SEMI MF576-0812 (Reapproved 1023)
TEST METHOD FOR MEASUREMENT OF INSULATOR THICKNESS AND REFRACTIVE INDEX ON SILICON SUBSTRATES BY ELLIPSOMETRY

This Standard was technically approved by the Silicon Wafer Global Technical Committee. This edition was approved for publication by the global Audits and Reviews Subcommittee on August 21, 2023. Available at www.semiviews.org and www.semi.org in October 2023; originally published by ASTM International as ASTM F576; previously published July 2018.

NOTICE: The section symbol (§) is used to reference a numbered section, paragraph, or header and all subordinate paragraphs, headers, and embedded material (i.e., EXCEPTIONs, lists) therein. The paragraph symbol (¶) is used to reference a particular numbered paragraph and embedded material therein. When duplicated (i.e., §§ and ¶¶) the symbols are used to reference more than one section or paragraph, respectively.

NOTICE: Paragraphs entitled 'NOTE:' are not an official part of this Standard or Safety Guideline and are not intended to modify or supersede the official Standard or Safety Guideline. These have been supplied by the global technical committee to enhance the usage of the Standard or Safety Guideline.

1 Purpose

1.1 Thin insulator films are used in semiconductor device fabrication for isolation, passivation, masking in diffusion processes, and in many 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.

1.1.1 The measurements are also useful for process control.

1.1.2 Because the multilaboratory tests that have been conducted and reported for this Test Method do not follow the conditions of present-day procedures, it is recommended that the test method be used for materials acceptance purposes only if the parties to the test establish correlation.

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

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

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

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

1.2 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 multilaboratory precision on silicon dioxide films from 20 nm to 280 nm thick using manual null ellipsometers, and of a test of multilaboratory precision of films of 5 nm to 550 nm using both manual null ellipsometers as well as automated ellipsometers of two types (refer to ¶ 2.5).

2 Scope

2.1 This Test Method covers the measurement by ellipsometry of the thickness and refractive index of an insulator grown or deposited on a silicon substrate.

2.2 This Test Method uses monochromatic light.

2.3 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.

Documents Document Sharing Hub

Full document details: SEMI MF576-0812-1023R

2.4 Two procedures for computing the results are provided. If the graphical procedure is used, the measuring wavelength shall be either 546.1 nm or 632.8 nm, and the angle of incidence shall be 70° ± 0.1°.

2.5 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. Since that time, 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 proprietary by the instrument manufacturers.

2.6 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.

NOTICE: SEMI Standards and Safety Guidelines do not purport to address all safety issues associated with their use. It is the responsibility of the users of the Documents to establish appropriate safety and health practices, and determine the applicability of regulatory or other limitations prior to use.

3 Limitations

3.1 Film thickness measurements determined by ellipsometry are not unique (refer to ¶ 13.2.1). When the film thickness is greater than that calculated from the expression:

$$t_{\max} = \frac{\lambda}{2\sqrt{n^2 - \sin^2 \phi_0}} \tag{1}$$

where:

$t_{\max}$ = maximum zero order thickness,

λ = measurement wavelength,

n = index of refraction, and

ϕ_0 = 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 (refer to ¶ 13.2.1).

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

3.3 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 (refer to ¶ 12.10.1), with the result that the precision of the measurement may be reduced.

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

3.5 When graphical methods are used in the calculations, the precision of the method is reduced when the angles Δ and Ψ (refer to ¶ 13.1) have a range of values from 140° to 180°, inclusive, and from 11.6° to 14.0°, inclusive, respectively.

4 Referenced Standards and Documents

4.1 *SEMI Standards and Safety Guidelines*

SEMI C19 — Specification for Acetone

Documents
Document Sharing Hub

Full document details: SEMI MF576-0812-1023R

SEMI C31 — Specification for Methanol

SEMI M59 — Terminology for Silicon Technology

4.2 *ASTM® Standards*[1]

ASTM D5127 — Guide for Ultra-Pure Water Used in the Electronics and Semiconductor Industries

ASTM E177 — Practice for Use of the Terms Precision and Bias in ASTM Test Methods

NOTICE: Unless otherwise indicated, all documents cited shall be the latest published versions.

5 Terminology

5.1 Terms relating to silicon and other semiconductor technology are defined in SEMI M59.

5.2 *Abbreviations and Acronyms*

5.2.1 *MOSFET* — metal-oxide semiconductor field-effect transistor

5.3 *Definitions*

5.3.1 *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.

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

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

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

5.3.4.1 *Discussion* — 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.

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

5.3.6 *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).

5.4 *Symbols*

5.4.1 T_c — transmission ratio

5.4.2 Δ_c — relative phase retardation angle

6 Summary of Test Method

6.1 The apparatus is assembled as shown in Figure 1. Light emitted from the monochromator is plane polarized after passing through the polarizer.

6.2 The compensator is set at −45° (or +315°) to convert the plane-polarized light to elliptically polarized light.

[1] ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, USA; Telephone: +1.610.832.9585, Fax: +1.610.832.9555, http://www.astm.org. ASTM trademark is owned by ASTM International.

