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Designation: F 110 – 00a

Standard Test Method for Thickness of Epitaxial or Diffused Layers in Silicon by the Angle Lapping and Staining Technique [1]

This standard is issued under the fixed designation F 110; 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 covers a procedure suitable for interlaboratory comparisons of layer thickness. This test method is applicable for layers of any resistivity so long as the layer differs from the silicon substrate under it either in conductivity type or in resistivity by at least one order of magnitude. The method described is destructive in nature but is more widely applicable than the alternate infrared method, Test Method F 95.

1.2 For layers with thicknesses between 1 and 25 µm, an interlaboratory precision as defined in Practice E 177, of $\pm(0.15\ T + 0.5\ \mu m)$ (3S) can be achieved where T represents thickness expressed in micrometres.

1.3 *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.* Specific hazard statements are given in Section 8.

2. Referenced Documents

2.1 *ASTM Standards:*

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods [2]

F 42 Test Methods for Conductivity Type of Extrinsic Semiconducting Materials [3]

F 95 Test Method for Thickness of Lightly Doped Silicon Epitaxial Layers on Heavily Doped Silicon Substrates Using a Dispersive Infrared Spectrophotometer [3]

2.2 *SEMI Standard:*

C 28 Specifications for Hydrofluoric Acid [4]

C 30 Specification for Hydrogen Peroxide[4]

C 35 Specification for Nitric Acid[4]

3. Terminology

3.1 *Definitions:*

3.1.1 *diffused layer*—a region of opposite conductivity type formed near the surface of a silicon crystal as a result of the introduction of impurities into the silicon crystal by means of solid state diffusion.

3.1.2 *epitaxial layer—in semiconductor technology*, a layer of semiconducting material having the same crystalline spacing as the host substrate on which it is grown.

3.1.3 *layer boundary—for the purpose of this test method*, the interface between the layer and substrate as revealed by this test method.

4. Summary of Test Method

4.1 The specimen is lapped to obtain a section inclined at a small angle to the original surface. The boundary of the layer is revealed by a chemical stain and illuminated with monochromatic light through a partially silvered optical flat to produce interference fringes. A photomicrograph of the fringe pattern and specimen is taken and the thickness is determined from the number of fringes.

5. Significance and Use

5.1 Epitaxial growth and dopant diffusion processes are used extensively in the manufacture of silicon electron devices. Measurement of the resulting layer thickness is a key factor in the control of these processes. Epitaxial and diffusion layer thickness measurements are also used to determine the suitability of silicon wafers for subsequent steps in electron-device manufacture.

5.2 The thickness as determined by various methods depends on the impurity profile in the layer. Therefore, the layer thickness determined by this test method may not be equal to the layer thickness determined in accordance with Test Method F 95 or to the depth of the electrical junction below the surface.

5.3 The test method is suitable for process control, research and development, and materials acceptance purposes.

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[1] This test method is under the jurisdiction of ASTM Committee F1 on Electronics and is the direct responsibility of Subcommittee F01.06 on Silicon Materials and Process Control.

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[2] *Annual Book of ASTM Standards*, Vol 14.02.

[3] *Annual Book of ASTM Standards*, Vol 10.05.

[4] Available from the Semiconductor Equipment and Materials International, 3081 Zanker Road, San Jose, CA 95135 (www. semi.org).

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6. Apparatus

6.1 *Angle Lapping*— A plug and plug holder which hold the specimen at an angle of 1 to 5° (Fig. 1), flat frosted-glass lapping plate, a hot plate, a diamond scriber, and tweezers are required.

6.2 *Etching*—Containers of polyethylene or polypropylene suitable for use with hydrofluoric acid. A low-power microscope is required for checking the progress of the etch in staining the specimen.

NOTE 1—Special consideration is necessary in choosing a microscope used in the staining procedure. The etches used contain hydrofluoric acid, the vapors of which will fog glass lenses. An objective lens with a long working distance is recommended to retard this effect.

