



sample. The method bias is estimated using the average range of the location deviations from the target for the round-robin results or 2.11 defects/cm².

12 Keywords

12.1 defect density; dislocation; grain boundary; microscopic; polycrystalline imperfection; preferential etch; silicon; slip

RELATED INFORMATION 1

SUMMARY OF MULTI LABORATORY ROUND ROBIN TEST

NOTICE: This related information is not an official part of SEMI MF1810. It was developed as part of the development of the document. This related information was approved for publication by full letter ballot on December 4, 2004.

R1-1 The multilaboratory round robin test was done over a period of 12 months and included 16 laboratories. The test required each laboratory to complete 11 diameter scan measurements of seven wafers. A “diameter scan” area is equal to the area defined by a microscope field of view and a length equal to the wafer diameter less the edge exclusion. Only one defect classification, OISF, was included in the count and described with pictures. The sequence and path of the measurement scans were defined so that the comparison of the individual measurements would be possible among data sets. Within the ability of each laboratory to align the sample on the microscope stage, the starting point and scan path was held constant.

R1-2 The complete repeatability and reproducibility study results are listed in Table R1-1 for the four scan method for the parameter, deviation from target, in units of defects/cm². The average range was 2.107 and the range of \bar{x} -bars was 12.130.

Table R1-1 Repeatability and Reproducibility for 16 Laboratories, Seven Samples, and Two Trials

	<i>Estimated Sigma</i>	<i>Estimated Variance</i>	<i>Percent of Total</i>
Repeatability	1.863	3.472	23.92
Reproducibility	3.323	11.045	76.08
Repeatability and Reproducibility	3.810	14.517	100.00

R1-3 A multiple range test by the 95% least squares deviation (LSD) method for the deviation from target by laboratory was done. Results are shown in Table R1-2. Differences among laboratories are also evident in the 95% confidence interval box and whisker plot provided in Figure R1-1, which shows the deviation from target for each laboratory. This plot is based on four diameter scans per test.

R1-4 Results from Laboratory F were significantly different from all other laboratories, but were included

in the reported Precision and Bias Section because no evidence pointed to a procedural mistake. The alternative repeatability and reproducibility study results are listed in Table R1-3 for the four scan method for the parameter, deviation from target, in units of defects/cm². In this case, the average range was 1.930 and the range of \bar{x} -bars was 7.018.

Table R1-2 Multiple Range Test for 16 Laboratories

<i>Laboratory</i>	<i>Count</i>	<i>Least Squares Mean</i>	<i>Homogeneous Groups</i>
H	77	-0.948022	X
C	77	-0.948002	X
A	77	-0.868478	XX
O	77	-0.635124	XXX
M	77	-0.469934	XX
J	77	-0.437819	X
I	77	-0.28686	XX
L	77	-0.261265	XX
N	77	0.064319	XX
P	77	0.176689	XX
B	77	0.403837	XX
E	77	0.406758	XX
K	77	0.425747	XXX
D	77	0.549704	XX
G	77	0.816097	X
F	77	2.01237	X

Table R1-3 Repeatability and Reproducibility for 16 Laboratories, Seven Samples, and Two Trials

	<i>Estimated Sigma</i>	<i>Estimated Variance</i>	<i>Percent of Total</i>
Repeatability	1.707	2.912	44.08
Reproducibility	1.922	3.695	55.92
Repeatability and Reproducibility	2.570	6.607	100.00

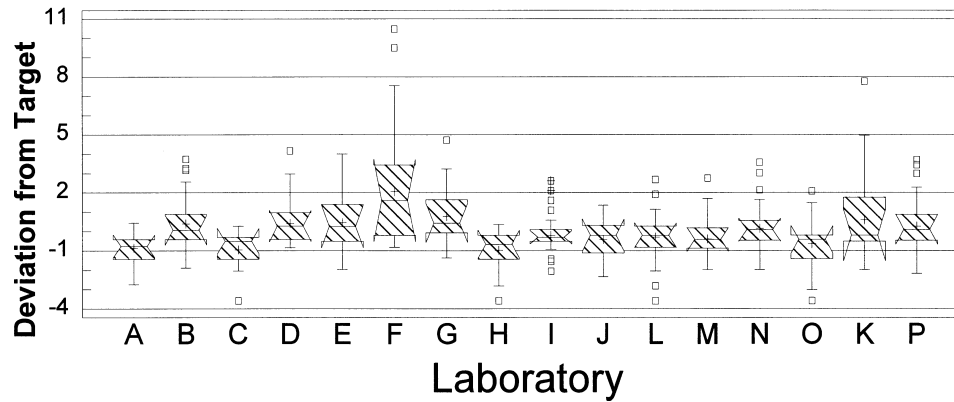


Figure R1-1
95% Confidence Interval Box and Whisker Plot for 16 Laboratories

NOTICE: SEMI makes no warranties or representations as to the suitability of the standards set forth herein for any particular application. The determination of the suitability of the standard is solely the responsibility of the user. Users are cautioned to refer to manufacturer' s instructions, product labels, product data sheets, and other relevant literature, respecting any materials or equipment mentioned herein. These standards are subject to change without notice.

By publication of this standard, Semiconductor Equipment and Materials International (SEMI) takes no position respecting the validity of any patent rights or copyrights asserted in connection with any items mentioned in this standard. Users of this standard are expressly advised that determination of any such patent rights or copyrights, and the risk of infringement of such rights are entirely their own responsibility.

SEMI MF1811-0704

GUIDE FOR ESTIMATING THE POWER SPECTRAL DENSITY FUNCTION AND RELATED FINISH PARAMETERS FROM SURFACE PROFILE DATA

This guide was technically approved by the Global Silicon Wafer Committee and is the direct responsibility of the North American Silicon Wafer Committee. Current edition approved for publication by the North American Regional Standards Committee on March 14, 2004. Initially available at www.semi.org May 2004; to be published July 2004. Original edition published by ASTM International as ASTM F 1811-97. Last previous edition SEMI MF1811-97(2002).

1 Purpose

1.1 There is some confusion in the roughness-measurement community concerning the use of estimators and the calculation of power spectral densities (*PSDs*) from discrete data sets. Use of this guide can eliminate these differences and result in the use of consistent units for the *PSD* and related parameters. It also provides a uniform reporting procedure for digital roughness data that can facilitate communication between different workers and different laboratories.

2 Scope

2.1 This guide defines the methodology for calculating a set of commonly used statistical parameters and functions of surface roughness from a set of measured surface profile data. Its purposes are to provide fundamental procedures and notation for processing and presenting data, to alert the reader to related issues that may arise in user-specific applications, and to provide literature references where further details can be found.

2.2 This guide is limited to the analysis of one-dimensional or profile data taken at uniform intervals along straight lines across the surface under test, although reference is made to the more general case of two-dimensional measurements made over a rectangular array of data points.

2.3 The data analysis procedures described in this guide are generic and are not limited to specific surfaces, surface-generation techniques, degrees of roughness, or measuring techniques. Examples of measuring techniques that can be used to generate profile data for analysis are mechanical profiling instruments using a rigid contacting probe, optical profiling instruments that sample over a line or an array over an area of the surface, optical interferometry, and scanning-microscopy techniques such as atomic-force microscopy. The distinctions between different measuring techniques enter this guide through various parameters and functions that are defined in Sections 4 and 5, such as their sampling intervals, bandwidths, and measurement transfer functions.

2.4 The primary interest here is the characterization of random or periodic aspects of surface finish rather than isolated surface defects such as pits, protrusions, scratches or ridges. Although the methods of data analysis described here can be equally well applied to profile data of isolated surface features, the parameters and functions that are derived using the procedures described in this guide may have a different physical significance than those derived from random or periodic surfaces.

2.5 The statistical parameters and functions that are discussed in this guide are, in fact, mathematical abstractions that are generally defined in terms of an infinitely-long linear profile across the surface, or the “ensemble” average of an infinite number of finite-length profiles. In contrast, real profile data are available in the form of one or more sets of digitized height data measured at a finite number of discrete positions on the surface under test. This guide gives both the abstract definitions of the statistical quantities of interest, and numerical procedures for determining values of these abstract quantities from sets of measured data. In the notation of this guide these numerical procedures are called “estimators” and the results that they produce are called “estimates”.

2.6 This guide gives “periodogram” estimators for determining the root-mean-square (rms) roughness, rms slope, and power spectral density (PSD) of the surface directly from profile height or slope measurements. The statistical literature uses a circumflex to distinguish an estimator or estimate from its abstract or ensemble-average value. For example, \hat{A} denotes an estimate of the quality *A*. However, some word-processors cannot place a circumflex over consonants in text. Any symbolic or verbal device may be used instead.

2.7 The quality of estimators of surface statistics are, in turn, characterized by higher-order statistical properties that describe their “bias” and “fluctuation” properties with respect to their abstract or ensemble-average versions. This guide does not discuss the higher-order statistical properties of the estimators given here since their practical significance and use are application-specific and beyond the scope of this document.

Details of these and related subjects can be found in the references listed in Section 7.

2.8 Raw measured profile data generally contain trending components that are independent of the microtopography of the surface being measured. These components must be subtracted before the difference or residual errors are subjected to the statistical-estimation routines given here. These trending components originate from both extrinsic and intrinsic sources. Extrinsic trends arise from the rigid-body positioning of the part under test in the measuring apparatus. In optics these displacement and rotation contributions are called “piston” and “tilt” errors. In contrast, intrinsic trends arise from deliberate or accidental shape errors inherent in the surface under test, such as a circular or parabolic curvature. In the absence of a-priori information about the true surface shape, the intrinsic shape error is frequently limited to a quadratic (parabolic) curvature of the surface. Detrending of intrinsic and extrinsic trends is generally accomplished simultaneously by subtracting a detrending polynomial from the raw measured data, where the polynomial coefficients are determined by least-squares fitting to the measured data.

2.9 Although surfaces and surface measuring instruments exist in real or configuration space, they are most easily understood in frequency space, also known as Fourier transform, reciprocal or spatial-frequency space. This is because any practical measurement process can be considered to be a “linear system”, meaning that the measured profile is the convolution of the true surface profile and the impulse response of the measuring system; and equivalently, the Fourier-amplitude spectrum of the measured profile is the product of that of the true profile and the frequency-dependent “transfer function” of the measurement system. This is expressed symbolically by the following equation:

$$A_{meas}(f_x) = A_{true}(f_x)T(f_x) \quad (1)$$

where:

A = the Fourier amplitudes,

$T(f_x)$ = instrument response function or the measurement transfer function, and

f_x = surface spatial frequency.

This factorization permits the surface and the measuring system to be discussed independently of each other in frequency space, and is an essential feature of any discussion of measurement systems.

2.10 Figure 1 sketches different forms of the measurement transfer function, $T(f_x)$:

2.10.1 Case (a) is a perfect measuring system, which has $T(f_x) = 1$ for all spatial frequencies, $0 \leq f_x \leq \infty$. This is unrealistic since no real measuring instrument is equally sensitive to all spatial frequencies.

2.10.2 Case (b) is an ideal measuring system, which has $T(f_x) = 1$ for $LFL \leq f_x \leq HFL$ and $T(f_x) = 0$ otherwise, where LFL and HFL denote the low-frequency and high-frequency limits of the measurement. The range $LFL \leq f_x \leq HFL$ is called the bandpass or bandwidth of the measurement, and ratio HFL/LFL is called the dynamic range of the measurement.

2.10.3 Case (c) represents a realistic measuring system, since it includes the fact that $T(f_x)$ need not be unity within the measurement bandpass or strictly zero outside the bandpass.

2.11 If the measurement transfer function is known to deviate significantly from unity within the measurement bandpass, the measured power spectral density (PSD) can be transformed into the form that would have been measured by an instrument with the ideal rectangular form through the process of digital “restoration.” In its simplest form restoration involves dividing the measured PSD by the known form of $|T(f_x)|^2$ over the measurement bandpass. Restoration is particularly relevant to measuring instruments that involve optical microscopes since the transfer functions of microscope systems are not unity over their bandpass but tend to fall linearly between unity at $T(0) = 1$ and $T(HFL) = 0$. The need for, and methodology of digital restoration is instrument specific and this guide places no requirements on its use.

2.12 This guide requires that any data on surface finish parameters or functions generated by the procedures described herein be accompanied by an identifying description of measuring instrument used, estimates of its low- and high-frequency limits, LFL and HFL , and a statement of whether or not restoration techniques were used.

2.13 In order to make a quantitative comparison between profile data obtained from different measurement techniques, the statistical parameters and functions of interest must be compared over the same or comparable spatial-frequency regions. The most common quantities used to compare surfaces are their root-mean-square (rms) roughness values, which are the square roots of the areas under the PSD between specified surface-frequency limits. Surface statistics derived from measurements involving different spatial-frequency ranges cannot be compared quantitatively except in an approximate way. In some cases measurements with partially or even nonoverlapping bandwidths can be compared by using analytic models

of the *PSDs* to extrapolate the *PSDs* outside their measurement bandwidth.

2.14 Examples of specific band-width limits can be drawn from the optical and semiconductor industries. In optics the so-called total integrated scatter or TIS measurement technique leads to rms roughness values involving an annulus in two-dimensional spatial frequencies space from 0.069 to 1.48 μm^{-1} ; that is, a dynamic range of $1.48/0.069 = 21/1$. In contrast, the range of spatial frequencies involved in optical and mechanical scanning techniques are generally much larger than this, frequently having a dynamic ranges of 512/1 or more. In the latter case the subrange of 0.0125 to 1 μm^{-1} has been used to discuss the rms surface roughness in the semiconductor industry. These numbers are provided to illustrate the magnitudes and ranges of *HFL* and *LFL* encountered in practice but do not constitute a recommendation of particular limits for the specification of surface finish parameters. Such selections are application dependent, and are to be made at the users' discretion.

2.15 The limits of integration involved in the determination of rms roughness and slope values from measured profile data are introduced by multiplying the measured *PSD* by a factor equal to zero for spatial frequencies outside the desired bandpass and unity within the desired bandpass, as shown in Case (b) in Figure 1. This is called a top-hat or binary filter function. Before the ready availability of digital frequency-domain processing as employed in this guide, bandwidth limits were imposed by passing the profile data through analog or digital filters without explicitly transforming them into the frequency domain and multiplying by a top-hat function. The two processes are mathematically equivalent, providing the data filter has the desired frequency response. Real data filters, however, frequently have Gaussian or *RC* forms that only approximate the desired top-hat form that introduces some ambiguity in their interpretation. This guide recommends the determination of rms roughness and slope values using top-hat windowing of the measured *PSD* in the frequency domain.

2.16 The *PSD* and rms roughness are surface statistics of particular interest to the optics and semiconductor industries because of their direct relationship to the functional properties of such surfaces. In the case of rougher surfaces these are still valid and useful statistics, although the functional properties of such surfaces may depend on additional statistics as well. The ASME Standard on Surface Texture, B46.1, discusses additional surface statistics, terms, and measurement methods applicable to machined surfaces.

2.17 The units used in this guide are a self-consistent set of SI units that are appropriate for many

measurements in the semiconductor and optics industry. This guide does not mandate the use of these units, but does require that results expressed in other units be referenced to SI units for ease of comparison.

NOTICE: This standard does not purport to address safety issues, 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 or other limitations prior to use.

3 Referenced Standards

3.1 SEMI Standards

SEMI ME1392 — Practice for Angle Resolved Optical Scatter Measurements on Specular or Diffuse Surfaces

SEMI MF1048 — Test Method for Measuring the Effective Surface Roughness of Optical Components by Total Integrated Scattering

3.2 ASTM Standard

E 284 — Terminology Relating to Appearance of Materials¹

3.3 ANSI Standard

ANSI/ASME B46.1 — Surface Texture (Surface Roughness, Waviness and Lay)²

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

4 Terminology

4.1 *Introduction* — This section provides the definitions of special terms used in this guide, and includes the mathematical definitions of different profile statistics in terms of continuous, infinitely-long profiles. The corresponding estimators of those statistics based on linear, sampled, finite-trace-length data are given in Section 5. Definitions of terms not included here can be found in SEMI ME1392, SEMI MF1048, ASTM Terminology E 284, or ANSI/ASME B46.1.

4.2 Definitions

4.2.1 *averaging, aperture or local* — smoothing of an estimate of the power spectral density function (*PSD*) by replacing its value at a given spatial frequency by its

1 *Annual Book of ASTM Standards*, Vol 6.01, ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428. Telephone: 610-832-9500, Fax: 610-832-9555, Website: www.astm.org

2 Available from the American National Standards Institute, New York Office: 11 West 42nd Street, New York, NY 10036, USA. Telephone: 212.642.4900; Fax: 212.398.0023 Website: <http://www.ansi.org>.

average over a local frequency range using a particular weighting function.

4.2.1.1 Discussion — The averaged quantities must include the same range of surface spatial frequencies.

4.2.2 averaging, data — numerical averaging of statistical estimates of the PSD, the mean-square surface roughness or the mean-square profile slope derived from different measurements, in order to obtain a single, composite result.

4.2.2.1 Discussion — For example, a rectangular or square array of measurements can be separated into a set of parallel profile measurements which can be analyzed separately and the results averaged. Again, the averaged quantities must include the same range of surface spatial frequencies.

4.2.3 bandwidth, bandwidth limits — range of surface spatial frequencies included in a measurement or specification.

4.2.3.1 Discussion — Bandwidth is specified by a high-frequency limit (*HFL*) and a low-frequency limit (*LFL*). The bandwidth and the measurement transfer function over the bandwidth must be taken into account when measurements or statistical properties are compared. Different measuring instruments are generally sensitive to different ranges of surface spatial frequencies; that is, they have different bandwidth limits. Real bandwidth limits are necessarily finite since no measuring instrument is sensitive to infinitely-low or to infinitely-high surface spatial frequencies.

4.2.4 bias error — average deviation between an estimate of a statistical quantity and its true value.

4.2.4.1 Discussion — The periodogram estimator of the power spectral density (*PSD*) given in this guide is a zero-bias or unbiased estimator of the *PSD*. On the other hand, local averaging of the periodogram can introduce bias errors in regions where the spectrum varies rapidly with frequency.

4.2.5 deterministic profile — surface profile that is a known function of surface position, with no random dependencies on position.

4.2.5.1 Discussion — In contrast, a random profile is known only in terms of a probability distribution function.

4.2.6 detrended profile, $Z_d(x)$ — the raw or measured profile after removing instrumental and surface trends. The detrended profile is the input for the statistical estimation routines described in Section 5.

4.2.6.1 Discussion — If the parametric form of the trend is known, its least-squares-fitted form can be subtracted from the measured profile data. Otherwise a

generic power-series form can be used. This guide describes the procedures for removing a zero-, first- or second-order polynomial in the trace distance. A zero-order polynomial removes piston; a first-order polynomial removes piston and tilt; and a second-order polynomial removes piston, tilt and quadratic curvature. In each case the detrended data set has zero mean. The coefficients of constant and linear terms correspond to the rigid-body orientation of the part being measured and need not be recorded. However, the coefficient of the quadratic term represents the intrinsic curvature of the surface being measured and should be recorded.

4.2.7 dynamic range — ratio of the high- to low-frequency limits of the bandwidth of a given measurement technique.

4.2.7.1 Discussion — The ratio *HFL/LFL* is a useful single-number characteristic of a measuring apparatus. It completely describes the measurement effects on surfaces with power-law power spectra.

4.2.8 ensemble — infinitely large collection (infinite ensemble) of quantities, the properties of which are governed by some statistical distribution law.

4.2.8.1 Discussion — For example, ensembles include surface profiles, and rms roughness values.

4.2.9 ensemble average value — value of a particular surface parameter or function averaged over the appropriate distribution functions.

4.2.9.1 Discussion — The ensemble average value of the quantity *A* is denoted by $\langle A \rangle$. Estimates of ensemble-average quantities based on a finite collection of measurements (finite ensemble) can deviate from their infinite-ensemble values by fluctuation and bias errors.

4.2.10 estimate — ensemble-average value of a roughness statistic from a finite set of measured profile data.

4.2.10.1 Discussion — In this guide, a circumflex is used to distinguish estimates from the corresponding ensemble-average quantities (see also Section 2.6).

4.2.11 estimator — algorithm or mathematical procedure for calculating an “estimate.”

4.2.12 fast fourier transform or FFT — algorithm for calculating the Fourier transform (discrete Fourier transform or *DFT*) of a set of numerical data.

4.2.12.1 Discussion — The discovery of the *FFT* is generally attributed to Cooley and Tukey, although it was used and reported in the earlier literature by a number of others, including Gauss, two centuries before. It is now ubiquitous and can be found in any

computer data analysis package (see Section 5.4.2 for details).

4.2.13 *finish parameters and functions* — numbers or functions that characterize surface height fluctuations.

