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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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# Standard Test Method for Measuring the Depth of Crystal Damage of a Mechanically Worked Silicon Slice Surface by Angle Polishing and Defect Etching [1]

This standard is issued under the fixed designation F 950; 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 describes a technique to measure the depth of damage, on or beneath the surface of silicon wafers prior to any heat treatment of the wafer. Such damage results from mechanical surface treatments such as sawing, lapping, grinding, sandblasting, and shot peening.

1.2 The principal application of this test method is for determining the depth of damage of the non-polished back surface that has had intentionally added work damage.

1.3 The measurement is destructive since a specimen is prepared from a section of a silicon wafer.

1.4 Depth of damage can be measured in the range of 5.0 to 200 µm using this method.

1.5 This test method is intended for use in process control where each individual location is resposible to determine the internal repeatability to its satisfaction.

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 1193 Specification for Reagent Water [2]

E 122 Practice for Choice of Sample Size to Estimate a Measure of Quality for a Lot or Process [3]

F 532 Test Methods for Measuring Width of Defects in Optical Surfaces, Using Nomarski Differential Microscopy [4]

F 672 Test Method for Measuring Resistivity Profiles Perpendicular to the Surface of a Silicon Wafer Using a Spreading Resistance Probe [5]

2.2 *SEMI Standard:*

C1 Specifications for Reagents [6]

## 3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 damage—a defect of the crystal lattice of a single crystal silicon specimen in the form of irreversible deformation. The damage is the result of mechanical surface treatments such as sawing, lapping, grinding, sandblasting, and shot peening at room temperature without subsequent heat treatments.

3.1.2 damage-free polishing—a method of preparing a surface of a silicon specimen without creating any mechanical damage detectable by this method.

3.1.3 bevel angle (α)—the smaller of the angles between the wafer surface and the section plane. (See Fig. 1.)

3.1.4 damage depth ($T_z$)—the maximum thickness of the damage region. The damage is revealed by a preferential etch that removes silicon in the region of the deformation. Preferential etching occurs because the chemical potential in the region of the deformation is changed by the stress fields associated with the deformation. The depth of damage is expressed in micrometers.

## 4. Summary of Test Method

4.1 A silicon specimen is coated with silicon nitride by a low-pressure plasma method to a minimum thickness of 1 µm. The specimen is then beveled at a small angle by a polishing technique that produces no additional mechanical damage. The bevel angle is measured. The beveled specimen is etched to reveal the damage. The length of the damage region is measured from the beveled edge on the beveled section. The depth of damage is then calculated from the relationship between the measured damage length and the sine of the bevel angle.

## 5. Significance and Use

5.1 This test method provides a means for measuring the

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

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

[4] Discontinued; see 1993 *Annual Book of ASTM Standards*, Vol 06.01.

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

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

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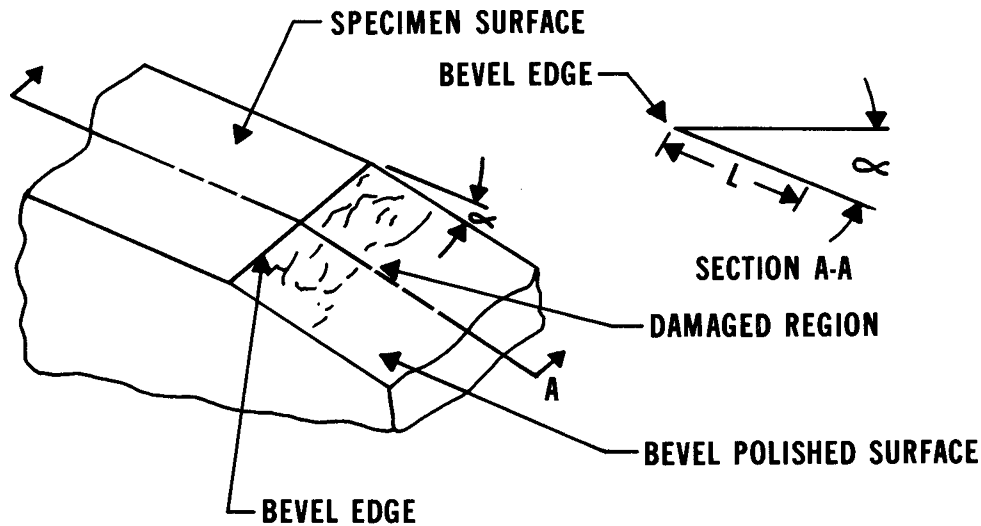



NOTE 1—A 1-μm thick LPCVD nitride film is deposited on the specimen surface prior to beveling.

