

IEEE Standard Test Procedures for High-Purity Germanium Crystals for Radiation Detectors

Sponsor

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of the
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Abstract: This standard applies to the measurement of bulk properties of high-purity germanium as they relate to the fabrication and performance of germanium detectors for gamma rays and x rays. Such germanium is monocrystalline and has a net concentration of fewer than 10^{11} electrically active impurity center per cm^3 , usually on the order of 10^{10} cm^{-3} .

Keywords: high-purity germanium (HPGe), radiation detectors

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Introduction

(This introduction is not a part of IEEE Std 1160-1993, IEEE Standard Test Procedures for High-Purity Germanium Crystals for Radiation Detectors.)

The subject of this standard is the measurement of the bulk properties of high-purity germanium (HPGe) intended for use in detectors of ionizing radiation.

Detector manufacturers desire numerical data that can be used to predict the performance of a detector having approximately coaxial geometry. However, because of the many variations in the physical characteristics, the final detector performance cannot be fully predicted from the crystal manufacturers' measurements. This standard defines terminology and measuring techniques for determining and communicating key crystal parameters.

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1. Overview

1.1 Scope

This standard applies to the measurement of bulk properties of high-purity germanium as they relate to the fabrication and performance of germanium detectors for gamma rays and x rays. Such germanium is monocrystalline and has a net concentration of fewer than 10^{11} electrically active impurity centers per cm^3 , usually on the order of 10^{10} cm^{-3} . Test and measurement procedures for fabricated germanium detectors are given in IEEE Std 325-1986 and IEEE Std 759-1984.

1.2 Purpose

The purpose of this standard is to establish uniform procedures for measurements and analyses in the determination and reporting of bulk properties relevant to germanium radiation detector fabrication and performance. These properties are net electrically active impurity concentrations, $|N_A - N_D|$, the concentration of isolated defects with deep electronic levels N_T , and certain crystallographic properties. The techniques described herein are those that have found general use in the industry, are practical, and provide verifiable and desired information to the detector fabricator.

As an aid to the reader, an annex is included for background material. Paragraph numbers in the annex are prefixed by the letter “A,” while the numerical parts of the numbers correspond to those in the main body of the text to which they pertain.

Not all tests described herein are mandatory; however, any tests that are intended to conform to this standard shall be performed according to its provisions.

Mandatory procedures are designated by the word “shall.” Recommended procedures are designated by the words “should” or “recommended.” Optional procedures are designated by the word “may.”

2. References

This document shall be used in conjunction with the standards listed below. References to material appearing in journals and in other publications are listed in clause 8.

IEC 759 (1983), Standard test procedures for semiconductor X-ray energy spectrometers.¹

IEC 973 (1989), Test procedures for germanium gamma-ray detectors.

IEEE Std 325-1986, IEEE Standard Test Procedures for Germanium Gamma-Ray Detectors (ANSI).²

IEEE Std 759-1984, IEEE Standard Test Procedures for Semiconductor X-Ray Energy Spectrometers (ANSI).

3. Letter symbols

Frequently used letter symbols are defined below. Infrequently used symbols are defined where they are introduced in the text.

B	magnetic flux density in tesla (see T)
C	coulombs
C	capacitance in farads
C_d	capacitance of the depleted region in a diode
DLTS	deep-level transient spectroscopy
Δ	change in
e	electronic charge, $1.60 \cdot 10^{-19}$ coulombs
e_r	emission rate from a localized electronic level in s^{-1}
$\exp(z)$	base of the natural logarithms (2.728) to the exponent, z
E	energy associated with an electronic level in the band gap
f	frequency in hertz
HPGe	high-purity germanium
k	Boltzman's constant, $8.617 \cdot 10^{-5}$ eV K ⁻¹
K	kelvin
LN	liquid nitrogen
$N_A - N_D$ ³	net electrically active impurity concentration per cm ³
N_T	deep-level impurity-center concentration per cm ³
Ω	ohm(s)
r	constant associated with measurement of $ N_A - N_D $ by Hall effect
R	resistance in ohms
R_H	Hall coefficient in cm ³ C ⁻¹
ρ	resistivity in $\Omega \cdot \text{cm}$
s	seconds
t	time in seconds

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²IEEE publications are available from the Institute of Electrical and Electronics Engineers, Service Center, 445 Hoes Lane, P.O. Box 1331, Piscataway, NJ 08855-1331, USA.

