**Designation: F 576 – 00**

# 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 ellipsometry 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, $\lambda$ the measurement wavelength, $n$ the index of refraction, and $\phi_0$ 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 ± 0.1°.

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 Industry[2]

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods[3]

E 284 Terminology of Appearance[4]

F 95 Test Method for Thickness of Lightly-Doped Silicon Epitaxial Layers on Heavily-Doped Silicon Substrates Using a Dispersive Infrared Spectrophotometer[5]

2.2 *SEMI Standard:*

C19 Specification for Acetone[6]

C31 Specification for Methanol[6]

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 $\chi$ 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.

3.1.2 *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°) 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

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

[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.

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

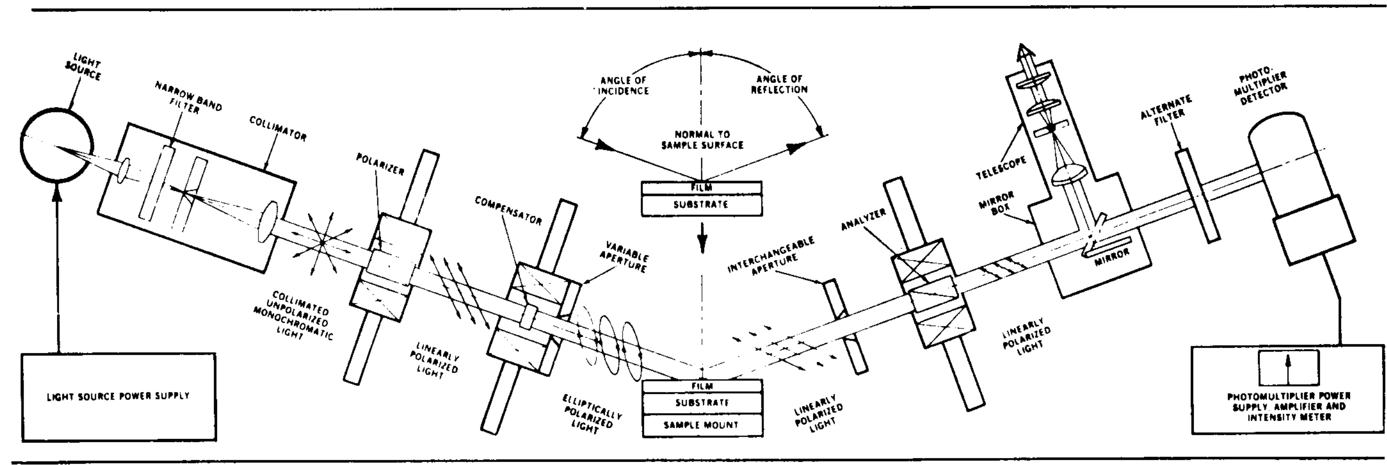


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**FIG. 1 Schematic of Ellipsometer Apparatus**

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 $\Delta$ and $\Psi$ (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 ±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 $\Delta_c$ (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 ±0.1°.

NOTE 4—If the constants $T_c$ and $\Delta_c$ 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 ±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 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 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.

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* $[(CH_3)_2CO]$, SEMI C19, grade 1.

8.4 *Methanol* $(CH_3OH)$, 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.

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

8.6 *Air or Nitrogen*, dry and oil-free.

## 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 5—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 (see Fig. 1) in accordance with instructions supplied with the instrument or following procedures given by Winterbottom **(2)** or McCracken, et al. **(3)**. Adjust the collimator and detector axes to be coplanar with the normal-to-thespecimen 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 **(3)** 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 crystals with respect to their divided circles so that the beam reflected from the glass plate is at a minimum intensity when the polarizer circle reads 0° and the analyzer circle reads 90°.

12.4 Mount the specimen on the specimen stage. Set collimator and detector optics at the desired angle of incidence and reflection. Use 70 ± 0.1° if the graphical method of computing results is intended.

NOTE 6—An angle of incidence (reflection) of 70° is recommended for insulator films on silicon.

12.5 Set the labelled "fast" axis of the compensator at – 45°. Consider angles to be positive when measured in a counterclockwise direction from the plane of incidence looking into the beam (toward the source).

12.6 Start all measurements with the polarizer and analyzer set at 0°. Make all angle changes by increasing the angle in the positive direction.

12.7 Increase the analyzer angle $A$ until a relative null is observed, but do not exceed $A = 90°$.

12.7.1 If a relative minimum is observed, proceed to 12.8.

12.7.2 If no relative minimum is observed (for $A$ less than 90°), increase the polarizer angle $P$ to some intermediate value between 0 and 10° and readjust $A$ (in the range from 0 to 90°) to obtain a relative minimum.