**FIG. 1 Bevel Polished Specimen**

depth of mechanical damage in silicon wafers in the range from 5 to 200 μm.

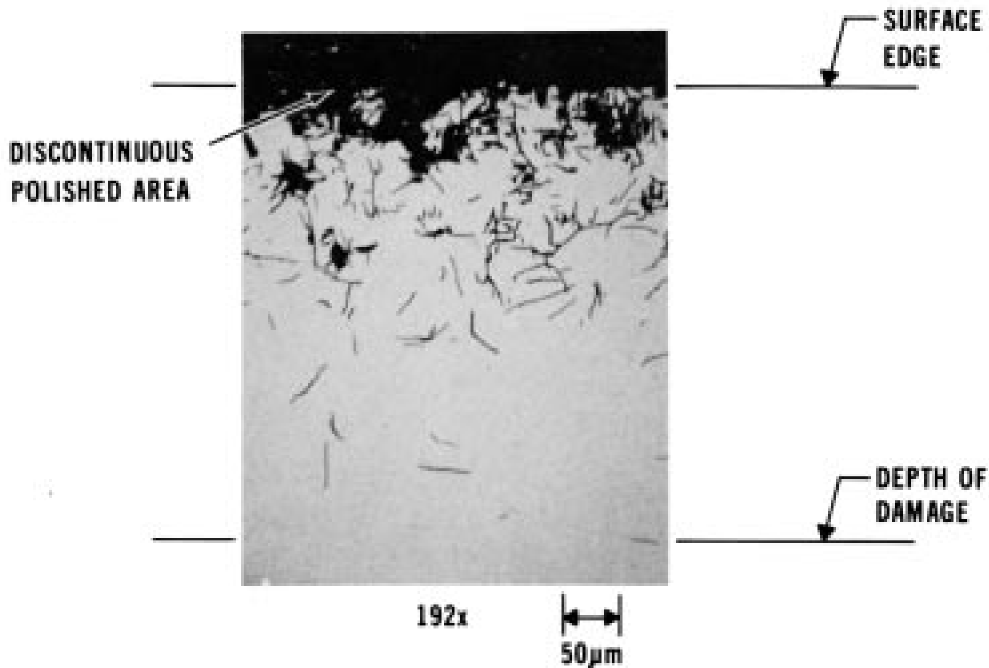
5.2 This test method can be used for process control or research and development purposes. It is not recommended for use in material acceptance.

## 6. Interferences

6.1 *Choice of Bevel Angle*—A bevel angle must be used such that a magnification of the depth of damage is at least a factor of 5, or the damage may not be detected. Bevel angles less than 5°44 min are not recommended because of difficulty in determining the surface edge due to the uneven surface topography generated by the damage. (See Fig. 2.) Table 1 lists the relationship of bevel angle (α), bevel length (*L*), and damage depth ($T_z$).

6.1.1 Even with a 5°44 min angle, there may be difficulty in determining the bevel edge for surface damage that generates a very rough surface. The bevel edge can be determined by the apparent "discontinuous" polished areas. (See Fig. 2.)

6.2 Damage depth may be nonuniform over a whole wafer




NOTE 1—The surface damaged from sandblasting.

NOTE 2—The 1-μm thick LPCVD nitride film is not visible in the photomicrograph.