6.3 *Optical Measurements*:

6.3.1 A research microscope adapted for use in this procedure or a special purpose two-beam interferometer is required. Several partially silvered optical flats or microscope slides of different transmission values, and a source of monochromatic light such as a mercury or sodium-vapor lamp are required if a microscope is to be adapted (Fig. 2).

6.3.2 A camera for photomicrography of the fringe pattern.

7. Reagents and Materials

7.1 *Purity of Reagents*—All chemicals for which specifications exist shall conform to Grade 1, SEMI specifications for those specific chemicals. Reagents for which SEMI specifications have not been developed shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, [5] where such specifications are available. Other grades may be used provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

[5] *Reagent Chemicals, American Chemical Society Specifications,* American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals,* BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary,* U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

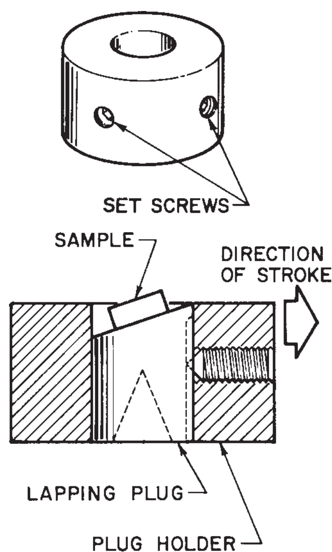

FIG. 1 Lapping Plug and Holder

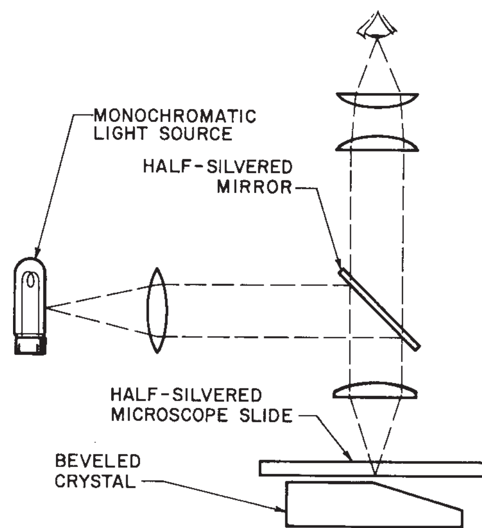

FIG. 2 Interference Microscope

7.2 *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.

7.3 *Compressed Air or Nitrogen*—Oil free and suitable for drying.

7.4 *Etch A*—Add 8 drops of nitric acid (HNO_3) to 50 mL of hydrofluoric acid (HF).

7.5 *Etch B*—Mix 30 mL of hydrofluoric acid (HF) and 15 mL of hydrogen peroxide (H_2O_2).

7.6 *Etch C*—Add to 10 mL of hydrofluoric acid (HF), 4 drops of nitric acid (HNO_3) and 2 drops of silver nitrate solution.

7.7 *Etch D*—Add 1 to 3 drops of nitric acid (HNO_3) to 20 mL of hydrofluoric acid (HF).

7.8 *Glycerin*.

7.9 *Lapping Compound*— Alumina or silicon carbide having particle sizes in the range from 6 to 12 µm.

7.10 *Mounting Wax*— Glycol phthalate or a suitable wax can be prepared from a mixture of 500 parts carnauba wax, 225 parts cherry-bark rosin, and 25 parts bee's wax, by weight.

7.11 *Polishing Compound*—Alpha-phase alumina having an average particle size of 0.3 µm.

7.12 *Silver Nitrate Solution*—Dissolve 2.0 g silver nitrate ($AgNO_3$) in water and dilute to 100 mL with water.

7.13 The recommended chemicals shall have the following nominal assays:

Hydrofluoric acid, %	49± 0.25
Hydrogen peroxide, stabilized %	30
Nitric acid, %	70 to 71

8. Safety Hazards

8.1 The acids used in this test method are extremely hazardous. All precautions normally used with these chemicals should strictly be observed. They should not be used by anyone who is not familiar with the specific precautions and first aid treatments given in the appropriate Material Safety Data sheets.