Documents Document Sharing Hub

Full document details: SEMI MF576-0812-1023R

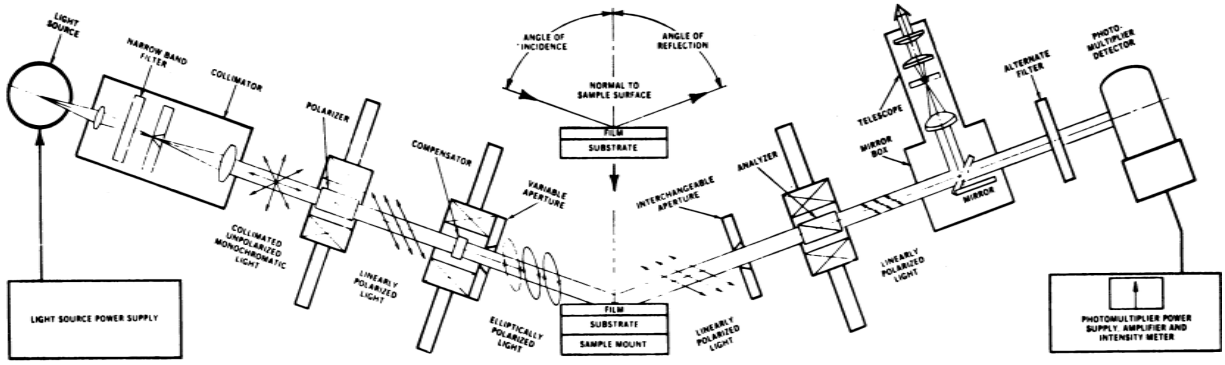

Figure 1
Schematic of Ellipsometer Apparatus[2]

6.3 The azimuth angle and degree of ellipticity of the light incident on the specimen are determined from the settings of polarizer and compensator.

6.4 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.

6.5 The film thickness and index of refraction are calculated either by a manual graphical method or by means of software included with the instrument.

7 Apparatus

7.1 *Light Source* — A laser or a polychromatic lamp with a collimator or a monochromator to produce a collimated beam of monochromatic light at the intended 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 ±0.1°.

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, used to convert plane-polarized light to elliptically polarized light, and mounted in a divided circle that can be accurately positioned to within ±0.1°.

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 ±0.1°. 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 Figure 1, including (1) a variable-aperture plate or several fixed-aperture plates having apertures ranging in diameter from 1 mm to 5 mm, inclusive, and used to define the size of the light-beam spot incident on the specimen, and (2) an interchangeable 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.

[2] Reprinted by permission of R. F. Spanier, from *Industrial Research*, September 1975, p. 75.

Documents
Document Sharing Hub
Full document details: SEMI MF576-0812-1023R

7.10 *Ultrasonic Cleaner* — With operating frequency in the nominal range from 18 kHz 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 specific chemicals. 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 Type E-3 or better water as described in ASTM D5127.

8.3 *Acetone* $[(CH_3)_2CO]$ — Grade 1, as specified in SEMI C19.

8.4 *Air or Nitrogen* — Dry and oil-free.

8.5 *Detergent Solution* — An aqueous, nonionic surfactant solution.

8.6 *Methanol* (CH_3OH) — Grade 1, as specified in SEMI C31.

9 Hazards

9.1 Acetone and methanol are flammable. Observe all precautions normally used with these solvents, including the avoidance of direct contact with the skin or inhalation of vapors.

10 Sampling

10.1 Since this Test Method is nondestructive, 100% testing of substrates is possible. However, the thickness or index of refraction, or both, of the insulator film may vary across an individual substrate. Sampling procedures shall be designed to reveal such variations.

10.1.1 The sampling procedures, both for the selection of individual substrates and with respect to the identification of measurement sites on a single substrate, shall be agreed upon by the parties to the test.

10.1.2 If sampling by lot is appropriate, the parties to the test shall agree on the definition of lot.

11 Test Specimen

11.1 Clean the specimen in warm water and detergent in an ultrasonic cleaner, rinse with water, and dry.

11.2 Ultrasonically degrease the specimen in acetone, rinse with methanol, and blow dry with oil-free air or nitrogen.

NOTE 1: If the insulator film is prepared just prior to the ellipsometer measurement, or is stored in a dry box that is purged with an inert gas, specimen cleaning may not be necessary.

12 Procedure

12.1 Assemble, level, and align the ellipsometer apparatus (refer to Figure 1) in accordance with instructions supplied with the instrument or following procedures given by Winterbottom[3] or McCracken, *et al*.[4] Adjust the collimator and detector axes to be coplanar with the normal-to-the specimen surface and to intersect at a common point on the specimen surface.

12.2 Mount the glass plate on the specimen stage.

12.3 Adjust the polarizer and analyzer in accordance with instructions supplied with the instrument or with published procedures[4] so that the polarizer circle reads 0° when the electric vector of the polarized light beam is vibrating in the direction parallel to the plane of incidence (*p*-wave mode). Make the adjustment by rotating the polarizer and analyzer

[3] Winterbottom, A. B. "*Optical Studies of Metal Surfaces*" I Kommisjon Hos F., Bruns Bokhamdel, Trondheim, 1956.

[4] McCracken, F. L., Passaglia, E., Stromberg, R. R., and Steinberg, H. L. "Measurement of the Thickness and Refractive Index of Very Thin Films and the Optical Properties of Surfaces." *J. Res. NBS* **67A**, No. 4 (1963): pp. 363–377.

Documents Document Sharing Hub

Full document details: SEMI MF576-0812-1023R