**FIG. 2 5°44′ Bevel Angle Polished Surface After 1-min Defect Etch**

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TABLE 1 Relation of Bevel Angle (α), Bevel Length ($L$), and Damage Depth ($T_z$)

| Angle (α) | Sine (α) | $T_z$, µm | 5 | 10 | 100 | 200 |
|---|---|---|---|---|---|---|
| | | | Bevel Length ($L$), mm | | | |
| 17 min 11 s | 0.005 | | 1 | 2 | 20 | 40 |
| 34 min 23 s | 0.01 | | 0.5 | 1 | 10 | 20 |
| 1°9 min | 0.02 | | 0.25 | 0.5 | 5 | 10 |
| 2°52 min | 0.05 | | 0.01 | 0.2 | 2 | 4 |
| 5°44 min | 0.10 | | 0.05 | 0.1 | 1 | 2 |
| 11°32 min | 0.20 | | 0.025 | 0.05 | 0.5 | 1 |

area. Because the sample specimens are relatively small with respect to the whole wafer area, depth of damage variations may not be detected; thus, the measurement of the damage depth may be ambiguous.

6.3 Measurement of the bevel angle must be done after lapping and *before* polishing, otherwise the correct angle may not be measured. The alkaline polishing compound may cause a slight surface perturbation near the silicon–nitride interface and at the edges of the specimen.

## 7. Apparatus

7.1 *Apparatus to Bevel Polish the Test Specimen*:

7.1.1 *Beveling Jig*, consisting of a solid cylinder that is free to move within a hollow cylinder. The specimen is wax mounted onto a beveled sample mount which is then attached to the free moving cylinder as shown in Fig. 3.

7.1.2 *Hot Plate, Diamond Scriber, and Tweezers*.

7.1.3 *Polishing Equipment* that can vary the polishing pressure and will not produce crystal damage.

7.2 *Cement Removal*—The usual chemical laboratory apparatus such as beakers. Adequate facilities for handling and disposing of chlorinated solvents and their vapors are essential.

7.3 *Optical Measurements*:

7.3.1 *Reflection-Light Microscope* with mechanical stage and Nomarski interference contrast optics capable of 100 to 500× magnification as specified in Test Methods F 532.

7.3.2 *Stage Micrometer*.

7.4 *Apparatus to Measure the Angle Beveled on Silicon Specimen*—See Appendix of Test Method F 672.

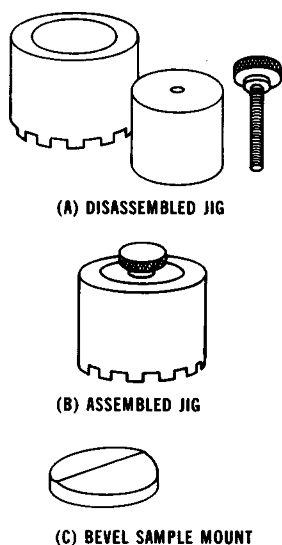


FIG. 3 Lapping/Polishing Jig and Sample Mount

7.5 *Hydrofluoric Acid*, proof chemical laboratory apparatus, such as fluorocarbon, polyethylene, or polypropylene beakers, graduates, pipets, and tweezers.

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

7.7 *Facility for Low-Pressure Plasma Nitride Deposition*—(Plasma enhanced CVD SiN capable of 1 µm thick film at ~330°C deposition temperature.)

## 8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. All reagents shall conform to the SEMI Specifications C1 where they exist. 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 Specification D 1193.

8.3 *Lapping Compounds*—0.1 µm diamond slurry or 3.0 µm silicon carbide wet/dry lapping paper.

8.4 *Polishing Compound*—Alkaline suspension of colloidal amorphous silica of sub-micron particle size with pH of 10 to 12.

8.5 *Polishing Pad*—Polyurethane based poromerics. These pads are commercially available from various metallurgical polishing supply companies.

8.6 *Mounting Cement*—Glycolphthalate or equivalent mounting cement with similar melting point and solubility in trichlorethane or perchlorethane. **Precaution**—These chlorinated solvents are on the suspected carcinogen lists of NIOSH.

8.7 *Mounting Cement Solvent*—Perchlorethane or trichlorethane.

