

INTERNATIONAL STANDARD

Nuclear instrumentation – High-purity germanium crystals for radiation detectors – Measurement methods of basic characteristics

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Nuclear instrumentation – High-purity germanium crystals for radiation detectors – Measurement methods of basic characteristics

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**NUCLEAR INSTRUMENTATION –
HIGH-PURITY GERMANIUM CRYSTALS FOR RADIATION DETECTORS –
MEASUREMENT METHODS OF BASIC CHARACTERISTICS**

FOREWORD

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International Standard IEC 61435 has been prepared by IEC technical committee 45: Nuclear instrumentation.

This second edition cancels and replaces the first edition published in 1996 and constitutes a technical revision.

The main technical changes with regard to the previous edition are as follows:

- Review the existing requirements.
- Update the terminology and definitions.

The text of this standard is based on the following documents:

FDIS	Report on voting
45/754/FDIS	45/760/RVD

Full information on the voting for the approval of this standard can be found in the report on voting indicated in the above table.

This publication has been drafted in accordance with the ISO/IEC Directives, Part 2.

The committee has decided that the contents of this publication will remain unchanged until the stability date indicated on the IEC web site under "<http://webstore.iec.ch>" in the data related to the specific publication. At this date, the publication will be

- reconfirmed,
- withdrawn,
- replaced by a revised edition, or
- amended.

A bilingual version of this publication may be issued at a later date.

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INTRODUCTION

Detector manufacturers demand 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 completed detector performance cannot be fully predicted from measurements of the crystal manufacturer. This standard defines terminology and test methods for determining basic crystal parameters such as net electrically active impurity concentrations, deep-level impurity-centre concentration and crystallographic quality of crystals.

Production of germanium crystals of the necessary size and defined purity for high-purity germanium (HPGe) detectors for detection of ionizing radiation has 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, and from difficulties in suitably describing the impurity distribution in the large volume that may form a single device. Existing standards do not cover these problems.

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. 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 method [1]¹ on lamellar samples immersed in liquid nitrogen (LN).

In this technique, ($N_A - N_D$) can be computed either from the resistivity or from the Hall coefficient. These in turn are obtained from a series of electrical measurements made on the sample.

¹ Numbers in square brackets refer to the Bibliography.

NUCLEAR INSTRUMENTATION – HIGH-PURITY GERMANIUM CRYSTALS FOR RADIATION DETECTORS – MEASUREMENT METHODS OF BASIC CHARACTERISTICS

1 Scope and object

This International Standard is applicable to high-purity germanium crystals used for radiation 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 of the order of 10^{10} cm^{-3} .

This International Standard specifies terminology and test methods for measurements of basic characteristics of high-purity germanium crystals. These characteristics are net electrically active impurity concentrations (hereinafter ($N_A - N_D$)), deep-level impurity-centre concentration and crystallographic quality of crystals.

These test methods are not mandatory but have found general use in the industry and provide verifiable and desired information to the detector manufacturer.

Test methods for completed assembled germanium detectors are given in IEC 60973 and IEC 60759.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

IEC 600050-393:2003, *International Electrotechnical Vocabulary (IEV) – Part 393: Nuclear instrumentation – Physical phenomena and basic concepts*

IEC 60050-394:2007, *International Electrotechnical Vocabulary (IEV) – Part 394: Nuclear instrumentation – Instruments, systems, equipment, and detectors*

IEC 60050-521:2002, *International Electrotechnical Vocabulary (IEV) – Part 521: Semiconductor devices and integrated circuits*

3 Terms, definitions, symbols and abbreviations

3.1 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1.1

semiconductor

substance whose total conductivity due to charge carriers of both signs is normally in the range between that of conductors and insulators and in which the charge carrier density can be changed by external means

Note 1 to entry: The term semiconductor generally applies where the charge carriers are electrons or holes.

[SOURCE: IEC 60050-521:2002, 521-02-01]

[SOURCE: IEC 60050-394:2007, 394-28-33]

3.1.2

high purity semiconductor detector

semiconductor detector using a high purity (e.g. high resistivity) semiconductor material

[SOURCE: IEC 60050-394:2007, 394-28-14]

3.1.3

Hall effect

production in a conductor or in a semiconductor of an electric field strength proportional to the vector product of the current density and the magnetic flux density

[SOURCE: IEC 60050-521:2002, 521-09-01]

3.1.4

Hall mobility

product of the Hall coefficient and the electric conductivity

[SOURCE: IEC 60050-521:2002, 521-09-02]

3.1.5

Hall coefficient

coefficient of proportionality R_H in the Hall effect quantitative relation:

$$\vec{E}_H = R_H(\vec{J} \times \vec{B})$$

\vec{E}_H is the resulting transverse electric field strength;

\vec{J} is the current density;

\vec{B} is the magnetic flux density.

Note 1 to entry: The sign of the majority carrier charge can usually be inferred from the sign of the Hall coefficient.

[SOURCE: IEC 60050-521:2002, 521-09-02]

3.1.6

mobility

drift mobility of a charge carrier

quantity equal to the quotient of the modulus of the mean velocity of a charge carrier in the direction of an electric field by the modulus of the field strength

[SOURCE: IEC 60050-521:2002, 521-02-58]

3.1.7

impurity

foreign atoms or either an excess or a deficiency of atoms with respect to the stoichiometric composition of a compound semiconductor

[SOURCE: IEC 60050-521:2002, 521-02-04]

3.1.8

resistivity

inverse of the conductivity when this inverse exists

[SOURCE: IEC 60050-121:1998, 121-12-04]

3.2 Symbols and abbreviations

3.2.1 Symbols

Frequently used symbols are defined below; infrequently used symbols are defined in the text.

