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**Standards for Organic Contaminant
Thickness Measurement by the
Ellipsometer**

By L. C. Jackson

Published March 1981

Topical Report

Prepared for the United States Department of Energy
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**Kansas City
Division**

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STANDARDS FOR ORGANIC CONTAMINANT THICKNESS
MEASUREMENT BY THE ELLIPSOMETER

By L. C. Jackson

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Topical Report
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Standards have been established for use in organic contamination thickness measurements by the ellipsometer. The standards are based on silicon oxide films of known thicknesses deposited on silicon wafers. A calibration curve was established correlating delta (a surface optical constant) with silicon oxide thicknesses ranging from 10 to 2700 Å. Linear regression curves were established for the 10- to 100-Å and the 10- to 200-Å thickness ranges; both curves had excellent coefficient of determination correlations of 0.996 and 0.991, respectively. A quadratic equation was established for thickness measurements from 200 to 1000 Å (coefficient of determination = 0.962). The relationship of the silicon oxide thickness standards and organic contaminant thickness measurement is discussed.

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SUMMARY

No film thickness standards have been established for the ellipsometer that can be used for determining the thickness of organic contaminants on electrical components. Thickness measurement of organic contamination on a surface is dependent on the refractive index of the film; knowledge of the refractive index of the film is required before thickness measurements can be made. Silicon oxide has a refractive index equivalent to most organic materials. Silicon oxide, therefore, can be used for developing thickness standards for use in ellipsometer measurement of organic film contamination.

A film thickness standard curve was established by correlating known silicon oxide thicknesses with their corresponding phase change (Δ , an optical constant of the surface). The thickness ranges of 10 to 100 Å and 10 to 200 Å yield linear regression curves with coefficient of determination (quality of fit) values of 0.996 and 0.991, respectively (1.0 is perfect). The coefficient of determination for the thickness range of 200 to 1000 Å is 0.962, using a quadratic equation. Thickness standards up to 2700 Å were established.

DISCUSSION

SCOPE AND PURPOSE

The purpose of this work was to establish film thickness standards for ellipsometer measurement of the thickness of organic contaminant films. The standards were necessary to provide thickness measurement of contaminated surfaces of electrical components.

ACTIVITY

Electrical components have varying degrees of tolerance to organic contamination, depending on the functional sensitivity.¹⁻⁴ The thickness of organic contaminant films that can cause electrical and adhesion failures in many electrical components is less than 200 Å. The ellipsometer can measure the thickness of such thin film contamination if it is present on a reflective metal surface. However, no film thickness standards have been established for use in evaluating the thickness of organic contamination. Silicon oxide, deposited in varying known thicknesses on reflective silicon wafers, can be used to develop standards for organic contamination thickness measurement by the ellipsometer. In selecting standards for organic contaminant thickness measurement, the refractive index of the standard must be equivalent to that of organic materials. Silicon oxide meets this requirement.

Measurement by the Ellipsometer

The ellipsometer can be used for the measurement of the thickness of thin films. The method is based on the measurement of optical changes in the polarization of light reflected from a surface. When polarized light is resolved into two components, one parallel (p wave) to and one normal (s wave) to the plane of incidence, the state of polarization is defined by the phase difference and amplitude ratio. Upon reflection from a surface, a change in the relative phase change (Δ) and amplitude ratio (ψ) results in elliptical polarization. Archer⁵ symbolically defined the relative Δ (Δ) and ψ (Ψ) change as follows.

$$\Delta = (B_p - B_s)_{\text{reflected}} - (B_p - B_s)_{\text{incident}}$$

$$\Psi = \arctan \left(\frac{R_p}{R_s} \cdot \frac{E_s}{E_p} \right)$$

The amplitudes E and R designate the incident and reflected beams and δ the phase angle. The optical properties of a surface are dependent on the angle of incidence, and, for a film-covered surface, the thickness and optical constants of the film. δ , however, can have any value between 0 and 360°, while ψ is limited to values between 0 and 90°. ⁵ The greatest sensitivity to thickness changes is obtained with δ , and, therefore, δ was used for thickness measurement in this work.

The Model L-119 ellipsometer (Gaertner Scientific Corporation, Chicago, IL) was used for this work. A helium-neon laser with a wavelength of 6328 Å was used as a monochromatic light source for the ellipsometer. The angle of incidence for maximum sensitivity is in the 70- to 80-degree range for most systems. ⁶ Most materials will provide a greater spread in data at these angles; 70 degrees, therefore, was used to provide the optimum data for many reflective surfaces.

Silicone Oxide Standards

One of the commonly used methods to determine the thickness of unknown films on surfaces by the ellipsometer is to obtain δ and ψ values for films of different thicknesses and then curve-fit the experimental data to δ and ψ plots, which are computer-generated for various assumed optical constants and thicknesses of silicon oxide films. ^{6,7} Greater accuracy, however, can be obtained by establishing thickness correlations with δ values obtained from known standards.