³The sign of the quantity indicates the type of carrier (n or p). Where only the magnitude is required, the expression will appear as $|N_A - N_D|$.

T	temperature in kelvins
T	tesla (one $T = 10^4$ gauss)
τ	emission time in seconds
μ	drift mobility in $\text{cm}^2 \text{V}^{-1} \text{s}^{-1}$
μ_H	Hall mobility in $\text{cm}^2 \text{V}^{-1} \text{s}^{-1}$
μ_n	electron drift mobility ($\text{cm}^2 \text{V}^{-1} \text{s}^{-1}$) in a semiconductor crystal
μ_p	hole drift mobility ($\text{cm}^2 \text{V}^{-1} \text{s}^{-1}$) in a semiconductor crystal
V	volt(s)
V_p	pulse amplitude (in volts) in deep-level transient spectroscopy
V_r	reverse bias in volts
ω	$2\pi f$ (see f)
W_p	pulse duration (in seconds) in deep-level transient spectroscopy

4. Introduction

Germanium fulfilling the necessary size and purity requirements for high-purity germanium (HPGe) detectors of ionizing radiation presents special problems in characterization resulting from the high resistivity of the material ($\sim 10 \text{ k}\Omega \cdot \text{cm}$ at 77 K), from the degree of impurity compensation that can be encountered, and from difficulties in suitably describing the distribution in the large volume that may form a single device (hundreds of cm^3). Material properties of HPGe have been reviewed in [B7] and [B10]. Other publications are also highly relevant. Existing standards are useful, but are not focused on the problems mentioned above.

One of the most important characteristics of HPGe is the net electrically active impurity concentration, $|N_A - N_D|$, because it determines the depletion voltage required for an operating detector. Although, in principle, the concentration can be extracted from a C^{-2} vs. V plot for a diode fabricated from the material to be characterized, such measurements may not readily yield carrier type, do not quantify nonuniformity, and are more time consuming than other techniques. The usual practice has been to determine $(N_A - N_D)$, with the sign indicating n-type or p-type on the basis of transport measurements using the van der Pauw technique (see [B25]) on lamellar samples immersed in liquid nitrogen (LN).

In the van der Pauw technique, $|N_A - N_D|$ can be computed either from the resistivity, ρ , or from the Hall coefficient, R_H . These, in turn, must be computed from a series of electrical measurements made on the sample.

Measurements made for the determination of ρ are made in the absence of a magnetic field; those for R_H are made with and without the field. The magnetic field, when used, must be oriented orthogonally to the plane of the sample. Details of the measurements are given in 5.2.4 and 5.2.5. The information so obtained also leads to the determination of charge mobility through the crystal.

This standard addresses procedures for characterizing HPGe in three essential areas: net impurity concentration, deep-level trap concentration, and crystallographic quality. Details of sample preparation and the measurement procedures for the determination of $|N_A - N_D|$ are given in clause 5 (see also [B2], Section 63042); the determination of deep-level trap concentration by deep-level transient spectroscopy (DLTS) is covered in clause 6 (see also [B4]); and crystallographic properties are covered in clause 7 (see also [B1] and [B3]).

Because the determination of $|N_A - N_D|$ is critically dependent on sample preparation, a round-robin test was conducted among four laboratories on a set of samples processed by the procedures described in clause 5. The findings are reported in annex A.

5. Sample preparation and measurement of net electrically active impurity concentration $|N_A - N_D|$

The size and shape of a sample upon which measurements will be made depends on the exact purpose of the test. Some characteristics, however, are common to all samples:

- a) Samples shall be lamellar, with the shortest linear dimension along a face being at least three times greater than the thickness.
- b) From the standpoint of measurement theory, samples may be square, rectangular, or circular; geometric perfection is not required.
- c) Surface roughness and strains (such as those caused by lapping) shall be removed by polish etching.
- d) Four contacts shall be attached, usually on the corners of square samples or at $\approx 90^\circ$ spacing on circular samples.