A	diode area, expressed in cm^2 ;
B	magnetic flux density, expressed in teslas (T);
C	capacitance, expressed in farads (F);
C_d	capacitance of the depleted region in a diode;
C_i	initial capacitance;
C_f	capacitance at voltage V_f ;
C_m	the capacitance of parallel equivalent circuit;
D_s	the series circuit dissipation factor;
D	Sample thickness, expressed in centimetres (cm);
e	electron charge, $1,60 \times 10^{-19}$ coulombs (C);
e_f	carrier emission rate from a localized electronic level, expressed in s^{-1} ;
E	energy associated with an electronic level in the band gap;
F	frequency, expressed in hertz (Hz);
f	is a factor dependent on ratio $\frac{R_{AB,CD}}{R_{BC,DA}}$;
K	Boltzmann constant, $8,617 \times 10^{-5} \text{ eV} \cdot \text{K}^{-1}$;
$(N_A - N_D)^*$	net electrically active impurity concentration per cm^3 ;
N_T	deep-level impurity-centre concentration per cm^3 ;
N_B	net concentration of all shallower levels;
m	slope of the $C^{-2}(V)$ plot, in $(\text{pF})^{-2} \cdot \text{V}^{-1}$;
Q	charge, expressed in coulombs (C);
r_H	Hall factor;
R	resistance, expressed in ohms (Ω);
R_H	Hall coefficient, expressed in $\text{cm}^3 \cdot \text{C}^{-1}$;
R_p	leakage resistance in parallel with the depleted region;
R_s	the series resistance of the sample, which includes the resistance of the undepleted region and of the contacts;
P	resistivity, expressed in $\Omega \cdot \text{cm}$;
T	temperature, expressed in kelvins (K);
T	time, expressed in seconds (s);
τ	emission time, expressed in seconds (s);
τ_{max}	the rate window at the peak temperature T_{max} ;

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

τ_r	the relaxation time of a carrier;
(τ_r^2)	average square of relaxation time;
μ	drift mobility, expressed in $\text{cm}^2 \text{V}^{-1} \text{s}^{-1}$;
μ_H	Hall mobility, expressed in $\text{cm}^2 \text{V}^{-1} \text{s}^{-1}$;
μ_n	electron mobility in a semiconductor crystal, expressed in $\text{cm}^2 \text{V}^{-1} \text{s}^{-1}$;
μ_p	hole mobility in a semiconductor crystal, expressed in $\text{cm}^2 \text{V}^{-1} \text{s}^{-1}$;
V	voltage;
V_{bi}	built-in potential of the diode;
V_p	filling pulse, expressed in volts (V);
V_r	quiescent reverse bias, expressed in volts (V);
W_p	duration of the filling pulse, expressed in seconds (s);
()	average value;
ΔC_d	the capacitance transient amplitude;
ΔV_c	the increase the reverse bias V_r needed for rising minimal capacitance to the final value.

3.2.2 Abbreviations

DC	direct current;
DLTS	deep-level transient spectroscopy;
HPGe	high-purity germanium;
LN	liquid nitrogen.

3.3 Quantities and units

In the present standard, units of the International System (SI) are used. The definitions of radiation quantities are given in IEC 60050-393, IEC 60050-394 and IEC 60050-521.

Nevertheless, the following non SI units may also be used:

- for energy: electron-volts (eV);
- for time: minutes (min);
- for volume: litres (l);
- for temperature: kelvins (K);
- for thickness: centimetres (cm), millimetres (mm).

Multiples and submultiples of SI units will be used, when practicable, according to the SI system.

4 Measurement of net electrically-active impurity concentrations

4.1 Sample preparation for Van der Pauw measurements

4.1.1 General

Accuracy in the determination of $(N_A - N_D)$ is critically dependent on sample preparation, which shall be carried out with great care.

The size and shape of a sample upon which measurements will be made depends on the exact purpose of the test, but some characteristics 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 90° 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 depend on ambient conditions. Surface type and uniformity may be affected by improper handling. Carriers that accumulate at the surface may contribute to conduction; if the surface is inverted, depletion will reduce the effective sample thickness. The purer the sample, the more important those effects become. For very pure material (impurity net concentration of approximately 10^{10} cm^{-3}), specialized etch and quench procedures may be required to achieve consistently satisfactory results, particularly for n-type material.

4.1.2 Equipment

The equipment for sample preparation shall be:

- a) a string saw or diamond-blade saw;
- b) supplies of semiconductor-grade reagents and access to a vented laboratory hood and sink;
- c) the surfaces of the sample shall be etched in a way that ensures acid contact on all surfaces; a beaker with a waffled bottom is useful for this 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 too small, the reaction will proceed too violently. The optimum size is slightly wider than the sample and deep enough to allow the etchant to extend 1 cm to 2 cm above the surface of the sample.

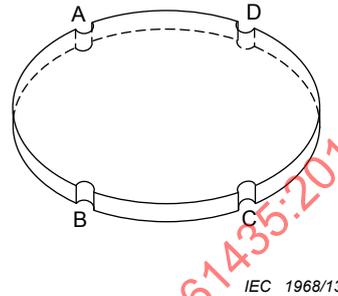
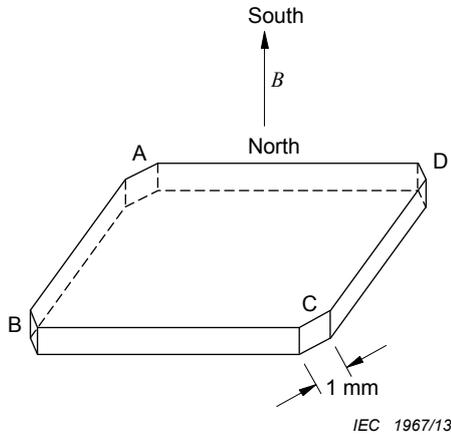
4.1.3 Dimensions and provisions for contacts

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

For small samples, the preferable forms are squares (or rectangles) with side from 1 cm to 2 cm. If contacts are made at the corners (that is preferred) the corners shall be beveled the minimum amount necessary for stable physical contact (~1 mm bevels; see Figure 1a). For circular samples, contacts should be made 90° apart around the perimeter, again minimizing contact width. Nicks (indentations) may be cut into the slice at the points where the contacts are to be made (see Figure 1b).

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

Type	$\Delta(V_C - V_A)$
p	> 0
n	< 0



Key

- B* magnetic field
- $\Delta(V_C - V_A)$ voltage difference between A and C

Figure 1a – Square

Figure 1b – Circular

Figure 1 – Samples

4.1.4 Etching

4.1.4.1 General

Three possible chemical procedures for sample preparation are listed below: A, B, and C. Procedure A is preferred, but the use of procedure B avoids mixing acid with methanol and presents fewer environmental problems. These procedures are used for the most accurate prediction of the characteristics of completed detectors.

Semiconductor-grade reagents shall be used.

4.1.4.2 Procedure A

- a) Clean the sample with methanol or deionized water to remove residual contact metal or other impurities. Allow to dry.
- b) Prepare chemical polish etch: three parts HNO₃ (70 % by weight) to one part HF (49 % by weight).
- c) 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 colour 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.
- d) 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 after the last rinse.
- e) Handling only with clean tweezers, dry the sample with a stream of clean dry nitrogen gas.
- f) At room temperature, apply four contacts with a eutectic mixture of either Ga-In or Hg-In.

The sample is now ready for measurement.

