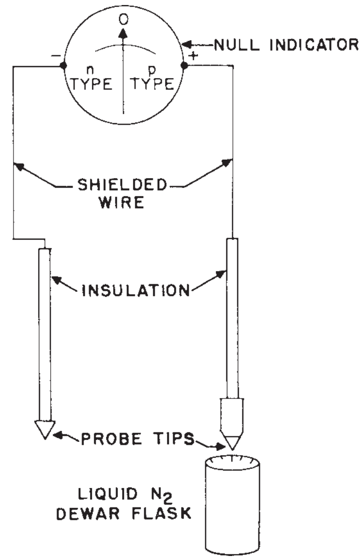

FIG. 2 Apparatus for Determination of Conductivity Type by Cold-Probe Thermal EMF

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

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).

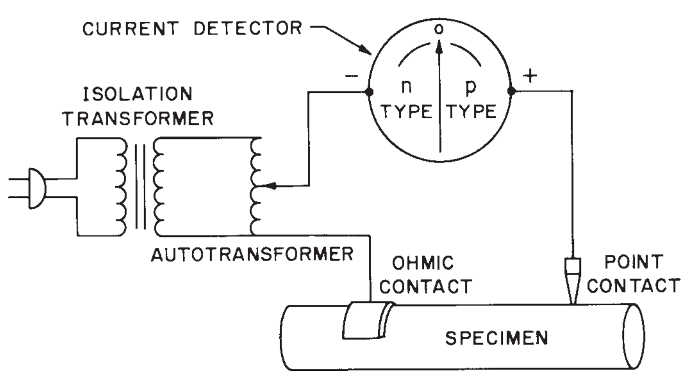

FIG. 3 Circuit for Determination of Conductivity Type by Point-Contact Rectification with the Use of a Current Detector