Designation: F 1212 – 89 (Reapproved 1996)ε1

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.

ε[1] NOTE—Keywords were added editorially in February 1997.

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:*

F 76 Test Methods for Measuring Resistivity and Hall Coefficient and Determining Hall Mobility in Single-Crystal Semiconductors[2]

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 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.

[1] This test method 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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[2] *Annual Book of ASTM Standards*, Vol 10.05.

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.)

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

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.

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

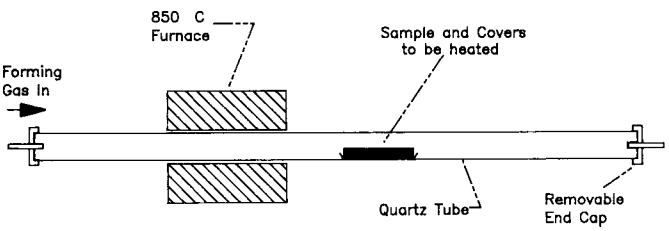

FIG. 1 Typical Heating Apparatus

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