



Designation: F 1389 – 00

Standard Test Methods for Photoluminescence Analysis of Single Crystal Silicon for III-V Impurities¹

This standard is issued under the fixed designation F 1389; 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.

INTRODUCTION

Several different methods of photoluminescence analysis are currently in practice worldwide. The following test methods address two of these, one in use primarily in Japan and the other primarily in the United States. Recently published works (1, 2)² describe other approaches that subcommittee F01.06 may incorporate into future versions of these test methods.

1. Scope

1.1 These test methods cover the simultaneous determination of electrically active boron, phosphorus, arsenic, and aluminum content in low-dislocation mono-crystalline silicon. These chemical species can also be determined by the procedure of Test Method F 1630.

1.2 These test methods can be used for samples that have majority dopant densities between approximately 1×10^{11} and 5×10^{15} atoms/cm³.

1.3 The concentrations obtained using these test methods are based on an empirically determined relationship of the logarithm of the concentration to the logarithm of specific luminescence line-intensity ratios.

1.4 The empirical relationship established assumes a constant sample excitation level for all measurements on a given instrument.

1.5 To accommodate differences in instrumentation, two methods are included in this proposal. “Test Method A” refers to procedures appropriate for dispersive infrared spectrophotometers operating under the high sample excitation conditions and “Test Method B” refers to procedures appropriate for Fourier transform instruments operating under low excitation conditions.

1.5.1 Typical calibration curves for each test method are provided. These curves are modified for each instrument using the analysis of standard samples as reference data. Once modified, the curves for a given instrument should produce sample dopant density values that agree with other similarly operated instruments using the same test method. Data obtained using Test Method A may not agree with data obtained

using Test Method B, hence values must be reported with reference to the test method used.

1.6 Many laboratories use photoluminescence to analyze epitaxial layers. However this application encounters many variables and the underlying physics is not fully understood; hence these test methods do not attempt to outline standard practices regarding such analysis.

1.7 *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:

F 416 Test Method for Detection of Oxidation Induced Defects in Polished Silicon Wafers³

F 723 Practice for Conversion Between Resistivity and Dopant Density for Boron-Doped and Phosphorus-Doped Silicon⁴

F 1630 Test Method for Low Temperature FT-IR Analysis of Single Crystal Silicon for III-V Impurities⁴

F 1723 Practice for Evaluation of Polycrystalline Silicon Rods by Float-Zone Crystal Growth and Spectroscopy⁴

3. Terminology

3.1 Definitions:

3.1.1 *defect luminescence lines*—those features arising from defect structures in the silicon.

3.1.2 *electron hole droplet (EHD)*—the condensed phase (liquid) of the excitonic gas generated by photoexcitation. The exciton population is dependent upon excitation intensity and can be raised to the point where the exciton density is sufficient to allow condensation of the exciton gas into a liquid like

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 June 10, 2000. Published August 2000. Originally published as F 1389–92. Last previous edition F 1389–99.

² The boldface numbers in parentheses refer to the list of references at the end of this test method.

³ Discontinued 1998; see 1997 *Annual Book of ASTM Standards*, Vol 10.05.

⁴ *Annual Book of ASTM Standards*, Vol 10.05.

exciton state (3). The existence of EHD luminescence can be an indirect measurement of excitation power density on the sample.

3.1.2.1 *Discussion—EHD onset*—the point in the sample excitation intensity curve where the electron-hole-droplet begins to form (4). The EHD luminescence includes a very broad feature underlying the TO region impurity lines which, with increasing excitation intensity, both intensifies and shifts to lower energies relative to the other silicon luminescence features.

3.1.3 *excitons*—the electron-hole pairs that give rise to the luminescence of interest upon recombination at either a free lattice site (free exciton) or an impurity atom site (bound exciton).

3.1.4 *extrinsic line* ($X_{TO}(BE)$ or $X_{NP}(BE)$)—the luminescence that arises from an exciton captured by an impurity site in the crystal lattice (a bound exciton). Its energy is lower than the intrinsic emission by an amount related to the exciton binding energy of the impurity at 4.2 K.

3.1.4.1 *Discussion—“X”* is the impurity element symbol and “BE” indicates bound exciton luminescence line. Extrinsic luminescence also includes features attributed to bound multi-exciton complexes (b_1 , b_2 , or b_3 would indicate the first, second and third bound multi exciton complex lines, respectively). In donor luminescence, these complexes give rise to two series of lines in the TO region, called the alpha and beta series. The weaker beta series features are denoted by an apostrophe after the line notation (that is, $P_{TO}(b'_1)$). See Table 1 and Table 2 for line locations.

3.1.5 *intrinsic line* ($I_{TO}(FE)$)—the luminescence that arises from the silicon itself, with no impurity species affecting the exciton recombination (5, 6).

