



Designation: F 418 – 77 (Reapproved 1996)<sup>ε1</sup>

## Standard Practice for Preparation of Samples of the Constant Composition Region of Epitaxial Gallium Arsenide Phosphide for Hall Effect Measurements<sup>1</sup>

This standard is issued under the fixed designation F 418; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

<sup>ε1</sup> NOTE—Editorial changes were made throughout in February 1997.

### 1. Scope

1.1 This practice covers a procedure to be followed to free the constant composition region of epitaxially grown gallium arsenide phosphide,  $\text{GaAs}_{(1-x)}\text{P}_x$ , from the substrate and graded region on which it was grown in order to measure the electrical properties of only the constant composition region, which is typically 30 to 100  $\mu\text{m}$  thick. It also sets forth two alternative procedures to be followed to make electrical contact to the specimen.

1.2 It is intended that this practice be used in conjunction with Test Methods F 76.

1.3 The specific parameters set forth in this recommended practice are appropriate for  $\text{GaAs}_{0.62}\text{P}_{0.38}$ , but they can be applied, with changes in etch times, to material with other compositions.

1.4 This practice does not deal with making or interpreting the Hall measurement on a specimen prepared as described herein, other than to point out the existence and possible effects due to the distribution of the free carriers among the two conduction band minima.

1.5 This practice can also be followed in the preparation of specimens of the constant composition region for light absorption measurements or for mass or emission spectrometric analysis.

1.6 This practice becomes increasingly difficult to apply as specimens become thinner.

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.* For hazard statement, see Section 9 and 11.9.2.4.

### 2. Referenced Documents

#### 2.1 ASTM Standards:

D 1125 Test Methods for Electrical Conductivity and Resistivity of Water<sup>2</sup>

F 76 Test Methods for Measuring Resistivity and Hall Coefficient and Determining Hall Mobility in Single-Crystal Semiconductors<sup>3</sup>

F 358 Test Method for Wavelength of Peak Photoluminescence and the Corresponding Composition of Gallium Arsenide Phosphide Wafers<sup>3</sup>

### 3. Terminology

#### 3.1 Definitions:

3.1.1 *constant composition region*—as applied to epitaxial  $\text{GaAs}_{(1-x)}\text{P}_x$ , the layer last grown in which the composition is held fixed at about the desired value of  $x$  in mole percent phosphorus;  $x$  is typically 0.38.

3.1.2 *graded region*—as applied to epitaxial  $\text{GaAs}_{(1-x)}\text{P}_x$ , the layer first grown in which the composition is changed from  $\text{GaAs}$  to  $\text{GaAs}_{(1-x)}\text{P}_x$  during the growth of the layer. The purpose of this layer is to minimize the lattice mismatch between the  $\text{GaAs}$  substrate and the  $\text{GaAs}_{(1-x)}\text{P}_x$  layers.

3.1.3 *Hall carrier density*— $1/R_{Hc}$  [ $\text{cm}^{-3}$ ]—the reciprocal of the product of the Hall coefficient and the electronic charge, a quantity related to the charge carrier density.

### 4. Summary of Practice

4.1 In this practice (1),<sup>4</sup> a specimen is cleaved from a full wafer of  $\text{GaAs}_{(1-x)}\text{P}_x$ , the substrate is partially removed by mechanical lapping, the remainder of the substrate and the graded region are removed by chemical lapping, and the specimen is contacted either by welding or by flip chip mounting.

<sup>1</sup> This practice is under the jurisdiction of ASTM Committee F-1 on Electronics and is the direct responsibility of Subcommittee F01.15 on Gallium Arsenide.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 11.01.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 10.05.

<sup>4</sup> The boldface numbers in parentheses refer to the list of references appended to this practice.

4.2 Complete removal of substrate and graded region is assured either by timed etching in an etchant with a compositionally dependent etch rate or by measurement of composition of the etched surface in accordance with Test Method F 358.

4.3 The thickness of the final specimen is measured microscopically on a small section cleaved out of the rest of the thinned specimen.

### 5. Significance and Use

5.1 The efficiency of light-emitting diodes is known to vary with the carrier density of the starting material. This procedure provides a technique to prepare specimens in which the Hall carrier density can be measured in a region typical of that in which devices are fabricated. This quantity, which is related to the carrier density, can be used directly as a quality control parameter.

