Designation: F 1049 – 00

Standard Practice for Shallow Etch Pit Detection on Silicon Wafers[1]

This standard is issued under the fixed designation F 1049; 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 practice is used to detect shallow etch pits, which may be related to the level of metallic impurities near the surface of silicon epitaxial or polished wafers.

1.2 This practice is not recommended for use in defect density evaluations, but as a subjective means of estimating defect densities and distributions on the surface of a polished or epitaxial wafer.

1.3 Silicon crystals doped either *p*- or *n*-type and with resistivities as low as 0.005 Ω·cm may be evaluated. This practice is applicable for silicon wafers grown in either a (111) or (100) crystal orientation.

1.4 This practice utilizes a thermal oxidation process followed by a chemical preferential etchant to create and then delineate shallow etch pits.

1.5 The values stated in acceptable metric units are to be regarded as the standard. The values in parentheses are for information only.

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.* Specific hazard statements are given in Section 9.

2. Referenced Documents

2.1 *ASTM Standards:*

D 5127 Guide for Electronic Grade Water[2]

F 154 Guide for Identification of Structures and Contaminants Seen on Specular Silicon Surfaces[3]

F 1725 Guide for Analysis of Crystallographic Perfection of Silicon Ingots[3]

F 1727 Practice for Dection of Oxidation Induced Defects in Polished Silicon Wafers[3]

F 1809 Guide for Selection and Use of Etching Solutions to Delineate Structural Defects in Silicon[3]

F 1810 Test Method for Counting Preferentially Etched or Decorated Surface Defects in Silicon Wafers[3]

2.2 *SEMI Standards:*

SEMI C 1 Specifications for Reagents[4]

M 17 Specification for a Universal Wafer Grid[4]

3. Terminology

3.1 *Definitions:*

3.1.1 *haze—on a semiconductor wafer*, non—localized light scattering resulting from surface topography (microroughness) or from dense concentrations of surface or near-surface imperfections. See also **laser light scattering event.**

3.1.1.1 *Discussion*—Haze due to the existence of a collection of imperfections of the type that result in haze cannot be readily distinguished by the eye or other optical detection system without magnification. In a scanning surface inspection system, haze and laser-light scattering events comprise the laser surface scanner signal due to light scattering from a wafer surface.

3.1.2 *shallow etch pits*—etch pits that are small and shallow in depth under high magnification, > 200×.

3.1.2.1 *Discussion*—Shallow etch pits on silicon wafers are shown in Guide F 154 .

3.1.3 *saucer pits*—same as **shallow etch pits**, see 3.1.2.

4. Summary of Practice

4.1 Silicon wafers, either epitaxial or polished, are thermally oxidized and preferential etched. This will reveal small etch pits, shallow in depth, when observed through an interference contrast microscope. The distribution of the etch pits on the surface of the wafer are determined by illuminating the wafer with a high intensity lamp.

5. Significance and Use

5.1 High levels of etch pits are reported[5] to indicate metallic contamination that is detrimental to wafer processing. This can be deduced from the density of etch pits on the surface of the wafer.

5.2 This practice is appropriate for process control, and research and development applications. Because its reproducibility has not been established by interlaboratory test, it is not recommended for use in materials acceptance unless the parties

---

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

Current edition approved June 10, 2000. Published August 2000. Originally published as F 1049 – 87. Last previous edition F 1049 – 95.

[2] *Annual Book of ASTM Standards*, Vol 11.01.

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

[4] Available from Semiconductor Equipment and Materials International, 805 E. Middlefield Rd., Mountain View, CA 94043.

[5] Pearce, C. W., and McMahon, R. G., "Role of Metallic Contamination in the Formation of 'Saucer' Pit Defects in Epitaxial Silicon," *Journal of Vacuum Science and Technology*, Vol 14, No. 1, 1977, p. 40.

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

to the test have conducted correlation experiments to establish the precision to be expected.

6. Interferences

6.1 Etch artifacts are the primary cause of difficulty in identifying shallow etch pits. Etch artifacts are generated in various ways such as gas bubble formation during etching, improperly cleaned surface prior to etching, or insufficient etch solution volume.