8.8 *Wipers*, residue-free, non-scratching.

8.9 *Brush* with soft nylon bristles.

8.10 *Detergent*, non-ionic 0.25% solution by volume.

8.11 The chemicals used for defect delineation shall have the following nominal assay: Chromium Trioxide >98%; Hydrofluoric Acid, concentrated 48.8 to 49.2%.

8.12 *Preferential Etch—Diluted Schimmel Etch* [7]:

8.12.1 Prepare 0.75 M Chromic Acid solution by placing 75 g chromium trioxide in a one liter volumetric flask and then add water to make a solution volume of 1 L. The solution may be stored in clean glass, polyethylene, propylene, or teflon bottles until use.

8.12.2 Mix 2 parts hydrofluoric acid, 1 part 0.75 $M$ chromic acid solution, and 1.5 parts water by volume in a hydrofluoric acid proof beaker immediately before using.

8.13 *Compressed Nitrogen or Air*—Filtered (1.0 µm or smaller) and oil free.

## 9. Safety Hazards

9.1 The chemicals used in this evaluation procedure are potentially harmful and must be handled in a fume hood, with

[7] Schimmel, D. G., "Defect Etch for ⟨100⟩ Silicon Evaluation", Journal of the Electrochemical Society, Vol 126, No. 2, 1979, p. 479.

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the utmost care at all times. **Warning**—Hydrofluoric acid solutions are particularly hazardous. **Precaution:** 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.

9.2 Chromic acid, that is contained in the preferential etch solution, 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 Since this procedure is destructive in nature, a sampling procedure must be used to evaluate the characteristics of a group of silicon wafers. No general sampling procedure is included as a part of this test method, because the most suitable sampling plan will vary considerably depending upon individual conditions. For referee purposes, a sampling plan shall be agreed upon before conducting the test. See Practice E 122 for suggested choices of sampling plans.

## 11. Test Specimen Preparation

11.1 *Mounting*:

11.1.1 Select an area of the silicon wafer in which the damaged depth is to be measured.

11.1.2 Coat the wafer surface with silicon nitride by a low-pressure plasma method to a minimum thickness of 1 µm, scribe the wafer, and break a piece approximately 10 by 10 mm in size from the area of interest.

NOTE 1—Alternatively, the wafer piece may be coated with silicon nitride after scribing and breaking.

11.1.3 Select a beveled sample mount with the desired bevel angle and heat on the hot plate to the melting point of the mounting cement. Apply a thin smooth coat of the cement to the beveled sample mount in the area where the specimen is to be mounted.

11.1.4 Firmly press the specimen into the cement with the damaged side upward. Position it with the edge to be sectioned parallel to the apex of the sample mount bevel surface.

11.1.5 Cool the sample to room temperature in air, taking care to handle it in such a way as to avoid position shifts.

NOTE 2—Alternatively, the sample may be cooled by lowering the sample mount into water at room temperature.

11.1.6 Remove the beveled sample mount from the water when the cement has solidified. Remove excess extruded cement with wipers dampened with solvent.

11.2 *Lapping*:

11.2.1 Secure the beveled sample mount with the sample to the free moving cylinder of the beveling jig with the clamping screw (see Fig. 3).

11.2.2 Lap the specimen using 0.1 µm diamond slurry or wet 3.0 µm silicon carbide lapping paper to achieve an exposed bevel length adequate for the expected damage depth in accordance with Table 1. For example, the bevel length should be in excess of 1 mm for an angle of 5°44 min and an expected damage depth of 100 µm.

11.2.3 After the desired bevel length is obtained, care must be taken to prevent residues from the lapping material from drying on the specimen. While it is still attached to the beveled mounting block, scrub the specimen surface immediately after lapping with a brush with detergent. After scrubbing, rinse the fixture and attached specimen thoroughly in running water and dry by gently wiping with wipers.

## 12. Procedure

12.1 *Measurement of Bevel Angle*:

12.1.1 After lapping, measure the angle $\alpha$ between the beveled surface and the original surface (Fig. 1). A number of different ways of making this measurement is given in the Appendix of Test Method F 672. Use the method that will give the needed accuracy for the angle being used.

