Designation: F 418 – 77 (Reapproved 2002)

Standard Practice for Preparation of Samples of the Constant Composition Region of Epitaxial Gallium Arsenide Phosphide for Hall Effect Measurements[1]

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 (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers a procedure to be followed to free the constant composition region of epitaxially grown gallium arsenide phosphide, $GaAs_{(1-x)}P_x$, 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 µm 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 $GaAs_{0.62}P_{0.38}$, 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:* [2]

D 1125 Test Methods for Electrical Conductivity and Resistivity of Water

F 76 Test Methods for Measuring Resistivity and Hall Coefficient and Determining Hall Mobility in Single-Crystal Semiconductors

F 358 Test Method for Wavelength of Peak Photoluminescence and the Corresponding Composition of Gallium Arsenide Phosphide Wafers

3. Terminology

3.1 *Definitions:*

3.1.1 *constant composition region*—as applied to epitaxial $GaAs_{(1-x)}P_x$, 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 $GaAs_{(1-x)}P_x$, the layer first grown in which the composition is changed from GaAs to $GaAs_{(1-x)}P_x$ during the growth of the layer. The purpose of this layer is to minimize the lattice mismatch between the GaAs substrate and the $GaAs_{(1-x)}P_x$ layers.

3.1.3 *Hall carrier density*—$1/R_He$ [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)**,[3] a specimen is cleaved from a full wafer of $GaAs_{(1-x)}P_x$, the substrate is partially removed by mechanical lapping, the remainder of the substrate and the

[1] This practice is under the jurisdiction of ASTM Committee F01 on Electronics and is the direct responsibility of Subcommittee F01.15 on Compound Semiconductors.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The boldface numbers in parentheses refer to the list of references appended to this practice.

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graded region are removed by chemical lapping, and the specimen is contacted either by welding or by flip chip mounting.

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 $GaAs_{(1-x)}P_x$ 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 $GaAs_{(1-x)}P_x$ 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*:

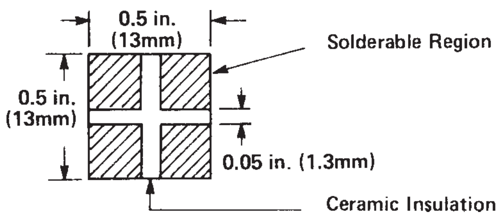
7.7.1.1 *Strip Heater Apparatus* capable of heating the specimen to 425 ± 25°C for a controlled time interval of a few seconds, and then to approximately 200°C for a controlled time interval of up to 1 min, in a forming gas atmosphere.

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).



NOTE 1—All dimensions are approximate.

FIG. 1 Specimen Mount.

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

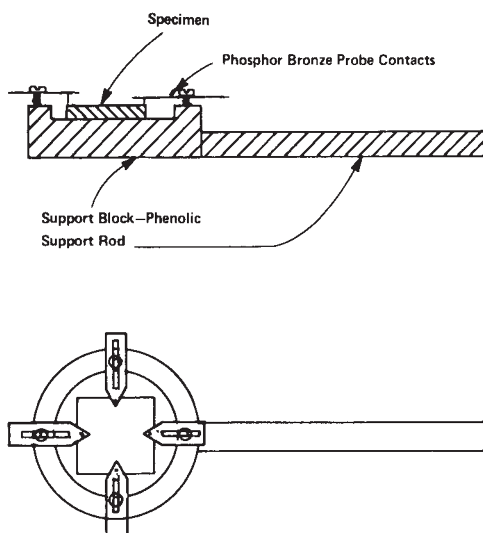


FIG. 2 Specimen Holder.

of *Reagent Chemicals*.[4] Other grades may be used provided it is first ascertained that the reagent is of sufficient purity to permit its use without lessening the accuracy of the determination.

8.2 *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.

8.3 *Acetone*, or other solvent for mounting wax (see 8.6).

8.4 *Etching Solution (1 + 2)*—Add 20 ml of ammonium hydroxide to 40 ml of hydrogen peroxide for each specimen to be thinned.

NOTE 1—Etching solutions containing hydrogen peroxide do not keep well and should be mixed fresh each day.

