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Standard Method for
MEASUREMENT OF OXIDE THICKNESS ON SILICON WAFERS AND METALLIZATION THICKNESS BY MULTIPLE-BEAM INTERFERENCE (TOLANSKY METHOD)[1]

This standard is issued under the fixed designation F 388; 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 destructive method covers the measurement of the thickness of a uniform layer formed on or deposited upon a flat surface. The method is appropriate for measurements of layers from 4 to 3000 nm thick.

1.2 The referee procedure is suitable for research laboratories and in process control applications where increased precision is required or to calibrate other thickness measurement methods. Two nonreferee procedures suitable for general use are also given.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary statements are given in Section 8.

2. Applicable Documents

2.1 *ASTM Standard:*

D 1125 Test Methods for Electrical Conductivity and Resistivity of Water[2]

2.2 *SEMI Standards:*

C1 Specifications for Reagents[3]

2.3 *Other Standard:*

Reagent Chemicals, American Chemical Society Specifications[4]

3. Summary of Method

3.1 A step in the layer whose thickness is to be measured is produced by etching or shadowing. This step and the adjacent plane areas are coated with a reflective metal (usually aluminum or silver). A partly reflecting flat glass plate is placed over the metallized area which is illuminated by monochromatic light through the glass plate. The plate is tilted slightly to produce an interference pattern of parallel lines. The direction of the tilt is adjusted to make these lines perpendicular to the step. A photograph of the interference pattern is made. The parallel interference lines are displaced at the step by an off-set distance proportional to the step thickness. This off-set distance and the spacing between adjacent parallel lines are measured. Since the spacing between successive parallel lines in the plane areas corresponds to a half wavelength of the monochromatic light, the thickness of the layer is the off-set distance multiplied by the half wavelength of the light divided by the spacing between interference lines.

4. Significance and Use

4.1 Thin silicon dioxide films are used in semiconductor device fabrication for isolation, for protection of surfaces (passivation), and for

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[1] This method is under the jurisdiction of ASTM Committee F-1 on Electronics and is the direct responsibility of Subcommittee F01.06 on Electrical and Optical Measurements.

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

[3] Available from the Semiconductor Equipment and Materials Institute, 625 Ellis St., Suite 212, Mountain View, CA 94043.

[4] Available from American Chemical Society, Washington, DC 20036.

masking in diffusion processes. A precise measurement of oxide thickness is not only necessary to provide optimum device yield and reliability, but also it is useful as a process control parameter.

4.2 The control of metallization thickness is required in semiconductor device fabrication in order to ensure optimum reliability and electrical conductivity within the design tolerance.

4.3 No interlaboratory precision has been established for this technique. It is not recommended that this method be used for the acceptance or rejection of material exchanged between suppliers and users.

5. Interferences

5.1 Since the basic measurement performed in this method is the location of a line, the precision is degraded by any broadening of the interference line width.

5.1.1 The line width can be reduced by the use of higher reflectivity silver rather than aluminum for metallization (see 6.8) but silver is more difficult to protect from tarnishing.

5.1.2 The line width can be reduced by increasing the reflectivity of the interferometer optical plate (see 6.3) to match that of the metallization, but further increase of reflectivity reduces the light intensity of the interference pattern. A reflectance of 90 to 95 % gives a satisfactory compromise between light intensity and line sharpness.

5.2 The precision of the method is also reduced by waviness, bowing, or other departures from flatness of the surfaces. Such conditions produce curved interference lines or uneven line spacings.

5.2.1 Nonflatness of the substrate, nonuniformity of the layer thickness, or nonuniformity of the metallization thickness distorts the interference pattern.

5.3 A rounding error less than 0.5 % is produced when the wavelength of sodium light is taken to be 589.0 nm. Present precision of this method does not force a decision on whether to use the stronger of the yellow sodium doublet lines at 588.9953 nm, or an intensity weighted average with the half-as-intense 589.592 nm lines, or an unweighted average of the two wavelengths. The rounding error is even smaller for the mercury light source when 546.074 nm is rounded to 546.0 nm for the green line and for the thallium light source when 535.046 nm is rounded to 535.0 nm.

6. Apparatus

6.1 *Metallurgical Microscope,* with camera and provisions for vertical illumination from an external source. Magnification should be sufficiently adjustable to ensure that six interference fringes (see 12.1.3) should fill at least 50 mm of the photograph. Magnification of 10× to 100× should be satisfactory for the usual specimens. Photographs of a nominal 2.75 by 3.75-in. (70 by 95-mm) size or larger are satisfactory.

6.1.1 *Graticule* with 100 subdivisions is required for alternate procedure C (see 12.3.2).

6.2 *Monochromatic Light Source,* usually a mercury or sodium vapor arc lamp (see 5.3 and 13.4).