4.1.4.3 Procedure B

- a) Clean the sample with methanol or deionized water to remove residual contact metal or other impurities. Allow to dry.
- b) Prepare chemical polishing etch: two parts HNO_3 (70 % by weight) to one part HF (49 % by weight).
- c) Etch for 30 s after the mixture begins to react vigorously (intense brown colour, large bubbles, and brown nitric oxide fumes). The sample shall be agitated during the reaction to ensure homogeneous etching.
- d) Slowly quench the reaction with deionized water (1 l/min to 2,5 l/min) from 15 s to 30 s. The sample shall not be exposed to air.
- e) Thoroughly clean the sample by flushing with deionized water for 1 min.
- f) Remove the sample with tweezers and immerse in methanol for 1 min.
- g) Handling only with clean tweezers, dry the sample with filter paper or with a stream of clean dry nitrogen gas.
- h) At room temperature, apply four contacts with a eutectic mixture of either Ga-In or Hg-In.

The sample is now ready for measurement.

4.1.4.4 Procedure C

- a) Clean the sample with methanol or deionized water to remove residual contact metal or other impurities. Allow to dry.
- b) Prepare a stock solution of chemical polishing etch: seven parts HNO_3 (70 % by weight), one part fuming HNO_3 , two parts HF (49 % by weight). Let age for at least one day.
- c) For already etched samples, tilt slowly back and forth for 1 min or for lapped samples tilt for 2 min.
- d) Quench the polishing solution with large quantities of fresh semiconductor-grade methanol.
- e) Rinse with methanol from a spray bottle.
- f) Handling only with clean tweezers, dry the sample with a stream of clean dry nitrogen gas.
- g) At room temperature, apply four contacts with a eutectic mixture of Ga-In.

The sample is now ready for measurement.

4.2 Measurements of $(N_A - N_D)$

4.2.1 General

Measurements are carried out on a square sample with four contacts, A through D (see Figure 1a; note that the contacts are labeled in counterclockwise order). Measurements for determination of resistivity (ρ) and Hall coefficient (R_H) shall be made and recorded in one continuous session. The measurements shall be repeated twice with reversed current and the current increased by a factor of 10. The purpose of the reversals and current increase is to uncover faults in the measuring instruments or in the preparation of the samples (see 4.2.3.2).

4.2.2 Equipment

The equipment for measurements of $(N_A - N_D)$ shall be:

- a) a suitable container for immersing the sample in LN during the measurement;
- b) a DC source;
- c) a voltmeter with an input resistance preferably more than 10 M Ω ;
- d) a calibrated, reversible magnetic field source with a magnetic flux density (B) $\geq 0,01$ T and uniformity to ± 5 % over the area of the sample;

e) appropriate electrical switching facilities.

4.2.3 Measurements of resistivity

4.2.3.1 Order of measurement

- a) Immerse the sample in LN and wait for temperature equilibration before recording data.
 b) Drive the current into contact A and out of B (I_{AB}) while measuring and recording the voltage difference (V_{CD}) and polarity between C and D. Define $R_{AB,CD}$ as

$$[R_{AB,CD}] = \frac{|V_{CD}|}{I_{AB}}$$

- c) Drive the current into contact B and out of C (I_{BC}) while measuring and recording the voltage difference and polarity between D and A (V_{DA}). Define $R_{BC,DA}$ as

$$[R_{BC,DA}] = \frac{|V_{DA}|}{I_{BC}}$$

- d) Repeat with the reversed current and with the current increased by a factor of 10.

4.2.3.2 Computation of resistivity

The resistivity ρ can be obtained from formula (1):

$$\rho = \frac{\pi d}{\ln 2} \left(\frac{R_{AB,CD} + R_{BC,DA}}{2} \right) \cdot f \quad (1)$$

where

d is the sample thickness, expressed in cm;

$R_{AB,CD}$ is defined as $[R_{AB,CD}]_{B=0} = \frac{V_{CD}}{I_{AB}}$ with the magnetic field

turned off (see Figure 1);

f is a factor shown in function $f \left(\frac{R_{AB,CD}}{R_{BC,DA}} \right)$ versus $\frac{R_{AB,CD}}{R_{BC,DA}}$ (see Annex B).

The quantities $R_{AB,CD}$, $R_{BC,DA}$, and $\Delta R_{BD,AC}$ shall not have a variation more than $\pm 5\%$ upon current reversal, magnetic field reversal, or increasing the current by a factor of 10 (the last artifact may be caused by using a voltmeter with insufficiently high input resistance). Also, the preceding quantities shall not have a variation more than $\pm 2\%$ over a 5 min period. If the preceding conditions are not met, the sample shall be reconditioned (re-etched, new contacts applied) and the measurements repeated.

4.2.4 Measurements of Hall coefficient

4.2.4.1 General

The procedure for obtaining the value of R_H is similar to the preceding one, but a different sequence of contacts shall be used. Also, these values shall be obtained with and without a magnetic field applied. The field, when applied, shall be perpendicular to the plane of the sample and shall be monitored during the measurements to be sure that the variation is $\pm 2\%$.

4.2.4.2 Order of measurement

- a) with the magnetic field off, drive current into contact B and out of D while measuring and recording the voltage difference between A and C ($[R_{BD,AC}]_{B=0}$);

- b) repeat step a) but with the magnetic field on. Note value of $[R_{BD,AC}]_{B=B}$;
- c) repeat steps a) and b) with the reversed current, and again with the current increased by a factor of 10.

4.2.4.3 Computation of the Hall coefficient

The Hall coefficient R_H in units of $\text{cm}^3 \cdot \text{C}^{-1}$ can be obtained from formula (2):

$$R_H = \frac{d}{B} \left\{ [R_{BD,AC}]_{B=B} - [R_{BD,AC}]_{B=0} \right\} \quad (2)$$

where

d is the sample thickness in cm;

B is magnetic flux density, expressed in teslas (T);

$[R_{BD,AC}]_{B=B}$ is the resistance $R_{BD,AC}$ in Ω with the magnetic field turned on;

$[R_{BD,AC}]_{B=0}$ is the resistance $R_{BD,AC}$ in Ω with the magnetic field turned off.

The required polarity of the magnetic field is shown in Figure 1a where R_H is positive in p-type material and is negative for n-type material.