12.7.3 Maintaining $A$ less than 90°, continue to readjust first $A$, then $P$, until the best null is obtained.

12.8 Starting with the null value as determined in 12.7.1 or 12.7.3, maintain $A$ fixed and change $P$ first to a slightly smaller angle and then to a slightly larger angle so that the detector reading in both cases is 10 % greater than the null reading. Record these two polarizer angle values.

12.8.1 Calculate the average of the two values to give the polarizer angle at extinction, $P_0$, in degrees. Record $P_0$.

12.9 Set $P$ at $P_0°$.

12.10 Determine two settings of $A$, one slightly smaller and the other slightly larger than the value in 12.8, that produce photodetector readings 10 % greater than the null reading. Record these two analyzer angle values.

12.10.1 Calculate the average of the two values to give the analyzer angle at extinction, $A_0$, in degrees. Record $A_0$.

12.11 Set $A$ at $180 - A_0°$.

12.12 Increase $P$ starting from $P_0$ to obtain the best null.

12.12.1 Make slight adjustments of $A$ (less than ±5°) with readjustments of $P$ to determine if a better null than that of 12.12 is obtained. Continue to readjust $A$, then $P$, until the best null is obtained.

12.13 Starting with the null as determined in 12.12.1, determine the polarizer extinction value as in steps 12.8 and 12.8.1 and record as $P_0^{\ +}$, in degrees.

12.14 Set $P$ at $P_0^{\ +°}$.

12.15 Determine the analyzer extinction and record as $A_0^{\ +}$, in degrees.

## 13. Calculation

13.1 Calculate the insulator thickness and refractive index from the measured ellipsometer values, $A_0$, $P_0$, $A_0^{\ +}$, $P_0^{\ +}$, the compensator constants $T_c$ (transmission ratio) and $\Delta_c$ (relative phase retardation angle), the angle of incidence, $\phi_0$, (equal to the angle of reflection), the wavelength of light used in the measurement, $\lambda$, and the index of refraction and extinction coefficient of the silicon substrate for the wavelength used.

13.2 Calculate $\Delta$ and $\Psi$ using the procedures of 13.2.1, 13.2.2, or 13.2.3, depending on the compensator constants.

13.2.1 If the compensator is a perfect quarter-wave plate (that is, $T_c = 1.0$, and $\Delta_c = 90 \pm 0.1°$), calculate $\Delta$ and $\Psi$ using the relations:

$$\Delta = P_0 + P_0^{\ +}$$

$$\Psi = 1/2\,[A_0 + (180 - A_0^{\ +})]$$

where $P_0$, $P_0^{\ +}$, $A_0$, and $A_0^{\ +}$ = the polarizer and analyzer settings, in degrees, determined in 12.8.1, 12.13, 12.10.1, and 12.15, respectively.

13.2.2 If the compensator is not a perfect quarter-wave

plate, but has a relative phase retardation, $\Delta_c \neq 90°$, and a transmission ratio, $T_c = 1$, calculate Δ and Ψ using the relations:

$$\tan \Delta = \sin \Delta_c \tan (P_0 + P_0^{+})$$

$$\tan^2 \Psi = - \tan A_0 \tan A_0^{+}$$

where $P_0$, $P_0^{+}$, $A_0$, and $A_0^{+}$ = the polarizer and analyzer settings, in degrees, determined in 12.8.1, 12.13, 12.10.1, and 12.15, respectively.

13.2.3 If the compensator is not a perfect quarter-wave plate with $T_c \neq 1$ and $\Delta_c \neq 90°$, calculate Δ and Ψ using the relations:

$$\tan \Psi \exp (i\Delta) = \frac{\tan A [\tan Q + \rho_c \tan (A - Q)]}{\rho_c \tan Q \tan (p - Q) - 1}$$

$$\rho = T_c \exp (-i\Delta_c)$$

where:

$Q$ = compensator setting = –45°,
$A$ = $A_0$ or $A_0^{+}$ as determined in 12.10.1 or 12.15, degrees, and
$P$ = $O_0$ or $P_0^{+}$ as determined in 12.8.1 or 12.13, degrees.

13.3 If a computer program **(1)** is used to calculate $t_{ins}$ and $n_{ins}$, substitute the values of Δ and Ψ calculated in 13.2.1, 13.2.2, or 13.2.3 and the constants listed in 13.1 into the program **(1).** [10] Use this procedure for the referee method.

NOTE 7—The maximum possible thickness, $t_{max}$, in nanometres, that can be calculated from the computer program is given by the relation:

$$t_{max} = \frac{\lambda}{2[n^2_{ins} - \sin^2 \phi_0]^{1/2}}$$

where:

λ = wavelength of light used in measurements, nm,
$n_{ins}$ = computed index of refraction of the insulator, and
$\phi_0$ = angle of incidence, degrees.