6.3 *Interferometer Jig* consisting of a partly reflecting optical (Fizeau) plate (see 5.1.2) supported in a manner which permits its plane to be rotated in two directions; an adjustable three-point support is satisfactory. The optical plate is commercially available and consists of multilayer dielectric films of zinc sulfides and hard oxides and with a reflectance matching that of the metallization (usually 0.9 to 0.95).

6.4 *Hot Plate,* thermostatically controlled, with a range up to 150°C and capable of maintaining a surface temperature within ±5°C.

6.5 *Plastic Beakers,* suitable for hydrofluoric acid and hot trichloroethylene.

6.6 *Toolmaker's Microscope* capable of resolving 2.5 μm and with a reticle consisting of either a single horizontal line or two horizontal lines separated by an apparent distance approximately equal to the apparent width of an interference line. (Not required for Procedures B or C).

6.7 *Rule,* metal, straight edge, graduated in hundredths inches or half millimetres. (Optional equipment for alternate Procedure B, see 12.2.3 and 12.2.4.)

6.8 *Metallization Facility* for vacuum evaporization of aluminum or silver films of uniform thickness of approximately 100 nm. The vacuum pressure should not exceed 10^{-5} Torr (1.3 mPa).

6.9 *Laboratory Facilities,* with hood and sink, suitable for conventional semiconductor materials processing.

7. Reagents and Materials

7.1 *Purity of Reagents*—All chemicals for which specifications exist shall conform to SEMI Specifications C1. 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, 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.

7.2 *Purity of Water*—Reference to water shall be understood to mean either distilled water or deionized water having a resistivity greater than 5 MΩ·cm at 25°C as determined by Method B of Test Methods D 1125.

7.3 *Hydrofluoric Acid* (HF).

7.4 *Trichloroethylene* ($CHClCCl_2$).

7.5 *Wax, Etch-Resistant*—A paraffin-based tissue embedding wax with a melting point of 56 to 57°C has been found to give a straighter line for the oxide step than the commonly used black waxes.

8. Safety Precautions

8.1 The hydrofluoric acid and the hot trichloroethylene used in this method are extremely hazardous. All precautions normally used with these chemicals should be strictly observed.

8.2 **Caution**—Hydrofluoric acid can cause painful and dangerous burns which sometimes leave bad scars.

8.2.1 Wear eye protection and acid-proof gloves at all times when handling HF. Instruct all immediate personnel in first aid measures for HF burns.

8.2.2 If HF comes in contact with the body, or it is suspected that HF might have come in contact with the body, the affected areas should be immediately washed thoroughly in water for at least 15 min. If this procedure is applied within a few seconds of the time the HF comes in contact with the skin, further treatment is rarely required. If, however, pain is noted after 1 h, the patient should see a physician for injection of calcium gluconate at the physician's discretion.

8.2.3 In cases where the affected areas are eyes, lips, under fingernails, or other soft tissues, the patient should be taken to a physician immediately after the affected area has been washed.

9. Sampling

9.1 Since this procedure is destructive in nature, a sampling procedure must be used. This sample should consist of a representative wafer or wafers from each group of wafers processed. For referee purposes, a sampling plan shall be agreed upon in advance.

10. Specimen Preparation

10.1 Prepare specimens for measurement of oxide thickness as follows:

10.1.1 Process the specimen wafer through the oxidation procedure.

10.1.2 Keep the specimen clean; or, if necessary, clean the specimen prior to measurement by any technique which does not affect the layer thickness, such as the following.

10.1.2.1 Clean ultrasonically in warm water and detergent solution, rinse in flowing water, ultrasonically degrease in acetone, rinse in methanol and air dry. Cushion the specimen with paper or place in a pliable plastic beaker during ultrasonic agitation in order to reduce the risk of breakage.

10.1.3 Mask part of the oxidized specimen by the following procedure:

10.1.3.1 Melt enough etch-resistant wax in a small beaker on a hot plate to provide a depth equal to one third to one half of the diameter of the specimen. Set the hot plate thermostat to a temperature of 15 ± 5°C above the melting point of the wax (see 7.5).

10.1.3.2 Hold the specimen with tweezers and dip one third to one half of the specimen into the molten wax for a few seconds. Keep the plane surfaces of the specimen perpendicular to the surface of the wax.

10.1.3.3 Remove the wafer from the beaker and touch the lower edge of the specimen with a paper towel to remove any accumulated excess molten wax.

10.1.3.4 Allow the wax to harden by cooling for a few seconds in air.

10.1.4 Etch the unmasked oxide from the specimen by the following procedure:

10.1.4.1 Immerse the specimen in a beaker of hydrofluoric acid (HF) for 10 to 15 s.

10.1.4.2 Remove the specimen from the etch.

10.1.4.3 Wash the specimen under flowing water for 5 to 10 s.

10.1.5 Inspect the specimen for complete re-