Designation: F 1404 – 92 (Reapproved 1999)

Test Method for Crystallographic Perfection of Gallium Arsenide by Molten Potassium Hydroxide (KOH) Etch Technique[1]

This standard is issued under the fixed designation F 1404; 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 test method is used to determine whether an ingot or wafer of gallium arsenide is monocrystalline and, if so, to measure the etch pit density and to judge the nature of crystal imperfections. To the extent possible, it follows the corresponding test method for silicon, Test Method F 47. Test Method F 47 also presents the definition of many crystallographic terms, applicable to this test method.

1.2 This procedure is suitable for gallium arsenide crystals with etch pit densities between 0 and 200 000/cm^2.

1.3 Gallium arsenide, either doped or undoped, and with various electrical properties, may be evaluated by this test method. The front surface normal direction of the sample must be parallel to the <001> within ± 5° and must be suitably prepared by polishing or etching, or both. Unremoved processing damage may lead to etch pits, obscuring the quality of the bulk crystal.

1.4 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.* Specific hazard statements are given in Section 8.

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 1125 Test Methods for Electrical Conductivity and Resistivity of Water

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

F 26 Test Methods for Determining the Orientation of a Semiconductive Single Crystal

F 47 Test Method for Crystallographic Perfection of Silicon by Preferential Etch Techniques [3]

3. Summary of Test Method

3.1 The determination of the etch pit density is only meaningful for monocrystalline material. After a mechanical or chemical polish, or both, of the sample surface, the sample is etched in molten KOH. This agent preferentially attacks the gallium arsenide surface in regions of crystal imperfections, such as low angle grain boundaries, twin lamellae, precipitates, slip lines, and dislocations. The etched surface is examined microscopically to characterize these imperfections, and determine their density.

3.2 Viewed through an optical microscope, etch pits appear as dark elongated hexagonal pits. The etch pit density (EPD) is determined by counting these pits at nine different standardized locations across the sample along <011> and <001> directions. A lens micrometer or a grid installed in the microscope is used to define the sampling area. The reported EPD is obtained by averaging the EPD values in the nine counted areas.

3.2.1 The orientation of the elongated KOH etch pits may also be used to determine the crystal orientation prior to the addition of flats to gallium arsenide (GaAs) wafers or crystals.

4. Significance and Use

4.1 The use of GaAs for semiconductor devices requires a consistent atomic lattice structure. However, lattice or crystal line defects of various types and quantities are always present, and rarely homogeneously distributed. It is important to determine the mean value and the spatial distribution of the etch pit density.

5. Characteristics of Revealed Imperfections

5.1 The KOH etch of the specimen surface reveals patterns that are characteristic for several of the crystalline defects described in detail in Test Method F 47.

5.1.1 Dislocations on {100} GaAs surfaces are characterized by microscopic anisotropic six-sided etch pits. The size of the pits depends on the consistency of the etch and the etching time and will be typically 25 to 50 μm for the procedure

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

Current edition approved May 15, 1992. Published July 1992.

[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] Discontinued; see 1997 *Annual Book of ASTM Standards*, Vol 10.05.

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described in Section 9. Because the sides of these pits are not normal to the incident light, they appear dark under vertical field illumination. The use of a Nomarski microscope is optional.

5.1.2 Lineage, a precursor to a low-angle boundary, appears as a linear array of etch pits with a density greater than 25 pits/mm. For this test method, linear arrays less than 0.5 mm in length are not considered lineage. The individual etch pits are aligned end to end, or side to side. The lineage does not necessarily follow a <110> direction.

5.1.3 Slip is evidenced by a pattern of one or more straight lines of etch pits that do not necessarily touch each other. The ends of the anisotropic etch pits will be on a common line. This line of etch pits will be in a <110> direction.

5.1.4 A grain boundary appears as a grooved line of any length in which individual etch pits cannot be resolved microscopically at 200× magnification. The grooved lines may enclose an area of the etched surface or extend to the periphery of the specimen.

5.1.5 A twin boundary appears as a straight line at the intersection of a crystallographic plane (usually a <111> plane) and the etched surface under examination. Two parallel twin boundaries that are separated by only a few crystal lattice planes form a twin lamella that appears as a straight grooved line.

6. Apparatus

6.1 *Slicing Equipment*—Typically an inside diameter (ID) saw. Such a saw produces a minimum amount of cutting damage.

6.2 *Wafer Preparation Equipment*—This equipment includes lapping and polishing facilities capable of removing a minimum of 12μ m from the surface to be characterized. A polishing etch may be used in place of the wafer polisher, but will require substantially more stock removal (50 μm minimum).

6.3 *Laboratory Equipment*—Nickel crucibles and tweezers are necessary to work with molten KOH. Platinum or zirconium have also been used successfully and can be substituted for the nickel tools.

6.4 *Device*, capable of heating the crucible with the samples to 500°C.

6.5 *Microscope*, provided with 10× and 20× magnification objective lenses, a 10× magnification eye piece, a 0.5-mm pitch micrometer, and a metric stage micrometer.

7. Reagents and Materials

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Where available, all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society. [4] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity that it will not reduce the accuracy of the test.

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

7.3 *Chemical Polish*— One of the following:

7.3.1 *Polishing Etch*, (such as bromine/methanol, or sulfuric acid/hydrogen peroxide).

7.3.2 *Sodium Hypochlorite*.

7.4 *Lapping Abrasive*— Alumina, Size 5 (0.06 to 0.3 μm).

7.5 *Degreasing Chemicals*—As required according to previous process such as:

7.5.1 *1,1,1–trichloroethane (TCA 1-1-1)*,

7.5.2 *Acetone*,

7.5.3 *Isopropanol* (2-propanol), and

7.5.4 *Other Wax-Removing Solvent*.

7.6 *Defect Etch*:

7.6.1 *Potassium Hydroxide (KOH)*, anhydrous.

8. Hazards

8.1 The chemicals used in this evaluation procedure are potentially harmful and must be handled with the utmost care at all times. Read the most current copy of the Material Safety Data Sheet (MSDS) for each chemical used. Wear protective gloves and a safety mask so that molten KOH cannot contact your skin. Safety glasses must be worn at all times. Observe common laboratory safety precautions. Dispose of all chemicals properly.

9. Sample

9.1 The wafer to be measured must be free of inclusions, large grains and twins. Those would interfere with the determination of the average EPD value.

9.2 The procedure applies to crystals grown by any method, such as Liquid Encapsulated Czochralski (LEC), Horizontal Bridgman (HB), and Vertical Gradient Freeze (VGF). The sample surface must be oriented within 5° parallel to a <100> plane.

10. Procedure

10.1 Orient the ingot so that the front surface normal direction of the sample is parallel to the <001> within 5°. Either the X-ray or the optical method of Test Methods F 26 can be applied. Cut a wafer at least 0.025 in.-thick from the crystal. If the crystal has no flats, notch a {110} edge of the wafer. This will later permit locating areas for etch pit counting. LEC crystals grown on <100> result in round wafers. HB wafers are D-shaped, unless processed into round wafers.

10.2 Polish the wafer. Afterwards, the wafer must be cleaned and dried. Make sure that a minimum of 0.0015 in. has been removed from each side. If the wafer appears contaminated or not fully polished, repeat the polishing process.

10.3 If the wafer was exposed to wax during previous processes, it must be fully degreased. Immerse the wafer for five min in hot (60°C) 1,1,1–trichloroethane, followed by 5 min in cold 1,1,1–trichloroethane, followed by an acetone dip and by an isopropanol dip. Finally, immerse the wafer for five min in hot (60°C) isopropanol; remove the wafer and allow 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."