3.1.5.1 *Discussion—“I”* indicates intrinsic silicon emission, TO indicates the transverse optical phonon associated with the transition, and FE refers to the free exciton recombination responsible for the emission (see Table 1 for line locations).

3.1.6 *phonon*—a quantum of lattice vibrational energy, as a photon is a quantum of electromagnetic energy.

3.1.6.1 *Discussion*—The recombination of excitons in sili-

TABLE 1 Photoluminescence Line Locations (Vacuum Wavenumbers)

NOTE 1—Formula to convert vacuum wavenumbers to air wavenumbers:

$$(\text{air wavenumber}) = 1.00030025 \times (\text{vacuum wavenumber})$$

NOTE 2—Formula to convert wavenumbers to electronvolts:

$$(\text{eV}) = 1.23985 \times 10^{-4} \times (\text{wavenumber})$$

Silicon Free Exciton (FE) Lines:

$I_{TO}(FE)$ at 8848 cm^{-1}

$I_{LO}(FE)$ at 8860 cm^{-1}

$I_{TA}(FE)$ at 9166 cm^{-1}

Major Shallow Impurity Bound Exciton (BE) Lines:

Element	TO region, cm^{-1}	NP region, cm^{-1}	NP/TO region BE line intensity ratio ^A
Boron	8812.6	9281.3	0.017
(Antimony)	(8810.5)	(9280.0)	(0.010)
Phosphorus	8806.8	9275.4	1.4
Aluminum	8803.4	9271.8	0.7
Arsenic	8801.0	9269.4	2.0

^A Instrument resolution = 0.5 cm^{-1} , sample = FZ silicon.

TABLE 2 Photoluminescence Line Locations (Vacuum Wavenumbers) Detailed Listing for Boron and Phosphorus

Boron Features, cm^{-1}	Phosphorus Features, cm^{-1}
$B_{NP}(BE) = 9281.3$	$P_{NP}(BE) = 9275.4$
$B_{NP}(b_1) = 9263.6$	$P_{NP}(b_1) = 9246.4$
$B_{NP}(b_2) = 9245.9$	$P_{NP}(b_2) = 9223.9$
	$P_{NP}(b_3) = 9208.4$
	$P_{NP}(b_4) = 9197.8$
$B_{TA}(BE) = 9130.1$	$P_{TA}(BE) = 9124.4$
$B_{TA}(b_1) = 9112.4$	$P_{NP}(BE-2e) = 8992.8^A$
$B_{TA}(b_2) = 9095.2$	
$B_{TO}(BE) = 8812.6$	$P_{TO}(b'_1) = 8812.7^B$
$B_{TO}(b_1) = 8795.0$	$P_{TO}(BE) = 8806.8^C$
$B_{TO}(b_2) = 8777.5$	$P_{TO}(b'_2) = 8790.4^B$
$B_{TO}(b_3) = 8763.2$	$P_{TO}(b_1) = 8778.0^C$
$B_{TO}(b_4) = 8752.1$	$P_{TO}(b'_3) = 8771.3^B$
$B_{TO}(b_5) = 8742.6$	$P_{TO}(b_2) = 8756.0^C$
	$P_{TO}(b'_4) = 8756.2^B$
	$P_{TO}(b'_5) = 8745.4^B$
	$P_{TO}(b_3) = 8740.2^C$

^A The two-electron transition is represented by 2e (see Ref (3)).

^B Beta series transition.

^C Alpha series transition.

con requires a momentum conserving mechanism owing to the indirect band gap of the crystal. Phonons provide such a mechanism. The principal phonon types of interest in silicon luminescence are the transverse acoustic (TA), transverse optical (TO), and longitudinal optical (LO) phonons. These test methods address the use of features associated with the TO phonon as well as those not including phonon emission in their momentum-conserving processes. These latter features are designated “NP” or no-phonon features.

4. Summary of Test Method

4.1 A sample of monocrystalline silicon is cooled to 4.2 K and photoexcited with greater-than-bandgap energy light at one of two intensities listed, depending upon the type of instrument used. The resulting luminescence is collected and recorded. Spectral features corresponding to intrinsic silicon and extrinsic impurity emissions are measured and related to calibration curves to yield dopant density.

5. Significance and Use

5.1 Photoluminescence analysis identifies and quantifies the electrically active dopant impurities in monocrystalline silicon. These test methods address boron, phosphorus, arsenic, and aluminum, found as impurities in electronic grade silicon.

5.2 These test methods can be applied to doped and undoped float-zoned or Czochralski material with dopant densities between approximately 1×10^{11} and 5×10^{15} atoms/ cm^3 .

5.2.1 Electronic-grade polycrystalline silicon producers and users require information regarding impurities for quality assurance as well as for research and development purposes. Polysilicon is float-zoned and a sample from the zoned rod is analyzed following these test methods to obtain impurity densities that can be related to the impurity content of the starting material (see Practice F 1723).

6. Interferences

6.1 *Variations in Excitation Intensity*—The extrinsic BE