13.3.1 The computed film thickness will lie between 0 and the $t_{max}$ value, and as such is a zero-order calculation result. If through known film growth rates, or an auxiliary measurement, the film thickness is determined to be greater than $t_{max}$, then the total film thickness, $t_{total}$, in nanometres, is given by the relation:

$$t_{total} = t_{ins} + \frac{\lambda N}{2 [n^2_{ins} - \sin^2 \phi_0]^{1/2}}$$

where:

$t_{ins}$ = computed thickness, nm, and
$N$ = integer determined from auxiliary information.

13.3.2 If graphical means (see Fig. 2 and Fig. 3) [7] are used to calculate $t_{ins}$ and $n_{ins}$, locate the values of Δ and Ψ calculated in 13.2.1, 13.2.2, or 13.2.3 on the appropriate chart and determine $\delta_{ins}$, in degrees, and $n_{ins}$ from linear interpolations. Calculate the film thickness, $t_{ins}$, in nanometres, using the relation:

$$t_{ins} = \frac{\lambda \delta_{ins}}{360 [n^2_{ins} - \sin^2 \phi_0]^{1/2}} \quad (1)$$

where:

$\delta_{ins}$, $n_{ins}$ = values obtained from the chart,
λ = wavelength of light used in measurements, nm, and
$\phi_0$ = angle of incidence, degrees.

NOTE 8—The value of $t_{ins}$ calculated by Eq 1 is of zero order. A relation expressing total film thickness is given in Note 6.

NOTE 9—Fig. 2 and Fig. 3 were plotted from values calculated using Ref **(1),** with the specific values of λ = 546.1 nm, $\phi_0$ = 70°, $n_{Si}$ = 4.050, and $K_{Si}$ = 0.028 for Fig. 2 **(4),** and λ = 632.8 nm, $\phi_0$ = 70°, $n_{Si}$ = 3.84, and $K_{Si}$ = 0.020 for Fig. 3 **(5).**

NOTE 10—Values of $n_{Si}$ = 3.875 and $K_{Si}$ = 0.018 are used in the certification of Standard Reference Materials for dry thermal oxides on silicon issued by NIST.

## 14. Report

14.1 The report for nonreferee measurements shall include the following:

14.1.1 Identification of operator,

14.1.2 Date of measurement,

14.1.3 Identification of specimen,

14.1.4 Insulator layer thickness $t_{ins}$, nm, and

14.1.5 Insulator refractive index, $n_{ins}$.

14.2 In addition to the material required in 14.1, the report for referee measurements shall include the following:

14.2.1 Wavelength of light used, nm,

14.2.2 Angle of incidence, deg,

14.2.3 Make and model of ellipsometer,

14.2.4 Constants of compensator,

14.2.5 Sampling plan,

14.2.6 Identification of computer program used,

14.2.7 Insulator thickness, $t_{ins}$, nm, ± its standard deviation (as calculated from the computer program), $\delta_{ins}$, nm, and

14.2.8 Insulator refractive index, $n_{ins}$, ± its standard deviation (as calculated from the computer program), $\delta_{ins}$.

## 15. Precision and Bias

15.1 The single-laboratory precision for the measurement of thickness is estimated to be ±0.1 to ±0.5 nm (1S as defined in Practice E 177) for nonabsorbing films ranging in thickness from a few nanometres to several thousand nanometres when angle measurements are made to ±0.01°.

15.2 The single-laboratory precision for the measurement of refractive index is estimated to be ±0.004 (1S).

15.3 A preliminary estimate of multilaboratory precision has been calculated from the results of the first three laboratories in a multilaboratory round robin. Specimens were $SiO_2$ films grown by a dry thermal process at five different thicknesses. Each laboratory took three measurements on different days for each specimen.

15.3.1 All measurements were made at 632.8 nm wavelength and an angle of incidence of 70°. However, two of the laboratories used settings of the quarter waveplate of + 45° instead of the required – 45°. All measurements were reduced to values of thickness and refractive index by the individual laboratories using a variety of computer software.

15.3.2 Values of estimated multilaboratory precision (R1S) of thickness and refractive index are given in Table 1 as computed from the average of the individual laboratory's reported values. For the four thinnest specimens, the precision

[10] A corrected computer program deck is available from F. L. McCracken, National Institute of Standards and Technology, Washington, DC 20402.

