



Standard Test Method for Wavelength of Peak Photoluminescence and the Corresponding Composition of Gallium Arsenide Phosphide Wafers¹

This standard is issued under the fixed designation F 358; 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.

^{ε1} NOTE—Keywords were added editorially in February 1997.

1. Scope

1.1 This test method covers the techniques used to determine the wavelength of the photoluminescence peak and the mole percent phosphorus content of gallium arsenide phosphide, GaAs_(1-x)P_x.

1.2 Photoluminescence measurements indicate the composition only in the illuminated region and only within a very short distance from the surface, a distance limited by the penetration of the radiation and the diffusion length of the photo-generated carriers, as contrasted to X-ray measurements which sample a much deeper volume.

1.3 This test method is limited by the surface preparation procedure to application to epitaxial layers of the semiconductor grown in a vapor-phase reactor on a flat substrate. It is directly applicable to *n*-type GaAs_(1-x)P_x with the wavelength, λ_{PL}, of the photoluminescence peak in the range from 640 to 670 nm, corresponding to mole percent phosphorus in the range from 36 to 42 % (*x* = 0.36 to 0.42). The calibration data provided for the determination of *x* from λ_{PL} is applicable to material doped with tellurium or selenium at concentrations in the range from 10¹⁶ to 10¹⁸ atoms/cm³.

1.4 The principle of this test method is more broadly applicable. Other material preparation methods may require different surface treatments. Extension to other dopants, doping ranges or composition ranges requires further work to relate λ_{PL} to the phosphorus content as determined by X-ray measurements of the precise dimensions of the unit cell upon which the calibration data are based. It is essential that calibration specimens have uniform composition in the volume sampled.

1.5 This test method is essentially nondestructive. It requires a light etching of the sample to be measured. The removal of a layer of material approximately 0.5 to 1.0 μm in thickness is required. This etching does not degrade the specimen in that devices can still be fabricated from it.

1.6 This test method is applicable to process control in the preparation of materials and to materials acceptance.

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. Specific hazard statements are given in Section 7.*

2. Referenced Documents

2.1 ASTM Standards:

D 1125 Test Methods for Electrical Conductivity and Resistivity of Water²

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods³

E 275 Practice for Describing and Measuring Performance of Ultraviolet, Visible, and Near-Infrared Spectrophotometers⁴

2.2 SEMI Standard:

C1 Specifications for Reagents⁵

3. Summary of Test Method

3.1 The photoluminescence spectrum is recorded for the wavelength range from 600 to 750 nm and the wavelength, λ_{PL}, at which maximum luminescence occurs is determined by means of a graphical construction. The phosphorus content is then determined by means of a calibration curve relating λ_{PL} to the amount of phosphorus as determined by X-ray measurement of the precise dimension of the unit cell.

4. Interferences

4.1 The apparent position of the photoluminescence peak can be distorted by the spectral response characteristics of the detection system, and, in particular, by the spectral response of the photomultiplier. Therefore, the detector to be used for measurements on a specific range of alloy compositions should be chosen so that the corresponding range of λ_{PL} falls in a region where the detector response is changing slowly.

¹ This test method is under the jurisdiction of Committee F-1 on Electronics and is the direct responsibility of Subcommittee F01.15 on Gallium Arsenide.

Current edition approved Nov. 28, 1983. Published July 1984. Originally published as F 358 – 72 T. Last previous edition F 358 – 73 (1983).

² Annual Book of ASTM Standards, Vol 11.01.

³ Annual Book of ASTM Standards, Vol 14.02.

⁴ Annual Book of ASTM Standards, Vol 03.06.

⁵ Available from Semiconductor Equipment and Materials Institute, 625 Ellis St., Suite 212, Mountain View, CA 94043.

4.2 The presence of strong background radiation and, in particular, of background radiation which changes rapidly with wavelength can displace the apparent position of the photoluminescence peak. Users should, therefore, assure themselves that the background radiation is small by replacing the sample with a mirror and scanning through the wavelength range of interest. The resulting trace should be a small fraction of the photoluminescence signal.