4.2.13.1 *Discussion* — The values and forms of finish parameters and functions may vary depending on the bandwidth of surface frequencies that they contain, and the shapes of the transfer functions of the measurement instruments involved. These quantities are represented by their ensemble-average values derived from measurements using specific estimation routines. In general, the finish parameters and functions of an area are different from those of profiles taken across the surface. In the case of surfaces that are statistically isotropic, however, the area and profile statistics have a one-to-one relationship. Except for incidental remarks, this guide is concerned exclusively with the properties of surface profiles.

4.2.14 *fluctuation error* — general term denoting the deviation of a quantity from its mean, average or detrended value.

4.2.14.1 *Discussion* — Fluctuation errors are usually measured in terms of their mean-square or rms values. For example, R_q is the rms fluctuation error in the surface height and Δ_q is the rms fluctuation error in the profile slope. In turn, the estimates of R_q and Δ_q have their own fluctuation errors. The magnitudes of these higher fluctuation errors not discussed in this guide.

4.2.15 *high-frequency limit (HFL)* [$1/\mu\text{m}$] — highest spatial frequency contained in a profile data set or specification.

4.2.15.1 *Discussion* — The *HFL* of a measurement is determined by the details of the measurement process, and its value in specifications is determined by the user. If the sampling interval in the measurement process is D , the extreme value of the *HFL* is given by the Nyquist criterion, $HFL = 1/(2D)$. However, other electrical, mechanical, or optical filtering mechanisms may further limit the *HFL*. Examples of such mechanisms are the stylus tip radius, projected measurement pixel size, optical resolution, and electrical and digital filters, all of which contribute to the high-frequency roll-off of the instrument transfer function. If the Nyquist frequency is used to determine the *HFL*, care should be taken to determine that the true *HFL* is not reduced by these additional mechanisms.

4.2.16 *intrinsic surface or finish parameters* — characteristics such as the rms roughness or rms slope that contain all surface spatial frequencies from zero to infinity.

4.2.16.1 *Discussion* — Intrinsic parameters are statistical abstractions that cannot be measured or estimated directly since real measurements are sensitive to only limited ranges of surface spatial frequencies. They can, however, be inferred from real measurements by augmenting measurements with *a-priori* information about very low and very high spatial frequencies contained in physically-based models of the *PSDs* of the surfaces involved. All measured finish parameters are finite, but their corresponding intrinsic values need not be. The important distinction between intrinsic and measured (band-width limited) finish parameters is not always made in the literature.

4.2.17 *impulse response, of a profile measuring system* — measured shape of an impulse or infinitely-sharp ridge lying perpendicularly to the profile direction.

4.2.17.1 *Discussion* — In the case of a linear measuring system the impulse response is the Fourier transform of the system transfer function. The impulse response of a perfect measuring system would be an infinitely sharp spike or delta function. In contrast, the impulse response of real measuring systems has a finite width.

4.2.18 *isotropic surface* — surface whose intrinsic finish parameters and functions are independent of the rotational position of the surface about its surface normal. Also known as *statistically-isotropic surface*.

4.2.18.1 *Discussion* — The rms roughness of profiles taken across an isotropically rough surface is independent of the profile directions, and equals the rms roughness of the surface area. The rms slope of an isotropically rough surface is also independent of the profile direction and equals $1/\sqrt{2}$ of the rms area gradient. The one-dimensional or profile power spectrum of an isotropic surface is also independent of the direction of the profile on the surface, and is related to the two-dimensional spectrum of the surface area by an integral transform. Examples of this are given in Section 4.2.41.

4.2.19 *linear systems, linear measurement system* — a signal-processing concept more precisely described as a linear, shift-invariant system.

4.2.19.1 *Discussion* — For the present purposes, a linear measurement of the surface profile is the true profile convolved with the impulse response of the measuring system, or equivalently, the Fourier amplitude spectrum of the measurement is the true amplitude spectrum times the measurement transfer function as indicated in Section 2.9. All practical measurement systems are taken to be linear over their operating ranges.

4.2.20 *low-frequency limit (LFL)* [$1/\mu\text{m}$] — lowest spatial frequency contained in a profile data set or specification.

4.2.20.1 *Discussion* — The minimum *LFL* in a profile measurement is the reciprocal of the length of the surface profile. The estimated value of the *PSD* at this value of the *LFL* is generally attenuated by the detrending process. To avoid this effect the lowest practical *LFL* is sometimes taken to be 3 to 5 times the reciprocal of the scan length. The *LFL* in surface specifications is determined by the user.

4.2.21 *mean-square profile roughness*, R_q^2 [nm^2] — ensemble-average value of the square of the height of the detrended profile.

4.2.21.1 *Discussion* — This value is given by the following relationship:

$$R_q^2 = \lim_{L \rightarrow \infty} \frac{1}{L} \int_{-L/2}^{+L/2} (Z_d(x))^2 dx = \int_0^{+\infty} S_1(f_x) df_x \quad (2)$$

where:

$Z_d(x)$ = detrended surface profile, and

$S_1(f_x)$ = its power spectral density.

The intrinsic value of the mean-square roughness of an isotropically-rough surface area equals the mean-square roughness of any profile across it. The rms roughness, R_q , is distinct from the arithmetic-average roughness, R_a . The two are only related through a specific height-distribution function. For example, for a Gaussian height distribution,

$$R_a = \sqrt{\frac{2}{\pi}} R_q = 0.798 R_q \quad (3)$$

Note that the optics literature uses the symbol σ for R_q .

4.2.22 *mean-square profile slope*, Δ_q^2 [units of choice] — average value of the square of the slope of the detrended profile.

4.2.22.1 *Discussion* — This expression, given by

$$\Delta_q^2 = \lim_{L \rightarrow \infty} \frac{1}{L} \int_{-L/2}^{+L/2} \left(\frac{dz_d}{dx} \right)^2 dx = \int_0^{+\infty} (2\pi f_x)^2 S_1(f_x) df_x \quad (4)$$

assumes that the average slope has been removed in the detrending process. The integrand in the frequency integral on the right can be viewed as the slope power spectral density. The mean-square surface slope of an isotropically-rough two-dimensional surface is half the mean-square gradient of the surface itself.

4.2.23 *measured profile parameters and functions* — quantities derived from detrended profile data that

include the bandwidth and transfer function effects of the particular measurement system used.

4.2.23.1 *Discussion* — Measured profile parameters and functions can be used for comparing surfaces quality providing the same measurement system is used in all cases. In order to compare quantitative measurements made by different measurement systems, or to estimate intrinsic surface properties, the system bandwidths and transfer functions must be taken into account. In the early literature, measurement systems were taken to be “perfect” in the sense of Section 2.10.1, and the effects of their bandwidths and transfer functions were ignored.

4.2.24 *Nyquist frequency* [$1/\mu\text{m}$] — spatial frequency equal to the reciprocal of twice the sampling interval. See Section 4.2.15.1.

4.2.24.1 *Discussion* — The Nyquist frequency represents the highest undistorted frequency involved in a series of uniformly-spaced profile measurements. Higher-frequency components in the surface appear at lower-frequencies through the process of aliasing. Unless the effects of aliasing are removed by anti-aliasing mechanisms in the measurement process, they corrupt the measured spectrum immediately below the Nyquist frequency. In that case the *HFL* should be taken to be a factor of 3 to 5 below the Nyquist frequency.

4.2.25 *periodic random roughness* — modified version of purely periodic roughness that has a definite fundamental spatial frequency, but random variations in its phase or amplitude (see Section 4.2.26).

4.2.26 *periodic roughness* — roughness with a definite fundamental spatial frequency.

4.2.26.1 *Discussion* — The power spectra of periodic and periodic random roughness appear as isolated peaks in the power spectral density function. This pattern is distinct from the broad variations appearing for purely random surfaces. Random surfaces can be viewed as periodic surfaces with a broad distribution of fundamental periods.

4.2.27 *periodogram estimate* — estimates of particular finish parameters that are derived from the periodogram estimator for the power spectrum.

4.2.28 *periodogram estimator* — a particular estimator for the power spectral density that is proportional to the square magnitude of the discrete Fourier transform of the detrended data set.

4.2.28.1.1 *Discussion* — The periodogram is the estimator for the PSD that is used in this guide. The signal-processing literature contains many different estimators of the power spectrum in addition to the

periodogram. In general, they differ from the periodogram in that they incorporate different types and degrees of *a-priori* physical or mathematical information about the original data set. The periodogram, in contrast, includes the maximum number of degrees of freedom and is always used for first-cut evaluation and analysis. Details of the correlation and other spectral estimation methods are discussed in the literature listed in Section 7. A mathematical variant of the periodogram estimator is the correlation method. This is a two-step process that requires the estimation of an intermediate function, the autocovariance function, which is then Fourier transformed to obtain the periodogram estimate of the power spectrum. This method is not discussed in this guide since it is indirect, but when properly applied gives identically the same results as the direct transform method recommended in this guide.

4.2.29 power spectral density (PSD) — statistical function that shows how the mean-square (rms)² of a given quantity is distributed among the various surface spatial frequencies inherent in the profile height. Also known as *power spectrum*.

4.2.29.1 Discussion — The two conventional measures of surface roughness, R_a and R_q do not carry any information about the transverse scale of the surface roughness. That is, they are independent of how much the surface profile is squeezed or stretched parallel to the surface plane. The *PSD* is the simplest statistic that carries that important additional information.

4.2.29.2 profile or one-dimensional PSD of the surface height, [μm^3] — a function of the spatial frequency, f_x , in units of inverse micrometers, μm^{-1} , defined as follows:

$$S_1(f_x) = \lim_{L \rightarrow \infty} \left(\frac{2}{L} \left| \int_{-L/2}^{+L/2} Z(x) e^{i2\pi f_x x} dx \right|^2 \right), f_x > 0 \quad (5)$$

4.2.29.2.1 Discussion — The subscript “x” on “ f_x ” corresponds to the direction of the profile on the surface and can be omitted if no confusion is involved. In this definition the spatial frequency, f_x , is always positive and greater than zero. The value at $f_x = 0$ corresponds to the average value of the profile height, which is zero for detrended profiles. The factor of 2 accounts for the equal contribution from negative frequencies and ensures that the area under the positive-frequency profile spectrum equals the rms-squared (mean-square) profile height.

4.2.29.3 area or two-dimensional PSD of the surface height [μm^4] — a function of the spatial frequencies in both the x and y directions on the surface, f_x and f_y , in units of inverse micrometers, μm^{-1} , defined as follows:

$$S_2(f_x, f_y) = \lim_{A \rightarrow \infty} \left(\frac{1}{A} \left| \iint_A Z(x, y) \exp\{i2\pi(f_x x + f_y y)\} dx dy \right|^2 \right), -\infty < f_x, f_y < +\infty \quad (6)$$

4.2.29.3.1 Discussion — The spatial frequency ranges included in this definition cover the entire frequency plane and are not limited to positive frequencies only as in the case of the profile spectrum. In the case of an isotropically rough surface the area spectrum is a function only of the magnitude of the two-dimensional frequency vector: $f = \sqrt{f_x^2 + f_y^2}$. The profile spectrum can be derived from the area spectrum, but the area spectrum cannot, in general, be derived from the profile spectrum. Uniaxial and isotropically rough surfaces are exceptions.

4.2.29.4 Discussion of Units: The surface height fluctuations of optical surfaces are usually measured in units of nanometers ($1 \text{ nm} = 10^{-3} \mu\text{m}$), or the non-SI units of Ångströms ($1 \text{ Å} = 10^{-4} \mu\text{m}$). Values of the *PSDs* estimated using height data in these units can be converted to the recommended units by multiplying by the following conversion factors:

- To convert S_1 in units of $\text{nm}^2 \mu\text{m}$ to units of μm^3 multiply it by 10^{-6} ,
- To convert S_1 in units of $\text{Å}^2 \mu\text{m}$ to units of μm^3 multiply it by 10^{-8} ,
- To convert S_2 in units of $\text{nm}^2 \mu\text{m}^2$ to units of μm^4 multiply it by 10^{-6} , and
- To convert S_2 in units of $\text{Å}^2 \mu\text{m}^2$ to units of μm^4 multiply it by 10^{-8} .

If the sample interval is given in millimeters instead of micrometers, the conversion factors for S_1 should be multiplied by an additional factor of 10^3 , and those for S_2 should be multiplied by an additional factor of 10^6 .

4.2.30 radius of curvature, \hat{R}_x — radius of a circle fitted to the measured surface profile.

4.2.30.1 Discussion — When the radius is large relative to the profile length its magnitude is most easily determined from the quadratic term in the detrending polynomial. If the average surface profile is written as $Z(x) = a + bx + cx^2$, the estimate of the radius of curvature in the x direction is $\hat{R}_x = 1/(2c)$. If Z and x are expressed in micrometers, \hat{R}_x is in micrometers. Since the radii of curvature of nominally flat surfaces can be quite large, other reporting units, such as meters or kilometers, may be more appropriate.

4.2.31 random surface profile — surface height profile that involves parameters that are distributed according to statistical distribution laws rather than having fixed

or deterministic values. Also known as *random roughness*.

4.2.31.1 *Discussion* — For example, the profile

$$Z(x) = A \cos(2\pi f_x x + \phi) \quad (7)$$

is deterministic if $\phi = \text{const.}$, but random if ϕ has a finite-width probability distribution function $P(\phi)$. Finish parameters and functions such as $Z(x)^2$, are then the values of those quantities averaged over $P(\phi)$.

4.2.32 *restoration* — signal-processing procedure in which measurements are compensated for a non-unit measurement transfer function by passing them through a digital filter that restores the effective measurement function to unity over its bandpass.

4.2.32.1 *Discussion* — The measured profile can be restored and the statistics of the restored profile can then be estimated. The most common spatial- and frequency-domain filters used for this purpose are “inverse” and “Wiener” filters. This guide does not discuss the details of such restoration processes, which may be found in standard signal-processing texts such as those listed in Section 7.

4.2.33 *RMS profile roughness*, R_q , [nm] — square root of the mean-square profile roughness.

4.2.34 *RMS profile slope*, Δ_q — square root of the mean-square profile slope.

4.2.34.1 *Discussion* — The slope is dimensionless, although the fundamental unit is the radian. In practice it may be convenient to express the rms slope of highly polished surfaces in microradians.

4.2.35 *sample interval*, D , [μm] — distance between adjacent measurements of the surface height along the x axis. Also known as *sampling interval*.

4.2.35.1 *Discussion* — The sample interval is usually chosen or recommended by the manufacturer of the profile instrument being used. The sample interval defines the Nyquist frequency and hence, the extreme *HFL* of the measurement. This guide does not address measurements with unequal sample intervals or those made along nonlinear traces over the surface.

4.2.36 *sampled profile*, $Z(x_n)$, [nm] — surface height, $Z(x_n)$, measured at N equally-spaced points along the x axis.

4.2.36.1 *Discussion* — This guide uses the following indexing convention for the position of the height samples,

$$x_n = (n-1)D, \quad n = 1, 2, \dots, N \quad (8)$$

Therefore the distance between the first and last points in the profile trace is $(N-1)D$.

4.2.37 *sampled slope*, $m(x_n)$ — surface slope, $m(x_n)$, measured at N equally-spaced points along the x axis using the same indexing convention as for the sampled profile.

4.2.37.1 *Discussion* — Some instruments measure the surface slope directly, while others, in effect, measure the surface height at $N+1$ points and generate N slope values using the equation:

$$m(x_n) = \frac{1}{D} [Z(x_{n+1}) - Z(x_n)], \quad n = 1, 2, \dots, N \quad (9)$$

4.2.38 *slope power spectrum*, $S'(f_x)$ [μm] — statistical function that shows how the mean-square profile slope is distributed over surface spatial frequencies as follows:

$$S'_1(f_x) = \lim_{L \rightarrow \infty} \left(\frac{1}{L} \int_{-L/2}^{+L/2} m(x) \exp(i2\pi f_x x) dx \right)^2 = (2\pi f_x)^2 S_1(f_x) \quad (10)$$

4.2.38.1 *Discussion* — This simple connection between the slope and roughness power spectra permits one to be determined immediately in terms of the other. The prime on S_1 , on the left denotes that this is the *PSD* of the slope, while the unprimed S_1 on the far right is the *PSD* of the height.

4.2.39 *spatial frequency*, f_x [$1/\mu\text{m}$] — frequency parameter in the Fourier transform of the surface profile $Z(x)$.

4.2.39.1 *Discussion* — The parameter f_x is related to the spatial wavelength, d_x through $f_x = 1/d_x$. Similar quantities are defined for the y component, and the magnitude of the two-dimensional spatial-frequency vector, $f = \sqrt{f_x^2 + f_y^2}$, that appears in the two-dimensional power spectral density of an isotropically-rough surface, $S_2(f)$.

4.2.40 *spatial wavelength*, d_x , [μm] — reciprocal of the spatial frequency, f_x .

4.2.40.1 *Discussion* — The mechanical-engineering community frequently uses the symbol λ for the spatial wavelength, while the optical community reserves that symbol for the radiation wavelength.

4.2.41 *spectral model* — analytic expression for the power spectral density which contains a number of adjustable parameters called finish parameters.

4.2.41.1 *Discussion* — The values of the finish parameters are obtained by fitting estimates of the *PSD* of the surface height or slope fluctuations to the model. The fitting process performs a number of important functions: it averages out the fluctuations appearing in

individual estimates of the power spectrum, it condenses the data into a few intrinsic surface parameters, and provides a mechanism for extrapolating the measured data outside the measurement bandwidth.

4.2.41.2 One example of a spectral model is the *ABC model*, which has the following form for the profile PSD:

$$S_1(f_x) = \frac{A}{[1 + (Bf_x)^2]^{C/2}} \quad (11)$$

and for the two-dimensional spectrum of an isotropically-rough surface:

$$S_2(f) = \frac{A'}{[1 + (Bf)^2]^{(C+1)/2}} \quad (12)$$

where:

$$A' = \frac{1}{2\sqrt{\pi}} \cdot \frac{\Gamma(C+1)/2}{\Gamma(C/2)} \cdot AB.$$

The finish parameters in this model are A , B and C , which have the dimensions of μm^3 , μm^1 , and μm^0 . This model is sometimes called the K-correlation model, and the quantity $B/(2\pi)$, the correlation length.

4.2.41.3 Another example of a spectral model is the *fractal model*, which has the following form for the profile PSD:

$$S_1(f_x) = \frac{K_1}{f_x^C} \quad (13)$$

and for the two-dimensional spectrum of an isotropically-rough surface:

$$S_2(f) = \frac{K_2}{f^{C+1}} \quad (14)$$

where:

$$K_2 = \frac{1}{2\sqrt{\pi}} \cdot \frac{\Gamma(C+1)/2}{\Gamma(C/2)} \cdot K_1.$$

The finish parameters in this model are K_1 and C , which have the dimensions of $\mu\text{m}^{(3-C)}$ and μm^0 . The dimensionless number C usually lies between 1 and 3 but need not be an integer. The quantity K_C is sometimes referred to as the spectral strength, and the parameter, C , the spectral index.

4.2.41.4 The fractal model is the limiting case of the *ABC model* when the finish parameter B becomes very large. The value of the intrinsic mean-square profile and area roughness of the *ABC model*, obtained by integrating the *ABC* spectrum over all frequencies, is as follows:

$$R_q^2 = \int_0^\infty S_1(f_x) df_x = 2\pi \int_0^\infty f S_2(f) df = \frac{2\pi}{C-1} \frac{A'}{B^2} \quad (15)$$

which is finite for $C > 1$. The intrinsic value of the mean-square roughness of the fractal model is always infinite because of its divergence at low spatial frequencies. In contrast, the measured roughness values, obtained by integrating only over the measurement bandpass, are finite for the *ABC model* for any value of C , and for the fractal model.

4.2.41.5 A third example of a spectral model is the *periodic model*, which is for a surface consisting of a periodic structure and has the following forms for the profile PSD:

$$S_1(f_x) = \frac{1}{2} \sum_{k=1}^{k=\infty} A_k^2 \cdot \delta\left(f_x - \frac{k}{d_0}\right) \quad (16)$$

and for the two-dimensional spectrum:

$$S_2(f_x, f_y) = \frac{1}{4} \sum_{k=-\infty}^{k=+\infty} A_{|k|}^2 \cdot \delta\left(f_x - \frac{k}{d_0}\right) \cdot \delta(f_y) \quad (17)$$

where the $k = 0$ is excluded. The finish parameters of this model are the A_k 's, the Fourier amplitudes of the periodic profile, and d_0 , the fundamental spatial wavelength of the periodicity, both expressed in μm . $A^2/2$ is the mean-square roughness of the k^{th} harmonic of the profile ($k = 1$ is the fundamental), and $\delta(F)$ is a unit-area function that is sharply peaked about the point $F = 0$. The value of the intrinsic mean-square profile and area roughness of the periodic model is as follows:

$$R_q^2 = \int_0^\infty S_1(f_x) df_x = \int_{-\infty}^{+\infty} df_x \int_{-\infty}^{+\infty} S_2(f_x, f_y) df_y = \frac{1}{2} \sum_{k=1}^{\infty} A_k^2 \quad (18)$$

In contrast, the measured value is the right-hand side summed over those spectral lines that fall within the measurement bandpass.