4.2.5 Calculation of $(N_A - N_D)$ from resistivity

The quantity $(N_A - N_D)$ is obtained from:

$$(N_A - N_D) = \frac{1}{\rho \cdot e \cdot \mu_{(n \text{ or } p)}} \quad (3)$$

where

ρ is the resistivity (see formula (1));

e is the electron charge, $1,6 \times 10^{-19}$ C;

μ is the drift mobility associated with the carrier type of the sample n or p according to whether;

R_H is negative or positive, respectively where in this standard:

$$\mu_n = 36\,000 \text{ cm}^2 \cdot \text{V}^{-1} \cdot \text{s}^{-1} \quad (4)$$

$$\mu_p = 42\,000 \text{ cm}^2 \cdot \text{V}^{-1} \cdot \text{s}^{-1} \quad (5)$$

The preceding drift mobilities for HPGe at 77 K are not universally agreed upon.

If magnetic field reversal and current reversal do not produce a consistent sign for R_H , or if the reversals produce values of $R_{BD,AC}$ that differ by more than $\pm 25\%$, a type shall not be assigned using this technique. If $\mu \leq 10^4 \text{ cm}^2 \cdot \text{V}^{-1} \cdot \text{s}^{-1}$ (as calculated in 4.2.6), the sign of the Hall coefficient shall not be used for specifying type.

4.2.6 Calculation of drift mobility from a Van der Pauw measurement

The data necessary to calculate drift mobility (μ) independently for each sample can be obtained from the Van der Pauw method [1]. The Hall mobility is defined by formula (6):

$$\mu_H = \frac{|R_H|}{\rho} \quad (6)$$

where

$|R_H|$ is the absolute value of the Hall coefficient in $\text{cm}^3 \cdot \text{C}^{-1}$;

ρ is the resistivity (obtained from formula (1)).

The Hall mobility is related to the drift mobility by formula (7):

$$\mu = \frac{\mu_H}{r_H} \quad (7)$$

where

μ_H is Hall mobility obtained from formula (6);

r_H is a Hall factor (~ 1) (see Annex A).

Values of μ obtained from Van der Pauw data may be lower than true values for several reasons:

- a) poor contacts;
- b) the effects of surface charge;
- c) macroscopic non-uniformity;
- d) microscopic inhomogeneities.

Low drift mobility caused by d) results from local shallow fluctuations in potential that impede carrier flow at electric fields less than $1 \text{ V} \cdot \text{cm}^{-1}$. Neutral impurity scattering may also reduce mobility, but this is an unusual problem in HPGe.

Low drift mobilities do not forecast lower carrier velocity at the high fields used in detector operation ($\sim 1 \text{ kV} \cdot \text{cm}^{-1}$), but they do present practical problems in determining $(N_A - N_D)$. Although this standard is restricted to material with $\mu > 25\,000 \text{ cm}^2 \cdot \text{V}^{-1} \cdot \text{s}^{-1}$ at 77 K (see 4.2.8.4), it should not be inferred that material with lower μ is unsuitable for nuclear detectors. For such material, determination of $(N_A - N_D)$ shall proceed along lines different from those specified herein.

NOTE Low values of mobility that persist through repeated etching and contacting of the sample may be taken as an indication of sample inhomogeneity.

4.2.7 Computation of $(N_A - N_D)$ from R_H

The quantity $(N_A - N_D)$ can be obtained from formula (8):

$$(N_A - N_D) = r_H / e \cdot R_H \quad (8)$$

where

r_H is a Hall factor;

e is the electron charge, $1,6 \times 10^{-19} \text{ C}$;

R_H is the Hall coefficient.

NOTE The sign of $(N_A - N_D)$ is the same as the sign of R_H .

For germanium at 77 K, this limit can be achieved only at magnetic field strengths beyond those conveniently available: $B \geq 10 \text{ T}$.

At lower field strengths,

$$r_H = (\tau_r^2)/(\tau_r)^2 \quad (9)$$

for an ideal semiconductor, where τ_r is the relaxation time of a carrier and $(\)$ indicates averaging over occupied states, (τ_r^2) is average square of relaxation time. In HPGe, τ_r depends on the details of the band structure of the carrier and on the scattering mechanisms. Also, r_H is different for n-type and p-type HPGe (see Annex A).

4.2.8 Spatial dependence of $(N_A - N_D)$

4.2.8.1 General

If the volume of an HPGe detector is much bigger than the volume of the Van der Pauw sample (as is usually the case), it is important to determine the axial and radial variations of $(N_A - N_D)$. Nonplanar growth interfaces, radial irregularities in microscopic growth rate, and diffusion from or towards the surface may cause $\Delta(N_A - N_D)$. Axial variations are caused by the non-unity distribution coefficients of the residual impurities. There are also inhomogeneities having sizes from 10 μm to 100 μm due to the microscopic growth rate dependence of the effective distribution coefficient. These inhomogeneities or striations are correlated with the rotation rate of the ingot during crystal growth or are induced by thermal fluctuations during the growth period. The inhomogeneities may cause low measured mobilities near p-n transitions in compensated samples.

4.2.8.2 Radial variations in $(N_A - N_D)$

For the analysis presented herein, it is assumed that $(N_A - N_D)$ is a linear function of radius, and that the average $(N_A - N_D)$ of interest, designated herein as $(N_A - N_D)$, is the arithmetic mean of the measurements made at the centre and edge of a whole crystal. The radial variation $\Delta(N_A - N_D)_{\text{rad}}$ in a slice of crystal is defined by formula (10):

$$\Delta(N_A - N_D)_{\text{rad}} = (N_A - N_D)_{\text{edge}} - (N_A - N_D)_{\text{centre}} \quad (10)$$

The following three techniques are acceptable for determining the radial variations.

4.2.8.3 Effective mobility technique

The effective mobility technique of crystal measurement is used when μ , given by formula (7), is within $\pm 10\%$ of the theoretical values. The measured value of $(N_A - N_D)$, as given by formula (8), may be considered as the true value if the variation $\Delta(N_A - N_D)$ is less than $\pm 15\%$.