6.2 Excessive silicon staining (very dark color) during the preferential etching may obscure or prevent the development of shallow etch pits on heavily doped p-type silicon material ($<0.2\Omega \cdot$ cm).[6]

NOTE 1—Light staining will not affect subsequent defect etch results. However, heavy stains are undesirable.

7. Apparatus

7.1 *Narrow Beam Light Source*—Tungsten filament with a concentrated beam intensity greater than 16 klx (1500 fc) and a beam diameter of 20 to 40 mm (0.8 to 1.6 in.) at a position 100 mm (4 in.) from the light-source housing. The light beam shall not be collimated and shall be capable of forming an image of the bulb filament at the lamp focus length.

NOTE 2—Some standard microscope illuminators meet these requirements.

7.2 *Hydrofluoric Acid-Proof Chemical Laboratory Apparatus*—Fluorocarbon, polyethylene, or polypropylene beakers, graduates, tweezers, eye protection, apron, gloves, and protective sleeves.

7.3 *Wafer Holders*—HF acid-proof wafer carriers which hold wafers. These are necessary if more than one wafer is to be etched at a time.

7.4 *Optical Microscope*—Equipped with interference contrast attachment. The eyepiece and objective lens in combination shall give 200 to 1000× magnification.

NOTE 3—Nomarski differential interference contrast is an example of interference contrast.

7.4.1 *Stage Micrometer*—, with divisions of 0.002 mm or finer, if an estimate of the shallow etch pit density is to be made.

7.5 *Acid Sink*—A fume hood and facilities for disposing of acids and their vapors.

7.6 *Spin Dryer*—Used to dry the wafers. This is not necessary, but provides a surface free of residue artifacts.

8. Reagents and Materials

8.1 *Purity of Reagents*—All chemicals for which such specifications exist shall conform to SEMI Specifications C 1. Reagents for which SEMI specifications have not been developed shall conform to the specifications of the Committee of Analytical Reagents of the American Chemical Society.[7] 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 distilled water, or deionized water having a resistivity equal to or greater than Type II water as defined by Guide D 5127.

8.3 *Schimmel Etch for (100) and (111) Surfaces.* [8]

8.3.1 *Chromic Acid Solution*—Make a 0.75 M solution of CrO_3 to a 1-L glass volumetric flask and add sufficient water to make a solution volume of 1 L, or 1000 mL. The solution may be stored up to 6 months in either clean glass, TFE-fluorocarbon, polyethylene, or polypropylene bottles.

8.3.2 *For Test Specimens with Resistivity Greater Than $0.2\Omega \cdot$ cm n- or p-Type*—Immediately before using, add 2 parts HF to 1 part chromic acid solution (see 8.3.1) by volume. Prepare and mix in HF-proof beakers (see 7.2).

8.3.3 *For Test Specimens with Resistivity Less Than 0.2 $\Omega \cdot$cm n- or p-Type*—Immediately before using, add 2 parts HF to 1 part chromic acid solution (see 8.3.1) and 1.5 parts water by volume. Prepare and mix in HF-proof beakers (see 7.2).

8.3.4 The specified chemicals shall have the following nominal assay:

Chemical	Assay, %
Chromium trioxide	>98.0
Hydrofluoric acid, concentrated	49 ± 0.25

9. Safety Hazards

9.1 The chemicals used in this evaluation procedure are potentially harmful and must be handled in an acid exhaust fume hood, with utmost care at all times.

9.2 **Warning:** Hydrofluoric acid solutions are particularly hazardous. 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.

9.3 Chromic acid, that is contained in the defect etch solutions, should not be released into drains that lead directly to domestic sewers. Chromates are an extreme eco-hazard and must be first treated by reduction to the trivalent form. Chromic acid is a strong oxidizing agent and should not be allowed to come into contact with organic solvents or other easily oxidized materials.

10. Sampling

10.1 Select wafers to represent the lot to be tested as specified in producer-consumer agreements.

11. Specimen Preparation

11.1 In most instances this test method may be used for polished or epitaxial wafers as they are received, but if

[6] Schimmel, D. G., and Elkind, M. J., "An Examination of the Chemical Staining of Silicon," *Journal of the Electrochemical Society*, No. 1, 1978, p. 152.

[7] 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.