With regard to etching, the surface type (n or p) and the surface stability usually are determined by the details of the wet-chemical processing and the subsequent ambient conditions. Surface type and uniformity may be affected by improper handling. Carriers at an accumulated surface may contribute to conduction. If the surface is inverted, depletion from it will reduce the effective sample thickness. The purer the sample, the more important those effects become and the more important the details of the handling become. For very pure material ($<10^{10} \text{ cm}^{-3}$), specialized etch and quench procedures may be required to achieve consistently satisfactory results, particularly for n-type material.

Further details are given in the following subclauses.

5.1 Sample preparation for van der Pauw measurements

As stated earlier, accuracy in the determination of $|N_A - N_D|$ is critically dependent upon sample preparation, which must be carried out with great care.

5.1.1 Equipment

Required equipment are:

- a) A string saw or diamond saw.
- b) Supplies of semiconductor-grade reagents and access to a vented laboratory hood and sink.
- c) The surfaces of the sample must be etched in a way that ensures acid contact on all surfaces; a beaker with a waffled bottom is useful for the purpose. The size of the beaker is important because self heating occurs during the etching process. If the beaker is too large, the reaction will proceed too slowly and a wasteful amount of reagent will be necessary. If the beaker is too small, the reaction will proceed too violently. The optimum size is slightly wider than the sample and deep enough to allow the surface of the etchant to be 1 to 2 cm above the top of the sample.

5.1.2 Dimensions and provisions for contacts

Samples may be cut and fashioned with a string saw or diamond-blade saw. The minimum lateral dimension of a sample used for determining $|N_A - N_D|$ shall exceed three sample thicknesses.

For small samples, squares (or rectangles) are the easiest to form. They should be 1 to 2 cm on a side. If contacts are made at the corners, which is the preferred practice, the corners shall be beveled the minimum amount necessary for stable physical contact [$\sim 1 \text{ mm}$ bevels, see figure 5.1 a)].

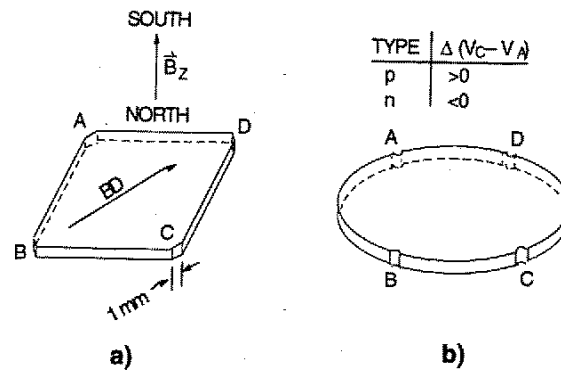


Figure 5.1—Samples: a) square, b) circular

For whole-slice samples, the contacts should be made $\sim 90^\circ$ apart around the perimeter, again minimizing contact width. To penetrate the as-grown skin, nicks (indentations) may be cut into the slice at the points where the contacts are to be made [see figure 5.1 b)].

The samples shall be etched before applying contacts. Material comprising the contacts is listed in the last step of each of the etching procedures.

5.1.3 Etching

Three chemical procedures for sample preparation are listed below: A, B, and C. All have been tested. Procedure A is preferred, but the use of procedure B avoids mixing acid with methanol and therefore presents fewer environmental problems. The procedure ultimately used, however, should be the one that yields the most accurate prediction of the characteristics of completed detectors.

Semiconductor-grade reagents shall be used. See 5.1.1 for the type of beaker to use.

5.1.3.1 Procedure A

- If necessary, clean the sample with methanol or deionized water to remove residual contact metal or other impurities. Allow to dry.
- Prepare chemical polish etch: three parts HNO_3 (70% by weight) to one part HF (49% by weight).
- If the sample already has a polished surface, etch for 45 s while agitating lightly. The etchant should change from clear to a light green color and should exhibit light bubble formation. If the sample has a sawed or lapped surface, etch for 3 min, agitating lightly or until a vigorous boiling-like reaction begins.
- Quench rapidly with semiconductor-grade methanol. Rinse rapidly and thoroughly in methanol without exposing the sample to air long enough for it to dry. Residual methanol should be thoroughly decanted from the beaker before the last rinse.
- Handling only with clean tweezers, dry the sample with a stream of clean dry nitrogen gas.
- At room temperature, apply four contacts with a eutectic mixture of either Ga-In or Hg-In.

The sample is now ready for measurement.