Designation: F 399 – 00a

Standard Test Method for Thickness of Heteroepitaxial or Polysilicon Layers [1]

This standard is issued under the fixed designation F 399; 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 the determination of thickness of silicon heteroepitaxial or polysilicon layers deposited under conditions such that the interface region between the deposited layer and the substrate is less than 20-nm thick. Interface regions 20 nm and thicker rarely occur, but are evidenced by a roughness in the originally smooth substrate which can be detected by a profilometer after the thickness measurement is completed and the layer has been etched away.

1.2 This test method is applicable to layers having a thickness in the range from 0.1 to 20 µm, inclusive. Other thicknesses may be measured by the same technique but the expected precisions have not been determined.

1.3 This test method is applicable to silicon layers deposited on any substrate, such as silicon dioxide, sapphire, spinel, etc., that is not significantly attacked by the recommended etch (see 8.3 and 8.4).

1.4 *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 9.

2. Referenced Documents

2.1 *ASTM Standards:*

D 5127 Guide for Ultra Pure Water Used in the Electronics and Semiconductor Industry [2]

2.2 *SEMI Standards:*

C 28 Specifications for and Guidelines for Hyrodfluoric Acid[3]

C 35 Specifications and Guideline for Nitric Acid[3]

C 36 Specifications for Phosphoric Acid[3]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *heteroepitaxial*—relating to the deposition of crystalline silicon on a substrate of a different material so that the crystallographic orientation of the deposited layer is predictably related to the substrate orientation. A typical example is the growth of <100>-oriented silicon films on single crystal <1102>-oriented sapphire or spinel.

3.1.2 *polysilicon (poly)*—polycrystalline silicon.

3.1.2.1 *Discussion*—For the purposes of this standard, include glassy or amorphous silicon layers.

3.1.3 *profilometer*—an instrument for measuring the profile of a surface by moving a stylus over the surface and recording the calibrated amplified motion of the stylus.

4. Summary of Test Method

4.1 A step is produced in the layer to be measured by etching the layer away in an area defined by a wax mask. After removing the wax, the depth of the step is measured by means of a surface profilometer.

5. Significance and Use

5.1 Epitaxially grown silicon films on sapphire, spinel, or other insulating substrates are used in the construction of integrated circuits. Polycrystalline silicon films are used as gates, field shields, conductors, contacts, etc. The thickness of such films is of key interest both for internal usage, as in process control, and for purposes of commercial exchange.

5.2 This test method may be used for process control, research, development, and materials acceptance purposes.

6. Interferences

6.1 Occasionally silicon films are produced in an apparatus that is not entirely leakproof, thereby introducing contamination into the film. The presence of a gross leak will typically produce a rough or cloudy appearance of the silicon film which is an obvious indication of the occurrence, but when only a minor leak is present, casual inspection of the film may not indicate that it is contaminated. Such contaminated films can exhibit etch rates that differ markedly from normal and are usually much slower. The end point in etching the step may then be very difficult to detect, and the step may be incompletely or excessively etched, thereby reducing the precision of the test method.

6.2 The etch rates of heavily doped films (resistivity less than 0.1Ω· cm) must be determined individually because they may differ markedly from those given in 13.5.2 for less heavily doped silicon and lead to over- or under-etching.

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[1] This test method is under the jurisdiction of ASTM Committee F01 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 11.01.

[3] Available from the Semiconductor Equipment and Materials International, 805 E Middlefield Rd., Mountain View, CA 94043, Web: www.semi.org

7. Apparatus

7.1 *Hotplate*, capable of maintaining a surface temperature of 100 ± 10°C.

7.2 *Surface Profilometer or Step Gage*, capable of resolving a 20-nm step and having a high magnification of at least 100 000 and a low magnification of not over 10 000. The instrument shall be supplied with step standards for calibration purposes.

NOTE 1—A magnification of 50 000 indicates that the stylus motion is 1/50 000 of the size of the magnified trace on the scale.

7.3 *Bath or Hotplate with Exhaust Hood*, suitable for use with trichloroethylene at a temperature of 50 to 60°C.

7.4 *Beakers*, or other containers, of polypropylene, polyethylene or fluorocarbon, suitable for use with hydrofluoric acid or hot trichlorethylene.