4.2.41.6 Finally, it is possible to develop a *composite model* that is made up of a sum of terms involving different models or models with different parameters, or both.

4.2.42 *trace length*, L [μm] — total length of the surface sampled by a linear profile measurement. Also known as *profile length*.

4.2.42.1 *Discussion*: In the indexing used in this guide

$$L = x_N = (N-1)D \quad (19)$$

where:

x_N = the position of the N^{th} or last point in the measurement (with x_1 assumed at zero), and

D = 1 the sampling interval (see Section 4.2.15.1).

The periodogram estimate is based on a Fourier representation of the surface profile. The basic periodicity of that expansion is ND rather than the literal profile length $L = (N-1)D$. Depending on the type of *FFT* used in the practical evaluation of the *PSD*, N may be required to be a power of 2, such as 1024, although in general, there is no restriction on N in this guide.

4.2.43 transfer function — function of spatial frequency having a magnitude between zero and one which describes the sensitivity of a linear measuring system to the amplitudes of different spatial-frequency components in the profile being measured. Also known as *measurement transfer function*.

4.2.43.1 Discussion — The ideal transfer function is unity within the measurement bandpass and zero for frequencies outside the bandpass. Real-world measurement transfer functions can deviate significantly from this. The transfer function is the Fourier transform of the impulse response function of the measuring apparatus.

4.2.44 uniaxial surface — surface whose roughness is confined to a particular direction or lay, so that it can be completely characterized by profile measurements perpendicular to the lay direction. Surfaces that display harmonic lines are frequently uniaxial. Also known as *grating-like surface*.

4.2.44.1 Discussion — In contrast, an isotropic surface can also be completely characterized by profile measurements made in one direction, but there is no preferred direction as there is for uniaxial surfaces. Surfaces that are neither uniaxial nor isotropic can be characterized using the procedures described in this guide, although profile measurements taken on many directions across the surface may be needed to generate a complete statistical description of the surface under test.

4.2.45 window function, $W(x_n)$ — bell-shaped or smooth-edged function that multiplies the detrended profile data set before it is inserted into the periodogram estimation routine. Also known as *data window*.

4.2.45.1 Discussion — The window function “smoothes out” possible discontinuities at the ends of the measured, finite-length data set in order to eliminate the spurious oscillations that those discontinuities would otherwise generate in the spectral estimate. As

long as the window function performs its function of reducing the contributions from the ends of the data record and has the proper normalization, its shape is of secondary importance.

4.2.46 zero padding — procedure of adding zero values to a data set to bring the total number of data points, N , to a power of two to facilitate the evaluation of the *FFT* appearing in the periodogram spectral estimate.

4.2.46.1 Discussion — The window functions should be applied to the data set before zero padding. Zero padding is less important with the ready availability of arbitrary- N *FFT* computing packages.

5 Calculations

5.1 Detrending

5.1.1 Introduction — The estimators defined in this section are based on the analysis of a data set $Z(n)$ consisting of N discrete values of the surface profile $Z(x_n = (n-1)D)$ measured at equally-spaced locations along a straight line of length L , where $n = 1$ to N . If $Z(n)$ is the measured profile, the detrended profile is given by:

$$\hat{Z}d(n) = Z(n) - [a + b \cdot n + c \cdot n^2] \quad (20)$$

where the quantity in the square bracket is the quadratic detrending polynomial. The estimated values of the polynomial coefficients a , b , and c , denoted by \hat{a} , \hat{b} , and \hat{c} , respectively, are determined by least-squares fitting of the polynomial to the measured profile data as now described. The degree of the detrending polynomial is chosen by the following considerations: Removing piston only (zeroth-order polynomial, a) is useful for instructional purposes but is inadequate in practice. It affects only the zero-frequency or “dc” term in the power spectral density. Removing piston and tilt (first-order polynomial, $a + b \cdot n$) is sufficient for removing uncertainties in the rigid-body positioning of a nominally flat sample in the measurement apparatus. Removing piston, tilt and curvature (second-order polynomial, $a + b \cdot n + c \cdot n^2$) removes an additional quadratic term in the profile that may result from instrumental (extrinsic) effects or true (intrinsic) curvature in the surface being measured.

5.1.2 Piston detrending is as follows:

$$\hat{Z}d(n) = Z(n) - \hat{a} \quad (21)$$

and

$$\hat{a} = +M_0 \quad (22)$$

where M_0 (and also M_1 and M_2) are evaluated using the general moment expression,

$$M_P = \frac{1}{N} \sum_{n=1}^N n^P Z(n) \quad (23)$$

where:

$$P = 0, 1, \text{ or } 2.$$

5.1.3 *Piston and tilt detrending* is as follows:

$$\hat{Z}d(n) = Z(n) - [\hat{a} + \hat{b}n] \quad (24)$$

where:

$$n = 1, 2, \dots, N,$$

$$\hat{a} = +\frac{2}{N-1} \cdot [(2N+1)M_0 - 3M_1], \text{ and}$$

$$\hat{b} = -\frac{6}{N-1} \left[M_0 - \frac{2M_1}{N+1} \right].$$

5.1.4 *Piston, tilt, and curvature detrending* is as follows:

$$\hat{Z}d(n) = Z(n) - [\hat{a} + \hat{b}n + \hat{c}n^2] \quad (25)$$

where:

$$n = 1, 2, \dots, N,$$

$$\hat{a} = +\frac{3}{(N-1)(N-2)} [(3N^2 + 3N + 2)M_0 - 6(2N+1)M_1 + 10M_2],$$

$$\hat{b} = -\frac{6}{(N^2-1)(N^2-4)} [(3N+1)(N+2)(2N+1)M_0 - 2(2N+1)(8N+11)M_1 + 30(N+1)M_2], \text{ and}$$

$$\hat{c} = +\frac{30}{(N^2-1)(N^2-4)} [(N+1)(N+2)M_0 - 6(N+1)M_1 + 6M_2].$$

NOTE 1: The estimated values of the coefficients depend on the degree of the polynomial being detrended. For example, the value of the coefficient \hat{a} for piston detrending and piston-plus-tilt detrending derived from the same data set are generally different.

NOTE 2: Despite these apparent differences, the mean values of each of the detrended profiles given by

$$\text{Mean value} = \frac{1}{N} \sum_{n=1}^N \hat{Z}d(n) = 0 \quad (26)$$

vanishes in all cases. This means that the “dc” value of the estimated power spectrum of the detrended profile is zero, which offers a convenient numerical check on the numerical processing routines used.

NOTE 3: Least-squares fitting routines are available in many computer packages. Analytic results are given above for reference and checking.

5.2 RMS Roughness

5.2.1 There are two different estimators for the rms roughness, R_q — one expressed in configuration space, and the other in frequency space, as follows:

$$\hat{R}_q^2(\text{Config}) = \frac{1}{N} \sum_{n=1}^N \hat{Z}d(n)^2 \quad (27)$$

and

$$\hat{R}_q^2(\text{Freq}) = \frac{1}{ND} \sum_{m=1}^{1+\frac{N}{2}} \hat{S}_1(m) \quad (28)$$

1 where $\hat{S}_1(m)$ is the periodogram estimate of the PSD based on $\hat{Z}d(n)$ (see Section 5.4).

NOTE 4: These two estimates of R_q^2 are mathematically identical if the periodogram is evaluated using a unit data window, $W(n) = 1$. Although a unit window function is not recommended for general use, the numerical identity of the Equation 27 and Equation 28 in that case offers a convenient check on the programming of the periodogram estimator.

NOTE 5: If a non-unit data window is used in the calculation of the PSD the two estimates of the rms roughness given will not, in general, be numerically identical for a particular profile measurement. On the other hand, the two estimates are identical for an ensemble average over a large number of profile measurements. In other words, the two estimates of R_q^2 are statistically the same.

NOTE 6: The first estimator has the advantage of familiarity and simplicity since it is expressed directly in terms of the detrended values of the measured profile data. Its disadvantage is that it involves, perforce, the transfer function of the measuring apparatus, and in a nonobvious way. In contrast, the frequency-space form may be more complicated to evaluate but has the advantage that it permits the bandwidth to be included in the rms value to be varied by selecting the range of m values included in the frequency sum. In addition, it permits the effects of a non-unit instrumental transfer function within that bandpass to be examined directly, and to be divided out by restoration processes, if its form is known independently.

NOTE 7: The spectra of real surfaces frequently tend to diverge at low spatial frequencies so that the values of the rms roughness obtained using either estimator may depend significantly on the value of the LFL of the measurement process, or chosen as a reference value. In some cases, the presence of a non-vanishing LFL can give a finite value of the profile roughness when its intrinsic value is infinite or undefined.

5.3 RMS Slope

5.3.1 There are two different estimators for the rms slope, Δ_q — one expressed in configuration space, and the other in frequency space, as follows:

$$\hat{\Delta}_q^2(\text{Config}) = \frac{1}{ND^2} \sum_{n=1}^{N-1} [\hat{Z}d(n+1) - \hat{Z}d(n)]^2 \quad (29)$$

and

$$\hat{\Delta}_q^2(\text{Freq}) = \frac{1}{ND} \sum_{m=1}^{1+(N/2)} \hat{S}_1(m) \left[\frac{2\pi(m-1)}{ND} \right]^2 \quad (30)$$

NOTE 8: The magnitudes of these two estimates are generally different since the first treats the profile as a collection of straight-line segments connecting the measurement points, while the second connects them with a bandwidth-limited interpolation curve and involves a smaller bias error.

NOTE 9: The notes for the rms roughness estimators just made generally apply to slope estimates as well. One difference is that the significant bandwidth effects on the slope occur principally at the HFL rather than the LFL as is the case for rms roughness measurements.

5.4 Periodogram Estimators of the Profile Power Spectral Density

5.4.1 Form for 1-dimensional Power Spectral Density for height-measuring profilometers is as follows:

$$\hat{S}_1(m) = \frac{2D \cdot |\text{FFT}(m)|^2 \cdot K(m)}{N} \quad (31)$$

The spatial frequency is evaluated at the discrete values of m , with $m = 1, 2, \dots, [1 + (N/2)]$:

$$f_x = \frac{m-1}{ND} \quad (32)$$

5.4.2 The symbol FFT in Equation 31 stands for discrete Fourier transform, which is always evaluated using some version of the fast Fourier transform as follows:

$$\text{FFT}(m) = \sum_{n=1}^N \exp[i2\pi(n-1)(m-1)/N] \cdot W(n) \cdot \hat{Z}d(n) \quad (33)$$

where:

$W(n)$ = window function (see Section 5.6) and

$K(m)$ = book-keeping factor equal to $1/2$ for $m = 1$ or $m = 1 + (N/2)$ and equal to 1 otherwise.

NOTE 10: Equation 33 applies for the conventional case of even N . Different forms apply for odd N .

NOTE 11: The case $m = 1$ corresponds to the zero-frequency or dc component of the detrended surface profile and $m = 2$ corresponds to the spatial frequency $1/(ND)$, which is essentially the reciprocal of the trace length, $(N-1)D$. On the opposite extreme, the frequency corresponding to $m = 1 + N/2$ is the Nyquist frequency, $1/(2D)$. The extreme range of surface wavelengths included in the measurement is therefore $1/(ND) < f < 1/(2D)$. In other words, the extreme LFL = $1/(ND)$, the extreme HFL = $1/(2D)$, and the dynamic range of the measurement is $N/2$.

NOTE 12: A convenient and readable reference to the FFT and its evaluation is Chapter 12 in *Numerical Recipes* by Press, Flannery, Teukolsky, and Vetterling (see Section 7.1).

NOTE 13: *The Brookhaven National Laboratory Report* (see Section 7.2) contains further background information on these procedures along with a computer program and numerical examples. (The BASIC routines used there involve different forms for the quantities M_p appearing in the expressions for the detrending polynomials than those discussed in this guide, although the numerical values of the detrending polynomials are identical in both cases.)

NOTE 14: The periodogram estimator just given is not the only method of estimating the power spectral density from a set of profile data, but it is the most direct and common method. It is sufficient for general use, and is a necessary first step to be taken before adding embellishments such as post-processing or considering more complicated estimators. This guide does not exclude the use of post-processing or alternative methods of analysis, but does require that the basic periodogram estimates just described be included in the discussion for comparative purposes.

5.4.3 Form for 1-dimensional Power Spectral Density for slope-measuring profilometers is as follows:

$$\hat{S}_1(m) = \left[\frac{2\pi(m-1)}{ND} \right]^{-2} \cdot \frac{2D \cdot |\text{FFT}(m)|^2 \cdot K(m)}{N} \quad (34)$$

where:

$$\text{FFT}'(m) = \sum_{n=1}^N e^{\frac{i2\pi(n-1)(m-1)}{N}} \cdot W(n) \cdot \hat{M}d(n)$$

and $\hat{M}d(n)$ is the value of the profile slope measurements detrended using either the least-squares piston or the piston-plus-tilt expressions (see Section 5.1).

NOTE 15: This estimate of the profile power spectrum is the power spectrum of the profile slope divided by $(2\pi f_m)^2$. The prime on the FFT on the left denotes that it involves slope rather than height data.

NOTE 16: The case $m = 1$ corresponds to zero spatial frequency and must be excluded in the use of the above expressions.

5.5 Periodogram Estimators of the Rms Profile Roughness and Slope

5.5.1 The periodogram estimator of the rms profile height, \hat{R}_q , is as follows:

$$\hat{R}_q = \sqrt{\frac{1}{ND} \sum_{m=2}^{1+(N/2)} \hat{S}_1(m)} \quad (35)$$

This quantity has the same dimension as the original height measurements and is independent of the magnitude and dimensions of the sampling interval, D .

5.5.2 The corresponding estimator for the rms profile slope, $\hat{\Delta}_q$, is as follows:

$$\hat{\Delta}_q = \sqrt{\frac{1}{ND} \sum_{m=2}^{1+(N/2)} \hat{S}_1(m)} = 2\pi \sqrt{\frac{1}{(ND)^3} \sum_{m=2}^{1+(N/2)} (m-1)^2 \hat{S}_1(m)} \quad (36)$$

In evaluating these quantities the height measurements, Z , and the sampling interval, D , must be expressed in the same length units. Although $\hat{\Delta}_q$ is in units of radians, its magnitude scales as $1/D$.

5.6 Window Functions

5.6.1 Window functions appear in a wide variety of signal-processing applications, with different shapes and normalizations. Although this guide recommends the use of the Hann or Blackman window, other forms are included for comparison. All are normalized so that

$\frac{1}{N} \sum_{n=1}^N W(n)^2 = 1$ in order to preserve the magnitudes of average values of the mean-square profile statistics.

5.6.2 Particular forms are:

5.6.2.1 *Rectangular or Daniell window:*

$$W(n) = 1 \quad (37)$$

5.6.2.2 Hann, or Raised Cosine window:

$$W(n) = \sqrt{\frac{2}{1728}} \left\{ 24 \left[1 - \cos\left(\frac{2\pi(n-1)}{N}\right) \right] \right\} \quad (38)$$

5.6.2.3 *Hamming window:*

$$W(n) = \sqrt{\frac{2}{1987}} \cdot \left[27 - 23 \cos\left(\frac{2\pi(n-1)}{N}\right) \right] \quad (39)$$

5.6.2.4 *Blackman window:*

$$W(n) = \sqrt{\frac{2}{1523}} \cdot \left[21 - 25 \cos\left(\frac{2\pi(n-1)}{N}\right) + 4 \cos\left(\frac{4\pi(n-1)}{N}\right) \right] \quad (40)$$

NOTE 17: The choice of window functions is of minor importance for randomly-rough surfaces as long as it smoothes the data at the ends of the data record. The rectangular or Daniell window does not do this, but is useful for numerical checking.

NOTE 18: In the case of profiles with a smooth PSD, the principal effect the window shape is to change the fine-scale fluctuations in the periodogram estimate without changing its ensemble-average value, except, perhaps, near the LFL.

NOTE 19: In the case of profiles involving periodicities, the window shape can change the shape of the sharp lines in the PSD, albeit without changing their areas. The choice of the window shape then involves a trade-off between line width and smoothness. The raised Hann or Blackman windows are recommended for general use.

NOTE 20: If the estimation routines are applied to deterministic profiles, such as individual steps, pits, or bumps, a data window must still be used to minimize effects of the finite data record, but the object should be placed in the center of the profile where the window function is relatively flat.

5.7 Zero Padding

5.7.1 The fastest FFT routines require the total number of data points to be a power of two, such as $N = 2^{10} = 1024$. If the number of measured points, N , is not a power of two but lies between 2^a and 2^b , the power-of-two routines can be used by dropping $N-2^a$ points from one end of the original data set, or by adding 2^b-N zeros and replacing N in the routines everywhere by 2^b .

5.7.2 The first method is wasteful of data, while the second uses the full set of measured data but requires that the estimated PSD be renormalized by multiplying it by the factor $2^b/N$.

5.8 Averaging of Statistical Quantities:

5.8.1 Power spectral density functions, the mean-square roughness, and slope values estimated from a number of individual profiles that have the same statistical properties can each be averaged together to obtain composite results. In the case of homogeneously and isotropically rough surfaces the profiles can lie in any position and direction on the surface under test. In the case of homogeneously but anisotropically rough surfaces they can lie anywhere on the surface but must lie parallel with each other, preferably perpendicular to the surface axis. Averaging data lowers the errors associated with individual measurements.

6 Numerical Test Sequences

6.1 Table 1 presents a set of numerical data for testing the execution of the users' implementations of algorithms discussed in Section 5.

6.2 Although these simulated profile data are in standard notation, they have been generated by a

random number generator corresponding to a constant PSD and are not the results of an actual measurement. In addition, the number of data points has been limited to $N = 32$ and the profile heights have been rounded to digits with magnitudes less than 100 to simplify their manual input into the users' programs.

6.3 Actual measured data sets would generally involve many more data points with height values involving a larger number of significant digits, and with different orders of magnitude than those used in this test sequence.

6.4 In order to provide a means for checking the proper inclusion of the sampling distance, D , in the spectral-estimation routines, the value $D = 0.1$ has been used.

6.5 Tables 2 and 3 give the values of the periodogram estimates of the profile power spectral density, $\hat{S}_1(m)$, of the data in Table 1 for the three different types of detrending described in Section 5.1. The values of these estimates depend on the data window used. Table 2 uses a rectangular window and Table 3 uses the Blackman window.

6.6 The dimensions of the power spectral densities in these tables is length-cubed = (units of Z)²·(units of D), and its magnitude at a given spatial frequency scales as the sampling interval, D .

6.7 The spatial frequency is given as follows:

$$f_m = \frac{m-1}{ND} \quad (41)$$

where:

$m = 1$ corresponds to the dc or piston part of the profile, and

$m = 1 + N/2 = 17$ is the Nyquist frequency in (units of D^{-1}).

6.7.1 Note that since the window function has been applied after the detrending process, the dc terms do not necessarily vanish for a non-rectangular window functions.

6.8 Table 4 gives values of \hat{R}_q derived from the spectra in Table 2 and Table 3 using the expression given in Table 4.

6.8.1 As mentioned, the unit of \hat{R}_q is the same as that of the height measurement since the magnitude and

dimensions of the sampling interval, D , cancels out in the evaluation of R_q .

6.9 The data in Table 2 through Table 4 are adequate for checking the users' implementation of the estimators described in Section 5, and further test data are not included in this guide.

7 Related Documents

7.1 Press, W. H., Flannery, B. P., Teukolsky, S. A., and Vetterling, W. T., *Numerical Recipes – the Art of Scientific Computing*, Cambridge University Press, Cambridge, 1986.

7.2 Church, E. L., and Takacs, P. Z., "BASIC program for power spectrum estimation", *Brookhaven National Laboratory Report BNL No. 49035*, May 1993 (revised May 1994).

7.3 Stover, J. C., *Optical Scattering: Measurement and Analysis*, SPIE Press, 1995.

7.4 Kay, S. M. *Modern Spectral Estimation; Theory and Application*, Prentice Hall, 1988.

7.5 Marple, Jr., S. L., *Digital Spectral Analysis with Applications* (Prentice Hall, 1987).

7.6 Harris, F. J., "On the Use of Windows for Harmonic Analysis With the Discrete Fourier Transform," *Proceedings IEEE* **66**, 51–83 (1978).