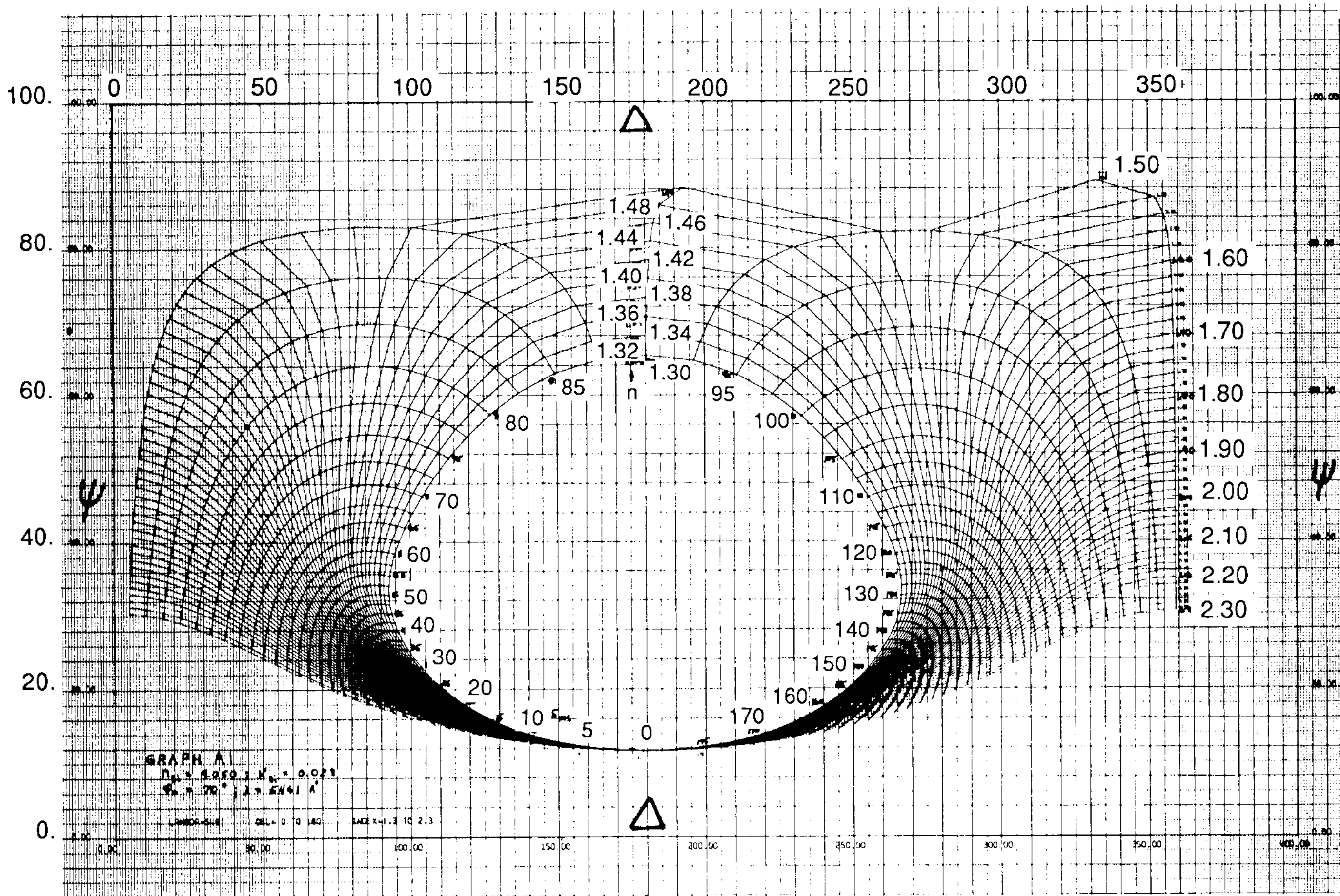


FIG. 2 Graph for Determining $n_{ins}$ and $\delta_{ins}$ from Δ and Ψ for λ = 546.1 nm and an Angle of Incidence = 70° [8]

of the thickness values ranged from 0.2 to 3.1 %, while the precision of refractive index values ranged from 0.08 to 2.2 %. The precision values for the thickest specimen were noticeably poorer, 15.6 % for thickness and 8.17 % for refractive index. This was due to the film thickness giving an optical path length nearly equal to one-half wavelength and consequently acting as a film of near zero thickness.

15.4 A more extensive multilaboratory study of thin layers of silicon dioxide was conducted in 1987–1988. The layers measured were in the thickness range 5 to 55 nm. While a number of the participating laboratories used ellipsometers that operated in modes other than the null mode detailed in this test method, their results are included in this analysis to give a more complete perspective of ellipsometric measurements of dielectric films on silicon. A detailed description of the study is given in Annex A1.

