Designation: F 1212 – 89 (Reapproved 2002)

# Standard Test Method for Thermal Stability Testing of Gallium Arsenide Wafers[1]

This standard is issued under the fixed designation F 1212; 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 destructive test method determines whether a given sample of semi-insulating gallium arsenide (GaAs) will remain semi-insulating after exposure to the high temperatures normally required for the activation of implanted layers.

1.2 The underlying assumption is that other wafers of GaAs, whose manufacturing history was the same as the wafer from which the test sample was taken, will respond to high temperatures in like manner.

1.3 The emphasis in this test method is on simplicity and safety of apparatus, and on securing a measurement that is independent of the apparatus used.

1.4 This test method is directly applicable to uncapped and unimplanted samples of GaAs. However, users of this test method may extend it to capped or implanted samples, or both, in which case a controlled test of capped versus uncapped samples, or implanted versus unimplanted samples, is recommended.

1.5 This test method detects impurities "from the bulk" (that is, from within the GaAs wafer) that will likely affect the electrical behavior of devices formed on the surface of the wafer. This test method is not sensitive to surface impurities or process-induced impurities, except as interferences (see Interferences).

1.6 *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:* [2]

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

## 3. Terminology

3.1 *Definitions:*

3.1.1 *annealing*—The process of heating a sample of GaAs in a furnace to a specific temperature, under a reducing atmosphere and with a means to reduce the loss of arsenic via sublimation from its surface.

3.1.2 *capped annealing*—The process of placing a protective layer (usually silicon nitride or silicon dioxide) on the GaAs sample surface, thereby reducing the loss of arsenic vapor from the sample's surface during annealing. It is not described further in this test method, since the capping process introduces several variables that can affect the test results.

3.1.3 *proximity annealing*—The process of placing the GaAs sample between two similar pieces of GaAs, thereby reducing the loss of arsenic vapor from the sample's surface during annealing.

3.1.4 *thermal stability*—The ratio between the sample's apparent bulk resistivity after the annealing test, and an identical sample's bulk resistivity without annealing.

## 4. Summary of Test Method

4.1 The sample is heated in a manner similar to the heating process that an ion-implanted wafer would undergo. Then the bulk resistivity of the sample is compared to the bulk resistivity of an identical sample (control) that did not undergo heat treatment. The difference between the resistivities, if any, is a measure of the sample's sensitivity to heat treatment, or in other words its "thermal stability".

## 5. Significance and Use

5.1 Devices that involve ion implantation into a monocrystalline semi-insulating GaAs wafer are designed with the assumption that the wafer will remain semi-insulating during manufacture. However, ion implantation always damages the crystal lattice of the wafer's surface, and the damaged surface layer tends to collect impurities from the bulk of the wafer when the wafer is heated. Those impurities can become unwanted dopants: they can render the surface layer conductive, or interfere with the implanted species in various ways. The net effect in either case is a nonfunctioning device.

5.2 No spectroscopic method is sensitive enough to detect all possible bulk impurities; their presence in the wafer itself cannot be predicted in advance. This test method serves to

---

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

Current edition approved Feb. 24, 1989. Published April 1989.

[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.

Copyright © ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States.

concentrate them in the surface layer of a sample taken from one of the wafers, so that a semiquantitative estimate of their electrical behavior may be made.

5.3 It is important to understand the main assumption that underlies this test method. By its use of Test Methods F 76 to measure the stability of the sample, this test method makes the tacit assumption that the resistivity in the bulk of the heat-treated test wafer is being measured. That is true, though only indirectly. After the heat treatment of this test method, it is the test sample's surface that typically contributes the most to the measured change in resistivity. That surface resistivity, in turn, is a measure of what conductive impurities were present in the bulk, prior to the anneal test.

5.4 Measurement units of ohms per square are the theoretically correct units for measuring the resistivity after this thermal conversion test. The alternative units of Ω-cm ("bulk'' as opposed to "sheet'' resistivity) imply that the thickness of the sample's conducting layer is known. Its thickness is known before the heat treatment of this test method, but not after. Nevertheless, this test method uses the units of Ω-cm after the heat treatment, as well as ohms per square, so the" apparent bulk'' resistivities before and after the test may be compared.

5.5 This test method is suitable for use in specifications, as well as in manufacturing control, research, and development.

## 6. Interferences

6.1 The chief interference in this test method is surface contamination on the specimens being measured for resistivity. Minute amounts of, for example, dried solvent residues or fingerprints may cause a heat-treated sample to appear thermally unstable when it actually is not. For this reason, the sample cleaning steps in 11.1.6-11.1.8 must be followed scrupulously.

6.2 A less common interference arises from using too long a cool down time in 11.2.5-11.2.7. The maximum allowable cool down time is not known, but a cool down time that brings the samples' temperature to under 200°C in 30 min or less is known to remove the potential for interference.

## 7. Apparatus

7.1 *Furnace*—A means to heat the test pieces to 850°C, maintain them at that temperature in a controlled atmosphere, and then cool them to below 100°C within 30 min [3], is required, consisting of: (One embodiment of the apparatus is shown in Fig. 1.)

[3] A Rapid-Thermal-Anneal (RTA) furnace may meet the requirements of this test method, but this has not yet been demonstrated.

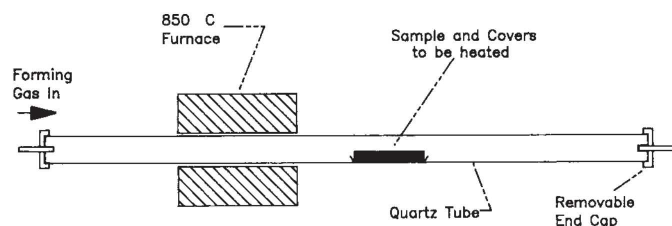


FIG. 1 Typical Heating Apparatus

7.1.1 A regulated heat source of the clamshell or tube furnace type, capable of maintaining 850 ± 3°C over a length corresponding to twice the length of the samples to be heated, when the forming gas is flowing;

7.1.2 A quartz tube more than twice the length of the total heat zone of the furnace, and of sufficient diameter to hold both the sample holder and all the samples that will be tested each time;

7.1.3 A means to slide the tube rapidly into and out of the hot zone of the furnace.

7.1.4 One removable end cap;

7.1.5 A supply of forming gas or Palladium-purified hydrogen, and of purified argon or nitrogen to one end;

7.1.6 A means to vent the exhaust gases (which are flammable, and which will also contain a trace of arsenic vapor) safely away from the work area.

7.2 *Sample Holder*—A small dish or boat which will hold the GaAs samples in the furnace is needed. It must be stable enough to hold the "stack'' of test samples with their protective cover pieces, and resistant to heat to the extent that it does not transfer impurities to the samples. Alumina or quartz is the recommended material for this sample holder. Three of the several possible types of sample holder are:

7.2.1 A simple rectangular tray, (Fig. 1) that will hold several small rectangular samples.

7.2.2 A larger tray, that will hold several half-wafer samples—if that is the size of sample that will be annealed.

7.2.3 A "leaky boat'' holder. This holder takes the form of a quartz capsule that sits within the furnace tube. Inside the capsule are (*a*) some pieces of Indium arsenide to create a few torr of local arsenic overpressure, and (*b*) the GaAs samples. The capsule is open enough to allow some of the furnace gas-flow to pass through.

7.3 *Resistivity Measurement Apparatus*—An apparatus of the type described in Test Methods E 76, sufficiently sensitive to measure the resistivity of the GaAs being tested shall be used.

7.4 *Miscellaneous*—Two clean plastic tweezers (of cleanliness suitable for the handling of semiconductor wafers) and a source of clean, submicron-filtered pressurized gas are also required for the drying step.

7.5 *Sampling Equipment*—A means to prepare samples both for the anneal furnace's holder, and later for the resistivity test kit shall be used. This apparatus normally consists of tools to scribe and cleave the GaAs wafers. It is also permissible to use a sandblaster. Refer to Test Methods F 76 for the required shapes of resistivity specimens.

## 8. Reagents and Materials

8.1 For the etching steps prior to the heating step itself, the following reagents are required, at ACS Grade [4] or better:

Concentrated sulfuric acid
30 % hydrogen peroxide
Methanol or 2-propanol

[4] ACS Grade is a specification set by the American Chemical Society of Washington, DC in "Reagent Chemicals, American Chemical Society Specifications.''

## 9. Hazards

9.1 Some of the chemical reagents and gases are extremely flammable or toxic, or both, and must be so treated at all times.

9.2 The effluent gas from the annealing furnace will contain traces of arsenic vapor, and so must be vented safely away from the work area.

## 10. Sampling

10.1 Since this test is destructive in nature, a sampling procedure must be used to evaluate the characteristics of a group of slices. The usual sampling plan is to perform this test on one sample from each end of a group of GaAs substrates. The ends in this case are the two wafers that were closest to the seed end and tail end respectively, of the GaAs single crystal when it was manufactured.

10.2 For greater confidence in the test results, duplicate samples may be used, as well as duplicate controls. It is recommended that a sample from a wafer that previously was proven to be thermally stable be annealed with the sample(s) under test, to demonstrate the validity of the results.

## 11. Procedure

11.1 *Sample Preparation*:

NOTE 1—The annealing furnace may be preheated at this time. Ensure that argon or nitrogen, but not hydrogen or forming gas, is flowing slowly through it. As shown in Fig. 1, place the quartz tube so that the end with the removable cap is as far away from the furnace as possible.

11.1.1 Cleave a sample from the GaAs wafer. This sample must be at least 2 mm wider and longer than the van der Pauw specimen that will be cut from it for the resistivity test to follow. Fig. 2 shows one possible sample configuration, one that allows for duplicate van der Pauw squares to be made from it after annealing.

11.1.2 Also prepare a pair of covers for the sample. These covers are pieces of GaAs that are the same length and width as, or larger than, the sample. Note that several test samples may be stacked together between on pair of covers. **Caution:** Handle samples and covers with clean tweezers only!

11.1.3 Wash the sample(s) in a nonreactive cleaning solution, then rinse and dry them so as to leave no stains or residue.

NOTE 2—Prepare the etch solution for the next step by mixing equal amounts of 30 % hydrogen peroxide and water, then carefully add the concentrated sulfuric acid. Allow the mix to cool to around 45°C before proceeding with the etch.

NOTE 3—Some workers modify the above procedure to add the hydrogen peroxide after the acid/water mix cools. Also, some workers prefer to prepare the etch solution 1 day ahead of time—in which case it must be kept in the dark.

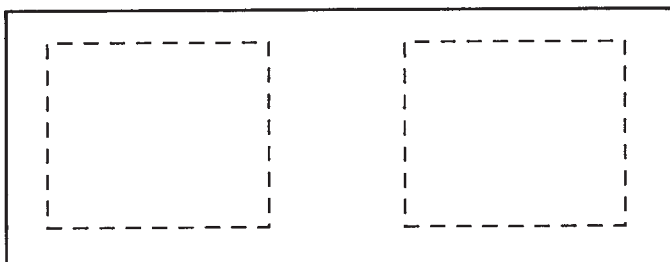

**FIG. 2 Typical GaAs Sample for Stability Test (approx 1.5 by 3.5 cm)**

11.1.4 Immerse the samples for at least two min in concentrated $H_2SO_4$. The intent is to remove any traces of organic matter prior to the etch which follows.

11.1.5 Etch the samples for at least five min in a 1:1:10 mixture of either $H_2O_2$:$H_2O$:$H_2SO_4$ or $NH_4OH$:$H_2O_2$:$H_2O$. The intent is to etch off about 1 to 2 µm of the surface, thus eliminating any interference from surface contaminants. Gentle agitation of this solution, or of the samples, is recommended.

11.1.6 Rinse the samples well with deionized (DI) water, and flowing 2-propanol (also known as isopropanol or isopropyl alcohol) or methanol, and keep them immersed in a small beaker under 2-propanol or methanol until the next step. Immersion in alcohol is the only safe storage medium for etched samples.

NOTE 4—Have the furnace tube open and have the sample holder, and GaAs cover pieces, ready to receive the samples you are preparing. One place to have them ready is just inside the open furnace tube.

NOTE 5—Ensure that during the critical washing and drying steps that follow, no other acid-cleaning or etching work is being done in the vicinity.

11.1.7 Place each individual sample in a separate small beaker, under flowing deionized water, for 1 to 1.5 min. Halfway through this washing period, turn the sample around so both sides of the sample are exposed equally to the flow of water.

11.1.8 Dry the wafers (ensure that they are thoroughly dry).

11.2 *Heat Treatment*:

11.2.1 Place the dried samples in the sample holder in the annealing apparatus. Stacks of up to eight samples can be accommodated, depending on the stability of the holder. The outer two pieces of GaAs serve as covers for the samples between them.

11.2.2 Slide the sample holder, with its stack of samples, to a point in the tube that is about halfway between the tube's cap and the end of the furnace, as shown in Fig. 1.

11.2.3 Replace the cap on the tube. Allow the purified inert gas (argon or nitrogen) to displace any residual air in the tube. This will require about 5 min, depending on tube geometry and gas flow rate.

11.2.4 Start the flow of hydrogen or forming gas. Then stop the inert gas flow. Ensure that the center of the hot zone has reached 850 ± 3°C.

NOTE 6—Forming gas refers to hydrogen that is diluted to below the explosion limit in air, using a purified inert gas such as nitrogen or argon.

11.2.5 Move the samples into the furnace, so that the samples come to sit in the center of the hot zone.

NOTE 7—Markmap edit s or notches on the tube can serve to indicate the proper positions for the samples and for the tube itself.

11.2.6 After 30 ± 5 min, slide the samples all the way out of the furnace to their starting position. Leave hydrogen or forming gas flowing for about 1 h while the samples cool.

NOTE 8—In the apparatus of Fig. 1, this is accomplished by sliding the entire quartz tube back to the illustrated starting position.

11.2.7 Once the samples are cool enough (quartz tube is cool to the touch), switch to inert-gas flow to remove them. If

the samples must remain in the furnace for a few h before the next steps, leave them in a slow flow of hydrogen or forming gas.

## 12. Evaluation of the Samples

12.1 Cleave or sandblast two van der Pauw specimensfrom each sample. No point of a specimen can come within 1 mm of the sample edge (See Fig. 2).

12.2 Measure their resistivity according to Test Methods F 76.

12.3 Also mount and measure the resistivity of two control specimens that have gone through all the steps of this test method.

## 13. Calculation

13.1 Let A1 and A2 be the resistivities of the two annealed van der Pauw specimens, and let C1 and C2 be the resistivities of the two control specimens, as determined by Test Methods F 76 and measured in Ω-cm.

13.2 Divide A1, A2, C1, and C2 by the sample thickness in centimetres to obtain the equivalent sheet resistivities A′1, A′2, C′1, and C′2 in units of ohms per square.

## 14. Report

14.1 Report the GaAs sample identification number, the annealing conditions, and the averages of:

A1 and A2
C1 and C2
A′1 and A′2
C′1 and C′2

## 15. Precision and Bias

15.1 *Single-Laboratory Test*—This test method was applied to 18 different semi-insulating GaAs wafers, by one operator using one apparatus, over a five-month period. For all but one of the wafers, the two individual thermal stability results A1 and A2 (as defined in Section 13) were within 22 % of their mean. The 95 % confidence limits are thus considered to be ± 25 % of the mean of A1 and A2. Further single laboratory tests are planned.

15.2 *Inter-Laboratory Test*—An interlaboratory test to determine the precision of this procedure is planned.

15.3 *Bias*—Since there is no accepted reference material suitable for determining the bias for the procedure in this test method for measuring thermal stability, no statement on bias is being made.

## 16. Keywords

16.1 gallium arsenide; Hal data; semi-insulating GaAs; stability; thermal anneal

*ASTM International 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 International 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, at the address shown below.*

*This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, 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 (www.astm.org).*