Designation: F 1725 – 97

AMERICAN SOCIETY FOR TESTING AND MATERIALS
100 Barr Harbor Dr., West Conshohocken, PA 19428
Reprinted from the Annual Book of ASTM Standards. Copyright ASTM

Standard Guide for Analysis of Crystallographic Perfection of Silicon Ingots [1]

This standard is issued under the fixed designation F 1725; 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 covers the analysis of the crystallographic perfection in silicon ingots. The steps described are sample preparation, etching solution selection and use, defect identification, and defect counting.

1.2 This practice is suitable for use if evaluating silicon grown in either [111] or [100] direction and doped either *p* or *n* type with resistivity greater than 0.005 Ωcm.

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.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 5127 Guide for Electronic Grade Water [2]

F 26 Test Method for Determining the Orientation of a Semiconductor Single Crystal [3]

F 523 Practice for Unaided Visual Inspection of Polished Silicon Wafers [3]

F 1241 Terminology of Silicon Technology [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 [2]

2.2 *SEMI Specifications:*

SEMI C-1 Specification for Reagents[4].

3. Terminology

3.1 Defect-related terminology may be found in Terminology F 1241.

4. Summary of Practice

4.1 The end portion of the silicon crystal, which solidified last, may contain dislocations or other defects such as slip. The portion containing the defects is removed by sawing the crystal. A specimen wafer from the end of the remaining ingot is obtained with a second cut.

4.2 This wafer is mechanically lapped, chemically polished, and then etched in a preferential defect etching solution.

4.3 The etched surface is examined under bright light illumination and examined microscopically to count and classify the imperfections highlighted by the preferential defect etching solution.

5. Significance and Use

5.1 The use of silicon wafers in many semiconductor devices requires a consistent atomic lattice structure. Crystal defects disturb local lattice energy conditions that are the basis for semiconductor behavior. These defects have distinct effects on essential semiconductor device-manufacturing processes such as alloying and diffusion.

5.2 This practice along with the referenced standards may be used for process control, research and development, and materials' acceptance purposes.

6. Apparatus

6.1 *Slicing Equipment*, suitable for removing wafers of varied thickness from ingots.

6.2 *Lapping or Grinding Equipment* (optional), suitable for removing saw damage.

6.3 *Laboratory Equipment*, suitable for use with hydrofluoric acid (fluorocarbon, polyethylene, or prolypropylene beakers, graduates, pipets, and nonmetallic wafer pickup tools).

6.4 *Acid Sink*, in a fume hood and facilities for disposing of acids and their vapors.

6.5 *Personnel Safety Equipment*, for handling acids, such as gloves, safety glasses, face shield, and gown.

7. Reagents and Materials

7.1 All chemicals for which such specifications exist shall conform to SEMI specifications C–1.

7.2 Reference to water shall be understood to mean either distilled water or deionized water, meeting the requirements of Type I water as defined by Guide D 5127.

7.3 A variety of etching solutions exist. They have been found to produce satisfactory results as illustrated in Table 1

7.4 An aqueous, nonionic surfactant detergent solution.

8. Hazards

8.1 The chemicals used in polishing etches are potentially harmful and must be handled in a chemical exhaust fume hood, with the utmost care.

---

[1] This guide 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, 1997. Published August 1997.

[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.

TABLE 1 Volume Proportions

Formulation	Nitric Acid, (Assay: > 99.7 %)	Hydrofluoric Acid, (Assay: 49 ± 0.25 %)	Acetic Acid (Assay: 70 to 71 %)
A	6	1	1
B	5	3	3
C	5	10	14
D	5	1	2

8.2 Hydrofluoric acid solutions are particularly hazardous and the specific preventive measures must be strictly observed.

8.3 Safety or protective gear should be worn while handling these acid solutions or their components. Safety requirements vary, but the essentials are: plastic gloves, safety glasses, face shield, acid gown, and shoe covers.

9. Procedure

9.1 *Sample selection*—Take the sample for evaluation from the crystal close to the discarded crystal portion found at the last of the solidified crystal. Other samples may be specified in producer-consumer relationships.

NOTE 1—Determination of the most logical point of sample selection may be established by inspection of the bottom taper of the crystal. If the crystal has a complete bottom taper, then the sample should be obtained from the last point of a full crystal diameter. If the crystal has lost zero dislocation growth before the formation of a tapered bottom, obtain the sample 1–crystal diameter above the point of lost zero dislocation structure.

9.2 Orient the ingot to be sliced with either the x-ray or optical method of Test Method F 26 so that the surface to be exposed is within 5° of the desired plane. Slice a wafer, 0.5 to 2-mm thick, from the crystal. Identify ingot growth lines on the sample by a mark or a ground flat for future reference in counting defects.

NOTE 2—Defects observed by preferential etching may be increasingly distorted as misalignment from the major crystallographic plane increases.

9.3 Remove the residual saw damage by mechanical lapping and chemical polishing or by chemical polishing alone.

9.3.1 Wash the as-cut or lapped wafer in a nonionic surfactant detergent solution and rinse thoroughly in water. Drying may be hastened by use of a lint-free paper towel. The surface must be uniformly matte in appearance with no scratches, wax, dirt or water stains.

9.3.2 *Chemical Polish*:

9.3.2.1 Place the sample in the bottom of a hydrofluoric acid resistant beaker with the side to be inspected facing upward. The beaker diameter need only be larger than the wafer diameter.

9.3.2.2 Pour the room temperature chemical polish etch (from 7.3) until the surface of the sample is covered with about 1 cm of solution.

9.3.2.3 Agitate during etching to reduce bubble formation and surface artifacts.

NOTE 3—The polish etch procedure in 9.3 describes a small sample size facility. More sophisticated facilities are used in commercial environments.

9.3.2.4 Rapidly dilute the etching solution with water and flush the solution from the beaker after the sample wafer develops mirror-polished surfaces.

NOTE 4—Staining may occur on heavily doped, *p*-type material with resistivity of ≤ 0.1 Ωcm, during dilution of the polishing etch. Rapid transfer to fresh polish etch for less than 30 s additional etching, followed by rapid flushing of the polish etch can reduce silicon staining. If necessary, dilution of the polishing etch with nitric acid flushing with water is also effective in reducing stains.

9.3.2.5 Dry with filtered air or nitrogen after thorough rinsing of the polish-etched sample.

9.4 Select an appropriate etching solution to decorate the defects.

9.4.1 Refer to Guide F 1809 to select an appropriate etching solution.

9.4.2 Etch the samples to remove an amount of silicon from the surface being evaluate, as agreed upon between the parties to the test. If no removal amount is defined, remove 5 to 15 µm of silicon from the surface being evaluated.

9.5 Evaluate the preferentially etched sample in two stages, macroscopic and microscopic.

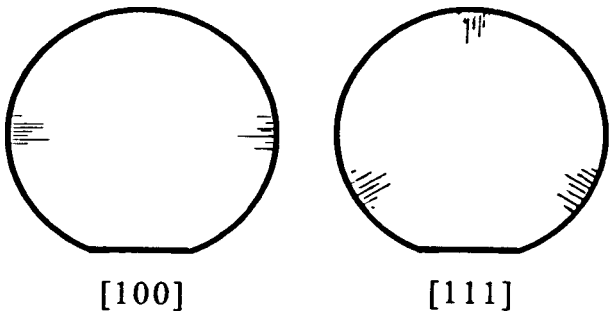
9.5.1 *Macroscopic Inspection*—Use a high intensity light to inspect the full sample surface. The characteristic patterns of slip defects as shown in Fig. 1 are easily identified in macroscopic inspection. If evidence exists of mechanically or handling induced damage or contamination, repeat 9.3 to 9.5.

NOTE 5—Use a high intensity light, such as that specified in Practice F 523, to inspect the full sample surface.

9.5.2 *Microscopic Defect Counting*—Count and report the density of observed defects using Test Method F 1810.

10. Keywords

10.1 dislocation; grain boundaries; ingot; polycrystalline imperfections; preferential etch; silicon; slip



NOTE 1—The orientation of the wafer defines the locations and direction of the line defects.

FIG. 1 Slip Defects as seen with Macroscopic High-Intensity Light Inspection.

F 1725

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, 100 Barr Harbor Drive, West Conshohocken, PA 19428.