15.4.1 Estimates of the reproducibility (R1s) of multilaboratory average values are as follows: for the parameter, Δ0.35 %, independent of thickness; for the parameter, Ψ0.6 % for films of 5 and 10 nm thickness, and 0.4 % for films that are 20 nm or thicker; for the calculated layer thickness (using a common preset index of refraction)- 3 % for films of 5 and 10 nm thickness, and 1 % for films that are 20 nm or thicker. The larger variability in calculated thickness values than in the ellipsometric parameters is predominantly due to differences in software used for the thickness analyses.

## 16. Keywords

16.1 dielectric thickness; ellipsometry; index of refraction; insulator thickness; refractive index; silicon dioxide

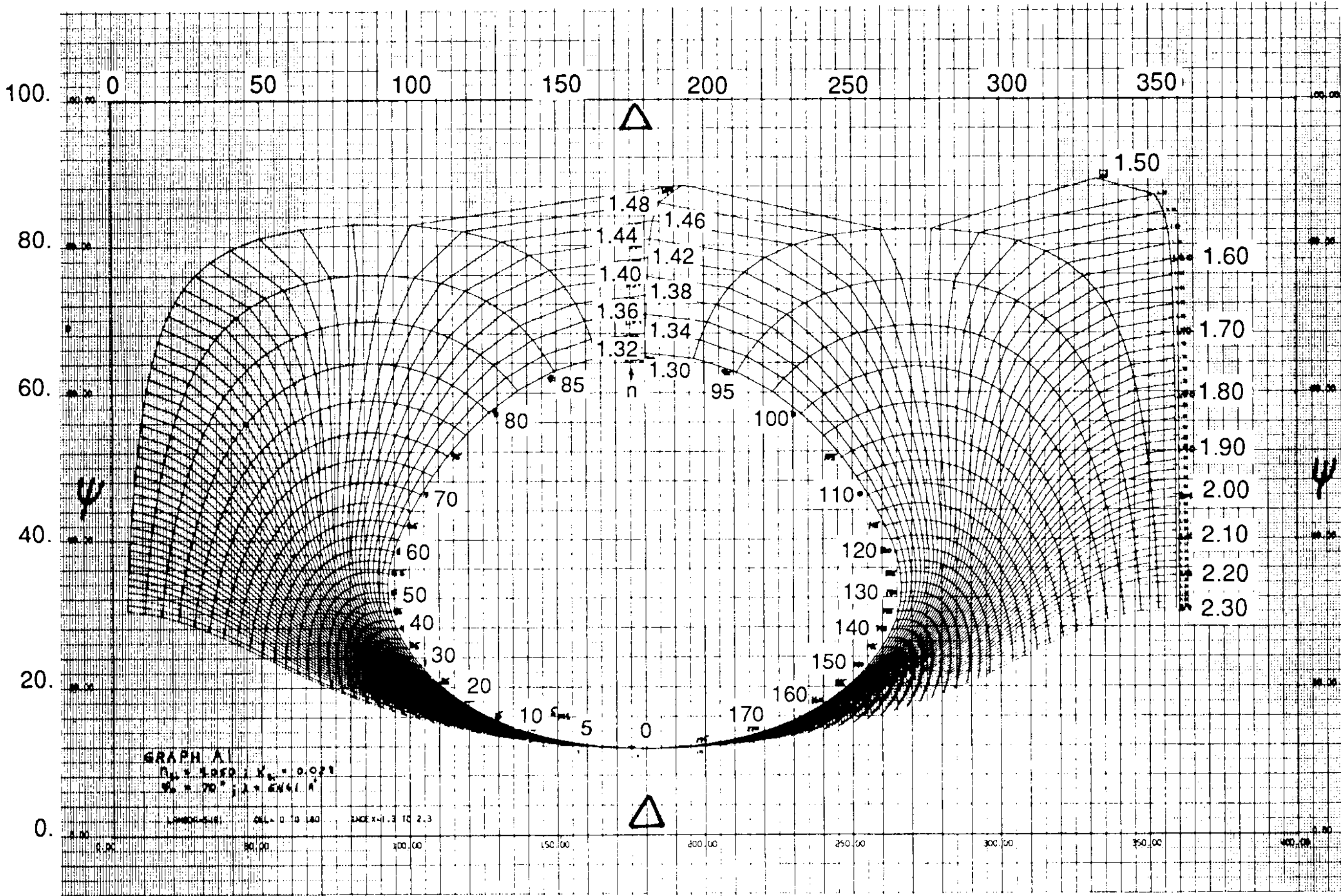


FIG. 3 Graph for Determining $n_{ins}$ and $\delta_{ins}$ from $\Delta$ and $\Psi$ for $\lambda$ = 632.8 nm and an Angle of Incidence = 70° [8]