7.7 Oppenheim, A. V., and Schaffer, R. W., *Digital Signal Processing*, Chapter 11, Prentice Hall, 1975.

7.8 Bendat, J. S., and Piersol, A. G., *Random Data; Analysis and Measurement*, Chapter 9 (Wiley, 1971).

7.9 Church, E. L., Vorbuerger, T. V., and Wyant, J. C., "Direct Comparison of Mechanical and Optical Measurements of the Finish of Precision Machined and Optical Surfaces," *Optical Engineering*, **24**, 388–395 (1985).

7.10 Blackman, R. B., and Tukey, J. W., *The Measurement of Power Spectra* (Dover, 1959).

8 Keywords

8.1 estimates; estimators; power spectral density; rms values; root mean square; roughness; slope; surface roughness; surface slope; surface statistics.

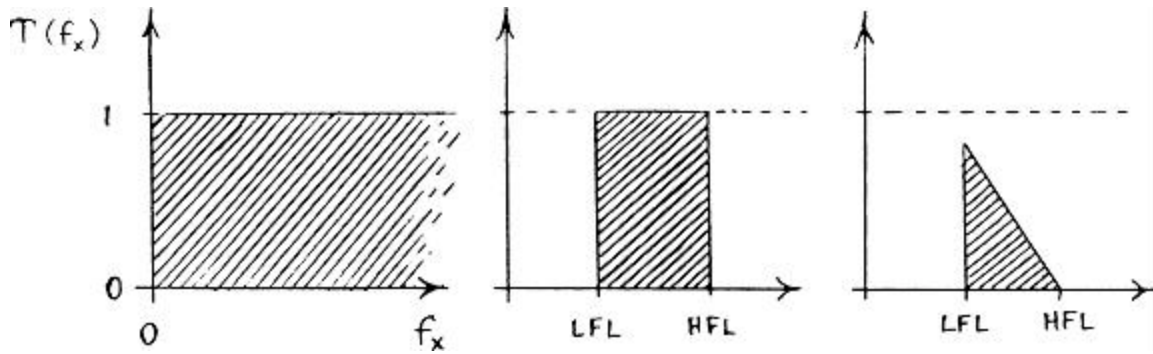


Figure 1

Different Forms of the Measurement-Transfer or Instrumental-Response Function as a Function of Spatial Frequency, f_x .

Table 1 Simulated Height Data ($N = 32$)

n	$Z(n)$	n	$Z(n)$	n	$Z(n)$	n	$Z(n)$
1	-38	9	-40	17	3	25	-35
2	15	10	45	18	6	26	23
3	36	11	20	19	17	27	4
4	22	12	3	20	20	28	-8
5	29	13	47	21	24	29	-45
6	-43	14	-18	22	16	30	26
7	-1	15	45	23	-5	31	1
8	-5	16	43	24	-17	32	6

Table 2 Periodogram Estimates $\hat{S}(m)$ for Different Types of Data Detrending Using a Rectangular Window

m	None	Piston	Piston+Tilt	Full Quadratic
1	120.0500	0	0	0
2	205.9506	205.9506	182.5409	14.81781
3	142.1861	142.1861	137.3580	204.3631
4	56.98463	56.98463	44.47469	53.08546
5	147.9039	147.9039	139.6268	126.5831
6	58.60630	58.60630	55.67305	50.13899
7	248.4120	248.4120	264.7417	266.1277
8	152.8321	152.8321	158.6038	154.5160
9	185.1250	185.1250	192.8103	190.0105
10	28.81090	28.81090	26.22179	25.40131
11	28.61438	28.61438	27.90264	28.74841
12	153.1641	153.1641	145.7812	145.2367
13	356.5711	356.5711	366.4113	365.4717
14	199.1965	199.1965	202.6268	203.4514
15	41.53757	41.53757	40.26207	40.50671
16	163.6550	163.6550	168.8428	169.0258
17	16.20000	16.20000	17.87214	17.87215

Table 3 Periodogram Estimates $\hat{S}(m)$ for Different Types of Data Detrending Using a Blackman Window

m	None	Piston	Piston+Tilt	Full Quadratic
1	312.4632	87.20874	90.11245	9.832211
2	536.6033	268.4217	260.2453	76.63136
3	195.1412	166.9871	162.0176	93.70938
4	1.046170	1.046172	1.041742	1.303189
5	30.14155	30.14156	30.16028	30.25176
6	54.87353	54.87350	54.91950	54.94658
7	188.9816	188.9818	188.8795	188.8293
8	74.45938	74.45934	74.50156	74.51955
9	45.28967	45.28967	45.27541	45.29324
10	59.40473	59.40471	59.41950	59.41210
11	94.24771	94.24768	94.23248	94.23342
12	202.7328	202.7328	202.7405	202.7253
13	289.7414	289.7414	289.7450	289.7637
14	130.1287	130.1287	130.1230	130.1162
15	76.22277	76.22275	76.22527	76.22248
16	62.42836	62.42840	62.42742	62.43071
17	5.585947	5.585948	5.585947	5.584926

Table 4 Values of the Estimates \hat{R}_q for Different Types of Detrending Followed by Different Types of Windowing

<i>Type of Window</i>	<i>Type of Data Detrending</i>			
	<i>None</i>	<i>Piston</i>	<i>Piston+Tilt</i>	<i>Full Quadratic</i>
Rectangular	26.13517	26.13517	26.05133	25.34362
Hann	25.49031	24.02299	23.91725	22.31003
Hamming	25.53997	24.26072	24.15406	22.58759
Blackman	25.29220	23.38999	23.30221	21.54917

NOTICE: SEMI makes no warranties or representations as to the suitability of the standards set forth herein for any particular application. The determination of the suitability of the standard is solely the responsibility of the user. Users are cautioned to refer to manufacturer's instructions, product labels, product data sheets, and other relevant literature, respecting any materials or equipment mentioned herein. These standards are subject to change without notice.

By publication of this standard, Semiconductor Equipment and Materials International (SEMI) takes no position respecting the validity of any patent rights or copyrights asserted in connection with any items mentioned in this standard. Users of this standard are expressly advised that determination of any such patent rights or copyrights, and the risk of infringement of such rights are entirely their own responsibility.

SEMI MF1982-1103

TEST METHODS FOR ANALYZING ORGANIC CONTAMINANTS ON SILICON WAFER SURFACES BY THERMAL DESORPTION GAS CHROMATOGRAPHY

This standard was technically approved by the Global Silicon Wafer Committee and is the direct responsibility of the North American Silicon Wafer Committee. Current edition approved for publication by the North American Regional Standards Committee on July 27, 2003. Initially available at www.semi.org October 2003; to be published November 2003. Originally published by ASTM International as ASTM F 1982-99. Last previous edition ASTM F 1982-99^{e1}.

1 Purpose

1.1 Organics are present in many materials, such as plastics, lubricants, cleansers, soaps, and living tissues. Some of these compounds are volatile and others can become airborne through chemical reactions, heating, abrasion, or outgassing. Also they can transfer to wafers by direct contact or be left behind from solvent residues. Once present in clean facilities, they can deposit on wafer surfaces. Organics deposited on wafers can cause degradation haze, wafer surface tension changes, irregular oxidation rates, and other effects, such as counter-doping by organophosphorus compounds. Identification of trace level organic contaminants is important in determining the source of the particular contamination. These test methods use the TD-GC technique to characterize and quantify organics deposited on wafer surfaces.

1.2 Monitoring of organic contamination on wafer surfaces also can be used to measure material outgassing for proper selection of cleanroom, construction, and wafer packaging materials.

2 Scope

2.1 These test methods cover the identification and quantification of organic contaminants on silicon wafer surfaces using a gas chromatograph interfaced to a mass spectrometer (GC-MS) or a phosphorus selective detector, or both.

2.2 These test methods describe the apparatus and related procedures for sample preparation and analyses by thermal desorption gas chromatography (TD-GC).^{1,2}

2.3 The range of detection limits of these test methods depends on the target organic compounds, for example, the range of detection limits is from the subpicogram to the nanogram level of hydrocarbons (C₈ to C₂₈) per square centimeter of silicon wafer surface.

2.4 These test methods can be used for polished silicon wafers, or silicon wafers with oxide films.

2.5 Two methods are described. Method A is performed on cleaved wafers. Method B is performed on full wafers. The detailed procedures of Method A and Method B as well as the differences between them, are described in Sections 5 and 7.

2.6 Suitable safety precautions must be followed when handling organic solvents and compounds, hot materials subjected to propane flame, the propane flame itself, wafer thermal desorption systems, rapid thermal annealer, or a high temperature furnace.

NOTICE: This standard does not purport to address safety issues, 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 or other limitations prior to use.

3 Referenced Standards

3.1 ASTM Standard

D 6196 — Practice for Selection of Sorbents and Pumped Sampling/Thermal Desorption Analysis Procedures for Volatile Organic Compounds in Air³

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

4 Terminology

4.1 Abbreviations and Acronyms

4.1.1 AED — atomic emission detector

4.1.2 C₁₆ — n-hexadecane, n-C₁₆H₃₄

4.1.3 FID — flame ionization detector

4.1.4 FPD — flame photometric detector

4.1.5 GC — gas chromatography

1 Ferguson, L.A., "Analysis of Organic Impurities on Silicon Wafer Surfaces," *Microcontamination*, 4(4), 33-37, April 1986.

2 Saga, K. and Hattori, T., "Identification and Removal of Trace Organic Contamination on Silicon Wafers Stored in Plastic Boxes," *Journal of Electrochemical Society*, 143, 3270-3284 (1996).

3 Published in Volume 11.03 of *Annual Book of ASTM Standards*. Available from ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, USA. Telephone: 610.832.9585, Fax: 610.832-9555, Web site: www.astm.org.

4.1.6 *MS* — mass spectrometer

4.1.7 *NPD* — nitrogen/phosphorus thermionic ionization detector

4.1.8 *TBP* — tributyl phosphate, $(C_4H_9O)_3PO$

4.1.9 *TCEP* — tris (2-chloroethyl) phosphate, $(ClCH_2CH_2O)_3PO$

4.1.10 *TD* — thermal desorption

4.2 Definitions of Term Specific to This Standard

4.2.1 *blank wafer* — a thermally-treated wafer desorbed of any surface organic contaminants.

4.2.1.1 *Discussion* — In Method A, surface organic contaminants are desorbed with a rapid thermal annealer or a high temperature furnace. In Method B surface organic contaminants are desorbed by purging them out in the heated quartz chamber unit while helium gas is flowing as a purge gas.

5 Summary of Test Methods

5.1 Method A

5.1.1 *Desorption and GC Analysis* — The volatile organic contaminants on a wafer surface are desorbed thermally from the wafer surface in a wafer desorption oven and swept into a sample thermal desorption tube. The sample thermal desorption tube then is heated for a set period in the thermal desorption unit and the volatile organic contaminants desorbed from the sample thermal desorption tube are swept by a stream of helium to a cold trap where they are preconcentrated. At the end of this period, the cold trap is heated rapidly to release the trapped organics to the GC column head. Sample components then are separated and eluted out of the GC column. Then, a portion goes to a phosphorus selective detector and the remainder goes to a mass spectrometer (MS). Blank wafers are prepared by purging out any surface organic contaminants in a rapid thermal annealer or a high temperature furnace while a purge gas is flowing.

5.1.2 *Identification of Contaminants* — Identification of individual unknown compounds is performed by correspondence of retention time of their peaks with that of known compounds. Correspondence of retention time on a single column should not be regarded as proof of identity. More precise identification of individual unknown compounds is performed with MS by matching their fragmentation patterns with mass spectra of known compounds in the spectral library.

5.1.3 *Quantification* — Quantification of total organics is based on the comparison of the integrated total peak area of the sample peaks with the area of the external standard compound, $n-C_{16}H_{34}$. Total organophosphorus

content in a sample is quantified by comparing the sample signal integrated from the phosphorus selective detector with the signal of the phosphorus standard compound, tris (2-chloroethyl) phosphate (TCEP) or tributyl phosphate (TBP). Specified range of standards is measured periodically, and the result is reported with blank wafer data.

5.2 Method B

5.2.1 *Desorption and GC Analysis* — The volatile organic contaminants on a wafer surface are desorbed thermally from the wafer surface in a quartz chamber unit and swept into a glass TD tube. The glass TD tube then is heated for a set period and a stream of helium sweeps the volatile organic contaminants desorbed from the glass TD tube to a cold trap where they are preconcentrated. At the end of this period, the cold trap is heated rapidly to release the trapped organics to the GC column head. Sample components then are separated and eluted out of the GC column. Then, a portion goes to a phosphorus selective detector and the remainder goes to a mass spectrometer (MS). Blank wafers are prepared by purging out any surface organic contaminants in the heated quartz chamber unit, while helium gas is flowing as a purge gas.

5.2.2 *Identification of Contaminants* — Identification of individual unknown compounds is performed by correspondence of retention time of their peaks with that of known compounds. Correspondence of retention time on a single column should not be regarded as a proof of identity. More precise identification of individual unknown compounds is performed with MS by matching their fragmentation patterns with mass spectra of known compounds in the spectral library.

5.2.3 *Quantification* — Quantification of total organics is based on the comparison of the integrated total peak area of the sample peaks with the area of the external standard compound, $n-C_{16}H_{34}$. Total organophosphorus content in a sample is quantified by comparing the sample signal integrated from a phosphorus selective detector with the signal of the phosphorus standard compound, tris (2-chloroethyl) phosphate (TCEP) or tributyl phosphate (TBP). Specified range of standards is measured periodically, and the result is reported with blank wafer data.

6 Apparatus

6.1 Method A

6.1.1 *GC Instrument* — Utilizes a capillary column to separate a wide variety of organic compounds coupled to a mass spectrometer (MS), or a phosphorus selective detector, or both. Examples of phosphorus selective detectors are flame photometric detector (FPD), and

atomic emission detector (AED). The nitrogen/phosphorus thermionic ionization detector (NPD) also responds to nitrogen containing compounds.

NOTE 1: A nitrogen/phosphorus thermionic ionization detector (NPD) may also be used as a phosphorus selective detector. This type of detector also responds to nitrogen containing compounds. If an NPD is used, the total organophosphorus reported should exclude any signals due to nitrogen containing compounds. Often, identification from the mass spectra can be used to determine whether compound contains nitrogen or phosphorus, or both.

6.1.2 Sample Thermal Desorption Tubes — Stainless-steel tubes packed with adsorbent medium, are used to trap compounds of interest and release them onto a thermal desorption unit.

NOTE 2: Note that stainless steel is catalytically active and can corrode with time, affecting recovery for some compounds. In this case, deactivated stainless steel, glass, or quartz tubes also can be used. Several adsorbent materials can be used for trapping organic compounds desorbed from silicon wafer samples. Some examples are activated carbon, graphitized carbon, and poly (2,6-diphenyl-p-phenylene oxide).

6.1.3 Thermal Desorption Unit — Used to desorb organics from sample thermal desorption tubes. The thermal desorption unit is coupled to the GC instrument via a heated transfer line, for example, heated to 225°C or above.

6.1.4 Thermal Annealer — Or a high temperature furnace used to obtain a blank wafer (see Section 7.1.1.2).

6.1.5 Temperature Controllable Wafer Desorption Oven — Used to hold and heat a wafer desorption tube.

6.1.6 Wafer Desorption Tube — Stainless-steel tube with an approximate dimension: 12.7 mm (0.5 in.) in outer diameter, 9.53 mm (0.375 in.) in inner diameter and 254 mm (10 in.) in length (see Figure 1), used to desorb organics from cleaved wafers. Larger tubes may be used for larger wafer sizes and to increase the sensitivity of the test (see Note 2).

6.1.7 Analytical Balance

6.1.8 Quartz Plate and Carbide-Tipped Scribe — Used to cleave wafer samples.

6.1.9 Quick Connect — Used to connect a nitrogen line to the wafer desorption tube.

6.2 Method B

6.2.1 GC Instrument — Utilizes a capillary column to separate a wide variety of organic compounds, coupled to a mass spectrometer (MS) or a phosphorus selective detector, or both. Examples of phosphorus selective

detectors are flame photometric detector (FPD), atomic emission detector (AED), or nitrogen/phosphorus thermionic ionization detector (NPD). The nitrogen/phosphorus thermionic ionization detector (NPD) also responds to nitrogen containing compounds (see Note 1).

6.2.2 Quartz Chamber Unit — Used to desorb organic contaminants from the wafer surface and transfer them to a glass TD tube (see Figure 2).

6.2.3 Quartz Chamber — May be used also to purge out any organic contaminants from a blank wafer.

6.2.4 Glass TD Tube — Packed with adsorbent medium used to trap compounds of interest and release them onto a cold trap. Several adsorbent materials can be used for trapping organic compounds desorbed from silicon wafer samples. Some examples are activated carbon, graphitized carbon, and poly (2,6-diphenyl-p-phenylene oxide).

6.2.5 Cold Trap — Used to concentrate organics desorbed from glass TD tube and release the organics to a GC instrument by rapid heating.

7 Procedure

7.1 Method A

7.1.1 Sample Handling and Preparation

7.1.1.1 At all times, avoid manual handling of samples for analysis to prevent any secondary contamination of samples. Use stainless steel tweezers for sample preparation. Use a propane torch to flame stainless steel tweezers, weigh boats, and other accessories that come into direct contact with the sample before their usage.

7.1.1.2 Thermally desorb any organics from blank wafers in a rapid thermal annealer or a high temperature furnace. In order to desorb any organics from blank wafers, the temperature of a rapid thermal annealer is kept at 900°C for 15 s, or the temperature of a high temperature furnace is kept at 700°C for 30 min. Heating under air or oxygen will make a consistent organic-free SiO₂ surface. Place the thermally treated blank wafers directly into petri dishes and wrap the dishes with organic-free aluminum foil.

7.1.2 Wafer Desorption

7.1.2.1 Clean and precondition sample thermal desorption tubes in accordance with ASTM Practice D 6196 prior to attaching to the wafer desorption tube.

7.1.2.2 Turn on the temperature controller for the wafer desorption oven and wait for the temperature to reach 275°C.

7.1.2.3 Cleave the wafer sample into strips about 5 to 7 mm wide by scribing a line on the wafer where one wants it to break and then placing it on a straight wire and breaking it along the scribed line. The portion of the wafer sample needed for the analysis changes with the size of the wafer. For a 100-mm wafer, about half of its surface area is needed. Larger samples (having larger surface area) may be used to increase the sensitivity of the test.

7.1.2.4 Place the wafer strips of wafer into the wafer desorption tube. Attach a sample thermal desorption tube to the side of the wafer desorption tube. Connect the other end of the wafer desorption tube to a nitrogen line with a flow of 15 to 100 mL/min.

7.1.2.5 Place the wafer desorption tube in the oven and heat it for 30 min at 275°C. This allows the volatile organics to be thermally desorbed from the sample wafer and transferred to the sample thermal desorption tube.

7.1.2.6 Seal both ends of the sample thermal desorption tube into the thermal desorption unit or by using caps if the thermal desorption-GC system is automated. Tubes must not be left open and exposed to the laboratory atmosphere. The end caps should be made of a material that has low outgassing and low reactivity properties, such as poly tetrafluoroethylene (PTFE), or stainless steel.

7.1.2.7 Analyze a blank wafer at least daily. Use a blank wafer treated with the same desorption procedure just described. Report blank data with the sample data.

7.1.3 GC Analysis

7.1.3.1 Place the sample thermal desorption tube, which contains organic contaminants desorbed from the wafer, onto the thermal desorption unit. Enter the sample information into the computer, and start the GC analysis.

7.1.3.2 Flow the helium carrier gas through the sample thermal desorption tube during primary desorption. Heat the sample thermal desorption tube and sweep out the target organics contained in the sample to a cold trap where they are preconcentrated. Two recommended conditions for heating the sample desorption tube are as follows. When graphitized carbon is used as adsorbent material, heat the tube to 400°C and hold at 400°C for 15 min. When poly (2,6-diphenyl-p-phenylene oxide) is used as adsorbent material, heat the tube to 270°C and hold at 270°C for 15 min. Cold trap parameters, that is, sorbent, sold temperature, etc., should be selected such that all target analytes are retained quantitatively throughout the tube desorption process, for example, using a cold trap temperature of -30°C. Refer to ASTM Practice D 6196

for a method for testing desorption efficiency and analyte recovery. At the end of this period, heat the cold trap rapidly to 300°C to release the target organics to the GC column head.

NOTE 3: For higher sensitivity analysis, larger sample sizes can be used, but clogging by water is possible. In this case, a subambient cold trap with an adsorbent may be used and maintained higher than 0°C during trapping to prevent freeze-up of trap.