4.3 Since the energy of the band gap of most semiconductors, and of GaAs_{(1-x)P_x} in particular, varies with temperature, the measurement of λ_{PL} can be perturbed if the incident power density from the illuminator is high enough to locally heat the specimen. Users of this technique should, therefore, assure themselves that they are not using too high a power density by measuring λ_{PL} as a function of incident power, by using neutral density filters or other means. There should be no variation if the power level is low enough; λ_{PL} will shift to longer wavelengths with increasing power if power is excessive.

5. Apparatus

5.1 *For Specimen Preparation*—Chemical laboratory apparatus such as plastic beakers, plastic-coated tweezers suitable for use with acids, and adequate facilities for handling and disposing of acids and their vapors must be provided.

5.2 *For Measurement of Specimen Photoluminescence* (see Fig. 1):

5.2.1 *Light Source*, a 200-W mercury or xenon arc lamp, a laser, or other source, with suitable filtration and focusing lens to illuminate the specimen with radiation at a wavelength shorter than 600 nm with a total incident energy of at least 1 mW in an area 1 mm² or less.

5.2.2 *Specimen Support*—A holder that can support the specimen in such a position that the incident radiation strikes it in a position that can be viewed by the collection optics of the monochromator. The holder should not damage the surface of the specimen and preferably should not touch the surface. It should also allow the controlled movement of the specimen in its own plane so that the luminescence of a desired portion of the specimen can be measured.

5.2.3 *Collection Optics*—A system of lenses and filters arranged to image the illuminated region of the specimen onto the entrance slits of the monochromator. It is important that the illuminating radiation be kept out of the monochromator either

by filtration or by positioning of the specimen with respect to the illuminating radiation so that the specularly reflected rays do not enter the collection system, or both. Fig. 1 shows a schematic diagram of a system in which the effects of the reflected illumination are minimized by suitable positioning.

5.2.4 *Monochromator*, designed to operate in the 600 to 750-nm wavelength range with wavelength accuracy and repeatability of 0.5 nm as determined in accordance with Practice E 275.

5.2.5 *Detector*—A photomultiplier tube with constant or slowly varying spectral sensitivity throughout the range of interest.

NOTE 1—In the absence of data to the contrary, a variation of no more than 10 % in sensitivity in any 10-nm region of the spectral range of interest as determined from the manufacturer’s published sensitivity curves for the tube shall be deemed acceptable.

5.2.6 *Detector Electronics*—Electronics capable of supplying the high voltage required by the photomultiplier and of detecting and amplifying the anode current from the photomultiplier so that it can drive the chart-recorder electronics.

5.2.7 *Detection System Sensitivity*—The detection system, consisting of collection optics, monochromator, detector, and detector electronics, should be capable of responding to a luminescence signal of 10⁻⁶ mW/nm or less as calculated from the following equation:

$$B = Sf^2/(WmTDG)$$

where:

- B = luminescence signal, mW/nm,
- S = minimum detectable signal at the output electronics, typically 10 times the detector dark current, mA,
- f = the lesser of the speeds (f numbers) of the collection optics or the monochromator,
- W = monochromator bandwidth, nm,
- m = efficiency of the grating (assume m = 0.5 in the absence of other data),
- T = mean transmission of the filters between the specimen and the monochromator in the band from 640 to 670 nm,
- D = mean detector sensitivity, mA/mW, and
- G = gain of the detector electronics, including the photomultiplier gain if this is not already included in D.