8.5 *For Contacting*, either*:*

8.5.1 *Solder Balls*, of nominal composition 98 % tin, 2 % tellurium, approximately 0.5 mm in diameter, or

8.5.2 *Solder Disks*, of nominal composition 98 % tin, 2 % tellurium, approximately 0.25 mm in diameter by 0.125 mm thick.

8.6 *Mounting Wax*—Glycol phthalate or other wax with a melting temperature between 50 and 100°C.

8.7 *Lapping Abrasive* commercially specified as 12-µm grade.

9. Hazards

9.1 The bases specified in this procedure are extremely hazardous. All precautions normally used with these chemicals should be observed.

9.2 The temperatures involved in wax-mounting or in soldering the specimens are high enough to cause injury, and the parts involved have sufficient specific heat to cause burns for some time after removal from the hot stage. Appropriate caution should therefore be exercised.

9.3 Since the voltage levels available from the capacitive discharge circuit can be high enough to cause personal injury, the normal safety precautions required when working with electrical apparatus should be followed.

10. Sampling

10.1 Since this procedure is destructive in nature, a sampling procedure must be used to evaluate the characteristics of a wafer or of a group of wafers. Because the most suitable sampling plan will vary considerably depending upon individual conditions, no general sampling procedure is included as a part of this test procedure. For referee purposes, a sampling plan shall be agreed upon before conducting tests.

11. Preparation of Test Specimen

11.1 If photoluminescence is to be used to determine completion of removal of the graded region, measure the composition of the original epitaxial layer in accordance with Test Method F 358.

11.2 Heat on the hot stage (7.1) the lapping fixture to approximately 10°C above the melting temperature of the mounting wax (8.6); mount the specimen on the lapping fixture with the epitaxial surface against the fixture, using mounting wax; remove the fixture from the hot stage; and cool to room temperature.

11.3 Mechanically lap the specimen to a thickness of 5 ± 1 mils (0.13± 0.03 mm) with 12-µm abrasive (8.7).

11.4 Heat the lapping fixture until the wax melts, remove the specimen from the lapping fixture, and transfer it to a glass microscope slide with the epitaxial surface against the slide; mount as described in 11.2 using mounting wax, if necessary.

11.5 Place the specimen and slide in etching solution (1 + 2) at room temperature for 30 min. At the end of this period, remove it from the solution and rinse it in running water.

11.6 Test for removal of the graded layer by either measuring the composition of the exposed surface using photoluminescence in accordance with Test Method F 358, or by looking through the specimen at a source of intense white light. The composition should agree with that found earlier (see 11.1), or red light should be transmitted uniformly through the specimen if the etching has removed all of the graded region.

11.7 If the specimen fails this test, etch for an additional 5 min in etching solution (1 + 2), rinse in running water, and retest.

11.8 Repeat the procedure of 11.7 until one of these tests indicates that the graded region has been removed.

11.9 Apply contacts to the specimen according to one of the following procedures:

11.9.1 *Flip Chip Mounting*:

11.9.1.1 Remove the specimen from the glass slide by heating on the hot plate, and cleave off a small section for thickness measurement (see 11.10).

11.9.1.2 Rinse the specimen carefully in acetone by immersion in a beaker using several changes of solvent, and pouring the solvent in and out slowly so as to disturb the specimen as little as possible. Then rinse it in the beaker in slowly flowing water. Remove it from the beaker and allow it to air dry.

[4] "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."

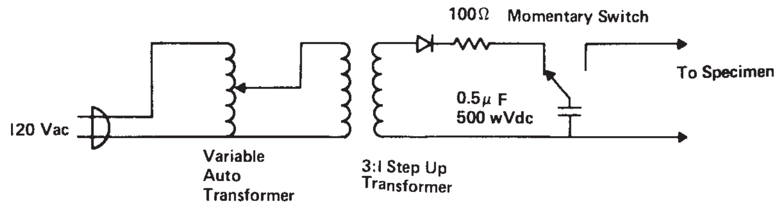


FIG. 3 Welded Contact Circuit.