7.1.3.3 Separate volatile organics desorbed from the sample thermal desorption tube by an appropriate column temperature program. Two recommended temperature programs are as follows. For high resolution analysis, using polydimethylsiloxane-coated column (60 m by 0.25 mm by 0.25-μm film thickness), heat the column from 40 to 280°C at a rate of 10°C/min and hold the temperature at 280 °C for 16 min. For rapid analysis, using polydimethylsiloxane-coated column (25 m by 0.32 mm by 0.52-μm film thickness), heat the column from 30 to 265°C at a rate of 12.5°C/min and hold the temperature at 265°C for 16 min.

7.1.3.4 Use n-Hexadecane, n-C₁₆H₃₄(C₁₆), as a standard for total organic analysis—4. Use tris (2-chloroethyl) phosphate, (ClCH₂CH₂O)₃PO (TCEP) or tributyl phosphate, (C₄H₉O)₃ PO (TBP), as a standard for organophosphorus analysis (see Section 6.1.1). These standards are introduced into the GC system from a precleaned sample thermal desorption tube. Two-point calibration method is recommended for checking the instrument performance. Recommended standards are two different concentrations of n-C₁₆H₃₄ (C₁₆) for total organic analysis; and, two different concentrations of tris (2-chloroethyl) phosphate (TCEP) for total organophosphorus analysis. Recommended frequency of these measurements is at least once a week.

NOTE 4: AED and FID are recommended for accurate total carbon quantification.

7.1.4 Quantification

7.1.4.1 Obtain each area below for the calculation of total organic contaminants. Obtain total organic contaminants of the sample wafer (A_s) by summing up all the peak areas integrated from GC-AED, FID, or MS chromatogram of the sample wafer. Obtain total area of the blank wafer (A_b) by summing up all the peak areas integrated from GC-AED, FID, or MS chromatogram of the blank wafer. Obtain the area of the standard peak (A_c) integrated from GC-AED, FID, or MS chromatogram of the standard sample.

7.1.4.2 Obtain each area below for the calculation of total organophosphorus (TP). Obtain total area of phosphorus compounds of the sample wafer (A_s) by

summing up all the peak areas integrated from GC-(phosphorus selective detector) chromatogram of the sample wafer. Obtain total area of the blank wafer (A_b) by summing up all the peak areas integrated from GC-(phosphorus selective detector) chromatogram of the blank wafer. Obtain the area of the standard peak (A_p) integrated from GC-(phosphorus selective detector) chromatogram of the standard sample.

7.1.4.3 Before starting analysis, weigh and get the total weight of each sample wafer to be tested, and also the weight of unused part of each wafer. These weights are used for the calculation of total organic contaminants and total organophosphorus (TP).

7.2 Method B

7.2.1 Sample Handling and Preparation

7.2.1.1 At all times, avoid manual handling of samples for analysis to prevent any secondary contamination of samples. Use stainless steel tweezers for sample preparation. Use a propane torch to flame stainless steel tweezers and other accessories that come into direct contact with the sample before their usage.

7.2.1.2 Thermally desorb any organics from blank wafers in the quartz chamber unit. Recommended conditions for preparing blank wafers are to heat them for 30 min in the quartz chamber kept at 700°C. Usually, helium is used as purge gas. Heating under air or oxygen will make a consistent organic-free SiO_2 surface.

7.2.2 Wafer Desorption

7.2.2.1 Enter the analytical information into the quartz chamber unit, cold trap and GC. Wait for the system to be stabilized. Place the sample wafer into the quartz chamber unit and start it. Supply helium gas into the quartz chamber and heat the chamber from initial temperature (40°C or lower) to 400°C at a rate of 10 to 30°C/min, and hold the temperature for 15 min. Sweep out the volatiles contained in the sample to a glass TD tube where they are adsorbed.

7.2.2.2 Analyze a blank wafer at least daily. Report blank data with the sample data.

7.2.3 GC Analysis

7.2.3.1 Heat the glass TD tube and sweep out the target organics adsorbed in the tube to a cold trap where they are preconcentrated. Two recommended conditions for heating the glass TD tube are as follows:

- When graphitized carbon is used as adsorbent material, heat the tube to 400°C and hold at 400°C for 15 min.

- When poly (2,6-diphenyl-p-phenylene oxide) is used as adsorbent material, heat the tube to 270°C and hold for 15 min.

7.2.3.2 Select cold trap parameters, that is, sorbent, cold temperature, etc., such that all target analytes are quantitatively retained throughout the tube desorption process, for example using a cold trap temperature of -130°C. Refer to ASTM Practice D 6196 for a method for testing desorption efficiency and analyte recovery. At the end of this period, heat the cold trap rapidly to 300°C to release the target organics to the GC column head.

7.2.3.3 Separate volatile organics desorbed from the glass TD tube by an appropriate column temperature program. Two recommended temperature programs are as follows. For high resolution analysis, use polydimethylsiloxane coated column (60 m by 0.25 mm by 0.25- μm film thickness), heat the column from 40 to 280°C at a rate of 10°C/min and hold the temperature at 280°C for 16 min. For rapid analysis, use polydimethylsiloxane coated column (25 m by 0.32 mm by 0.52- μm film thickness), heat the column from 30 to 265°C at a rate of 12.5°C/min and hold the temperature at 265°C for 16 min.

7.2.3.4 Use n-Hexadecane, $\text{n-C}_{16}\text{H}_{34}(\text{C}_{16})$, as a standard for total organic analysis (see Note 4). Use tris (2-chloroethyl) phosphate, $(\text{ClCH}_2\text{CH}_2\text{O})_3\text{PO}$ (TCEP) or tributyl phosphate, $(\text{C}_4\text{H}_9\text{O})_3\text{PO}$ (TBP), as a standard for organophosphorus analysis (see Section 6.1.1). These standards are introduced into the GC system from a precleaned wafer. A two-point calibration method is recommended for checking the instrument performance. Recommended standards are two different concentrations of $\text{n-C}_{16}\text{H}_{34}(\text{C}_{16})$ for total organic analysis; and two different concentrations of tris (2-chloroethyl) phosphate (TCEP) for total organophosphorus analysis. Recommended frequency of these measurements is at least once a week.

7.2.4 Quantification

7.2.4.1 Obtain each area below for the calculation of total organic contaminants. Obtain total area of organic contaminants of the sample wafer (A_s) by summing up all the peak areas integrated from GC-AED, FID, or MS chromatogram of the sample wafer. Obtain total area of the blank wafer (A_b) by summing up all the peak areas integrated from GC-AED, FID, or MS chromatogram of the blank wafer. Obtain the area of the standard peak (A_c) integrated from GC-AED, FID, or MS chromatogram of the standard sample.

7.2.4.2 Obtain each area below for the calculation of total organophosphorus (TP). Obtain total area of phosphorus compounds of the sample wafer (A_s) by summing up all the peak areas integrated from GC-

(phosphorus selective detector) chromatogram of the sample wafer. Obtain total area of the blank wafer (A_b) by summing up all the peak areas integrated from GC-(phosphorus selective detector) chromatogram of the blank wafer. Obtain the area of the standard peak (A_p) integrated from GC-(phosphorus selective detector) chromatogram of the standard sample.

8 Calculation

8.1 Method A

8.1.1 Calculation of Total Organic Contaminants

8.1.1.1 When AED or FID is used:

$$TC = \frac{A_s - A_b}{A_c} \times \frac{W_c \times 10^{-9}}{MW_c} \times 6.02 \times 10^{23} \times \frac{1}{S_w \times F} \times 16 \quad (1)$$

where:

TC = total organic carbon, C atoms/cm²,
 A_s = total area integrated from GC-AED or FID chromatogram of the sample wafer for carbon compounds,
 A_b = total area integrated from GC-AED or FID chromatogram of the blank wafer,
 A_c = total area integrated from GC-AED or FID chromatogram of the standard peak,
 W_c = weight of C₁₆ standard injected onto the standard tube, ng,
 MW_c = molecular weight of n-C₁₆H₃₄ = 226.45,
 S_w = total area of the wafer sample, cm²,
 F = conversion factor = $(W_T - W_U)/W_T$,
 W_T = total weight of the wafer, g, and
 W_U = weight of unused part of the wafer, g.

NOTE 5: In this calculation, blank subtraction ($-A_b$) is optional.

8.1.1.2 When MS is used:

$$TO = \frac{A_s - A_b}{A_c} \times W_c \times \frac{1}{S_w \times F} \quad (2)$$

where:

TO = total organic contaminants, ng C₁₆ (n-C₁₆H₃₄) equivalent/cm²,
 A_s = total area integrated from GC-MS chromatogram of the sample wafer,
 A_b = total area integrated from GC-MS chromatogram of the blank wafer,
 A_c = total area integrated from GC-MS chromatogram of the standard peak,
 W_c = weight of C₁₆ standard injected onto the standard tube, ng,
 S_w = total area of the wafer sample, cm²,
 F = conversion factor = $(W_T - W_U)/W_T$,
 W_T = total weight of wafer, g, and

W_U = weight of unused part of the wafer, g.

See Note 5.

NOTE 6: Recommended mass range to be scanned is 33 to 700 amu.

8.1.2 Calculation of Total Organophosphorus (TP):

$$TP = \frac{A_s - A_b}{A_p} \times \frac{W_p \times 10^{-9}}{MW_p} \times 6.02 \times 10^{23} \times \frac{1}{S_w \times F} \quad (3)$$

where:

TP = total organophosphorus, P atoms/cm²,
 A_s = total area integrated from GC-(phosphorus selective detector) chromatogram of the sample wafer for phosphorus compounds only,
 A_b = total area integrated from GC-(phosphorus selective detector) chromatogram of the blank wafer,
 A_p = total area integrated from GC-(phosphorus selective detector) chromatogram of the standard peak,
 W_p = weight of phosphorus standard (TCEP or TBP) injected onto the standard tube, ng,
 MW_p = molecular weight of phosphorus standard (TCEP = 285.49, or TBP = 266.3),
 S_w = total area of the wafer sample, cm²,
 F = conversion factor = $(W_T - W_U)/W_T$,
 W_T = total weight of the wafer, g, and
 W_U = weight of unused part of the wafer, g.

See Note 5.

8.2 Method B

8.2.1 Calculation of Total Organic Contaminants

8.2.1.1 When AED or FID is used:

$$TC = \frac{A_s - A_b}{A_c} \times \frac{W_c \times 10^{-9}}{MW_c} \times 6.02 \times 10^{23} \times \frac{1}{S_w} \times 16 \quad (4)$$

where:

TC = total organic carbon, C atoms/cm²,
 A_s = total area integrated from GC-AED or FID, chromatogram of the sample wafer for carbon compounds,
 A_b = total area integrated from GC-AED or FID chromatogram of the blank wafer,
 A_c = total area integrated from GC-AED or FID chromatogram of the standard peak,
 W_c = weight of C₁₆ standard injected onto the standard wafer, ng,
 MW_c = molecular weight of n-C₁₆H₃₄ = 226.45,
 S_w = one side area of the wafer sample, cm².

See Note 5.

8.2.1.2 When MS is used:

$$TO = \frac{A_s - A_b}{A_c} \times W_c \times \frac{1}{S_w} \quad (5)$$

where:

TO = total organic contaminants, ng C_{16} ($n-C_{16}H_{34}$) equivalent/cm²,

A_s = total area integrated from GC-MS chromatogram of the sample wafer,

A_b = total area integrated from GC-MS chromatogram of the blank wafer,

A_c = total area integrated from GC-MS chromatogram of the standard peak,

W_c = weight of C_{16} standard injected onto the wafer, ng,

S_w = one side area of the wafer sample, cm².

See Notes 5 and 6.

8.2.2 Calculation of Total Organophosphorus (TP):

$$TP = \frac{A_s - A_b}{A_p} \times \frac{W_p \times 10^{-9}}{MW_p} \times 6.02 \times 10^{23} \times \frac{1}{S_w} \quad (6)$$

where:

TP = total organophosphorus, P atoms/cm²,

A_s = total area integrated from GC-(phosphorus selective detector) chromatogram of the sample wafer for phosphorus compounds only,

A_b = total area integrated from GC-(phosphorus selective detector) chromatogram of the blank wafer,

A_p = total area integrated from GC-(phosphorus selective detector) chromatogram of the standard peak,

W_p = weight of phosphorus standard (TCEP or TBP) injected onto the wafer, ng,

MW_p = molecular weight of phosphorus standard (TCEP = 285.49 or TBP = 266.3),

S_w = area of one surface of the wafer sample, cm².

See Note 5.

9 Report

9.1 Report the following information:

9.1.1 Sample identification, sample size, sample history including how received or packaged if it has been shipped, side of wafer desorbed (front, back, or

both). (Shipping often can add or remove organic compounds). If the wafer is a witness wafer, report how prepared and exposure conditions, location whether horizontal or vertical, time, flow, etc.

9.1.2 Analysis date, analysis method, wafer desorption time and temperature, purge gas used.

9.1.3 Manufacturer, types, and models of instruments.

9.1.4 Identification of operator.

9.1.5 Testing laboratory.

9.1.6 Software version of instrument.

9.1.7 Analysis results.

9.1.7.1 Total organic contaminants, C atoms/cm² (when AED or FID is used), or ng C_{16} ($n-C_{16}H_{34}$) equivalent/cm² (when MS is used).

9.1.7.2 Total organophosphorus, P atoms/cm².

9.1.8 Blank wafer data. The blank wafer should be treated with the same desorption procedure as the sample wafers.

9.1.9 Calibration data.

10 Precision and Bias

10.1 *Precision* — The precision of these test methods can be determined from the reproducibility measurement of standards. The accuracy of these test methods can be measured from the recoveries of standards.

10.2 *Bias* — Bias for these test methods has not been determined.

11 Keywords

11.1 atomic emission detector (AED); flame ionization detector (FID); flame photometric detector (FPD); gas chromatography; mass spectrometer (MS); nitrogen/phosphorus thermionic ionization detector (NPD); organic contamination; organophosphorus compounds; phosphorus selective detector; silicon wafer surfaces; thermal desorption

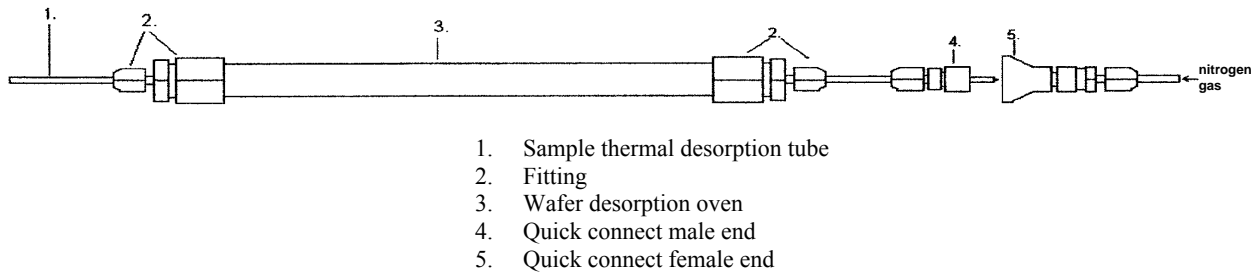


Figure 1
Schematic of Wafer Desorption Setup

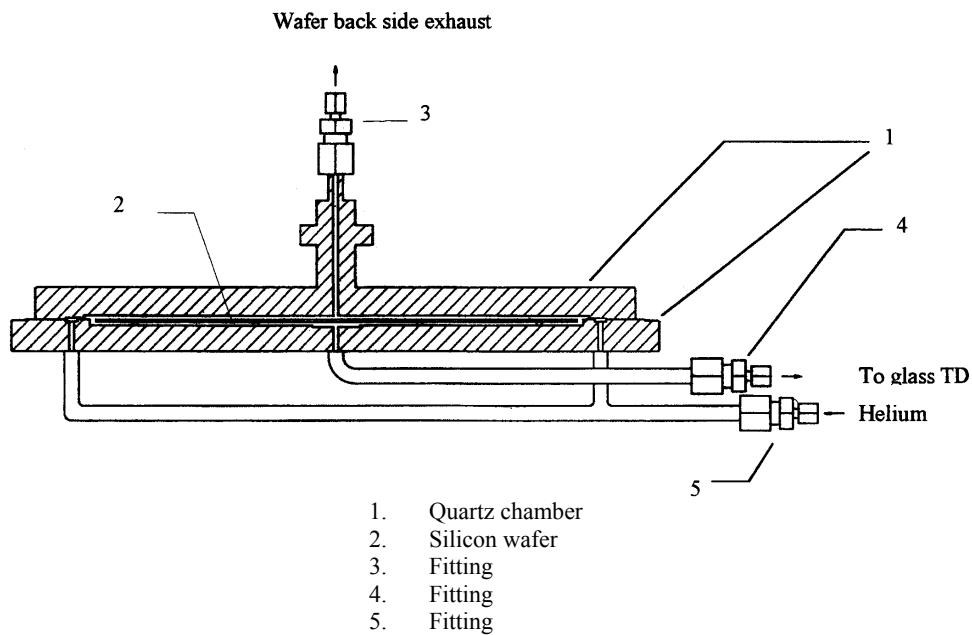


Figure 2
Schematic of Quartz Chamber

NOTICE: SEMI makes no warranties or representations as to the suitability of the standards set forth herein for any particular application. The determination of the suitability of the standard is solely the responsibility of the user. Users are cautioned to refer to manufacturer's instructions, product labels, product data sheets, and other relevant literature, respecting any materials or equipment mentioned herein. These standards are subject to change without notice.

By publication of this standard, Semiconductor Equipment and Materials International (SEMI) takes no position respecting the validity of any patent rights or copyrights asserted in connection with any items mentioned in this standard. Users of this standard are expressly advised that determination of any such patent rights or copyrights, and the risk of infringement of such rights are entirely their own responsibility.

SEMI MF2074-1103

GUIDE FOR MEASURING DIAMETER OF SILICON AND OTHER SEMICONDUCTOR WAFERS

This standard was technically approved by the Global Silicon Wafer Committee and is the direct responsibility of the North American Silicon Wafer Committee. Current edition approved for publication by the North American Regional Standards Committee on September 3, 2003. Initially available at www.semi.org October 2003; to be published November 2003. Originally published by ASTM International as ASTM F 2074-00. Last previous edition ASTM F 2074-00.

1 Purpose

1.1 The diameter of semiconductor wafers is an important parameter in microelectronic fabrication. Wafer diameters must conform to the limits specified in SEMI M1; or other agreed upon limits, or product that is otherwise suitable may not fit correctly in process equipment.

1.2 Cam-follower edge rounding usually results in wafers that are circular. However, wafers ground with edge-follower edge rounding equipment may be elliptically shaped. Measurements made at the three positions specified in this guide do not provide complete information about the roundness of the wafer.

2 Scope

2.1 This guide defines standardized positions for measuring diameter of circular wafers of silicon and other semiconducting materials that contain flats or notches (fiducials) on the periphery. It was developed for use with silicon wafers with standard diameter and fiducial positions as given in SEMI M1.

2.2 It may be applied to other semiconductor wafers if the flat locations are properly taken into account.

2.3 No recommendations are given regarding the test instrumentation to be used, but either contacting (taking suitable precautions to avoid edge chipping) or non-contacting gauges have been found to be satisfactory. Wafers of any size can be measured provided that suitable test jigs and instruments are available.

2.4 Roundness of wafers cannot be determined from measurements made solely at the positions defined in this guide. No information is provided concerning the diameter of the wafer at points other than those measured.

NOTICE: This standard does not purport to address safety issues, 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 or other limitations prior to use.

3 Limitations

3.1 The presence of an indent or uneven edge on the periphery of the wafer at the measurement position can cause the resulting diameter measurement to be in error.

3.2 If the wafer is not circular, the measured diameter may differ from that found by measuring at different positions or based on a different number of measurements.

4 Referenced Standards

4.1 SEMI Standards

SEMI M1 — Specifications for Polished Monocrystalline Silicon Wafers

SEMI M20 — Specification for Establishing a Wafer Coordinate System

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

5 Terminology

5.1 Definitions

5.1.1 *diameter* — of a semiconductor wafer, the linear dimension across the surface of a circular wafer that contains the wafer center and excludes flats or other peripheral fiduciary geometries.

5.1.2 *indent* — of a semiconductor wafer, an edge defect that extends from the front surface to the back surface.

6 Measurement Positions

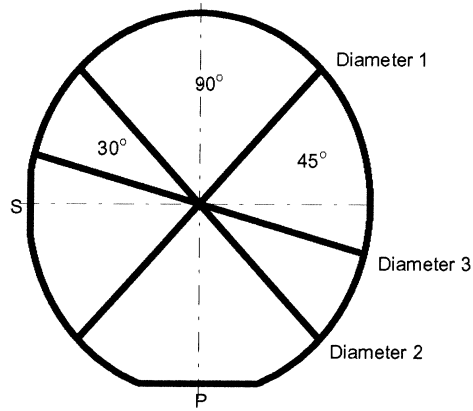
6.1 Measure the diameter at the three positions listed in Table 1 for the appropriate fiducial position(s) (see Figure 1). The angular positions in Table 1 are given in degrees from the *x*-axis as defined in SEMI M20.