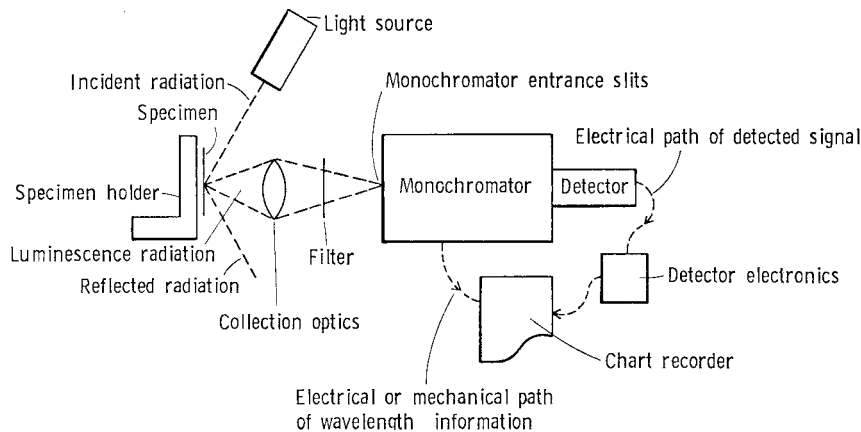


FIG. 1 Schematic Diagram of Photoluminescence Apparatus

5.2.8 *Chart Recorder*, synchronized with the monochromator drive is usually most convenient. If the chart recorder is not electrically or physically synchronized with the monochromator drive, it should have an event marker that is triggered by the monochromator to mark the position of the paper every 10 nm. The ratio of chart speed to wavelength scan speed should be such that there is a span of no more than 0.8 nm/mm (20 nm/in.).

6. Reagents

6.1 *Purity of Reagents*—All chemicals for which such specifications exist shall conform to SEMI Specifications C 1. Reagents for which SEMI specifications have not been developed shall conform to the specifications in Reagent Chemicals.⁶ Other grades may be used provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Purity of Water*—Reference to water shall be understood to mean either distilled water or deionized water having a resistivity greater than 2 MΩ·cm at 25°C, as determined by the Nonreferee Method of Test Methods D 1125.

6.3 *Etching Solution 5 + 1 + 1*—For each specimen, add 25 mL of sulfuric acid (H₂SO₄) to 5 mL of water. When this solution has cooled to approximately room temperature, add 5 mL of hydrogen peroxide (H₂O₂).

6.4 The recommended chemicals shall have the following nominal assay:

H ₂ O ₂ , %	29–32, incl
H ₂ SO ₄ , %	95–98, incl

7. Hazards

7.1 Under no circumstances look directly into the illuminator as ultraviolet radiation from arc sources or the high intensity of laser radiation can damage the eye. Take precaution also to prevent specular reflections of the source light from striking the eye.

7.2 Observe normal chemical laboratory safety precautions including the wearing of protective clothing and gloves to prevent the reagents from coming into contact with any portion of the body.

8. Preparation of Test Specimen

8.1 Etch the specimen in etching solution 5 + 1 + 1 for approximately 30 s at room temperature, rinse it several times in water, and allow it to air dry.

9. Procedure

9.1 Place the specimen in the specimen holder and adjust it so that it is illuminated approximately in the geometrical center (Note 2) and so that the surface of the wafer is at a distance from the collection optics such that the illuminated region is focused onto the entrance slits of the monochromator.

NOTE 2—The geometrical center may be taken to be at the midpoint of the perpendicular erected at the center of the crystallographic flat on one

side of the specimen if the specimen is an unbroken wafer.

9.2 Quickly scan the wavelength region to find the peak, and adjust the sensitivity of the system to yield a peak reading of from 40 to 90 % of full scale.

9.3 Slowly scan through the wavelength region of interest, recording the photoluminescent spectrum, and mark (manually or automatically) the position of every 10 nm. Scan speed is sufficiently slow if reducing the scan rate by a factor of two changes the apparent position of the peak by less than 0.5 nm.

10. Interpretation of Results

10.1 *Determination of Peak Wavelength:*

10.1.1 Draw a straight line that has the longest possible segment tangent to a side of the peak on each side of the peak. Extend these two lines until they intersect (Fig. 2).

10.1.2 Interpolate between the nearest marked divisions to find the position of the wavelength scale of this intersection. Record this wavelength as λ_{PL}, in nanometres.

10.2 *Determination of Mole Percent Phosphorus*—Find the mole percent phosphorus corresponding to λ_{PL} from Table 1.