[8] Schimmel, D. G., "Defect Etch for ⟨100⟩ Silicon Evaluation," *Journal of the Electrochemical Society*, Vol 126, No. 3, 1979, p. 479.

cleaning is required, the parties using this test method must establish a uniform cleaning procedure prior to etching.

12. Procedure

12.1 Use the furnace cycle shown in Table 1 and execute the thermal oxidation and preferential etching as described in Guides F 1725, F 1809, Practice F 1727, and Test Method F 1810.

NOTE 4—Large diameter furnaces may have difficulty in duplication of this process. Rapid thermal processing is an acceptable alternative.

13. Evaluation

13.1 View the wafer at 1× magnification under the narrow beam light source (see 7.1) in a dark hood. If haze is observed, further examine the etched wafer under a minimum of 200× magnification to establish if the haze is due to shallow etch pits. If no shallow etch pits are seen, record the wafer as being free of shallow etch pits.

NOTE 5—The percentage of the wafer covered may be determined with the use of the universal wafer grid specified in SEMI M17 that divides the wafer into 1000-area elements. If the universal wafer grid is used, record the size of the edge exclusion or the fixed quality area used. A1.6-mm peripheral ring on a 125-mm diameter wafer represents 5 % of the wafer area.

13.2 With the use of the following table, determine the level of haze present on the surface.

Level	% Area of Wafer
A	0–5
B	5–25
C	25–75
D	75–100

13.3 *Optional Estimation of Shallow Etch Pit Density*—If it is desired to estimate the shallow etch pit density, examine the wafer under magnification in the range from 200 to 1000× to distinguish between etching artifacts and shallow etch pits.

13.3.1 Place the wafer on the microscope stage.

13.3.2 Position the specimen so as to view the area of interest on the etched wafer surface. Choose the area to be viewed to include a high density of haze.

TABLE 1 Shallow Pit Oxidation Procedure

Shallow Pit Oxidation	
Push	
Ambient	1 % O_2/99 % N_2
Temperature	950°C
Rate	60 cm/min
Oxidation	
Ambient	1 % O_2/99 % N_2
Temperature	950°C
Time	7 min
Pull	
Ambient	1 % O_2/99 % N_2
Temperature	950°C
Rate	60 cm/min

13.3.3 Adjust the magnification so that up to 100 shallow etch pits are seen in the field of view. If more than 100 shallow etch pits are in the field of view at maximum magnification, report the shallow etch pit density as “Too high to count”.

13.3.4 Calculate the area of the field of view from its diameter as determined to ±1 µm with a stage micrometer.

13.3.5 Count and record the number of shallow etch pits in the field of view. Count as one defect, those defects that converge or overlap except when the etch pits are well defined and are individually distinguishable.

13.3.6 Determine the estimate of the shallow etch-pit density by dividing the number of etch pits counted by the area of the field of view.

13.3.7 If more than one area is counted, compute the average shallow etch-pit density for the wafer by dividing the sum of the shallow etch-pit densities estimated by the total number of counting positions.

14. Report

14.1 Report the following information:

14.1.1 Date of test, laboratory and operator identification,

14.1.2 Identification of wafer lot,

14.1.3 Identification of the test wafer(s) (including conductivity type, orientation, diameter, growth method, and back surface condition),

14.1.4 Level of haze (see 13.2) for each wafer tested, and

14.1.5 A diagram showing the location and distribution of areas of high shallow etch-pit density, and, if estimates of shallow etch-pit density are made, locations of the count positions.

14.2 If the shallow etch-pit density was estimated on one or more wafers, also report the following information of each wafer tested:

14.2.1 Magnification used in the test,

14.2.2 Average estimated shallow etch-pit density, and

14.2.3 Maximum and minimum measured shallow etch-pit density, if more than one count position was employed.

15. Precision and Bias

15.1 *Precision*—Because this practice is intended for use only for qualitative estimates of the area of a wafer covered by haze due to shallow etch pits and the shallow etch-pit density, no interlaboratory evaluation of this practice has been conducted for the purposes of determining the expected repeatability or reproducibility.

15.2 *Bias*—No standards exist against which the bias of this practice can be evaluated.

16. Keywords

16.1 epitaxial; oxidation; preferential etch; saucer pit; shallow etch pit; silicon

The American Society for Testing and Materials 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 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, 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).