4.2.8.4 Cloverleaf technique

Cloverleaf shape of sample may be used if the Van der Pauw measurement on a sample produces a value of $\mu > 25\,000\text{ cm}^2\cdot\text{V}^{-1}\cdot\text{s}^{-1}$ (see Annex A). Such samples may be machined to produce notches between the contacts extending inward by at least half the radius of the sample (an example of such a form is shown in Figure 2a). Then sample shall be re-etched and re-measured (see 4.2.5). In the new geometry, only the central area significantly contributes to the measurement result. In these instance formulas (11) and (12) apply:

$$((N_A - N_D)) = (N_A - N_D)_{\text{slice}} \quad (11)$$

$$\Delta(N_A - N_D)_{\text{rad}} = 2[(N_A - N_D)_{\text{slice}} - (N_A - N_D)_{\text{cloverleaf}}] \quad (12)$$

4.2.8.5 Dice technique

A slice also may be analyzed by taking small disks or squares from the edge and centre (examples of such forms are shown in Figure 2 (b, c)). The maximum lateral dimension of those pieces shall be less than 1/4th the diameter of the sample. The edge sample should be within 1 mm of the periphery. In this case, $\mu > 25\,000\text{ cm}^2\text{V}^{-1}\text{s}^{-1}$, formula (13) applies:

$$((N_A - N_D)) = ((N_A - N_D)_{\text{edge}} + (N_A - N_D)_{\text{centre}})/2 \quad (13)$$

and $\Delta(N_A - N_D)_{\text{rad}}$ is given by formula (10). Note that the radial variation is positive for acceptor concentration increasing or donor concentration decreasing outward along a radius.

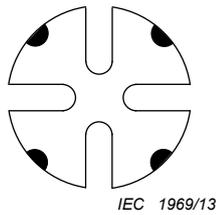


Figure 2a

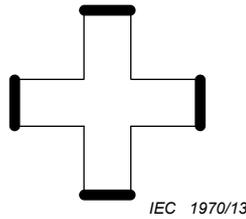


Figure 2b

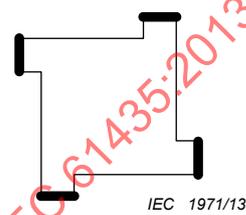


Figure 2c

Figure 2 – Examples of sample shapes

4.2.9 Axial variations in $(N_A - N_D)$

Axial variations are obtained by measuring $(N_A - N_D)$ on slices at different axial locations.

With a slice intended for a planar detector (the diameter exceeds the length), $(N_A - N_D)$ is obtained from whole-slice measurements.

For a piece intended for a coaxial detector (the diameter approximately equals to the length), measurements of $(N_A - N_D)$ and $\Delta(N_A - N_D)$ shall be made on slices adjacent to each end of the crystal. For sections of an as-grown crystal which is long enough to produce several coaxial detectors, interpolation is required from measurements made on slices from the two ends.

5 Deep level transient spectroscopy for the determination of impurity-centre concentration

5.1 General

When HPGe is fabricated into a nuclear radiation detector, electrically active defects may occur at levels deep enough to prevent trapped carriers from being re-emitted in a relatively short time compared with the shaping time of the linear amplifier. The effect may cause peak broadening and tailing to occur in recorded spectra. In this standard, a deep level is one considered to have an emission rate less than 10^6 s^{-1} at 77 K. Capacitance transient measuring techniques, particularly DLTS or Lang's method [2] have proved useful in identifying and quantifying several harmful deep acceptor levels.

5.2 Equipment for DLTS method

The equipment for the Lang method shall be:

- a) a capacitance meter capable of measuring from 1 pF to 100 pF with $\pm 2\%$ precision. A three-terminal instrument is required to guard against the effects of stray capacitance from the sample contacts to the surroundings. The instrument shall allow the application to the

sample of a sustained DC bias of at least 10 V, and the instrument shall respond with a rise time much less than the smallest emission time constant to be measured. Also, a mean limiting the large transient during the filling pulse V_p may be required. A capacitance bridge is not recommended because of its slow response time and inferior signal-to-noise ratio;

- b) a pulse generator capable of changing the bias voltage from one level to a different one, with a switching time much less than the smallest emission time constant to be measured. It may be necessary to connect an additional voltage source in series with the pulse generator in order to extend the range of reverse bias;
- c) a double boxcar integrator (or equivalent) with adjustable gate duration, two separately controllable gate delay times, and adjustable sampling time;
- d) a recording system capable of digitally or continuously measuring the difference in capacitance between the two gate delay times as a function of temperature;
- e) an oscilloscope that allows measurement of the gate delay times and observation of the capacitance transient;
- f) a cryostat in which the sample temperature can be varied from 15 K to 220 K;
- g) a thermometry system that allows measurements of sample temperature within $\pm 0,5$ K while the temperature is varying.

The measurements described here are based on the boxcar-integrator method of Lang [2], but several other valid and acceptable instrumentation schemes may be used.

5.3 Sample selection and preparation for DLTS

The size of the sample and its location in the as-grown crystal are critically important because deep-level concentrations may have strong radial and axial gradients [4]. For DLTS characterization of an HPGe crystal which is to be made into a coaxial detector, the following selection procedure is specified: for p-type, a sample shall be taken from a slice adjacent to the seed end; for n-type, from a slice adjacent to the tail end. The sample shall be located midway between the "flats" formed during crystal growth, and its edge shall be ± 2 mm of the slice's perimeter. If the sample is square, it shall be about 6 mm on a side; if a disc, the diameter shall be about 6 mm.

To determine $(N_A - N_D)$, Van der Pauw measurements shall be made (see Clause 4) prior to junction formation in the sample.

The junction may be formed by any technique that provides rectification: diffusion, implantation or metallization. To prevent diffusion of Cu-related centres into the sample, processing temperatures shall be kept below 550 K, and the thermal processing time shall be less than 2 h. Uncontacted surfaces shall be etched immediately before measurement as a precaution against unwanted surface effects.

5.4 Measurements for the determination of impurity-centre concentration

5.4.1 General

In DLTS, the diode is held at a quiescent reverse-bias voltage V_r . At that voltage, the diode will exhibit a capacitance C_f . Then, a voltage pulse V_p is applied that reduces the bias, thereby introducing majority carriers into a region previously depleted. At the end of the pulse (when the voltage returns to V_r , the majority carrier levels that were filled cause the diode to exhibit a capacitance C_i , but those levels immediately begin emptying, and the capacitance returns exponentially to its original value C_f .

This change is analyzed with a double boxcar averager that produces a signal proportional to $C(t_2) - C(t_1)$, which is the difference between the capacitance at time t_1 and the capacitance at time t_2 [2]. This change is the DLTS signal that is shown in Figure 3.