TABLE 1 Estimates of Multilaboratory Precision Based on the Measurements of Three Laboratories

| | Nominal Film Thickness, nm | | | | |
|---|---|---|---|---|---|
| | 30 | 50 | 70 | 90 | 280 |
| Average thickness, (R1S) | 30.12 (3.1%) | 50.37 (0.2%) | 72.55 (0.7%) | 94.73 (0.2%) | 284.0 (15.6%) |
| Average index, (R1S) | 1.447 (2.2%) | 1.458 (0.08%) | 1.468 (0.38%) | 1.465 (0.18%) | 1.418 (8.17%) |

# ANNEX

## (Mandatory Information)

## A1. DESCRIPTION OF ROUND ROBIN ON 5 TO 55 NM OXIDES

A1.1 Five specimens were used in this study. Four were unpatterned dry thermal oxides on 100 mm (100) *p*-type silicon substrates. These had nominal oxide thicknesses of 5, 10, 20 and 30 nm and were measured at the wafer centers only. The fifth was a nominal 50 nm dry thermal oxide on a 3-in. (100) *p*-type silicon substrate. This specimen was to be measured in the centers of each of two photolithographically defined, 1.25 by 2.0 cm windows (noted as "left" and "right" in the data tables).

A1.2 The multilaboratory test was a spoke and hub design. The specimens were returned to the coordinating laboratory for a standardized cleaning process prior to shipping to the next laboratory. The cleaning consisted of immersion in a solution of 5 g of ammonium persulfate in 2 L of concentrated sulfuric acid heated to 90°C for the cleaning. The immersion was followed by multiple rinsing in deionized water and blow drying in a jet of filtered nitrogen. This cleaning was done just prior to shipment; the laboratories were requested to complete measurement within 2 weeks of receipt of wafers. In the event measurements could not be completed within 2 weeks, laboratories were requested to flush the specimens with fresh ethanol, then deionized water, then to blow dry with filtered nitrogen before starting the first measurement. Each specimen

was to be measured four times with a separate mounting on the ellipsometer for each measurement. The required angle of incidence was 70°; the specified measurement wavelength was 632.8 nm. Both nulltype and rotating analyzer type ellipsometers were allowed.

A1.3 For each set of measurements the values of the ellipsometric parameters, Δ and Ψ were reported. These parameters were used to analyzed with the software normally used by that laboratory to calculate the oxide thickness and index of refraction with an assumed index of refraction for the silicon substrate of $n = 3.866$ and $K = 0.020$. A second calculation of oxide thickness was also done using an assumed index of refraction for the oxide of 1.462.

A1.4 Two laboratories used null ellipsometers with manually adjusted optical components, three laboratories used computer-driven null ellipsometers, four laboratories used computer driven rotating analyzer ellipsometers (no quarter wave plate needed). One laboratory made rotating analyzer type measurements at 70° angle of incidence on a custom-built automated ellipsometer, and also made measurements in the rotating analyzer mode with the angle of incidence set equal to the principal angle for each specimen. (In this latter mode, the reflected beam is circularly polarized, Δ = 90°)

A1.5 Table A1.1 lists the averages and percent standard deviations of the four values of Δ measured by each laboratory. The notation in the column marked "INST" is a key to the instrument used. The numbers 1 to 4 indicate one of the four types described in A1.4, the letter following the number designates differences in model numbers for instrument types 1, 2 and 3, and indicates differences in angle of incidence for the custom-built instrument, Number 4.

A1.6 Table A1.2 lists, for the parameter, Ψ, the same type of information as Table A1.1 lists for Δ.

A1.7 Table A1.3 lists the average thickness values, in nm, and the percent standard deviation for the four thickness values calculated under the assumption of fixed index of refraction (1.462) for the oxide layer. Attempts to calculate both thickness and index values resulted either in "no solution" or in wildly varying results for all software for the thinner oxides.

A1.8 Table A1.4 lists the overall averages and percent standard deviations of the individual laboratory averages. For these calculations, the results of the principal-angle measurements (Laboratory 7) are not included for Δ or Ψ, but are included for the oxide thickness.

A1.9 The variations, from laboratory to laboratory, in the parameters Δ and Ψ are caused both by instrument differences and by changes in the oxide surface condition due to varying time delays after the standard clean and the possible use of the "in-situ" cleaning procedure. The variations in the oxide thickness values are due to these factors and also to differences in software; in particular, users of instrument Type 3 noted that it did not appear possible to input the requested indices of refraction to the third decimal digit specified.