11.9.1.3 Set the strip heater to reach 425 ± 25°C, allow it to cool, and place the specimen on it.

11.9.1.4 Slightly flatten four solder balls and place one on the center of each edge.

11.9.1.5 Cover the stage, allow the forming gas to flow into the covered volume at a rate of 0.1 to 0.2 litres/s for a few seconds to displace the captured air, then heat the specimen to 425 ± 25°C for 10 ± 1 s and allow it to cool to room temperature.

11.9.1.6 Test the bond strength by lifting the specimen by each ball in turn. If any bond breaks, repeat 11.9.1.5 and retest.

11.9.1.7 Reset the strip heater to reach 180°C.

11.9.1.8 Cut a piece from a microscope cover slide narrow enough to fit between two opposite pairs of solder balls, but long enough to extend beyond the specimen.

11.9.1.9 Place the specimen mount on the stage, and place the specimen on it so that one solder ball lies in each of the four solderable pads.

11.9.1.10 Place the cut section of microscope cover slide under the specimen, and a microscope cover slide on top of the specimen, and cover the stage.

11.9.1.11 Flow forming gas as in 11.9.1.5 to displace the air, and gradually heat the specimen mount and the specimen while watching the specimen under the microscope.

11.9.1.12 Observe until, at some time after the start of the cycle, one to three of the balls soften and partially collapse under the weight of the cover slide, allowing the fourth ball, which usually will not have touched the specimen mount before this, to come in contact with the mount.

11.9.1.13 Continue to heat the specimen and mount for just a few seconds longer, then terminate the heat cycle (Note 2).

11.9.1.14 Allow the specimen and specimen mount to cool to room temperature before removing them from the strip heater.

NOTE 2—This timing is critical and must be determined experimentally for the specific apparatus used. The time interval must be long enough to allow all four balls to wet the solderable pads, but not so long as to cause the first-to-melt solder ball to be completely drawn away from the semiconductor by its greater affinity for the solderable pad as compared with its affinity for the semiconductor.

11.9.2 *Welded Contact Procedure*:

11.9.2.1 Gently heat the specimen until the wax softens, and then, leaving the remainder of the specimen on the slide, cleave off and remove from the slide a small section of the specimen to be used for a thickness measurement (see 11.10).

11.9.2.2 Remove the slide with the remainder of the specimen from the hot plate and allow it to cool.

11.9.2.3 Place the slide and specimen on the specimen holder (Fig. 1), and place a pressure contact on the center of each edge of the specimen with a solder disk under each contact.

11.9.2.4 Discharge the 0.5-μF capacitor, charged to 100 V, sequentially through each pair of adjacent contacts, until each contact has served as both the anode and the cathode for this discharge. **Warning:** see 9.3.

11.9.2.5 Test the contacts for ohmic quality as described in Note 6 of Test Methods F 76. If curvature is observed in the I-V trace, repeat 11.9.2.4 with the capacitor charged to 25 V more than in the previous welding sequence.

11.9.2.6 Repeat 11.9.2.5 until curvature is not observed.

NOTE 3—If the capacitor is charged to too high a voltage, the entire specimen may be destroyed or it may be severely burned or cracked. It is therefore best to proceed with gradual increases in voltage until satisfactory contacts are achieved.

11.10 Measure the thickness of the specimen by measuring the thickness of the cleaved section using the microscope with the calibrated eyepiece.

12. Keywords

12.1 epitaxial GaAsP; gallium arsenide phosphide; Hall coefficient; Hall data; Hall mobility; resistivity; sample preparation

REFERENCES

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(**2**) Wolfe, C. M.; Holonyak, N., Jr.; Nuese, C. J.; Stillman, G. E.; Sirkis, M. D.; and Hill, D.; "Effect of the Direct-Indirect Transition on the Hall Effect in GaAs $_{(1-x)}P_x$,'' *Journal of Applied Physics,* JAPIA, Vol 37, 1966, p. 434.

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(**4**) Petritz, R. L., "Theory of an Experiment for Measuring the Mobility and Density of Carriers in the Space Charge Region of a Semiconductor Surface,'' *Physical Review,* PHRVA, Vol 110, 1958, pp. 1254–1262.

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