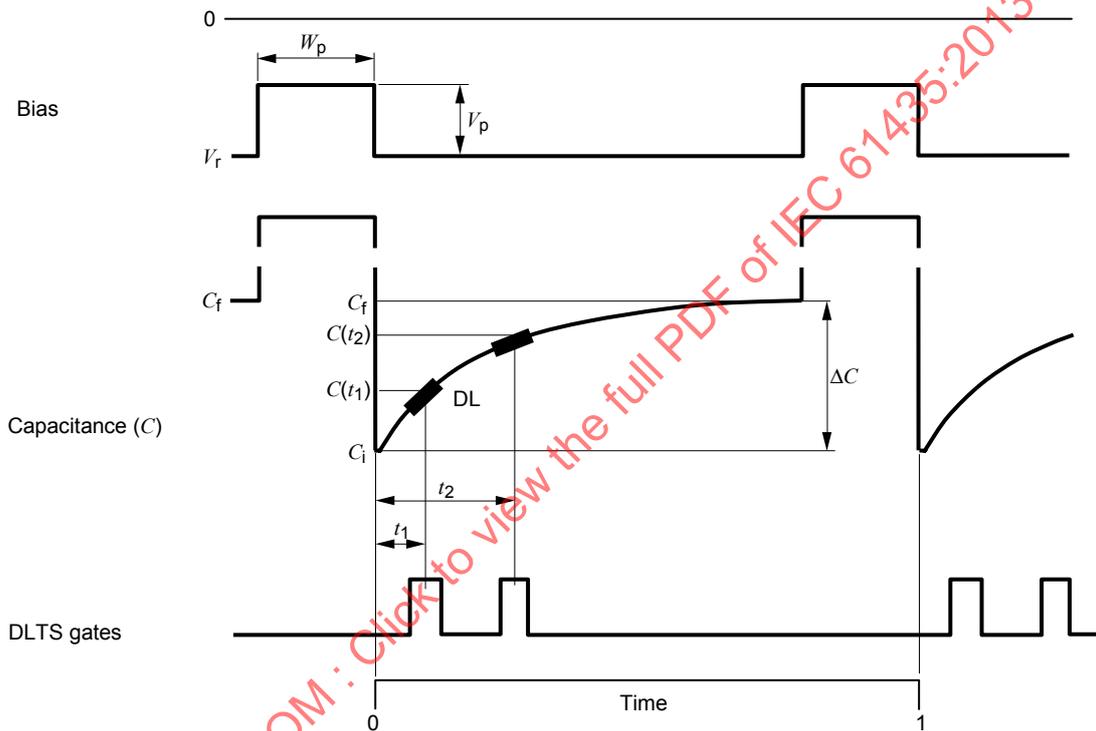
In DLTS, $C(t_2) - C(t_1)$ is plotted as function of temperature. $C(t_2) - C(t_1)$ depends on the carrier emission rate e_r , which increases strongly with temperature. The DLTS signal from a particular level is a maximum at a unique temperature (see 5.4.2) for which the emission time $\tau = e_r^{-1}$ meets the condition of formula (14) [2].

$$\tau_{\max} = \frac{(t_2 - t_1)}{\ln(t_2 - t_1)} \tag{14}$$

where

τ_{\max} is the rate window at the peak temperature T_{\max} ;

t_1 and t_2 refer to the times shown in Figure 3.



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Figure 3 – DLTS waveforms and gate timing

There is a difficulty with DLTS of HPGGe: at low values of $(N_A - N_D)$, the resistance of undepleted germanium may be not only high, but also a strongly increasing function of temperature over the range of interest from 15 K to 220 K.

Depending on $(N_A - N_D)$, sample thickness, bias, frequency of the capacitance-measuring circuit, and temperature, the resistance may be comparable to the capacitive reactance, particularly above 100 K. The foregoing affects the spectrometer sensitivity and the reliability of peak identification. Corrections for equivalent circuit effects are presented in 5.4.4.

Occasionally, extended defects are also observed in DLTS of HPGGe [3], [4], but quantitative interpretation of the data is unclear because there is no generally accepted model for electronic occupation of states associated with them. For this reason, the characterization of extended defects by DLTS is not included in this standard.

The examination of minority-carrier deep levels requires the injection of minority carriers. Since saturation of these is difficult to obtain and demonstrate, the method is more qualitative than quantitative. Such tests will not be dealt with in this standard.

5.4.2 DLTS signal as a function of temperature

The temperature of the sample shall be varied at a rate less than $0,1 \text{ K}\cdot\text{s}^{-1}$ while recording the DLTS signal. The results shall be graphed. To obtain the level parameters: factor K_n and activation energy ΔE_n in Table 1, several DLTS temperature scans are required in which the gate separation time ($t_2 - t_1$) is varied while the ratio t_2/t_1 is held constant. Typically, ($t_2 - t_1$) is from 0,5 ms to 10 ms while the ratio $t_2/t_1 \approx 5$.

5.4.3 Calculation of ($N_A - N_D$)

The temperature shall be stabilized below the lowest temperature DLTS peak appearing in the spectrum from 10 K to 20 K to ensure freeze-out of all deep-level carriers. It shall be verified that the sample is undepleted at V_r . For an abrupt junction with a flat-band surface near the edge of the junction, a least-squares fit of $C^{-2}(V)$ shall approximate a straight line with a slope m that may then be used to estimate the concentration ($N_A - N_D$) from formula (15):

$$(N_A - N_D) = \frac{8,86 \times 10^6}{A^2 m} \quad (15)$$

where

($N_A - N_D$) is in units of cm^{-3} ;

A is the diode area, in cm^2 ;

m is the slope of the $C^{-2}(V)$ plot, in $(\text{pF})^{-2}\cdot\text{V}^{-1}$

The commonly used rectifying contact for p-type HPGe is made by lithium diffusion, resulting in a poor approximation to an abrupt junction. Surface states at the edges of the sample also contribute distortions, as do stray capacitances, non-uniform impurity distributions, and other effects. The use of formula (15) assumes that the space charge region terminates abruptly. This will not be true if more than one majority level is present and may not be justified in extremely pure materials at higher temperatures. Because of these difficulties, the C^{-2} versus V curve shall be used only to check the qualitative behavior of the diode; the concentration of shallow levels shall be obtained from a Van der Pauw measurement (see 4.2.5). The concentration obtained from formula (15) (by taking the $C^{-2}(V)$ slope at V_r) shall be within $\pm 20\%$ of that deduced from the Van der Pauw measurement; corrections shall be made for the concentration of levels that are ionized in thermal equilibrium at the temperature of that measurement, but which are frozen out during the lower temperature $C^{-2}(V)$ measurement.