7.5 *Microscope Illuminator*, capable of producing a collimated high-intensity light beam of at least 20-mm diameter for observation of the chemical etch process.

7.6 *Lint-Free Absorbent Tissue*, for drying the specimen.

7.7 *Scriber*, to break the specimen in half.

7.8 *Metallization Apparatus*, for preparing secondary step height standards.

7.9 *Filter*, having a 0.45-µm or smaller pore size for filtering compressed air or nitrogen.

8. Reagents and Materials

8.1 *Purity of Reagents*—All chemicals for which specifications exist shall conform to the assay and to impurity levels of Grade 1 SEMI specifications for the appropriate chemical. 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 [4], 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.

8.2 *Purity of Water*— Reference to water shall be understood to mean either deionized water meeting the requirements of Grade E.1 of Guide D 5127.

8.3 *Chemical Etch A*— Mix by volume 3 mL of hydrofluoric acid (HF) and 247 mL of nitric acid (HNO_3).

8.4 *Chemical Etch B*— Mix by volume 20 mL of hydrofluoric acid (HF) and 230 mL of nitric acid (HNO_3).

8.5 *Compressed Air or Nitrogen*, oil-free and suitable for drying. Moisture content of 10 ppm or less is acceptable.

8.6 *Dewaxing Agent (Water-based)*, used in conjunction with the masking wax specified in 8.7.

8.7 *Masking Wax* resistant to Chemical Etches A and B; softening range from 60 to 90°C; and completely soluble in the dewaxing agent of 8.6.

8.8 *Phosphoric Acid*, (H_3PO_4).

[4] *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.

9. Safety Hazards

9.1 The chemicals used in this test method are potentially harmful and must be handled with the utmost care at all times.

9.2 **Warning**—Hydrofluoric acid solutions are particularly hazardous. **Warning:** They should not be used by anyone who is not familiar with the specific preventive measures and first aid treatments given in the appropriate Material Safety Data Sheet.

10. Sampling

10.1 The thickness of polycrystalline or heteroepitaxial layers may vary across a wafer or among wafers of a particular batch. Initial material sampling of a batch should be such that the details of the variation are revealed; statistically selected samples may then be taken to characterize subsequent wafers. For materials acceptance purposes, a sampling plan should be agreed upon by the parties concerned.

10.2 Sampling on an individual specimen is discussed in 13.12.

11. Specimen Preparation

11.1 Chemically treat the specimen to remove layers that may be present on the surface of the silicon as a result of processing; for example, use hot (180 ± 5°C) phosphoric acid to remove a silicon nitride or an aluminum oxide layer, or hydrofluoric acid to remove a silicon dioxide layer.

12. Calibration

12.1 Calibrate the profilometer using the step standards supplied by the manufacturer and adjust it if necessary to bring it to within the manufacturer's specifications.

13. Procedure

13.1 Scribe and break the specimen to be measured into two halves; retain one half for later verification if desired and use the other half for the test specimen.

13.2 Remove dirt or fingermarks from the specimen by swabbing with trichloroethylene and then place the specimen on the hotplate, set to 100 ± 10°C, with the layer to be measured up.

13.3 Cover the entire specimen except for a 5 to 10-mm wide strip parallel to the straight edge, at the break, with masking wax. Allow the wax to flow so that it is smooth and uniform.

13.4 Cool the specimen to room temperature.

13.5 Etch the specimen in the following manner using Chemical Etch A for specimens having 0.1 to 2.5-µm thick heteroepitaxial or polysilicon films or Chemical Etch B for specimens having 2.0 to 20-µm thick films:

13.5.1 Immerse the specimen in 20 to 30 mL of the appropriate etch in a plastic beaker at 25 ± 3°C.

13.5.2 Gently swirl the solution until all the exposed silicon has been etched off. Observe under the light from a microscope illuminator.

NOTE 2—The silicon is etched away when the etched area assumes the uniform color of the substrate. Chemical Etch A attacks polysilicon at the rate of 0.2 to 0.3 µm/min and silicon dioxide at the rate of 14 to 16 nm/min. Chemical Etch B attacks polysilicon at a rate of about 2.2µ m/min and silicon dioxide at the rate of about 90 nm/min (see 6.2). It is advisable