The refractive index of organic materials differs little from one another, or from a common value of 1.5, because of the carbon-to-carbon backbone. ⁸ This value is equivalent to the refractive index of silicon oxide (1.55). A known thickness of silicon oxide, therefore, can be used to represent the thickness of organic contamination when measured by the ellipsometer.

Vacuum deposition of silicon oxide on silicon wafers was performed by Evaporated Metal Films, Ithaca, NY. A quartz crystal, used as a vacuum deposition monitor, measures the mass of material deposited according to the frequency of the crystal during its oscillation. The frequency change (and a knowledge of the density of the material) is translated into thickness. Repeatability by this method is better than actual thickness accuracy, the latter being 5 percent or less in the 10- to 200-Å range. The ellipsometer data which follows tend to verify this estimate.

Electrical components perform different functions and are subject to adhesion or electrical failures. Component protection is dependent on surface cleanliness to achieve adequate adhesion of the epoxy or urethane encapsulant. Electrical functions, however,

are subject to failure by less amounts, or thickness, of organic contamination than are adhesion functions (encapsulation). Many problems, such as arc over and electrical surface leakage of printed circuit boards; excessive contact loop resistance of electromechanical devices; and poor adhesion in chip, beam lead, and lid bonding in hybrid microcircuits, will occur when organic contamination thickness is less than 100 or 200 Å.

Each class of contacts (static connectors, sliding contacts, relays) have their own operational design and wear characteristics, but all three have the potential common major problem of high contact resistance. In critical contact application, surface films greater than 10 Å may affect reliability while films of 100 Å are completely insulating to contacts operating at less than 1 V.⁴ Within this range, electrical continuity is dependent on the amount of pressure during the mechanical wiping action on the contacts.³ Organic surface films have been shown to play a dominant role in solid phase welding of gold.⁴ The thermocompression gold bonding process is highly temperature dependent when 20 Å of organic films are present; these films can significantly degrade the bonding of hybrid microcircuit lead frames.

This thin film contamination range has been evaluated using the ellipsometer on silicon oxide-coated silicon wafers ranging from 10 to 100 Å in 10-Å increments. A plot of the data is given in Figure 1. The coefficient of determination for a linear regression (straight line) fit for the 10- to 100-Å range is 0.996. The coefficient of determination is an indication of the quality of fit of data to specific types of curves; a perfect fit of data is 1.0, a poor fit is zero. The resultant inverse equation for use in organic contaminant thickness measurements in the 10- to 100-Å range on silicon wafers, therefore, is:

$$X = 496.080 - 3.084 (\Delta).$$

where X = angstroms. The standard deviation found within the 10- to 100-Å range is ± 2.09 Å.

The linear regression curve also can be applied to the expanded range of 10 to 200 Å, but with less precision. The standard deviation found within the 10- to 200-Å range is ± 5.99 Å, based on the inverse equation

$$X = 584.732 - 3.635 (\Delta)$$

which has a coefficient of determination of 0.991.

Included in these formulations are the adjustments for bias of the slope of the lines to compensate for data failing to coincide with the zero point on the x-y axis.

Figure 1. Changes in Phase Differences (Delta) With Known Silicon Oxide Thicknesses

The potential of the ellipsometer to measure thicknesses greater than 200 Å is shown in Figure 2. The nature of an elliptical curve is to reverse itself and describe positive and negative quadrants. The disconnected curve in Figure 2 is related to the nature of trigonometric functions; a continuous, but reversed, curve could be obtained by subtraction from 360 degrees.⁹ However, such a bowl-type curve always will yield two thickness values for one delta value obtained within the range of 10 to 1800 Å. This problem is avoided by using the technique shown in Figure 2. Above 1800 Å, the cycle repeats itself. A quadratic equation is needed for the 200 to 1000 Å portion of the curve.

$$\Delta = A + B_X + C_X^2$$

Figure 2. Correlation of Silicon Oxide Thickness Variations With Delta Values

where

X = thickness in angstroms,

A = 140.036262,

B = -0.158713, and

C = +0.0000847411.

The inverse equation is:

$$X = \frac{-B - \sqrt{B^2 - 4C(\Delta - A)}}{2C}$$

which provides a coefficient of determination of 0.962.

Each metal has its own optical constants of delta and psi values (Figure 3). Many metals will not have as high a refractive index as silicon wafers; these metals therefore will have delta values which are lower than those obtained with silicon surfaces. The difference between the nominal refractive index of organic contaminants and the lower refractive index of various metals (other than silicon) reduces the delta scale and, therefore, the thickness range.

ACCOMPLISHMENTS

Silicon oxide standards have been established for use in measuring the thickness of organic contamination on electrical component surfaces by the ellipsometer. Formulas for calculating organic contaminant thickness have been established for the 10- to 100-Å, 10- to 200-Å, and 200- to 1000-Å ranges.

Figure 3. Optical Constants of Various Metals (Delta-Psi)

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