Standard Test Method for Measurement of Insulator Thickness and Refractive Index on Silicon Substrates by Ellipsometry ¹

This standard is issued under the fixed designation F 576; 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.

INTRODUCTION

When this test method was developed in the mid-1970's, manual-null ellipsometers, which are the basis of this test method, were in routine use. More recently, faster, automated instruments have replaced manual-null ellipsometers for all common use in the semiconductor industry. There are two basic types of such automated instruments commonly used: the rotating element null ellipsometer and the rotating element photometric ellipsometer. For each of these, microprocessors or microcomputers are used to operate the instrument and to analyze the data. Details of the procedures utilized in these instruments are usually considered to be proprietary by the instrument manufacturers.

Despite the fact that this test method is not commonly used in its present form, it embodies all the basic elements of this test method and a simple analysis of data. Thus, it provides useful guidance in the fundamentals and application of ellipsometry to film thickness measurements. Until a test method, or test methods, can be developed that cover the newer, automated instruments, this test method provides the only such information that is available in a standard test procedure. It also contains results of a test of interlaboratory precision on silicon dioxide films from 20 to 280 nm using manual null ellipsometers, and of a test of interlaboratory precision of films of 5 to 550 nm using both manual null ellipsometers as well as automated ellipsometers of both types just mentioned.

Two major changes have occurred since this test method was initially adopted. First, reference materials certified for the thickness of silicon dioxide layers on silicon are available both from the National Institute of Standards and Technology and from commercial sources. These can be used to evaluate the performance of automated ellipsometers. Second, significantly improved materials and procedures have been developed for storage of reference wafers needed for long term testing of baseline performance of ellipsometers. It is not uncommon for reference wafers simply to be stored "clean" with no further wafer-cleaning utilized. If cleaning steps are in fact, utilized, they are not those described in this test method. The cleaning steps detailed in this test method are retained, however, to provide background information on procedures used for the first of the interlaboratory tests.

1. Scope

1.1 This test method covers the measurement by ellipsometry of the thickness and refractive index of an insulator grown or deposited on a silicon substrate.

1.2 This test method uses monochromatic light.

1.3 This test method is nondestructive and may be used to measure the thickness and refractive index of any film not absorbing light at the measurement wavelength on any substrate (1) not transparent to light at the measurement wavelength, and (2) of a material for which both the refractive index and the absorption coefficient are known at the measurement wavelength.

1.4 The precision of this test method is reduced by variations, over regions smaller than the light-beam spot size, in substrate flatness, insulator thickness, and index of refraction.

1.5 Film thickness measurements determined by ellipsom etry are not unique. When the film thickness is greater than that calculated from the expression $N\lambda/[2(n^2 - \sin^2\phi_0)^{1/2}]$, where *N* is an integer, λ the measurement wavelength, *n* the index of refraction, and ϕ_0 the angle of incidence, the thickness value determined by this expression must be added to the thickness value determined by ellipsometry to obtain the correct film thickness. The value of *N* must be obtained by another procedure.

1.6 Two procedures for computing the results are provided. If the graphical procedure is used, the measuring wavelength shall be either 546.1 or 632.8 nm, and the angle of incidence shall be $70 \pm 0.1^{\circ}$.

1.7 This test method may be used for referee measurements

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

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with computer calculations.

1.8 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 Ultra Pure Water Used in the Electronics and Semiconductor Industry²
- $E\ 177\ Practice \ for \ Use \ of \ the \ Terms \ Precision \ and \ Bias \ in \ ASTM \ Test \ Methods^3$

E 284 Terminology of Appearance⁴

F 95 Test Method for Thickness of Lightly-Doped Silicon Epitaxial Layers on Heavily-Doped Silicon Substrates Using a Dispersive Infrared Spectrophotometer⁵

2.2 SEMI Standard:

C19 Specification for Acetone⁶

C31 Specification for Methanol⁶

2.3 ASTM Adjuncts:

Large size figures 7

3. Terminology

3.1 Definitions:

3.1.1 *ellipticity—in optics, of elliptically polarized light*, the angle χ given by the inverse tangent of the ratio of the minor to the major axis of the ellipse described by the electric vector of the light.

- ³ Annual Book of ASTM Standards, Vol 14.02.
- ⁴ 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 Institute, 625 Ellis St., Suite 212, Mountain View, CA 94043.

3.1.2 *fast axis—in optics, of a doubly refracting crystal*, that direction in which the velocity of light is a maximum.

3.1.3 *optic axis—of a doubly refracting crystal*, that direction through the crystal along which no double refraction occurs.

3.1.4 *polarization—in optics*, the term used to describe the orientation of the time-varying electric field vector in an electromagnetic wave.

NOTE 1—If the electric field vector is confined to a plane containing the direction of propagation of the wave, the wave is said to be plane polarized. If the vector rotates around the direction of propagation as an axis but remains constant in magnitude, the wave is said to be circularly polarized. If the amplitude does not remain constant, so that the end of the vector traces out an ellipse, the wave is said to be elliptically polarized.

3.1.5 *polarized light—in optics*, light exhibiting different properties in different directions at right angles to the line of propagation.

3.1.6 *relative minimum—in optics*, a minimum in the amount of light transmitted through a polarizer and analyzer combination that results from varying either the polarizing angle or the analyzing angle (with the other angle fixed).