**TABLE A1.1 Averages and Percent Standard Deviation for Delta ( Δ)**

| Laboratory | INST | No. 13 | No. 01 | No. 07 | No. 10 | Left | Right |
|---|---|---|---|---|---|---|---|
| 1 | 2C | 162.900 | 152.610 | 126.090 | 115.330 | 94.455 | 93.465 |
| | | 0.01 | 0.01 | 0.02 | 0.00 | 0.06 | 0.57 |
| 2 | 3B | 164.013 | 153.653 | 125.725 | 115.243 | 94.105 | 93.598 |
| | | 0.02 | 0.11 | 0.03 | 0.10 | 0.03 | 0.18 |
| 3 | 3B | 163.918 | 153.913 | 125.565 | 115.520 | 94.543 | 94.068 |
| | | 0.07 | 0.16 | 0.12 | 0.19 | 0.05 | 0.04 |
| 4 | 2A | 163.910 | 153.900 | 125.390 | 115.430 | 93.940 | 93.430 |
| | | 0.02 | 0.02 | 0.43 | 0.06 | 0.07 | 0.02 |
| 5 | 1A | 164.380 | 154.208 | 126.413 | 115.990 | 94.410 | 94.003 |
| | | 0.11 | 0.05 | 0.08 | 0.04 | 0.06 | 0.05 |
| 6 | 4A | 164.063 | 153.858 | 126.218 | 115.820 | 94.448 | 94.098 |
| | | 0.13 | 0.31 | 0.22 | 0.23 | 0.05 | 0.03 |
| 7 | 4B | 90.373 | 90.160 | 90.065 | 90.155 | 90.098 | 89.763 |
| | | 0.46 | 0.19 | 0.06 | 0.06 | 0.05 | 0.03 |
| 8 | 1B | 163.848 | 153.698 | 125.895 | 115.510 | 94.368 | 93.770 |
| | | 0.06 | 0.06 | 0.08 | 0.02 | 0.03 | 0.02 |
| 9 | 2B | 163.643 | 153.613 | 125.795 | 115.548 | 94.305 | 93.703 |
| | | 0.04 | 0.03 | 0.04 | 0.07 | 0.01 | 0.01 |
| 10 | 1B | 164.253 | 154.443 | 125.185 | 115.175 | 93.838 | 93.323 |
| | | 0.09 | 0.29 | 0.04 | 0.14 | 0.03 | 0.02 |
| 11 | 2B | 163.595 | 153.238 | 125.523 | 115.275 | 93.850 | 93.453 |
| | | 0.08 | 0.05 | 0.02 | 0.05 | 0.02 | 0.02 |

**TABLE A1.2 Averages and Percent Standard Deviation for psi ( Ψ)**

| Laboratory | INST | No. 13 | No. 01 | No. 07 | No. 10 | Left | Right |
|---|---|---|---|---|---|---|---|
| 1 | 2C | 10.775 | 11.340 | 14.325 | 16.445 | 23.340 | 23.498 |
| | | 0.09 | 0.10 | 0.07 | 0.06 | 0.09 | 0.09 |
| 2 | 3B | 10.740 | 11.248 | 14.253 | 16.465 | 23.448 | 23.685 |
| | | 0.00 | 0.09 | 0.07 | 0.16 | 0.04 | 0.34 |
| 3 | 3B | 10.880 | 11.313 | 14.363 | 16.448 | 23.378 | 23.665 |
| | | 0.20 | 0.31 | 0.15 | 0.25 | 0.20 | 0.08 |
| 4 | 2A | 10.680 | 11.200 | 14.250 | 16.300 | 23.290 | 23.560 |
| | | 0.00 | 0.00 | 0.14 | 0.14 | 0.09 | 0.00 |
| 5 | 1A | 10.768 | 11.265 | 14.278 | 16.345 | 23.295 | 23.493 |
| | | 0.05 | 0.05 | 0.07 | 0.04 | 0.11 | 0.06 |
| 6 | 4A | 10.770 | 11.243 | 14.268 | 16.333 | 23.293 | 23.453 |
| | | 0.13 | 0.15 | 0.47 | 0.49 | 0.13 | 0.11 |
| 7 | 4B | 3.083 | 5.135 | 11.878 | 15.058 | 23.265 | 23.438 |
| | | 0.67 | 0.61 | 0.22 | 0.25 | 0.11 | 0.07 |
| 8 | 1B | 10.770 | 11.280 | 14.355 | 16.460 | 23.320 | 23.593 |
| | | 0.21 | 0.18 | 0.20 | 0.00 | 0.09 | 0.11 |
| 9 | 2B | 10.743 | 11.255 | 14.330 | 16.415 | 23.300 | 23.575 |
| | | 0.05 | 0.05 | 0.06 | 0.08 | 0.06 | 0.02 |
| 10 | 1B | 10.648 | 11.135 | 14.260 | 16.315 | 23.228 | 23.475 |
| | | 0.12 | 0.27 | 0.06 | 0.23 | 0.04 | 0.02 |
| 11 | 2B | 10.718 | 11.238 | 14.305 | 16.390 | 23.435 | 23.638 |
| | | 0.09 | 0.22 | 0.04 | 0.09 | 0.02 | 0.04 |

