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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Practice for Preparing Silicon for Spreading Resistance Measurements¹

This standard is issued under the fixed designation F 674; 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 surface preparation of silicon samples prior to measurement of resistivity variations by the spreading resistance technique.

1.2 Separate procedures are given for preparation of large-area specimens for measurement of lateral resistivity variations and for preparation of bevel-sectioned specimens (usually small chips) for measurement of vertical variations of resistivity (depth profiling).

NOTE 1—Benefits derived from diamond polishing are (1) stability and reproducibility of spreading resistance values on large area or beveled specimens, and (2) acuity of beveled surface geometry. The benefits of stability and reproducibility are likely to apply to both conductivity types and all resistivity values; however, they have been demonstrated extensively only for (111) *n*-type above 1 Ω -cm. Enhanced bevel acuity is independent of conductivity-type or resistivity value.

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. Summary of Practice

2.1 Silicon specimens are polished using fine-grain diamond compound in a non-aqueous fluid. Polishing of silicon wafers or other large-area specimens is done against a nonwoven polishing cloth; bevel polishing of small specimens is done against a frosted lapped glass surface. When polishing is complete, residual polishing compound is removed by an organic solvent.

3. Significance and Use

3.1 Resistivity is probably the single most important parameter for the characterization of silicon starting material for semiconductor device fabrication. Spreading resistance measurements are used to measure resistivity variations in raw silicon crystals and completed semiconductor devices. The reproducibility of spreading resistance measurements on silicon specimens is known to depend on the manner of specimen

preparation. The interpretation of spreading resistance measurements depends in turn on the reproducibility of test specimen measurements and on the reproducibility of calibration specimen measurements.

3.2 The procedures given are intended to confer a high degree of reproducibility to spreading resistance measurements, and offer improvement over other preparation techniques.²

4. Interferences

4.1 Polishing of silicon with diamond causes light but controllable and uniform scratch damage to the silicon surface. Nevertheless, such uniform damage is compatible with spreading resistance measurement data having very low scatter. Contamination of the polishing medium with hard foreign particles can cause random heavy scratch damage to a specimen. If encountered by the spreading resistance probes, heavily scratch-damaged regions may yield erratic measurement results.

4.2 Contamination of the specimen with water subsequent to polishing may adversely affect the reproducibility of spreading resistance measurements.

5. Front-Surface Diamond Polishing

5.1 Apparatus:

5.1.1 *Polishing Machine*—Oscillating-tubpolisher or other similar small laboratory-scale polishing machine capable of providing randomized motion of the silicon specimen over the polishing pad.

5.1.2 *Mounting Block and Fixture*, to support and apply vertical load to the silicon specimen during polishing.

5.1.3 *Polishing Pad*—Nonwoven cloth pad of a texture specified as being compatible with the grain size of diamond used during polishing. The polishing pad should be adhesive-backed for attaching to a support plate.

NOTE 2—The preferred material is of a type identified as a “chemotextile.”

5.1.4 *Support Plate*, of glass or other similar hard material compatible with the chosen polishing machine and capable of

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² Ehrstein, J. R., Ricks, D. R. and Robinson, L. A., “Spreading Resistance Measurements, Measurement Techniques for High Power Semiconductor Materials and Devices,” Annual Report, Oct. 1, 1977 to Sept. 30, 1978, NBSIR 79-1756, F.F. Oettinger, ed.

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providing a flat support for the polishing pad during polishing.

5.1.5 Microscope—Optical microscope having a total magnification of at least 30× and provision for oblique illumination of the specimen.

5.1.6 Hot Plate capable of heating the sample mounting block and wax to 150°C.

5.2 Reagents and Materials:

5.2.1 Diamond Slurry—Synthetic or natural diamond with grain size in the range 0.5 to 3 μm, inclusive, suspended in a nonaqueous liquid or paste carrier.

NOTE 3—The predominant causes of variation in the surface finish of the silicon specimen are expected to result from (1) the uniformity of particle size in the diamond grit, (2) the inclusion of a large fraction of needle-shaped grains (fines) in addition to the preferred symmetric grains (blocky diamond), and (3) in the case of diamond suspended in paste, the uniformity of the diamond distribution in the paste. For a fixed diamond grain size, whether the diamond is natural, single-crystal synthetic, or polycrystalline synthetic should make little difference in the resulting surface finish. However, the abrasive breakdown mechanisms differ somewhat for the different types of diamond. Consequently, the size and type of diamond should be chosen to give an acceptable cutting rate for the specimen and machine conditions that will be used.