5.4.4 Corrections for equivalent circuit effects

A simplified equivalent circuit that is appropriate for DLTS measurements of HPGe samples [5], [6] consists of three components that can be ascribed to the depleted and non-depleted regions of the sample:

- The capacitance of the depleted region, C_d , is the component of interest in DLTS measurements. It depends on the applied voltage, ($N_A - N_D$) in the depletion region, the thickness of the depleted region, and the area of the contacts.
- The leakage resistance in parallel with the depleted region, R_p , usually is so high in good material operating at cryogenic temperatures that its effect can be neglected.
- The series resistance of the sample, R_s , (includes the resistance of the undepleted region and of the contacts), if present, affects the determination of C_d . If R_s is due predominantly to the contacts, reworking them may reduce the resistance to a level not requiring corrections. If R_s is due predominantly to the resistance of the undepleted region of the diode, it often is possible to test the sample in a temperature range where R_s is too low to

require corrections. Corrections are discussed below for situations where they cannot be avoided.

Capacitance meters used in the semiconductor industry are three-terminal devices; the samples are connected to the meter through shielded cables. The shields act as guard electrodes to prevent the desired depletion-region capacitance from being affected by the capacitance from the sample contacts to the surroundings. Capacitance can be read directly from a panel-mounted voltmeter, and there is an output connector that allows the capacitance signal V_c to be observed on an oscilloscope. The operating frequency ($f \approx 1$ MHz) is chosen by the manufacturer of the meter to give a good signal-to-noise ratio. That frequency also has the attribute of making the series time constant of the sample considerably less than $(2\pi f)^{-1}$ and the parallel time constant considerably greater than $(2\pi f)^{-1}$.

The meter sees the equivalent of a capacitance and resistance in parallel [5] across its input terminals. Because the true circuit is that of a series time constant $R_s C_d$, it was suggested [5] that in measurements where the capacitance does not change with time formula (16) can be used to compute the capacitance of the depleted region (C_d) from the measured parallel capacitance:

$$C_d = C_m (1 + D_s^2) \quad (16)$$

where

C_m is the measured parallel equivalent capacitance;

D_s is the dissipation factor of the sample with series resistance (R_s) and the angular velocity (ω)

$$D_s = \omega R_s C_d$$

where

$$\omega = 2\pi f$$

f is frequency.

To determine the value of D_s at a particular operating temperature, a temperature plot of $C(T)$ shall be made for the sample. A plateau can be expected somewhere in the range from 20 K to 100 K, indicating that D_s has been reduced to considerably less than 1 and that C_m equals C_d . If the DLTS peak falls on the plateau, no corrections are necessary, but if it is off the plateau on the high temperature side, C_m will decrease. D_s can then be inferred from formula (16). This scheme assumes that the contact resistance is low compared with the resistance of the undepleted region of the diode and that the change in capacitance due to the emptying of deep levels is small compared with the correction term in formula (16).

In DLTS measurements involving capacitance changes, the relative change in series capacitance can be obtained from the measured relative change in parallel capacitance by applying the correction [5], [6] given in formula (17). This correction is valid in the case of small signal, $\Delta C_m < 0,2 C_m$.

$$\frac{\Delta C_d}{C_d} = \frac{\Delta C_m}{C_m} \exp\left[\frac{1 + D_s^2}{1 - D_s^2}\right] \quad (17)$$

where

$\frac{\Delta C_d}{C_d}$ is the corrected fractional change in capacitance of the depleted region;

$\frac{\Delta C_m}{C_m}$ is the measured fractional change in parallel equivalent capacitance.

There is a singularity in formula (17) when $D_S = 1$. The formula shall not be used if $D_S > 0,8$. The correction is unnecessary if $D_S < 0,2$.

5.4.5 Corrections for high trap concentrations and for voltage pulse height

The calculated deep-level concentration N_T shall also be corrected for high trap concentrations and for the height of the voltage pulse. In the classical method [5], formula (18) is applied. A quicker method is given in 5.4.6.

$$N_T = N_B \times \frac{\Delta C_d}{C_d} \times \frac{2 - \frac{\Delta C_d}{C_d}}{\left(1 - \frac{\Delta C_d}{C_d}\right)^2} \times \frac{V_r + V_{bi}}{V_p} \quad (18)$$

where

N_B is the net concentration of all shallower levels derived from a Van der Pauw measurement at 77 K and DLTS analysis;

V_{bi} is the built-in potential of the diode (a value of 0,5 V may be assumed).

5.4.6 $\frac{\Delta V_c}{V_p}$ technique for measuring N_T

This alternative technique [7] is used for determining the relative high deep-level concentrations in high-resistivity material after qualitative analysis for DLTS. It has the merit that overt capacitance measurements are not required (although the capacitance meter shall remain part of the set-up). Also, no corrections are needed for high trap concentrations, series-resistance effects, or voltage pulse height, and inaccuracy of the capacitance meter does not affect the result.

The procedure is as follows:

- a) with the oscilloscope connected to the output of the capacitance meter, obtain an entire capacitance transient waveform on the oscilloscope screen as described in this clause. The exponentially increasing portion of the waveform should resemble the upper one in Figure 4;
- b) record the height of the filling pulse V_p ;
- c) note the vertical position of the encircled point 1, which corresponds to the initial capacitance C_i ;
- d) increase the reverse bias V_r by an amount ΔV_c such that the signal corresponding to the final capacitance C'_f at the encircled point 2 is at the same vertical level as point 1, and record the ΔV_c necessary to establish this condition. Care shall be taken to ensure that the bias does not completely deplete the diode at C_i or C'_i . If that occurs, a smaller initial bias voltage shall be chosen;
- e) compute N_T from formula (19):

$$N_T = \frac{\Delta V_c}{V_p} N_B \quad (19)$$

where

ΔV_c is the increase the reverse bias V_r needed for rising minimal capacitance to the final value.

5.5 Majority-carrier deep levels in p-type HPGe

Majority deep levels that have been reported in p-type HPGe are listed in Table 1 with the identifications suggested by the authors.

Because the capture cross-sections of the defects differ, the concentration of each defect (or the upper limit of the concentration) shall be reported.

The first ionization levels of Cu and Cu-H_x are listed, but are not considered as trapping for the purpose of this standard. If interferences do not exist, these ionization levels shall be used to determine the concentration of the centre.

Deep levels observed but not listed in Table 1 may be identified by the level parameters K_n and ΔE_n , or more simply by the emission rate (e_r) at a particular temperature (see the formula for e_r below).