11. Report

11.1 Report the following information:

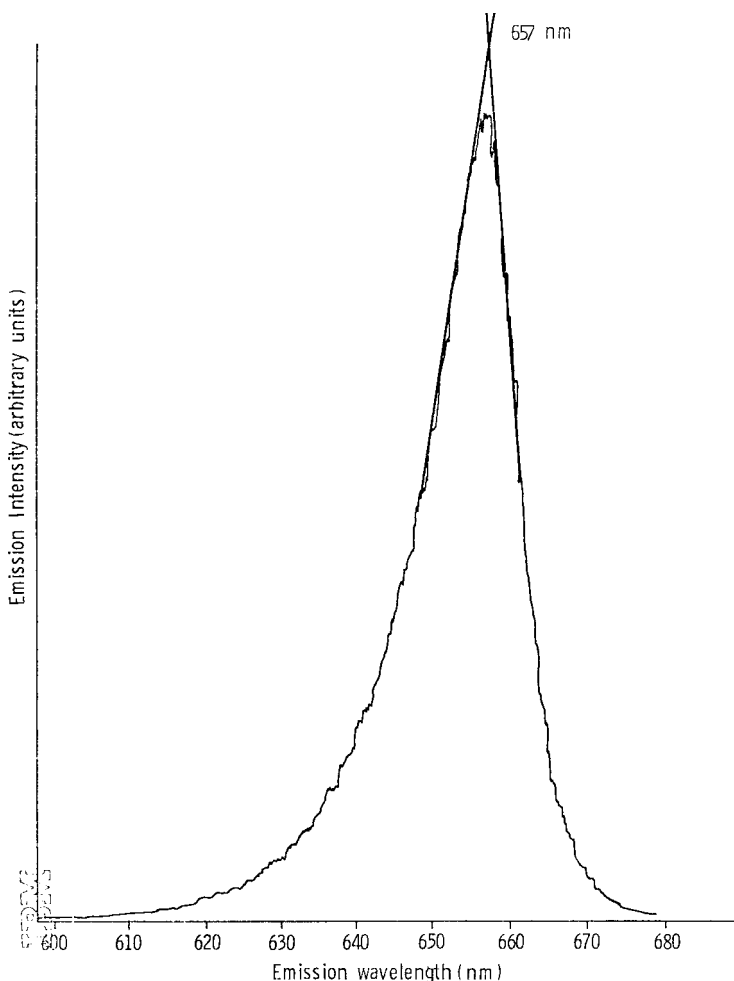


FIG. 2 Photoluminescence Response of a Gallium Arsenide Phosphide Specimen

⁶ "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

TABLE 1 Phosphorus Content of Gallium Arsenide Corresponding to λ_{PL}

λ_{PL} nm	Phosphorus, mole %	λ_{PL} nm	Phosphorus, mole %	λ_{PL} nm	Phosphorus, mole %
640	42.02	651	40.04	661	38.24
641	41.84	652	39.86	662	38.06
642	41.66	653	39.68	663	37.88
643	41.48	654	39.50	664	37.70
644	41.30	655	39.32	665	37.52
645	41.12	656	39.14	666	37.34
646	40.94	657	38.96	667	37.16
647	40.76	658	38.78	668	36.98
648	40.58	659	38.60	669	36.80
649	40.40	660	38.42	670	36.62
650	40.22				

11.1.1 Specimen identification,

11.1.2 Approximate position on the specimen at which the measurement was taken; a sketch may be used for this purpose,

11.1.3 Wavelength of peak photoluminescence, λ_{PL} ,

11.1.4 The mole percent phosphorus corresponding to λ_{PL} , and

11.1.5 For referee tests, also report the following:

11.1.5.1 Nature of the light source,

11.1.5.2 Approximate band of radiation used for illumination,

11.1.5.3 Whether the monochromator used was linear in wavelength or wavenumber, and

11.1.5.4 Spectral response type of the detector.

12. Precision

12.1 The multilaboratory precision of this test method was established by a round-robin experiment in which seven laboratories made one measurement each on each of five

samples. With the results from one laboratory excluded from the analysis because of an apparent temporary systematic error, λ_{PL} was determined with a multilaboratory precision, as defined in Practice E 177, of ± 2.26 nm (2S). This corresponds to a precision of ± 0.43 mole % phosphorus (2S) in the determination of the amount of phosphorus in the material.

12.2 In either experiments, λ_{PL} was determined with a single instrument precision, as defined in Practice E 177, of ± 1.0 nm (2S). This corresponds to a precision of ± 0.2 mole % phosphorus (2S) in the determination of the amount of phosphorus in the material.

13. Keywords

13.1 composition; gallium arsenide phosphide; mole percent phosphorus content; photoluminescence; wavelength

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 100 Barr Harbor Drive, West Conshohocken, PA 19428.

This standard is copyrighted by ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (<http://www.astm.org>).