12.2 *Polishing*:

12.2.1 Place the assembled lapping/polishing jig with the attached lapped sample onto the polishing pad containing the alkaline polishing compound. Continue polishing until all the lapping damage has been removed. (See Appendix X1.)

12.2.2 After polishing, care must be taken to prevent residues from the polishing compound from drying on the specimen surface.

12.2.2.1 Immediately after polishing, rinse the polished specimen while it is still attached to the beveled mounting block with running water and dry the specimen by gently wiping with wipers.

12.2.2.2 Place the beveled mounting block with the sample on the hot plate and heat to the melting point of the mounting cement. Demount the beveled polished specimen from the beveled mounting block and completely remove the mounting wax from all the specimen surfaces using the mounting cement solvent.

12.3 *Damage Delineation*:

12.3.1 Etch the beveled sample as follows:

12.3.1.1 Place beveled specimen in plastic beaker and add sufficient preferential etch solution (8.12.2) to cover to a depth of approximately ½ in.

12.3.1.2 Etch for 1 min with manual agitation.

12.3.1.3 Decant etch solution into a container for chromic acid waste and rinse the specimen with running water.

NOTE 3—If adequate chromic acid disposal facilities are available, the etch may be quenched in running water.

12.3.1.4 Blow the specimen dry with dry, filtered air or nitrogen.

12.4 *Measurement of Length of Damage Region*:

12.4.1 Place the specimen on a clean beveled mounting block on the stage of the microscope.

12.4.2 Adjust the interference microscope for maximum contrast at a magnification of 100 to 500×.

12.4.3 Locate the damaged region. (See Fig. 2 for typical damaged region.) Search the bevel plane for the maximum damage penetration.

12.4.4 Measure the length, $L$, of the damage region. The length, $L$, is measured from the intersection of the specimen surface and the bevel plane to the maximum damage level along a direction perpendicular to the intersection.

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## 13. Calculation of Damage Depth

13.1 Calculate the damage depth ($T_z$) from the measured damage region length ($L$) and the bevel angle ($\alpha$) by the relationship as follows:

$$T_z = (L \times 1000 / \text{photographic magnification}) \times \sin \alpha$$

where:

$T_z$ = damage depth, µm, and
$L$ = damage region length as measured from photomicrograph to one decimal place, mm.

NOTE 4—If not known, the magnification may be determined in accordance with the section on Calibration of Method A, of Methods F 532.

13.2 Record each $T_z$ value in micrometres.

## 14. Report

14.1 Report the following information:

14.1.1 Operator identification,

14.1.2 Date of test,

14.1.3 Specimen's identification and location on wafer,

14.1.4 Bevel angle ($\alpha$) measurements and measurement technique used,

14.1.5 Length ($L$) of damage region as measured on beveled surface,

14.1.6 Magnification and type microscope used,

14.1.7 Photograph of typical damaged region as observed at magnification of measurement of $L$, and

14.1.8 Damage depth ($T_z$).

## 15. Precision and Bias

15.1 The precision and bias of this test method are not reported for interlaboratory use. This test method is intended for use in process control where each individual location is responsible to determine the internal repeatability to its satisfaction.

## 16. Keywords

16.1 bevel polish; damage-depth; defect; preferential etch; silicon

# APPENDIX

**(Nonmandatory Information)**

## X1. Minimum Polishing Time

X1.1 The amount and depth of damage which results from angle lapping will vary with different laboratories and facilities. Thus, there is no absolute polish time that can be specified to guarantee complete damage removal. However, residual lapping damage is recognizable by straight lines of dislocations usually extending over most, if not the full width, of the polished/etched surface.

X1.2 In order to establish the polishing time required to fully remove lapping damage, etch-polish-lapped control samples for varying lengths of time and etch them in accordance with 14.1. The sample with no defect etch pits will be the minimum polish time required to achieve removal of the lapping damage.

NOTE X1.1—The control samples are cleaved from a polished wafer that has no intentional surface damage.

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