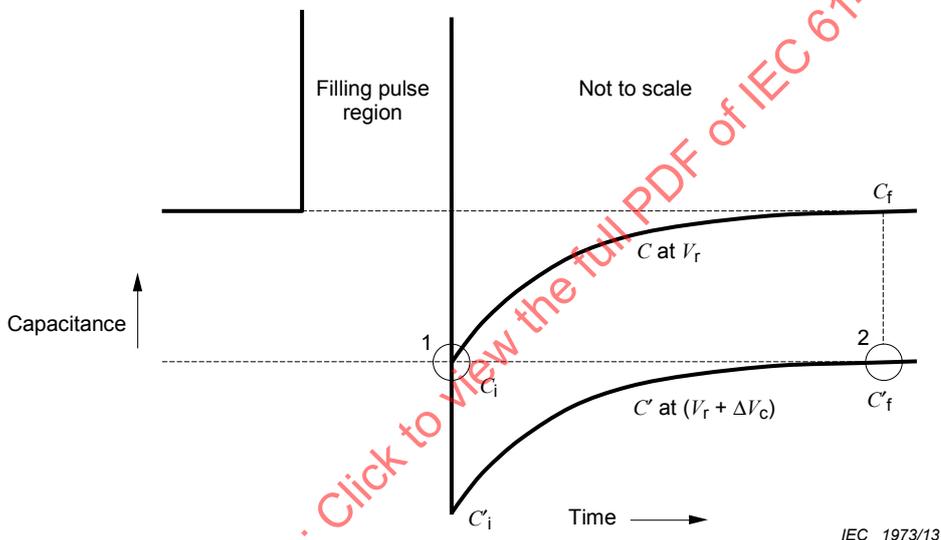


Figure 4 - $\frac{\Delta V_c}{V_p}$ waveforms

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Table 1 – Majority-carrier deep levels in p-type HPGe

Impurity centre*	Ionization level**	K_n ($10^7 \cdot \text{K}^{-2} \cdot \text{s}^{-1}$)	ΔE_n meV	Reference
Cu _s	0/-	6,0	37	[10]
Cu-H	0/-	8,6	62	[10]
V ₂ -H	?	6,2	71	[8]
Cu-H-Li	?	21,8	160	[10]
Cu-H	-/2-	4,5	185	[8]
Cu _s	-/2-	146	322	[8]

NOTE 1 $-e_r = K_n T^2 \exp(-\Delta E_n / kT)$
where
 e_r is the emission rate,
 K_n and ΔE_n are parameters of the level n,
 k is Boltzmann's constant,
 T is temperature, K.

NOTE 2 It may be assumed that the Cu-H_x (0/-) level and all shallower levels are ionized when thermal equilibrium is established at 77 K,
where
* Cu_s refers to substitutional copper,
** 0/- refers to (neutral)/(singly ionized) before/after hole emission
-/- refers to (singly)/(doubly ionized) before/after hole emission.

It shall be verified that previously unpublished defect levels really are bulk effects – rather than surface effects or contact-related phenomena – by measuring the magnitude of the DLTS signal as a function of pulse height V_p at the same V_r . For a uniformly distributed bulk defect, there is a linear relationship between the magnitude of the DLTS signal and V_p . It shall be verified that the emission from the level is exponential. The methods of either references [3] or [4] shall be used. It also shall be verified that capture by such a level is exponential. This shall be done by observing the strength of the DLTS signal or $\Delta C(W_p)$, where W_p is the duration of the filling pulse.

For an isolated defect level, there exists emission time (τ_c) such that signal strength complies with formula (20):

$$\frac{[\Delta C_d]_{(W_p=\infty)} - [\Delta C_d]_{(W_p)}}{[\Delta C_d]_{(W_p=\infty)}} = \exp(-W_p / \tau_c) \quad (20)$$

where

ΔC_d is the capacitance transient amplitude.

5.6 Majority-carrier deep levels in n-type HPGe

Majority-carrier deep levels reported in n-type high-purity germanium exhibit broader peaks than expected for single isolated-defect levels [11]; in some cases capture kinetics similar to those observed in plastically deformed samples were reported [12]. As indicated previously, this standard does not address DLTS of extended defects. Isolated-defect trapping levels shall be characterized and reported in the same manner as in p-type material. Also, both exponential emission from and exponential capture by such levels shall be demonstrated.

5.7 Report

If the measurement technique is different from that of Lang [2] the results shall be preceded by a description of the method that was used.

In all cases, the operating frequency of the capacitance bridge or meter shall be stated. If the Lang method is used, the value of τ_{\max} shall be stated as well. Observed levels that can be quantified according to this standard shall be tabulated with their concentrations. Upper limits on the concentration at the centres listed in Table 1, which are not observed, may be deduced from the minimal detectable capacitance transient at the appropriate temperature. If a DLTS peak is observed but cannot be quantified (e.g. because of high D_s) the reason shall be stated.

6 Crystallographic properties

6.1 General

There have been many reports concerning the effects of crystallographic defects on detector performance. It is noteworthy that absolute crystalline perfection usually is undesirable because as-grown dislocation-free germanium contains an unacceptably high concentration of electrically active vacancy-related point defects [13]. However, dislocations themselves may possess electrical activity due to any of several mechanisms (which of those are important is presently unclear). The effect of dislocations on charge collection has received attention [13], [14], [15], [16], [17] and in one study the DLTS peaks could be related to the type of dislocation [18]. For adequate charge collection in particular detector geometries, references [14] and [17] cite upper limits on etch-pit density. However, since the type of dislocation and its electrical activity appear to depend strongly on growth conditions [15], [16] these limits cannot be assumed universally applicable. Complete crystallographic information is never available, and if it were, the state of knowledge in this field is such that device properties cannot be predicted from these data. However, there is legitimate interest from the device standpoint in some general crystallographic properties as discussed below. For example, apart from dislocations, certain types of large-scale crystallographic defects (twin planes, grain boundaries, and some lineage) frequently generate excess leakage current.

6.2 Crystallographic orientation

Crystalline germanium has a diamond structure, and the crystallographic orientation is referred to as the conventional unit cube. Electronic-transport, mechanical, and etching characteristics are generally anisotropic. The crystallographic orientation shall be determined either optically or by X-ray diffraction. Useful guidance in using those techniques is found in [18], which contains semiconductor single-crystal descriptions of both optical and X-ray diffraction orientation. It is accepted practice to assume the axial orientation of the crystal from the previously measured axial orientation of the seed, providing that no twins or grain boundaries are present in the crystal.

6.3 Sample preparation

6.3.1 General

The as-grown ingot shall be visually examined in strong light for twin planes or grain boundaries. Sections of the crystal displaying either feature are not considered to be monocrystalline. Slices for etch-pit evaluation shall all be cut adjacent to the tail end of the piece being characterized and normal to the growth direction unless other positions are specified by the user.

6.3.2 Preferential etching

Crystallographic perfection is most conveniently assessed by preferential etching techniques that produce pits at the intersection of a dislocation with the surface being etched. The procedure consists of: