

SEMI M64-0915 (Reapproved 0422) TEST METHOD FOR THE EL2 DEEP DONOR CONCENTRATION IN SEMI-INSULATING (SI) GALLIUM ARSENIDE SINGLE CRYSTALS BY INFRARED ABSORPTION SPECTROSCOPY

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1 Purpose

1.1 The purpose of this Test Method is to specify a method to measure the concentration of the deep donor EL2 in SI GaAs by infrared absorption.

2 Scope

2.1 This Test Method covers a procedure for measuring the concentration of the deep donor EL2 in SI GaAs. This method focuses on improving the accuracy and repeatability of the measurement by standardizing the test conditions and reporting and by routine calibration of the measurement.

2.2 This Test Method is intended to cover SI GaAs samples with an electrical resistivity in the range from 1×10^6 to $5 \times 10^8 \Omega \cdot \text{cm}$, determined by doping with carbon. The concentration of EL2 must be greater than $5 \times 10^{15} \text{ cm}^{-3}$.

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3 Referenced Standards and Documents

3.1 SEMI Standards and Safety Guidelines

SEMI M39 — Test Method for Measuring Resistivity and Hall Coefficient and Determining Hall Mobility in Semi-Insulating GaAs Single Crystals

NOTICE: Unless otherwise indicated, all documents cited shall be the latest published versions.

4 Terminology

4.1 Definitions

4.1.1 *transmittance* — the ratio of the intensity of transmitted to incident light at a certain wavelength.

4.1.2 *reflectance* — the ratio of the intensity of reflected to incident light at a certain wavelength.

4.1.3 *ionization degree* — the ratio of the concentration of singly positive charged to total EL2 defects.

5 Properties of EL2

5.1 SI GaAs crystals, grown under arsenic overpressure, contain the double donor EL2. EL2 is an intrinsic point defect, consisting of an arsenic antisite atom. By adding a controlled amount of a compensating shallow acceptor (usually carbon, C_{As}), the Fermi level is pinned at the first electronic level of EL2. At 300 K, this level is at 0.69 eV below the conduction band.

5.2 By variation of crystal growth parameters and by thermal treatment of the grown crystal, the concentration of the EL2 defect can be controlled in the range between 5×10^{15} and $3 \times 10^{16} \text{ cm}^{-3}$. To obtain SI GaAs, complete ionization of shallow acceptors and shallow donors (if present) and partial single ionization of EL2 must be achieved, requiring that:

$$0 < c_A - c_D < c_{EL2} \quad (1)$$

where c_A , c_D , and c_{EL2} are the concentration of shallow acceptors, shallow donors, and EL2, respectively.

5.3 The determination of c_{EL2} is carried out by measuring the near-infrared (NIR) absorption due to the electronic transitions from the first level of EL2 to the conduction band (CB) and valence band (VB). At room temperature, these transitions cause a broad absorption band, starting at about 1700 nm and increasing in strength towards shorter wavelengths.

5.4 The partial ionization of EL2 leads to two contributions to the NIR absorption:

1. The electron transition from the occupied level to the CB
2. The hole transition from the unoccupied level to the VB

5.5 The contributions of both transitions are dependent on the ionization degree f_i of the EL2 defect. f_i is related to the electron concentration n by:

$$f_i = \left[1 + \frac{n}{2N_{CB}} \exp\left(\frac{E_{EL2}}{k_B T}\right) \right]^{-1} \quad (2)$$

where N_{CB} is the effective density of states in the CB ($4.21 \times 10^{17} \text{ cm}^{-3}$ at 300 K), E_{EL2} the activation energy of the electron transition from the first level of EL2 to the CB (0.69 eV at 300 K), k_B the Boltzmann constant, and T the temperature. By measuring n according to SEMI M39 (s.o.), the ionization degree f_i can be determined for a given temperature T .

5.6 The NIR absorption coefficient α_{EL2} at the wavelength λ is given by:

$$\alpha_{EL2}(\lambda) = c_{EL2} \left[(1 - f_i) \cdot \sigma_n(\lambda) + f_i \cdot \sigma_p(\lambda) \right] \quad (3)$$

where σ_n and σ_p are the optical absorption cross sections for the electron transition to the CB and the hole transition to the VB, respectively. At the wavelength $\lambda = 1000 \text{ nm}$, the cross sections, used for this Standard, have the values $\sigma_n = 1.29 \times 10^{-16} \text{ cm}^2$ and $\sigma_p = 0.35 \times 10^{-16} \text{ cm}^2$. For further information, refer to ¶¶ 13.1 through 13.3.

5.7 The concentration c_{EL2} is calculated with the help of Equations 2 and 3:

$$c_{EL2} = \alpha_{EL2} \frac{1 + xn}{xn \cdot \sigma_n + \sigma_p} \quad (4)$$

with

$$x = \frac{1}{2N_{CB}} \times \exp\left(\frac{E_{EL2}}{k_B T}\right) \quad (5)$$

5.8 In the case of a low ionization degree ($f_i \ll 1$ or $xn \gg 1$), Equation 4 can be simplified:

$$c_{EL2} \approx \frac{\alpha_{EL2}}{\sigma_n} \quad (6)$$

6 Interferences

6.1 *Electron Concentration n* — Is strongly dependent on the sample temperature (near room temperature the change is about 10% per 1°C). Therefore, for calculation of c_{EL2} from Equation 4, n must be measured at the same temperature as α_{EL2} .

6.2 *Optical Absorption Cross Sections σ_n and σ_p* — There is some discussion in the literature about the proper value for σ_p (refer to ¶¶ 13.4 and 13.5). Any error of this value leads to bias of the EL2 concentration, becoming increasingly important for large ionization degree.

6.3 *Multiple Internal Reflections* — Multiple internal reflections at the surfaces of the sample can lead to errors in the measured transmittance. This effect can be avoided if the coherence length of the measuring NIR light is small compared to the sample thickness. The proposed spectral width of 40 nm limits the coherence length to about 25 μm .

NOTE 1: Laser light is obviously not appropriate for this purpose.

6.4 *External Reflections* — The tilt of the sample surface with respect to the optical axis must be selected carefully in order to hinder any external reflections at the surfaces of the sample from hitting the detector directly or indirectly.

6.5 *Beam Shading* — Can have a deleterious effect on the accuracy for the determination of the sample transmittance. In particular, the area of the sample must be larger than the beam impinging on the sample.

6.6 *Stray Light* — Coming from the NIR light source or from external sources in the laboratory can lead to bias of the measured sample transmittance if it hits the detector. Appropriate shielding of the apparatus is necessary to block stray light.

7 Apparatus

7.1 *NIR Light Source* — Broadband quartz halogen lamp with electrical power of (typically) 100 W.

7.2 *Optical Imaging System* — Lens system to generate a parallel light beam between light source and sample and an image on the detector.

7.3 *NIR Detector* — Photodiode made from silicon, germanium or indium gallium arsenide with a high linearity and homogeneity. The detector area must be large enough to collect the measuring light completely. Optional: Array or matrix detector for simultaneous measurement of one- or two-dimensional EL2 distributions.

7.4 *Sample Holder* — Capable of maintaining an angle of 2° to 5° between the optical axis and the sample surface. Optional: Translation stage in one or two directions vertically to the optical axis for scanning purposes.

7.5 *1000-nm Optical Band Pass Filter* — With a bandwidth of 40 nm. Used to select the wavelength band of NIR light: $980\text{ nm} < \lambda < 1020\text{ nm}$. Must be placed between light source and sample.

7.6 *Aperture Stop* — Optional; used to increase the spatial resolution. Must be placed in front of the sample.

7.7 *Long-Wavelength (1800 to 2000 nm) Band Pass Filter* — Optional; used to calibrate the apparatus according to procedure A. The filter must be exchangeable with the 1000 nm filter for the measuring light.

7.8 *Calibrated Grey Filter* — Optional; used to calibrate the apparatus according to procedure B. The filter must have a calibrated transmittance τ_G at the wavelength of 1000 nm in the range between 0.45 and 0.55. τ_G must be known within ± 0.005 .

7.9 *SI GaAs Reference Sample* — Optional; used to calibrate the apparatus according to procedure C. The reference sample must have the same thickness as the sample to be measured (maximum deviation: 2%) and the same optical surface quality. The EL2 concentration must be between 1.0 and $1.5 \times 10^{16}\text{ cm}^{-3}$. The transmittance τ_R at the wavelength of 1000 nm must be known within ± 0.005 .

8 Specimen Preparation

8.1 The sample must be prepared from SI GaAs single crystals with an electrical resistivity in the range from 1×10^6 to $5 \times 10^8\ \Omega\cdot\text{cm}$, determined by doping with carbon. The measurement of the electrical resistivity is performed according to SEMI M39.

8.2 The sample must have parallel surfaces and a thickness between 0.5 and 5.0 mm.

8.3 The surfaces must be polished mirror like.

8.4 The sample should be cleaned with organic solvent and deionized water and dried.