TABLE A1.3 Averages and Percent Standard Deviation for Thickness

| Laboratory | INST | No. 13 | No. 01 | No. 07 | No. 10 | Left | Right |
|---|---|---|---|---|---|---|---|
| 1 | 2C | 57.425 | 96.625 | 224.000 | 296.000 | 514.250 | 520.250 |
| | | 0.09 | 0.16 | 0.00 | 0.00 | 0.10 | 0.10 |
| 2 | 3B | 53.000 | 92.500 | 226.250 | 297.750 | 519.500 | 527.250 |
| | | 0.00 | 0.62 | 0.22 | 0.32 | 0.11 | 0.45 |
| 3 | 3B | 53.250 | 90.500 | 225.750 | 293.500 | 510.750 | 517.750 |
| | | 0.94 | 1.10 | 0.42 | 0.59 | 0.10 | 0.10 |
| 4 | 2A | 53.475 | 90.950 | 224.500 | 293.250 | 515.500 | 523.500 |
| | | 0.18 | 0.11 | 0.45 | 0.17 | 0.19 | 0.11 |
| 5 | 1A | 52.073 | 90.360 | 222.450 | 292.348 | 514.245 | 520.423 |
| | | 1.22 | 0.33 | 0.24 | 0.06 | 0.16 | 0.11 |
| 6 | 4A | 53.100 | 91.375 | 222.850 | 292.625 | 513.600 | 518.800 |
| | | 1.40 | 1.76 | 0.35 | 0.28 | 0.12 | 0.09 |
| 7 | 4B | 52.775 | 90.325 | 222.550 | 292.650 | 513.375 | 518.525 |
| | | 0.78 | 0.63 | 0.26 | 0.26 | 0.12 | 0.09 |
| 8 | 1B | 53.900 | 92.150 | 224.750 | 295.000 | 513.750 | 522.500 |
| | | 0.66 | 0.34 | 0.22 | 0.00 | 0.10 | 0.11 |
| 9 | 2B | 54.488 | 92.305 | 225.288 | 294.928 | 514.493 | 523.258 |
| | | 0.40 | 0.15 | 0.06 | 0.16 | 0.07 | 0.03 |
| 10 | 1B | 52.500 | 89.000 | 229.000 | 297.500 | 521.750 | 529.500 |
| | | 1.10 | 1.83 | 0.00 | 0.34 | 0.10 | 0.11 |
| 11 | 2B | 54.750 | 94.000 | 227.750 | 297.750 | 515.750 | 522.500 |
| | | 0.91 | 0.00 | 0.22 | 0.17 | 0.19 | 0.11 |

TABLE A1.4 Multilaboratory Averages and Percent Standard Deviation

| Parameter | No. 13 | No. 01 | No. 07 | No. 10 | Left | Right |
|---|---|---|---|---|---|---|
| Delta | 163.852 | 153.713 | 125.780 | 115.484 | 94.226 | 93.691 |
| | 0.25 | 0.33 | 0.30 | 0.22 | 0.29 | 0.30 |
| $\Psi$ | 10.749 | 11.252 | 14.309 | 16.392 | 23.333 | 23.563 |
| | 0.58 | 0.51 | 0.30 | 0.39 | 0.30 | 0.35 |
| Thickness | 53.703 | 91.826 | 225.013 | 294.845 | 515.178 | 522.205 |
| | 2.74 | 2.28 | 0.93 | 0.72 | 0.59 | 0.70 |

## REFERENCES


(**1**) McCracken, F. L.," A FORTRAN Program for Analysis of Ellipsometer Measurements," *NBS Technical Note 479*, April 1969.

(**2**) Winterbottom, A. B., *Optical Studies of Metal Surfaces* , I Kommisjon Hos F. Bruns Bokhamdel, Trondheim, 1956.

(**3**) 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," *Journal of Research of the National Bureau of Standards* , JRNBA, Vol 67A, No. 4, 1963, pp. 363–377.

(**4**) Archer, R. J., "Determination of the Properties of Films on Silicon by the Method of Ellipsometry," *Journal of the Optical Society of America*, JOSAA, Vol 52, 1962, pp. 970–977.

(**5**) Philipp, H. R., and Taft, E. A., "Optical Constants of Silicon in the Region 1 to 10 eV," *Physical Review*, PHRVA, Vol 120, 1960, pp. 37–38.



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