NOTE 4—For use with large-area specimens, the appropriate size diamond grain and, in part, the type of diamond to be used should be compatible with (1) the starting surface texture of the silicon, which may range from as-sawn to prepolished, and (2) the load that is applied during polishing.

5.2.2 Solvent—Suitable nonaqueous solvent for removing diamond slurry subsequent to polishing.

NOTE 5—The choice of solvent is governed in part by the composition of the carrier liquid or paste. The supplier of the diamond slurry should be consulted regarding the appropriate solvent. Acetone ((CH₃)₂CO) and methanol (CH₃OH) are known to work well for removing many commercial diamond compounds.

5.2.3 Wax—Glycol phthalate or other similar wax having a melting temperature of less than 150°C.

5.2.4 Dry Air or Nitrogen—Source of clean, dry air or nitrogen suitable for drying the specimen.

5.3 Procedure:

5.3.1 If not previously done, attach the polishing pad to the rigid plate and place several drops of diamond polishing slurry at random locations on the polishing pad surface. Spread drops of slurry in a reasonably uniform manner over the pad so that the pad surface becomes damp and so that there are no freestanding layers of slurry. If the diamond is suspended in paste, be sparing in the amount of paste applied to the pad.

NOTE 6—Some polishing slurry adheres to the specimen and mounting fixture and is lost every time a specimen is removed and cleaned. Replenish the slurry on the pad regularly with a few drops of fresh slurry.

5.3.2 With the hot plate, heat the mounting block to the melting temperature of the wax. Mount the silicon specimen to the block with the wax. Allow the block to cool to room temperature.

5.3.3 Assemble the mounting block and attach specimen to the sample mounting fixture and place this assembly on the polishing pad in the polishing machine.

5.3.4 In accordance with the manufacturer's instructions for the polishing machine, polish until the specimen surface exhibits a uniform density of random-direction scratches comparable to the pattern shown in Fig. 1. For this test, remove the polishing slurry from the specimen surface and examine that surface with the microscope.

NOTE 7—The time required to reach a uniform surface finish will depend on specimen surface area, size of diamond grains, static load applied, rate of movement of the specimen surface over the polishing pad, and previous surface finish.

NOTE 8—The coarseness of the scratch damage on the specimen surface is related to the size of diamond grit used.

5.3.5 When an acceptable surface finish has been reached, thoroughly swab or flush the specimen with the solvent to remove all diamond slurry residues. Use dry air or nitrogen to blow the specimen surface dry prior to carrying out any spreading resistance measurements.

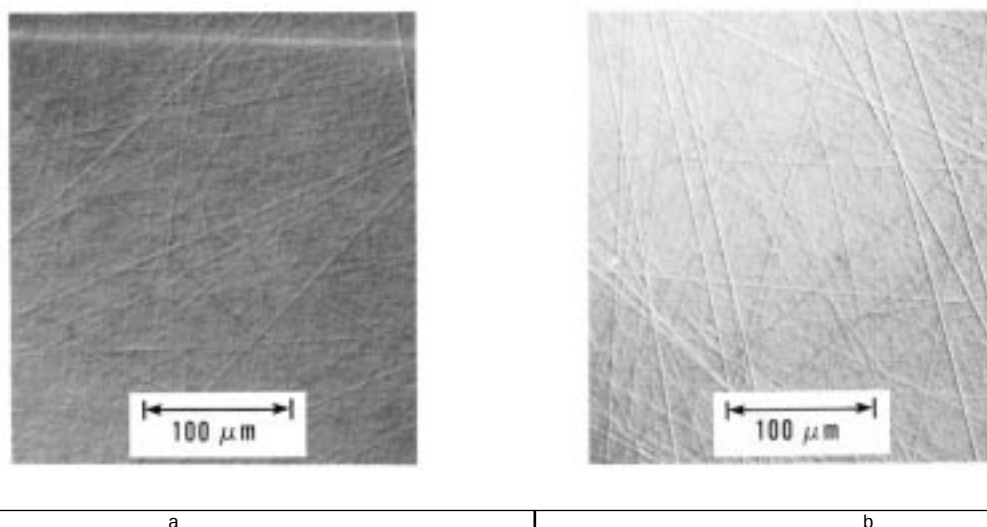


FIG. 1 Surface Texture of Large-Area Silicon Specimen Polished with Diamond Against Chemotextile Pad
(a) 0.5-μm Diamond (b) 3-μm Diamond