5.2 Mobility is a function of a number of parameters of a semiconductor, including ionized impurity density, compensation, and lattice defects, some or all of which may be relatable to material quality as reflected in device quality. Use of this procedure makes the measurement of the mobility of the constant composition region possible.

5.3 Since in GaAs<sub>(1-x)</sub>P<sub>x</sub> with *x* near 0.38, as is most often used for light-emitting diodes, the direct (000 or Γ) minimum and the indirect (100 or X) minima are within a few millielectronvolts in energy of each other, both are populated with current-carrying electrons. The mobility in the two bands is significantly different, and the relative population of the two is dependent upon the precise composition (*x* value), doping level, and temperature. Therefore, both Hall coefficient and Hall mobility must be interpreted with care (2,3). In particular, a measurement of Hall carrier density will not agree with a carrier density measurement on the same specimen made by capacitance-voltage techniques. Nevertheless, if the intent of measuring the carrier density of purchased or grown specimens is to find those which are optimum for diode fabrication, Hall measurements can be of value because a curve of efficiency versus Hall carrier density can be derived for the device process to be used based upon data taken on specimens prepared in accordance with this procedure.

### 6. Interferences

6.1 Incomplete removal of the graded region can lead to spurious results, since the Hall coefficient and Hall mobility of the graded layer will add to those of the constant composition region in the usual multilayer fashion (4).

6.2 Erroneous measurement of the specimen thickness will lead directly to an erroneous determination of the Hall coefficient,

since the Hall coefficient is the product of the measured Hall voltage and the thickness.

6.3 Nonuniform thickness can also lead to erroneous measurements (see 6.2).

6.4 GaAs<sub>(1-x)</sub>P<sub>x</sub> can be highly photoconductive; hence Hall measurements on this material must be made in the dark.

### 7. Apparatus

7.1 *Hot Stage* capable of heating the lapping fixture or a glass microscope slide to a temperature approximately 10°C above the melting point of the mounting wax (8.6).

7.2 *Lapping Fixture* capable of limiting the removal by lapping of material to a final thickness of 5 ± 1 mil (0.13 ± 0.03 mm).

7.3 *Glass Plate* for lapping. A sheet of plate glass ¼ by 8 by 10 in. (or 7 by 200 by 250 mm) is convenient, but the exact size is not critical.

7.4 *Chemical Laboratory Apparatus* such as beakers, graduates, and tweezers suitable for handling the reagents and solvents used in the etching and mounting steps.

7.5 *Microscope* with cross hair and calibrated eyepiece capable of measuring the specimen thickness (typically 30 to 100 μm) to ±1 % or ±0.5 μm, whichever is greater.

7.5.1 *Microscope Lamp* or other source of intense white light to illuminate the specimen by transmitted light.

7.5.2 *Microscope Slides and Microscope Cover Slides*.

7.5.3 *Diamond Scribe and Metal Straight-edge* or other apparatus for cutting microscope cover slides.

7.6 *Specimen Mounts*, each comprising an insulating ceramic substrate composed of 96 % alumina with four solderable regions composed of fired silver palladium paste or other solderable material (see Fig. 1).

7.7 *Contacting Apparatus* as described in 7.7.1 or 7.7.2.

7.7.1 *For Flip Chip Mounting*:

7.7.1.1 *Strip Heater Apparatus* capable of heating the specimen to 425 ± 25°C for a controlled time interval of a few seconds, and then to approximately 200°C for a controlled time interval of up to 1 min, in a forming gas atmosphere.

7.7.1.2 *Low-Power Microscope* suitably positioned over the strip heater to observe the specimen during the heating cycle.

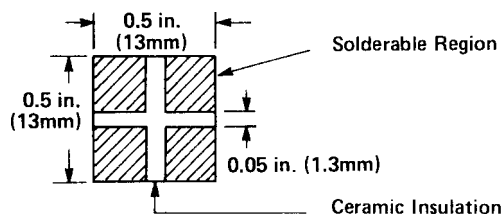
7.7.2 *For Welding*:

7.7.2.1 *Fixture* capable of supporting the specimen when waxed onto a glass slide while making pressure contact to the specimen at four points on its perimeter (see Fig. 2).

7.7.2.2 *Electrical Apparatus* capable of charging a 0.5-μF capacitor to 100 to 200 V and of discharging that capacitor through an arbitrary pair of the four contacts to the specimen (see Fig. 3).

### 8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. All reagents shall conform to the requirements



NOTE 1—All dimensions are approximate.

FIG. 1 Specimen Mount.