6.2 Calculate the diameter of the wafer as the average of the diameters measured at the three positions.

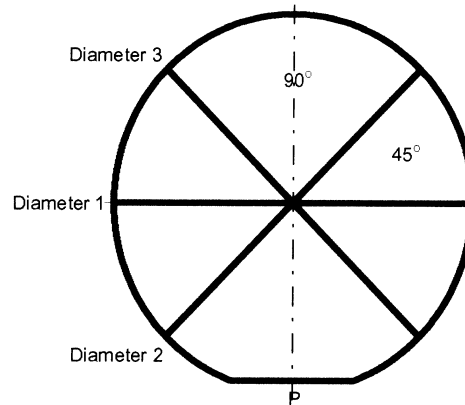
6.3 The relationships between wafer configurations and wafer conductivity types and orientations as defined in SEMI M1 are given in Table 2.

7 Keywords

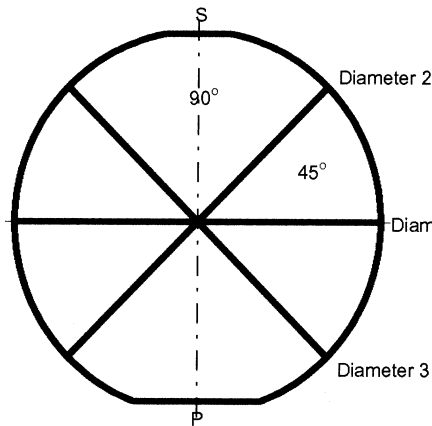
7.1 diameter, semiconductor, silicon, wafer



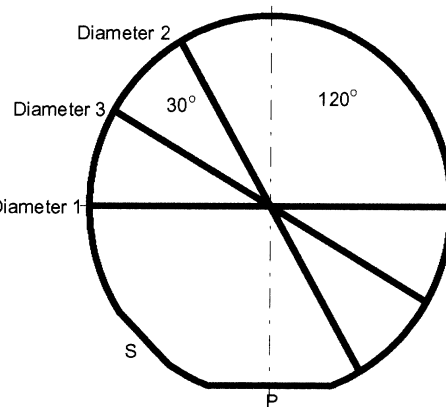
Configuration 1: flatted p [100] wafer with secondary flat



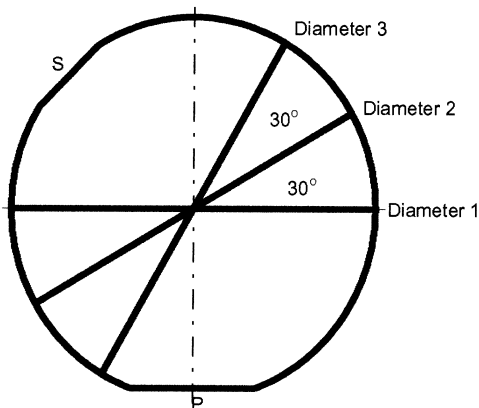
Configuration 2: any notched or flatted wafer without a secondary flat regardless of type or orientation (including flatted p [111] wafer)



Configuration 3: flatted n [100] wafer with secondary flat (diameter 125 mm and smaller)



Configuration 4: flatted n [111] wafer with secondary flat



Configuration 5: flatted n [100] wafer with secondary flat (diameter 150 mm only)

P = primary flat or notch location
S = secondary flat location

Figure 1
Diameter Measurement Positions by Wafer Fiducials Category

Table 1 Positions for Measurement of Wafer Diameter

<i>Configuration</i>	<i>Primary Fiducial</i>	<i>Secondary Flat</i>	<i>Diameter 1</i>	<i>Diameter 2</i>	<i>Diameter 3</i>
1	270°	180°	45°	135°	165°
2	270°	none	0°	45°	135°
3	270°	90°	0°	45°	135°
4	270°	225°	0°	120°	150°
5	270°	135°	0°	30°	60°

Table 2 Wafer Types and Orientations Corresponding to Measurement Configuration of Table 1

<i>Configuration</i>	<i>Conductivity Type</i>	<i>Orientation</i>	<i>Notes</i>
1	<i>p</i>	[100]	Applies only to [100] <i>p</i> -type wafers, 150 mm and smaller, with secondary flats
2	<i>p</i>	[111]	Applies both to [111] <i>p</i> -type wafers, 150 mm and smaller, (without secondary flats) and to all other notched and flatted wafers without secondary flats regardless of conductivity type and orientation
3	<i>n</i>	[100]	Applies only to [100] <i>n</i> -type wafers, 125 mm and smaller, with secondary flats
4	<i>n</i>	[111]	Applies only to [111] <i>n</i> -type wafers, 150 mm and smaller, with secondary flats
5	<i>n</i>	[100]	Applies only to [100] <i>n</i> -type 150 mm wafers with secondary flats

NOTICE: SEMI makes no warranties or representations as to the suitability of the standards set forth herein for any particular application. The determination of the suitability of the standard is solely the responsibility of the user. Users are cautioned to refer to manufacturer' s instructions, product labels, product data sheets, and other relevant literature, respecting any materials or equipment mentioned herein. These standards are subject to change without notice.

By publication of this standard, Semiconductor Equipment and Materials International (SEMI) takes no position respecting the validity of any patent rights or copyrights asserted in connection with any items mentioned in this standard. Users of this standard are expressly advised that determination of any such patent rights or copyrights, and the risk of infringement of such rights are entirely their own responsibility.

SEMI MF2139-1103

TEST METHOD FOR MEASURING NITROGEN CONCENTRATION IN SILICON SUBSTRATES BY SECONDARY ION MASS SPECTROMETRY

This standard was technically approved by the Global Silicon Wafer Committee and is the direct responsibility of the North American Silicon Wafer Committee. Current edition approved for publication by the North American Regional Standards Committee on July 27, 2003. Initially available at www.semi.org October 2003; to be published November 2003. Originally published by ASTM International as ASTM F 2139-01. Last previous edition ASTM F 2139-01.

1 Purpose

1.1 Secondary ion mass spectrometry (SIMS) can measure in un-annealed, polished Czochralski (CZ) silicon substrates the nitrogen concentration that may be intentionally introduced to: (1) increase the V/G tolerance for grown-in defects free region, where V is the pull rate and G is the crystal temperature gradient at the solid-liquid interface;¹ (2) increase the void-free denuded zone depth and the bulk micro-defect density after annealing in hydrogen or argon;^{2,3} (3) reduce the crystal originated particle (COP) size after annealing;^{2,3} or (4) enhance the precipitation of oxygen in epitaxial substrates under reduced temperature processing.⁴

1.2 SIMS can measure total bulk nitrogen in CZ-silicon, whereas infrared spectroscopy is negatively affected by the chemical state in oxygen-containing silicon.⁵ In addition, SIMS can measure the total bulk nitrogen in $p^+(B)$ and $n^+(Sb)$ substrates used for epitaxial silicon, whereas infrared spectroscopy cannot due to free electron absorption interferences.

1.3 SIMS can measure in un-annealed, polished Float-zoned (FZ) silicon substrates the nitrogen concentration

that may be introduced to strengthen low oxygen substrates.

1.4 The SIMS method can be used for process check of crystal doping, and for research and development.

2 Scope

2.1 This test method covers the determination of total nitrogen concentration in the bulk of single crystal substrates using secondary ion mass spectrometry (SIMS).^{6,7}

2.2 This test method can be used for silicon in which the dopant concentrations are less than 0.2% (1×10^{20} atoms/cm³) for boron, antimony, arsenic, and phosphorus.

2.3 This test method is for bulk analysis where the nitrogen concentration is constant with depth.

2.4 This test method can be used for silicon in which the nitrogen content is 1×10^{14} atoms/cm³ or greater. The detection capability depends upon the SIMS instrumental nitrogen background and the precision of the measurement.

2.5 This test method is complementary to infrared spectroscopy, electron paramagnetic resonance, deep level transient spectroscopy, and charged particle activation analysis.⁸ The infrared spectroscopy method detects nitrogen in specific vibrational states, rather than total nitrogen, and is limited to silicon with doping concentrations less than about 1×10^{17} atoms/cm³. The

1 Iida, M., Kusaki, W., Tamatsuka, M., Iino, E., Kimura, M., and Muraoka, S., "Effects of Light Element Impurities on the Formation Grown-In Defects Free Region of Czochralski Silicon Single Crystal," in *Defects in Silicon III*, edited by W. M. Bullis, W. Lin, P. Wagner, T. Abe, and S. Kobayashi, The Electrochemical Society Proceedings Series PV99-1 (The Electrochemical Society, Pennington, NJ, 1999) pp. 499-510.

2 Tamatsuka, M., Kobayashi, N., Tobe, S., and Masui, T., "High Performance Silicon Wafer with Wide Grown-in Void Free Zone and High Density Internal Gettering Site Achieved via Rapid Crystal Growth with Nitrogen Doping and High Temperature Hydrogen and/or Argon Annealing," *ibid.*, pp. 456-467.

3 Minami, T., Takeda, R., Saito, H., Hirano, Y., Suzuki, O., Nitta, S., Kashima, K., and Matsushita, Y., "Influence of Void Size on the Formation of Defect Free Regions in Hydrogen Annealed CZ Silicon Wafers," ECS Extended Abstract No. 514, 197th Meeting of the Electrochemical Society, (The Electrochemical Society, Pennington, NJ, 2000).

4 Shimura, F., and Hockett, R. S., "Nitrogen effect on oxygen precipitation in Czochralski silicon," *Appl. Phys. Lett.* **48**, 224-226 (1986).

5 Abe, T., Kikuchi, K., Shirai, S., and Muraoka, M., in *Semiconductor Silicon 1981*, edited by H. R. Huff, R. J. Kriegler and Y. Takeishi, (The Electrochemical Society, Pennington, NJ, 1981) pp. 54-71.

6 Hockett, R. S., Evans, Jr., C. A., and Chu, P. K., "The SIMS Measurement of Nitrogen in Nitrogen-Doped CZ-Silicon," in *Secondary Ion Mass Spectrometry SIMS VI*, edited by A. Benninghoven, A. M. Huber, and H. W. Huber, (John Wiley & Sons, New York, 1988) pp. 441-444.

7 Hockett, R. S. and Sams, D. B., "The Measurement of Nitrogen in Silicon Substrates by SIMS," in *High Purity Silicon VI*, edited by C. L. Claeys, P. Rai-Choudhury, M. Watanabe, P. Stallhofer, and H. J. Dawson, ECS Proceedings Vol PV 2000-17 (The Electrochemical Society, Pennington, NJ, 2000) pp. 584-595.

8 Stein, Herman J., "Nitrogen in Crystalline Si," in *Materials Research Society Symposia Proceedings Vol 59, Oxygen, Carbon, Hydrogen and Nitrogen in Crystalline Silicon*, edited by J. C. Mikkelsen, Jr., S. J. Pearton, J. W. Corbett, and S. J. Pennycook, (Materials Research Society, Pittsburgh, PA, 1986) pp. 523-535.

charged particle activation analysis detection capability is limited by an interference from boron.

NOTICE: This standard does not purport to address the safety issues, 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 or other limitations prior to use.

3 Limitations

3.1 Nitrogen on or in the surface silicon oxide can interfere with the bulk nitrogen measurement.

3.2 Nitrogen adsorbed on the test specimen surface from the SIMS instrument chamber and fixtures interfere with the bulk nitrogen measurement by raising the background signal. The vacuum quality of the SIMS instrument can be used to minimize this.

3.3 Nitrogen in the SIMS primary Cs beam may be implanted into the silicon specimen as CsN and thereby increase the nitrogen background concentration. A primary beam mass filter may be used to reduce this interference, but in this case, a reduced Cs beam current density is needed to maximize the sputter rate.

3.4 Anomalous nitrogen intensity spikes can interfere with the averaging of signal intensity which is assumed to be random (see Section 6.9).

3.5 Carbon introduces an interference as $^{12}\text{C}^{30}\text{Si}$ at mass 42 for detecting nitrogen as $^{14}\text{N}^{28}\text{Si}$. This can be avoided by detecting the nitrogen as $^{14}\text{N}^{29}\text{Si}$ at mass 43, but the signal rate is reduced greatly, about a factor of 20 when the minor isotope of silicon is used. There are methods to measure the carbon interference, and subtract this interference. One of the methods to measure the carbon interference can have its own interference from high levels of boron dopant.

3.6 The specimen surface must be flat in the specimen holder window so that the inclination of the specimen surface with respect to the ion collection optics is constant from specimen to specimen. Otherwise, the accuracy and precision can be degraded.

3.7 The bias and precision of the measurement significantly degrade as the roughness of the specimen surface increases. This degradation can be avoided by using chemical-mechanical polished wafers.

3.8 Variability of nitrogen in the calibration specimen can limit the measurement precision.

3.9 Variability from the calibration measurement may increase the measurement precision of the test specimen.

3.10 Bias in the assigned nitrogen concentration of the calibration specimen can introduce bias into the SIMS measured nitrogen.

3.11 Thermal processing above 800° C of the silicon substrate may cause diffusion of the nitrogen, so that the nitrogen concentration is not constant with depth, a key assumption of this test method.

3.12 Thermal processing of the silicon substrates in a nitrogen-containing ambient can introduce large amounts of nitrogen from the ambient deep into the silicon crystal.⁶

4 Referenced Standards

4.1 ASTM Standards⁹

E 122 — Practice for Calculating Sample Size to Estimate, with a Specified Tolerable Error, the Average for a Characteristic of a Lot or Process

E 673 — Terminology Relating to Surface Analysis

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

5 Terminology

5.1 *Definitions* — all terms in this test method are in accordance with those given in ASTM Terminology E 673.

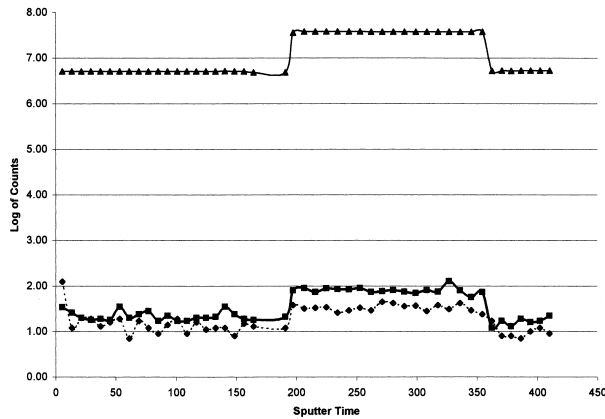
6 Summary of Test Method

6.1 SIMS is utilized to determine the bulk concentration of nitrogen in single crystal silicon substrate. Specimens of single crystal silicon (one calibration specimen and the test specimens) are loaded into a sample holder. The holder with the specimens is baked at 100° C in air for 1 h and then transferred into the analysis chamber of the SIMS instrument.

6.2 The calibration sample is analyzed using a cesium (Cs) primary ion beam and negative ion spectrometry of $^{14}\text{N}^{28}\text{Si}$, or $^{15}\text{N}^{28}\text{Si}$, depending upon the nitrogen isotope in the calibration sample, to determine a relative sensitivity factor (*RSF*) of nitrogen in silicon.

6.3 Each test specimen in the sample holder is sputtered by the primary cesium ion beam, without analyzing secondary ion intensities, in order to reduce the nitrogen background. The amount of time required for this pre-analysis sputtering depends upon the instrumentation and the desired background nitrogen concentration.

⁹ Available from ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, USA. Telephone: 610.832.9585, Fax: 610.832-9555, Web site: www.astm.org. ASTM E 122 is published in Volume 14.02 of *Annual Book of ASTM Standards* while ASTM E 673 is published in Volume 3.06.



NOTE: Top curve (▲) Mass 30 (^{30}Si); middle curve (■) Mass 42 ($^{14}\text{N}^{28}\text{Si}$), bottom curve (◆) Mass 40 ($^{12}\text{C}^{28}\text{Si}$)

Figure 1

SIMS Signals in Nitrogen-Doped Silicon, Log Scale

6.4 For the analysis of the test specimens, a cesium primary ion beam is used to bombard each test specimen at two different sputter rates, by reducing the beam raster area for the second sputter rate, while at the same time maintaining a constant detected area. This beam raster change technique is used for two reasons. Firstly, this technique reduces the background nitrogen contribution from the sample surface. Secondly, this technique allows the determination of both the instrumental nitrogen background and the real bulk nitrogen concentration in each test specimen measurement without the use of a separate blank specimen, and due to averaging is capable of separating the bulk nitrogen from the instrumental nitrogen even if the bulk nitrogen is less than the instrumental nitrogen.¹⁰

6.5 The two sputter rates, and the amount of time they are applied in each measurement, are chosen to optimize the detection capability; the values depend on the instrumentation being used. However, the second sputter rate is typically the maximum sputter rate of the instrumentation, and the first sputter rate is typically less than half that of the second sputter rate.

6.6 The negative secondary ion of $^{14}\text{N}^{28}\text{Si}$ is mass analyzed by a mass spectrometer, and detected by an electron multiplier (EM) or equivalent high-sensitivity ion detector as a function of time. The matrix negative secondary ion count for silicon (^{28}Si , ^{29}Si , ^{30}Si , or a

dimer such as $^{30}\text{Si}_2$) is measured by a Faraday cup (FC) or appropriate detector during the profile. If multiple detectors are used during the test, the relative sensitivities of the detectors are determined by measuring standard ion signals (either the same negative secondary ion count rate or ion count rates of known relative intensity such as natural $^{28}\text{Si}/^{30}\text{Si}$) on each detector. Additional negative secondary ions of $^{11}\text{B}^{28}\text{Si}$, ^{12}C , and $^{12}\text{C}^{28}\text{Si}$ may also be detected depending upon the boron and carbon concentration in the test specimen.

6.7 For illustration purposes an example data profile is shown in Figure 1 where the log of counts of $^{12}\text{C}^{28}\text{Si}$, ^{14}N , ^{28}Si , and ^{30}Si is plotted versus sputter time. At about 200 s, the Cs beam raster area was changed from $250\text{ }\mu\text{m} \times 250\text{ }\mu\text{m}$ down to $50\text{ }\mu\text{m} \times 50\text{ }\mu\text{m}$, resulting in an increase in signal intensities, because the volume of material analyzed per second has increased. At about 350 s, the Cs beam raster area was reversed back to the first condition, and the signal intensities are seen to be reduced back to the original levels. Since the surface analysis area was kept constant during the raster change (that is, the actual surface area detected is independent of the raster area), the signal intensities have two separate contributions, one from elements that are adsorbed to the surface and one from elements in the volume of material analyzed each second. There is some nitrogen (and carbon in this case) that is adsorbed to the surface from the local environment outside of the silicon, and there is some nitrogen that is from the bulk silicon. A key point is that if there is no nitrogen in the bulk silicon, the nitrogen signal intensity does not change as a result of the change in sputter rate, because the adsorbed nitrogen (that is, instrumental nitrogen signal) is only dependent on the analysis area, which has been held constant. This understanding leads to the following equations:

$$\frac{I_n}{I_{si}} \times RSF = [N_b] + [N] \quad (1)$$

$$\frac{I_N}{I_{SI}} \times RSF = [N_B] + [N] \quad (2)$$

$$[N_b] = \frac{I_B}{I_{si}} \times RSF \quad (3)$$

$$[N_B] = \frac{I_B}{I_{SI}} \times RSF \quad (4)$$

where:

I_n = secondary ion intensity of $^{14}\text{N}^{28}\text{Si}$ under the higher sputter rate conditions,

I_N = secondary ion intensity of $^{14}\text{N}^{28}\text{Si}$ under the lower sputter rate conditions,

¹⁰ Ishitani, A., Okuno, K., Karen, A., Karen, S. and Soeda, F., "Improvement of Oxygen Detection Limit in Silicon by Use of the Secondary Ion Energy Distribution and Background Subtraction," in *Proceedings of the International Conference of Materials and Process Characterization for VLSI, 1988 (ICMPC'88)*, edited by X-F Zong, Y-Y Wang, and J. Chen, (World Scientific, New Jersey, 1988) pp. 124-129.

$[N_b]$ = contribution of background nitrogen to the observed nitrogen concentration under the higher sputter rate conditions,
 $[N_B]$ = contribution of background nitrogen to the observed nitrogen concentration under the lower sputter rate conditions,
 I_B = secondary ion intensity from the adsorbed background nitrogen under both sputter rate conditions,
 I_{si} = secondary ion intensity of the silicon matrix (e.g., ^{30}Si) under the higher sputter rate conditions,
 I_{sl} = secondary ion intensity of the silicon matrix (e.g., ^{30}Si) under the lower sputter rate conditions,
 RSF = relative sensitivity factor for converting the ion intensity ratio to concentration, and
 $[N]$ = bulk nitrogen concentration in the test specimen.