3.1.7 Other terms used in this method are defined in Terminology E 284, Test Method F 95.

4. Summary of Test Method

4.1 The apparatus is assembled as shown in Fig. 1. Light emitted from the monochromator is plane polarized after passing through the polarizer. The compensator is set at -45° (or $+315^{\circ}$) to convert the plane-polarized light to elliptically polarized light. The azimuth angle and degree of ellipticity of the light incident on the specimen are determined from the settings of polarizer and compensator. The incident light undergoes a change in degree of ellipticity and azimuth when reflected from the specimen. The system is adjusted for signal extinction at the detector by alternately changing the polarizer and analyzer settings with the result that the incident light on the specimen surface is elliptically polarized and the reflected light is plane polarized. The film thickness and index of refraction are calculated either by a manual graphical method



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FIG. 1 Schematic of Ellipsometer Apparatus

² Annual Book of ASTM Standards, Vol 11.01.

⁷ Also available as large-size figures from ASTM Headquarters, 100 Barr Harbor Drive, West Conshohocken, PA 19428. Order Adjunct ADJF0576.

or by means of a computer program (1).⁸

5. Significance and Use

5.1 Thin insulator films are used in semiconductor device fabrication for isolation, passivation, masking in diffusion processes, and in some applications as a part of the device. Precise knowledge on the part of the device designer and fabricator of actual insulator thickness or index of refraction, or both, provides information useful for the optimization of quantities such as device operating parameters, yield, and reliability. The measurements are also useful for process control. Since the interlaboratory precision and accuracy of this test method have not yet been determined (see 15.3), it is not recommended that the test method be used for materials acceptance purposes.

5.1.1 The threshold voltage for a MOSFET device is related to the thickness of the gate insulator.

5.1.2 The capacitance of a capacitor is inversely proportional to the insulator thickness.

5.1.3 The maximum voltage possible across a MOSFET gate is proportional to the insulator thickness.

5.1.4 The effectiveness of a diffusion mask is proportional to insulator thickness.

NOTE 2-MOSFET is an acronym for Metal-Oxide Semiconductor Field-Effect Transistor.

6. Interferences

6.1 The presence of fingerprints or other foreign contamination on the surface may give erroneous results.

6.2 If the substrate is not flat, the thickness of the layer is not uniform, or the index of refraction is not uniform over regions comparable in dimension to the diameter of the light beam, it may not be possible to obtain complete extinction (see 12.10.1), with the result that the precision of the measurement may be reduced.

6.3 If the film is partially absorbing or scattering at the measurement wavelength, a unique solution may not be obtainable.

6.4 When graphical methods are used in the calculations, the precision of the method is reduced when the angles Δ and Ψ (see 13.2.1) have a range of values from 140 to 180°, inclusive, and from 11.6 to 14.0°, inclusive, respectively.

7. Apparatus

7.1 *Light Source*, producing a collimated beam of monochromatic light at the intended measurement wavelength.

NOTE 3—The source may consist of either (1) a laser, or (2) a polychromatic lamp with collimator and filters or monochromator for selecting the measurement wavelength.

7.2 *Polarizer*—Doubly refracting crystal used to convert the unpolarized monochromatic radiation from the light source to plane-polarized light. The crystal shall be rotatably mounted in a divided circle that can be read to within $\pm 0.1^{\circ}$.

7.3 *Analyzer*—Doubly refracting crystal of similar construction to that of the polarizer and with the same type of mounting. 7.4 Compensator— Doubly refracting plate, with known constants T_c and Δ_c (see 13.1), used to convert plane-polarized light to elliptically polarized light, and mounted in a divided circle that can be accurately positioned to within $\pm 0.1^\circ$.

Note 4—If the constants T_c and Δ_c are not known, they may be determined experimentally in accordance with Section 12 provided that the calculations are performed by means of a computer program (1). For this purpose, the test specimen is replaced by a metal specimen known to be free of any film. The ellipsometer parameters calculated in 12.8.1, 12.10.1, 12.13, and 12.15 are used as input data for the computer program, and the compensator constants are calculated by the program.

7.5 Specimen Table— Specimen mounting table with graduated circle for measuring the angles of incidence on and reflection of light from the specimen to within $\pm 0.1^{\circ}$. At its center, the table shall incorporate an X-Y stage suitable for mounting the specimen and capable of positioning different regions of the specimen in the light beam for the measurements.

7.6 *Detector*—Photoelectric detector, for determining the minimum of the reflected light signal.

7.7 Aperture Plates, as required by the apparatus shown in Fig. 1, including (1) a variable-aperture plate or several fixed-aperture plates having apertures ranging in diameter from 1 to 5 mm, inclusive, and used to define the size of the light-beam spot incident on the specimen, and (2) an inter-changeable aperture-plate assembly.

7.8 *Chemical Laboratory Apparatus*, such as plastic beakers and plastic-coated tweezers suitable for use with solvents.

7.9 *Ventilated Hood*— Working space with means for limiting the concentration of solvent vapors to acceptable levels and for exhausting air containing vapors in a manner consistent with safe practice.

7.10 *Ultrasonic Cleaner*, with operating frequency in the nominal range from 18 to 45 kHz and with adequate power to clean test specimens.

7.11 Glass Plate, suitable for use in 12.2.

7.12 Supports, Mounts, and Other Fixtures, as required.

8. Reagents and Materials

8.1 *Purity of Reagents*—All chemicals for which SEMI specifications exist shall adhere to Grade 1 specifications for those chemicals. 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,⁹ 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.

8.2 *Purity of Water*— References to water shall be understood to mean Type I or II water as specified in Guide D 5127.

8.3 Acetone [(CH $_3$)₂ CO], SEMI C19, grade 1.

8.4 Methanol (CH $_3$ OH), SEMI C31, grade 1.

⁸ The boldface numbers in parentheses refer to the list of references at the end of this test method.

⁹ 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. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.