6.8 Equations (1) through (4) can be used to determine $[N]$ and $[N_b]$ from measured data as follows:

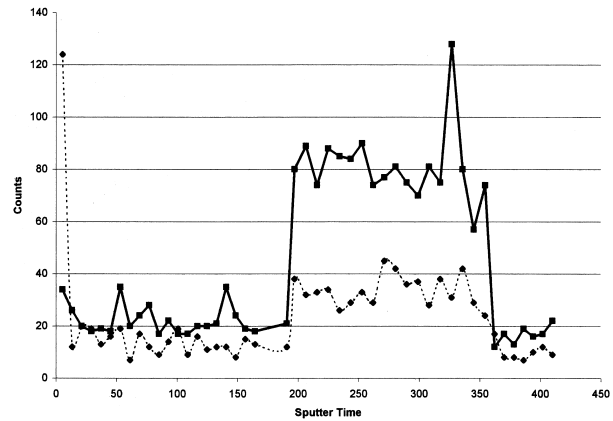
$$[N] = RSF \times \frac{I_n}{I_{si}} \times \frac{1 - (I_N / I_n)}{1 - (I_{sl} / I_{si})} \quad (5)$$

$$[N_b] = RSF \times \frac{I_n}{I_{si}} - [N] \quad (6)$$

In these equations, the secondary ion intensities used are averages of these signal intensities. In the example shown in Figure 1, the lower sputter rate was used at the beginning of the profile, and the higher sputter rate in the middle of the profile. For this example, $[N]$ was determined to be $8 \times 10^{14}/\text{cm}^3$ and $[N_b]$ was $2 \times 10^{14}/\text{cm}^3$. The use of averages for I_n and I_N is particularly critical for detecting low levels of nitrogen where the signal intensity count rates can be low and therefore noisy. By using averages, it is possible to detect an $[N]$ level that is less than $[N_b]$ in the analysis.

6.9 The SIMS measurement of nitrogen in bulk-doped silicon sometimes reveals anomalous intensity spikes that are greater than a random fluctuation of signal intensity, even in silicon that has not been thermally processed beyond crystal growth.^{6,7} These nitrogen intensity spikes are not well understood, but are suspected to be associated with oxygen defects, possibly a co-aggregation of nitrogen and oxygen in a precipitate form. Similar SIMS effects have been well known for oxygen and even carbon in thermally-processed silicon.¹¹ Figure 2 shows an expanded view of the carbon and nitrogen data taken from Figure 1.

The vertical scale is linear. The unusually high nitrogen signal at about 325 s may be one of these anomalous intensity spikes. The presence of such non-random signals, if significant, can make the averaging method used here questionable.



NOTE: Top curve (■) Mass 42 ($^{14}\text{N}^{28}\text{Si}$), bottom curve (◆) Mass 40 ($^{12}\text{C}^{28}\text{Si}$)

Figure 2
SIMS Signals from Nitrogen-Doped Silicon, Linear Scale

6.10 Depending upon the carbon concentration in the test specimen and the level of nitrogen, it may be necessary to correct the $^{14}\text{N}^{28}\text{Si}$ signal for an interference from $^{12}\text{C}^{30}\text{Si}$. This correction is done by co-detecting $^{12}\text{C}^{28}\text{Si}$ at mass 40, and calculating the contribution of $^{12}\text{C}^{30}\text{Si}$ from isotopic ratios. The contribution to mass 42 from a carbon-related signal at mass 40 is 0.0341 times the signal at mass 40. In the example in Figure 1, where the actual carbon concentration is about $1.5 \times 10^{15}/\text{cm}^3$ by SIMS calibration, the carbon interference is insignificant. A carbon concentration of $5 \times 10^{15}/\text{cm}^3$ (the detection limit of most Fourier Transform Infrared (FTIR) measurements of carbon in silicon) corresponds to an interference equivalent to about $8 \times 10^{13}/\text{cm}^3$ of nitrogen. Thus, in detecting nitrogen in the low $10^{14}/\text{cm}^3$ level, a co-measurement of the carbon interference is critical.

6.11 Depending upon the boron concentration in the test specimen, the procedure to correct for the carbon interference may need to be modified by detecting $^{11}\text{B}^{28}\text{Si}$ at mass 39, and calculating the contribution of $^{11}\text{B}^{29}\text{Si}$ and $^{10}\text{B}^{30}\text{Si}$ at mass 40. This procedure becomes important for test specimens with boron concentrations at $1 \times 10^{17}/\text{cm}^3$ and greater. In the example in Figure 1, the boron concentration is on the order of $10^{15}/\text{cm}^3$ and does not interfere.

11 Hockett, R. S., Fraundorf, P. B., Reed, D. A., and Wayne, D. R., "Oxygen and Carbon Defect Characterization in Silicon by SIMS," in *Oxygen, Carbon, Hydrogen and Nitrogen in Crystalline Silicon*, edited by J. C. Mikkelsen, Jr., S. J. Pearton, J. W. Corbett, and S. J. Pennycook, Materials Research Society Symposia Proceedings Vol 59 (Materials Research Society, Pittsburgh, PA, 1986) pp. 433-438.

7 Apparatus

7.1 SIMS Instrument — Equipped with a cesium primary ion source, electron multiplier detector, and Faraday cup detector, capable of measuring negative secondary ions. The SIMS instrument should be adequately prepared (that is, baked) so as to provide the lowest possible instrumental background, since the instrumental background can affect the detection capability. High quality vacuum is desired. A liquid nitrogen- or liquid helium-cooled cryopanel, which surrounds the test specimen holder in the analysis chamber, may be helpful.

7.2 Test Specimen Holder

7.3 Oven — For baking the test specimen holder.

7.4 Stylus Profilometer — Or equivalent device to measure SIMS crater depths. This device is required to calibrate depth scale for concentration profiles of calibration standard samples.

8 Reagents and Materials

8.1 No reagents or materials are required for this test method.

9 Safety Precautions

9.1 The preparation of silicon test specimens from silicon substrates requires the crystal to be cleaved or broken. This procedure may generate very sharp silicon shards. Care must be taken to protect the body and particularly the eyes from these shards.

10 Sampling, Test Specimens, and Test Units

10.1 Since this procedure is destructive in nature, a sampling procedure must be used to evaluate the characteristics of a group of silicon wafers. No general sampling procedure is included as part of this test method, because the most suitable sampling plan depends upon individual conditions. For referee purposes, a sampling plan shall be agreed upon before conducting the test. See ASTM Practice E 122 for suggested choices of sampling plans.

10.2 The calibration or reference material shall be silicon crystal with boron concentration below 2×10^{17} atoms/cm³ and implanted with ions of ¹⁴N or ¹⁵N; an ion implant of energy 200 keV and dose of about 1×10^{14} atoms/cm² is preferred.

10.3 Sample specimens must have a chemo-mechanical polished surface on the side used for analysis.

11 Calibration and Standardization

11.1 Calculate the *RSF* for an element in a matrix experimentally from the SIMS profile of a reference

material containing a known areal density of the impurity of interest as follows:

$$RSF = \frac{D n I_m t}{d(SI_i - I_b n)} \quad (7)$$

where:

- D* = a real density of the impurity, atoms/cm²,
- n* = number of data cycles in the profile,
- I_m* = matrix isotope secondary ion intensity, counts/s,
- d* = sputtered depth, cm,
- SI_i* = sum of the impurity isotope secondary ion (counts over the depth of the profile),
- I_b* = constant background intensity of the impurity isotope, counts/cycle and
- t* = analysis time for the species of interest, s/cycle.

11.2 Calculate the calibration of concentration in the SIMS profile as follows:

$$C_i = \frac{I_i}{I_m} \times RSF \times \frac{FC}{EM} \quad (8)$$

where:

- C_i* = impurity atomic concentration, atoms/cm³,
- I_i* = isotope secondary ion intensity, counts/s,
- I_m* = matrix isotope secondary ion-intensity, counts/s,
- RSF* = relative sensitivity factor, atoms/cm³, and
- $\frac{FC}{EM}$ = ratio of matrix intensities on the Faraday cup (FC) to the electron multiplier (EM), when two detectors are used.

11.3 Complete the conversion of data cycles or time into depth by measuring the crater depth and the total time (or data cycles) of the sputtering that formed the crater.

NOTE 1: The depth scale is assumed to be linear with time or data cycles.

12 Procedure

12.1 Cleave or break specimens from the silicon substrates in order to fit within the SIMS sample holder. Specimens must be handled under normal analytical laboratory practices, but no special ambient is required.

12.2 Load the specimens into the SIMS sample holder, checking to see that the specimens are flat against the backs of the windows and cover the windows as much as is possible. A specimen load includes one calibration specimen, and one or more test specimens.

12.3 Bake the loaded sample holder at $100 \pm 10^\circ$ C for a minimum of 1 h in air.

12.4 Turn on the instrument in accordance with the manufacturer' s instructions.

12.5 If a cryopanel is used, fill the liquid nitrogen or helium cold trap.

12.6 For the calibration specimen use a cesium primary ion beam and detect the negative ion of $^{14}\text{N}^{28}\text{Si}$ or $^{15}\text{N}^{28}\text{Si}$, depending upon the nitrogen isotope used in the ion implantation; detect during or at the end of the profile a matrix negative ion (^{28}Si , ^{29}Si , or ^{30}Si). After the entire sample load is analyzed, measure the SIMS crater depth in this sample using a profilometer.

12.7 Position the specimen holder to sputter a crater in a test specimen near the center of the window. Without detecting secondary ion intensities, begin a SIMS profile.

12.8 Repeat Section 12.7 for each test specimen.

12.9 If the boron level in the test specimen is below $1 \times 10^{17}/\text{cm}^3$, detect the negative ions of $^{12}\text{C}^{28}\text{Si}$, $^{14}\text{N}^{28}\text{Si}$, and a matrix signal (^{28}Si , ^{29}Si , ^{30}Si , or $^{30}\text{Si}_2$). If the boron level is at or above $1 \times 10^{17}/\text{cm}^3$, include the detection of the negative ion of $^{11}\text{B}^{28}\text{Si}$.

12.10 For the analysis of the test specimens, select two raster conditions, one to maximize the sputter rate, and one that provides a slower sputter rate. Choose apertures or other means to maintain the analysis area under raster change and to keep the negative ion count rates on the electron multiplier detector below 1×10^5 counts/s for the test specimen.

12.11 Position the specimen holder to sputter a new crater near the crater created in Section 12.7 or Section 12.8. Do not make the second crater in the same crater created in Section 12.7 or Section 12.8.

12.12 Center the primary ion beam and begin a SIMS profile with the raster area that produces the lower sputter rate. Continue the profile until all signals provide relatively constant intensity for at least 20 data points.

12.13 Change the raster area to maximize the signal intensity. Continue the profile under this raster condition for at least 20 data points.

12.14 Reduce the raster area back to the original condition.

12.15 If the boron content is less than $1 \times 10^{17}/\text{cm}^3$, subtract 0.0341 times the measured mass 40 signal (counts/s) from the measured mass 42 signal (counts/s) to obtain the corrected mass 42 signal corresponding to real nitrogen ($^{14}\text{N}^{28}\text{Si}$).

NOTE 2: If mass 40 carbon ($^{12}\text{C}^{28}\text{Si}$) is defined = 1, mass 42 ($^{12}\text{C}^{30}\text{Si} + ^{13}\text{C}^{29}\text{Si}$) = $0.0335 + 0.0006 = 0.0341$.

NOTE 3: A hydride correction should be negligible for H on the order of 10^{19} atoms/ cm^3 or less.

12.16 If the boron concentration is at or greater than $1 \times 10^{17}/\text{cm}^3$, subtract 0.0583 times the mass 39 signal from the carbon correction to correct for the boron interference.

NOTE 4: The mass 39 boron ($^{11}\text{B}^{28}\text{Si} + ^{10}\text{B}^{29}\text{Si}$) is defined = 1, and mass 40 ($^{11}\text{B}^{29}\text{Si} + ^{10}\text{B}^{30}\text{Si}$) = 0.0583.

12.17 Calculate the bulk nitrogen concentration [N] and the background nitrogen concentration [N_b] for each test specimen using Equations 5 and 6.

12.18 Record the specimen identification.

13 Report

13.1 Report the following information:

13.2 The instrument used, the operator, and the date of the measurements,

13.3 Identification of the test and calibration specimens,

13.4 The bulk nitrogen concentration values for the test specimens,

13.5 The nitrogen instrumental background concentration during the faster sputter rate, and

13.6 Whether anomalous nitrogen spikes were present, and if so, whether the averaging included or excluded these spikes.

14 Precision and Bias

14.1 The precision of the nitrogen determination has not yet been determined either by pilot study or by interlaboratory test.

14.2 There is no accepted reference material for determining the bias for the procedure in this test method. Therefore, no statement on bias can be made.

15 Keywords

15.1 nitrogen concentration; secondary ion mass spectrometry; silicon; SIMS



NOTICE: SEMI makes no warranties or representations as to the suitability of the standards set forth herein for any particular application. The determination of the suitability of the standard is solely the responsibility of the user. Users are cautioned to refer to manufacturer's instructions, product labels, product data sheets, and other relevant literature, respecting any materials or equipment mentioned herein. These standards are subject to change without notice.

By publication of this standard, Semiconductor Equipment and Materials International (SEMI) takes no position respecting the validity of any patent rights or copyrights asserted in connection with any items mentioned in this standard. Users of this standard are expressly advised that determination of any such patent rights or copyrights, and the risk of infringement of such rights are entirely their own responsibility.

SEMI MF2166-0304

PRACTICES FOR MONITORING NON-CONTACT DIELECTRIC CHARACTERIZATION SYSTEMS THROUGH USE OF SPECIAL REFERENCE WAFERS

This standard was technically approved by the Global Silicon Wafer Committee and is the direct responsibility of the North American Silicon Wafer Committee. Current edition approved for publication by the North American Regional Standards Committee on October 16, 2003. Initially available at www.semi.org February 2004; to be published March 2004. Originally published by ASTM International as ASTM F 2166-02. Last previous edition SEMI MF2166-02.

1 Purpose

1.1 Non-contact dielectric characterization systems (NCDCS) that supply surface charge by means of a corona instead of an electrode are frequently used to determine the oxide characteristics of MOS device structures. These instruments can measure a number of characteristic parameters, including:

1.1.1 initial surface voltage (V_{surf} or V_{cpd}),

1.1.2 flatband voltage (V_{fb}),

1.1.3 effective charge (Q_{eff}),

1.1.4 density of interface traps (D_{it}),

1.1.5 mobile charge (Q_m), and

1.1.6 electrical dielectric thickness (T_{ox}).

1.2 To determine that the NCDCS is performing correctly, it is necessary to test the tester. This practice provides procedures for testing these systems with the use of special reference wafers with specified characteristics.

1.3 The procedures in these practices are designed to ensure that the NCDCS being evaluated provides reliable data for selected device-related parameters.

1.4 Flatband voltage is an important parameter in the fabrication of MOS devices. Its value is dependent on the charge structure within and on the dielectric as well as at the silicon-dielectric (Si-SiO₂) interface. These charges consist of interface and bulk trapped and fixed charge, mobile charge distributed within the dielectric, and charge residing on the top of the dielectric. Although the total charge in and on the oxide determines flatband voltage, the charge near the silicon has the greatest effect.

1.4.1 Most device applications require that the mobile charge be minimized to provide stable threshold voltage. Stability of the charges at elevated temperatures is a measure of mobile charge density within the dielectric. The mobile charge changes location under temperature and electric field stress. The mobile charge density can be calculated from the resulting change in flatband voltage.

1.4.2 Equipment used to produce dielectrics can become contaminated in various ways. When such equipment becomes contaminated, it usually introduces an increased level of mobile ions into the dielectric.

1.4.3 Measuring V_{surf} or V_{cpd} , the contact potential between the probe and the semiconductor, with a Kelvin or Monroe probe allows a quantitative measurement of the charges associated with the semiconductor surface and the dielectric, independent of top (contact) electrode variations as in MOS C-V measurements. A special reference wafer has the mobile ions confined to the top half of the dielectric. It is therefore a good check of the Kelvin or Monroe probe, which is most sensitive to charges near the top of the dielectric.

1.5 The procedures in this practice can be used to ascertain that the dielectric characterization system(s) meet quality control requirements. They can be used as a training tool for tester applications.

1.6 The results obtained by these measurements can also be used to expose and troubleshoot tester problems including wafer handling and software issues.

2 Scope

2.1 These practices describe the use of wafers with special electrical and physical characteristics for controlling and monitoring performance of non-contact dielectric characterization systems (NCDCS) that employ corona. They apply in general to dielectrics with charge structure that can be measured by the characterization system. Their applicability to dielectrics of less than 3-nm electrical oxide thickness depends on the capability of the NCDCS. A thickness correlation between the reference wafer(s) and thin-dielectric monitor wafers must be established.

2.1.1 The reference wafers must be designed to check the repeatability and functionality of all the parts and subsystems of the characterization system. Thermally oxidized wafers are not sufficient for this purpose because of parametric drift due to temperature-bias stressing and exposure to the atmosphere.

2.2 Three methods are described to cover various experimental arrangements (see Related Information 1 for a discussion on the two different types of corona).

2.2.1 *Method A* — For systems that employ point source corona,

2.2.2 *Method B* — For systems that employ line source corona and can be programmed to obtain a full set of parameters, and

2.2.3 *Method C* — For systems that employ line source corona but can only determine mobile ion density.

2.3 For each of the three methods, the total testing time can be reduced by using a short-cycle-time modification.

2.4 Special reference wafers are used to determine selected performance characteristics of these dielectric characterization systems, including initial surface voltage (V_{surf} or V_{cpd}), flatband voltage (V_{fb}), effective charge (Q_{eff}), density of interface traps (D_{it}), mobile charge (Q_m), and electrical dielectric thickness (T_{ox}).

2.5 The parameters determined relate to the properties of the NCDCS subsystems in the following way:

2.5.1 V_{fb} and Q_{eff} data are used to check the surface photovoltage (SPV) probe, SPV strobe and corona biasing subsystems, by monitoring behavioral changes through accumulation and around the flat band condition.

2.5.2 D_{it} data are used to check the same subsystems as in Section 2.5.1, by monitoring behavioral changes through the depletion region of the Q_{bias} vs SPV curve.

2.5.3 Q_m data are used to check the temperature-stress subsystem, the corona subsystem, and the Kelvin or Monroe probe.

2.5.4 T_{ox} data are used to check the corona stepping and voltage bias and the Kelvin or Monroe probe air-gap setting.

NOTICE: This standard does not purport to address safety issues, 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 or other limitations prior to use.

3 Limitations

3.1 The presence of light on the wafer surface during the measurements can affect the results obtained and should be controlled according to the system manufacturer's recommendations.

3.2 Relative humidity in the measurement environment above 60% may affect measurement values.

3.3 Stray electromagnetic fields external to the NCDCS may affect measurement results and should be limited according to the system manufacturer's recommendations.

3.4 Wafer handling should be done in accordance with accepted clean room practices, because surface contamination can affect the measurement results.

3.5 Change in the characteristics of the special reference wafers due to handling and other effects may affect measurement results. The special reference wafers must be checked at least once every six months, to assure consistent performance.

4 Referenced Standards

4.1 SEMI Standards

SEMI M20 — Specification for Establishing a Wafer Coordinate System

SEMI MF1241 — Terminology of Silicon Technology

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

5 Terminology

5.1 General terms related to testing of semiconductor materials may be found in SEMI MF1241.

5.2 Definitions

5.2.1 *accumulation condition* — the region of the capacitance-voltage (C-V) curve for which a 5-V increment toward a more negative voltage for *p*-type material, or toward a more positive voltage for *n*-type material, results in a change of less than 1% in the maximum capacitance, C_{max} .

5.2.2 *bias temperature stress, (BTS)* — a voltage applied to the top surface of a dielectric on a wafer whose temperature is maintained at a defined level for a specified time.

5.2.3 *contact potential difference, (V_{cpd})* — the potential measured by a Kelvin or Monroe probe that results from the potential difference between two metals or between a semiconductor and a metal, due to their difference in work function. Also called surface voltage (V_{surf}); see Section 5.2.16.

5.2.4 *corona temperature stress, (CTS)* — a BTS in which bias voltage is supplied by a corona discharge of known charge and voltage applied to the top surface of a dielectric on a wafer whose temperature is maintained at a defined level for a specified time.

5.2.5 *density of interface traps, (D_{it})* — the calculated interface charge per unit area that accounts for the difference in gradient between the measured and

theoretical Q_{bias} vs. SPV curve or equivalent “ Q - Q ” plot, in the band gap region.

5.2.6 *depletion condition* — the condition that exists in the semiconductor surface region when the free carrier density is insufficient to neutralize the fixed dopant charge density of donors and acceptors.

5.2.7 *dielectric (oxide) electrical thickness, (T_{ox})* — the silicon dioxide equivalent thickness (when the silicon dioxide dielectric constant is assumed) calculated from electrical measurement of Q_{bias} vs V_{surf} (or V_{cpd}) for the capacitor.

5.2.8 *effective charge, (Q_{eff})* — the oxide charge/cm² in the capacitor that gives the measured flatband voltage.

5.2.9 *flatband condition* — the point at which an external applied voltage causes there to be no internal potential difference across the MOS structure.

5.2.9.1 *Discussion* — In the flatband condition, the SPV is zero.

5.2.10 *flatband voltage, (V_{fb})* — the applied voltage necessary to produce the flatband condition.

5.2.11 *inversion condition* — the condition in which a minority carrier layer is formed at the semiconductor surface separated from the bulk by a depletion region.

5.2.12 *Kelvin probe* — a reference electrode that vibrates perpendicular to the wafer surface and generates an AC signal by varying the electrode-wafer capacitance.

5.2.13 *mobile charge density, (Q_m)* — the calculated charge/cm² that moves in the oxide under temperature and electrical field stress, creating shifts in SPV, V_{fb} , and V_{cpd} (or V_{surf}).

5.2.14 *Monroe probe* — a reference electrode that is stationary and generates an AC signal by the horizontal vibration of a grounded fork which shields the probe from the wafer.

5.2.15 *surface photovoltage, (SPV)* — the change of the electrostatic potential of the silicon surface caused by illumination.

5.2.16 *surface voltage, (V_{surf})* — the potential measured by the Kelvin or Monroe probe that results from the potential difference between two metals or between a semiconductor and a metal, due to their difference in work function. Also called contact potential difference (V_{cpd}); see Section 5.2.3 .

6 Summary of Practices

6.1 Method A

6.1.1 A special reference wafer is loaded into the NCDCS.

6.1.2 A series of tests, which must include CTS, is executed to position mobile ions into a consistent pre-test condition. All CTS times must be at least 4 minutes to allow full movement of the mobile ions.

6.1.3 Three additional identical test sequences are executed to collect data to evaluate performance of the instrument.

NOTE 1: The bias, temperature and time used for the last CTS prior to carrying out the above tests should be employed for these test sequences.

6.1.4 Performance of the NCDCS and its selected components is checked by measuring the following characteristics: initial surface voltage (V_{surf}), flatband voltage (V_{fb}), effective charge (Q_{eff}), density of interface traps (D_{it}), mobile charge (Q_m), and electrical dielectric thickness (T_{ox}).

6.1.5 Measurements are made at three points on the surface of the wafer and the mean, standard deviation and relative standard deviation of the results obtained in the three identical sequences (Section 6.1.3) are computed and recorded.

6.1.6 The results are compared with the values supplied with the special reference wafer to evaluate the NCDCS performance.

6.1.7 If it is desired to check the system over its full range of operation, this procedure is repeated using two or more additional special reference wafers with parameters that cover the typical parameter ranges in Section 7.4 .

6.1.8 *Short-cycle Time Modification of Method A* — The short-cycle time modification is performed in the same manner except that the measurement of Q_m is carried out first, in order to assure consistent alignment of the mobile ions prior to the other measurements. The Q_m measurement is followed by the measurement of the other parameters. No repetitions are required; the test sequence in this modification is only run once.

6.2 Method B

6.2.1 First any remaining residual charge on a special reference wafer is neutralized by immersing it in DI water.

6.2.2 Then the special reference wafer is loaded into the NSCDS and the surface voltage (contact potential difference) is zeroed to within ± 0.3 V.

6.2.3 Then the steps in Sections 6.1.2 –6.1.4 and 6.1.6 are followed.

6.2.4 If it is desired to check the system over its full range of operation, this procedure is repeated using two or more additional special reference wafers with

parameters that cover the typical parameter ranges in Section 7.4 .

6.2.5 Short-cycle Time Modification of Method B — The short-cycle time modification is performed in the same manner except that the measurement of Q_m is carried out first, in order to assure consistent alignment of the mobile ions prior to the other measurements. The Q_m measurement is followed by the measurement of the other parameters. No repetitions are required; the test sequence in this modification is only run once.

6.3 Method C

6.3.1 The steps in Sections 6.2.1 and 6.2.2 are followed.

6.3.2 Then a dummy mobile ion run is performed to position the mobile ions into a consistent pre-test condition.

6.3.3 Then three additional mobile ion runs are made and the average, standard deviation, and relative standard deviation of the mobile ion density are determined.

6.3.4 The results are compared with the mobile ion density data supplied with the special reference wafer to evaluate the system performance.

6.3.5 If it is desired to check the system over its full range of operation, this procedure is repeated using two or more additional special reference wafers with parameters that cover the typical parameter ranges in Section 7.4 .

6.3.6 Short-cycle Time Modification of Method C — The short-cycle time modification is performed in the same manner except that the first CTS time is 4 min and the second is 8 min. Only a single mobile ion run is carried out.

7 Special Reference Wafers

7.1 Fabricate the special reference wafers (SRW) from *p*-type silicon with a multi-layer dielectric including an embedded layer of mobile ions confined to the top portion of the dielectric. These mobile ions drift during temperature-bias stressing. Since the drift occurs only in the top of the dielectric, the Kelvin (Monroe) probe easily senses a shift in V_{surf} (V_{cpd}) for the mobile ion concentration calculation. Confining the mobile ions to the top of the dielectric also ensures that measurements associated with the silicon-dielectric interface are left unchanged after the mobile ion test.

7.2 Limit the thickness of the dielectric on the special reference wafers to between 30 and 70 nm, to provide sufficient distance for the mobile ions to drift while remaining clear of the interface.

7.3 Ensure the dielectric is rugged enough so that repeated use, for example, once a day for six months, of the same test sites does not result in shifts in electrical parameters.

7.3.1 Check parameter drift every 6 months. Parameter drift over time should be less than 10% in six months. If drift exceeds this value, replace the special reference wafers.

7.3.2 Test wafers must be re-calibrated and documented semi-annually by testing the wafers on two documented NCDCSs that have been maintained using this Practice. The results must match within 10%. If the results exceed this value, replace the special reference wafers.

7.4 Test Parameter Ranges — *p*-type special reference wafers should have parameters appropriate for the test application. Parameters in the following ranges cover all anticipated applications of the SRWs.

7.4.1 $V_{fb} = -0.1$ to -5.0 V,

7.4.2 $Q_{eff} = 0.5$ to 10×10^{11} charge/cm²,

7.4.3 $Q_m = 0.7$ to 20×10^{10} charge/cm²,

7.4.4 $D_{it} = 0.5$ to 50×10^{11} charge/cm², and

7.4.5 $T_{ox} = 30$ to 200 nm.

7.5 Supporting Data

7.5.1 Data collected using the appropriate method shall be supplied with each special reference wafer.

7.5.2 Provide appropriate reference data for each wafer on a data sheet similar to that in Figure 1 (Methods A or B) or Figure 2 (Method C).

7.5.3 These values are the targets against which the system is evaluated.

7.6 To determine the suitability of the system over its range of operation, select a set of at least three special reference wafers with values of each parameter that span the ranges given in Section 7.4 .

8 Calibration Data Sheet

8.1 A test plan shall be agreed upon among all parties to the test. Data shall be recorded on a Test Certification Sheet similar to that supplied with the special reference wafers. Examples are shown in Figures 1 and 2.

9 Procedure

9.1 Observe and record the temperature and relative humidity of the room where the testing is taking place.

9.2 **Method A —** for NCDCDs that employ a point source corona:

9.2.1 Place a special reference wafer in the NCDCS.

9.2.2 Program the NCDSCS to measure the following parameters of *p*-type wafers: initial V_{surf} , T_{ox} , Q_{eff} , V_{fb} , D_{it} , and Q_m .

9.2.2.1 Program the CTS portion of the Q_m measurement to run for at least 4 min at a temperature from 170 to 200°C.

9.2.3 Align the point probe over position A, then execute a dummy measurement to stabilize the mobile ions. Repeat this procedure at two other locations. An example would be the locations (0,0; -40,40; and 40, -40), where the *x* and *y* locations are expressed in millimeters, relative to the wafer center, using a Cartesian wafer coordinate system as defined in SEMI M20. (See Figure R1-1.)

9.2.4 Execute three additional, identical runs. Determine and record the parameters listed in Section 9.2.2.

9.2.5 Calculate and record the average, standard deviation, and % standard deviation for each parameter measured at each location in runs 2 through 4. Enter the data on a data sheet such as the example in Figure 1.

9.2.6 To check the NCDSCS over its full range of operation, repeat Sections 9.2.1 –9.2.5 using at least two additional special reference wafers with parameter values that span the typical parameter ranges in Section 7.4.

9.3 Method A, Short-cycle Modification

9.3.1 Execute the procedure in Section 9.2.1.

9.3.2 Next, execute the procedure in Section 9.2.2, except program the NCDSCS to measure Q_m first, followed by the other listed measurements.

9.3.2.1 Run the CTS portion of the measurements for at least 4 minutes at a temperature from 170–200°C.

9.3.3 Then, execute the procedure in Section 9.2.3.

9.3.4 Enter the data on a data sheet such as the example in Figure 1. Use the “Mean” columns, leaving the “Std Dev” and “% Std Dev” columns blank.

9.4 Method B — for NCDSCSs that employ a line source corona and can be programmed to obtain a full set of parameters:

9.4.1 Rinse a special reference wafer in a DI water bath for 10 min then dry for 10 min to naturalize any residual charge.

9.4.2 Place the special reference wafer in the NCDSCS.

9.4.3 Measure the voltage on the wafer surface (V_{cpd}).

9.4.4 Zero the voltage on the wafer (V_{cpd} should be between -0.3 V and +0.3 V). Use positive or negative

corona sweeps as appropriate to erase the wafer voltage. Re-measure and record the voltage on the wafer surface to verify it has been adjusted to zero.

NOTE 2: To determine the voltage change for one corona sweep, measure the voltage on the wafer. Then sweep the wafer with one positive sweep. Re-measure the voltage on the wafer. The difference in voltage on the wafer is the voltage change per corona sweep.

9.4.5 Program the NCDSCS to measure the following parameters of *p*-type wafers: T_{ox} , Q_{eff} , V_{fb} , D_{it} , and Q_m .

9.4.5.1 Program the CTS portion of the Q_m measurement to run for at least 4 min at a temperature from 170 to 200°C.

9.4.6 Choose three test locations such as (0,0; -40,40; and 40,-40). Execute a dummy measurement to stabilize the mobile ions.

9.4.7 Execute three additional, identical runs. Determine and record the parameters listed in Section 9.4.5.

9.4.8 Calculate and record the average, standard deviation, and % standard deviation for each parameter measured at each location in runs 2 through 4. Enter the data on a data sheet such as the example in Figure 1.

9.4.9 To check the NCDSCS over its full range of operation, repeat Sections 9.3.1–9.3.7 using at least two additional special reference wafers with parameter values that span the typical parameter ranges in Section 7.4.

9.5 Method B, Short-cycle Modification

9.5.1 Execute the procedures in Sections 9.2.1 –9.4.4.

9.5.2 Next, execute the the procedure in Section 9.4.5, except program the NCDSCS to measure Q_m first, followed by the other listed measurements.

9.5.2.1 Run the CTS portion of the measurement for at least 4 minutes at a temperature from 170–200°C.

9.5.3 Execute the procedure in Section 9.4.6.

9.5.4 Enter the data on a data sheet such as the example in Figure 1. Use the “Mean” columns, leaving the “Std Dev” and “% Std Dev” columns blank.

9.6 Method C — for NCDSCSs that employ a line source corona but can only determine mobile ion density.

9.6.1 Execute the procedures in Sections 9.4.1 through 9.4.4.

9.6.2 Perform four mobile ion runs as follows:

9.6.2.1 Run (–) Corona sweeps to place –1.2 V on the special reference wafer while measurements are being taken. (Note 2)

9.6.2.2 Run (–) CTS (2 min at 170–200°C) on the special reference wafer while measurements are being taken.

9.6.2.3 Run (+) Corona sweeps to place +2.4 V on the special reference wafer while measurements are being taken.

9.6.2.4 Run (+) CTS (4 min at 170–200°C) on the special reference wafer while measurements are being taken.

9.6.2.5 Calculate mobile ion density (Q_m).

9.6.2.6 Zero the voltage on the special reference wafer using the method outlined in Section 9.4.4 .

9.6.3 Using the data from runs 2 through 4, calculate and report on a data sheet such as the example in Figure 2 the average, standard deviation, minimum, and maximum for each parameter tested. Record the following:

- (+) Corona,
- (+) CTS,
- (–) Corona,
- (–) CTS,
- Corona Voltage/Sweep, and
- mobile charge density (Q_m).

9.6.4 To check the NCDCS over its full range of operation, repeat Sections 9.6.1 –9.6.3 using at least two additional special reference wafers with parameter values that span the typical parameter ranges in Section 7.4 .

9.7 Method C, Short-cycle Modification:

9.7.1 Carry out the procedures in Sections 9.4.1 –9.4.4 .

9.7.2 Perform one mobile ion run as follows:

9.7.2.1 Run (–) Corona sweeps to place –1.2 V on the special reference wafer while measurements are being taken. (Note 2)

9.7.2.2 Run (–) CTS (4 min at 170–200°C) on the special reference wafer while measurements are being taken.

9.7.2.3 Run (+) Corona sweeps to place +2.4 V on the special reference wafer while measurements are being taken.

9.7.2.4 Run (+) CTS (8 min at 170–200°C) on the special reference wafer while measurements are being taken.

9.7.2.5 Calculate mobile ion density (Q_m).

9.7.2.6 Zero the voltage on the special reference wafer using the method outlined in Section 9.4.4 .

9.7.3 Report the data listed in Section 9.6.3 in the “Mean” column of a data sheet such as the example in Figure 2. Ignore the columns labeled “Std Dev,” “Minimum,” and “Maximum.”

10 Interpretation

10.1 Regardless of method used, compare the measured results with the target values supplied with the special reference wafer(s).

10.2 Record as “In tolerance”/“No repair required” any NCDCS for which the results of all measured values are within 10% of the target values.

10.3 Record as “Out of tolerance”/“Repair required” any NCDCS for which any measured value differs from the target value by more than 10%. Repair or recalibrate the system before returning it to production use.

11 Report

11.1 Report the following information for each test carried out in accordance with Method A or Method B (see Figure 1 for an example data sheet that contains all the information to be reported):

11.1.1 Tester identification, including supplier, model number, corona method, and software Rev number,

11.1.2 Date of test,

11.1.3 Test operator identification,

11.1.4 Temperature and relative humidity of the test room,

11.1.5 Characteristics of the special reference wafer used in the test, including serial number,

11.1.6 Three-run average, standard deviation, and relative standard deviation for each parameter value in the list below across all locations,

11.1.6.1 Initial value of surface voltage, V_{surf} (or V_{cpd}), V,

11.1.6.2 Dielectric thickness, T_{ox} , nm,

11.1.6.3 Flatband voltage, V_{fb} , V,

11.1.6.4 Effective charge, Q_{eff} , ions/cm²,

11.1.6.5 Density of traps, D_{it} , ions/cm², and

11.1.6.6 Mobile ion density, Q_m , ions/cm².

11.2 Report the following information for each test carried out in accordance with Method C (see Figure 2 for an example data sheet that contains all the information to be reported):

11.2.1 Tester identification, including supplier, model number, corona method, and software Rev number,

11.2.2 Date of test,

11.2.3 Test operator identification,

11.2.4 Temperature and relative humidity of the test room,

11.2.5 Characteristics of the special reference wafer used in the test, including serial number,

11.2.6 Stress voltages and temperature,

11.2.7 Duration of CTS, min, and

11.2.8 Three-run average, standard deviation, and relative standard deviation of mobile ion density, Q_m , ions/cm².

12 Key Words

12.1 dielectric tester; dielectric trap; effective charge; electrical dielectric thickness; flatband voltage; interface trap; line corona; mobile charge; point corona.

Special Reference Wafer Calibration Data Sheet For Non-Contact Dielectric Characterization System									
System Supplier:		System Model #:		System Software Rev:			Test Date:		
Calibration Organization		Operator ID		Method A — Point Source Corona []			Method B — Line Source Corona []		
Measurement Data Statistics									
	Mean			Std Dev (1 sigma)			% Std Dev (1S%)		
Location	(0,0)	(-40,40)	(40,-40)	(0,0)	(-40,40)	(40,-40)	(0,0)	(-40,40)	(40,-40)
Parameter									
V_{surf} (V) (Method A)									
V_{cpd} (V) (Method B)									
T_{ox} (nm)									
V_{fb} (V)									
Q_{eff} ($\times 10^{11}$ charge/ cm ²)									
D_{it} ($\times 10^{11}$ charge/ cm ²)									
Q_m ($\times 10^{11}$ charge/ cm ²)									
Environmental Data				Special Reference Wafer Used					
Temperature ____ []°F []°C		Relative Humidity		Serial Number		Mfg.	Wafer Dia.	Calibration Dates	
								Last	Next
Equipment Performance Summary				Comments					
In Tolerance []		Out of Tolerance []							
Repair Required Yes [] No []									

Figure 1
Example Data Sheet for Methods A and B

Special Reference Wafer Calibration Data Sheet For Non-Contact Dielectric Characterization System						
System Supplier:		System Model #:		System Software Rev:		Test Date:
Calibration Organization		Operator ID		Method C — Line Source Corona []		
Measurement Data Statistics						
Parameter	Mean	Std Dev (1 sigma)	Minimum	Maximum		
V_{cpd} , (V)						
Corona Voltage per Sweep						
(-) CTS Voltage						
(-) CTS Time						
(-) CTS Temperature						
(+) CTS Voltage						
(+) CTS Time						
(+) CTS Temperature						
Q_m ($\times 10^{11}$ charge/ cm^2)						
Environmental Data			Special Reference Wafer Used			
Temperature _____ []°F _____ []°C		Relative Humidity		Serial Number	Mfg.	Wafer Dia.
Equipment Performance Summary			Comments			
In Tolerance []		Out of Tolerance []				
Repair Required Yes [] No []						

Figure 2
Example Data Sheet for Method C

RELATED INFORMATION 1

CORONA SUBSYSTEMS

NOTICE: This related information is not an official part of SEMI MF2166. It was developed during the original development of the document. This related information was approved for publication by full letter ballot on October 16, 2003.

R1-1.1 Two types of coronas are used in NCDCSs:

R1-1.2 *Point Source Corona* — This corona type applies charge at discrete locations on the wafer surface. The area of the corona is defined by a mask. (See Figure R1-1.)

R1-1.3 *Line Corona* — In this corona, a wire applies a charge uniformly across the entire wafer. The charge level is applied in increments that cause a V_{cpd} change of between 0.3 and 0.6 V. (See Figure R1-2.)

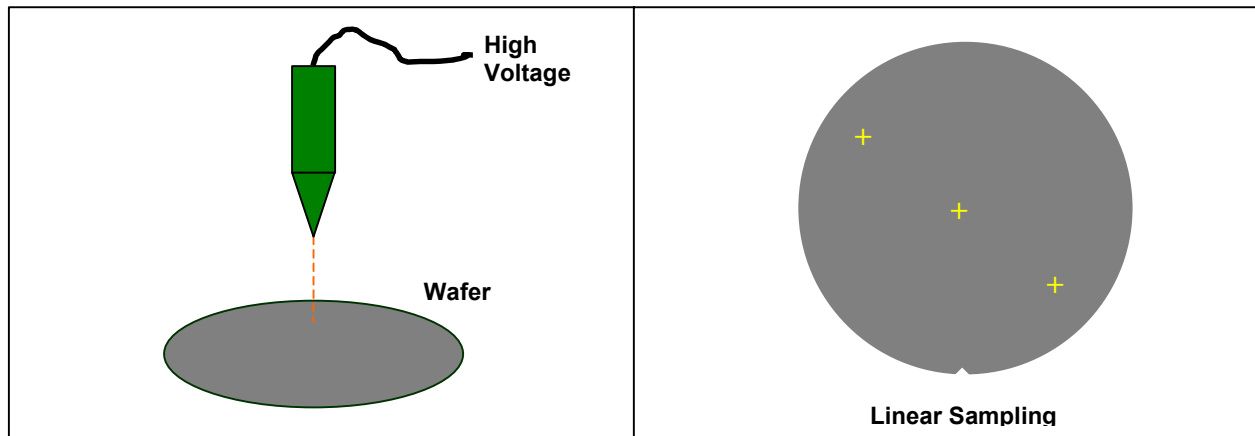


Figure R1-1
Point Corona – Method A

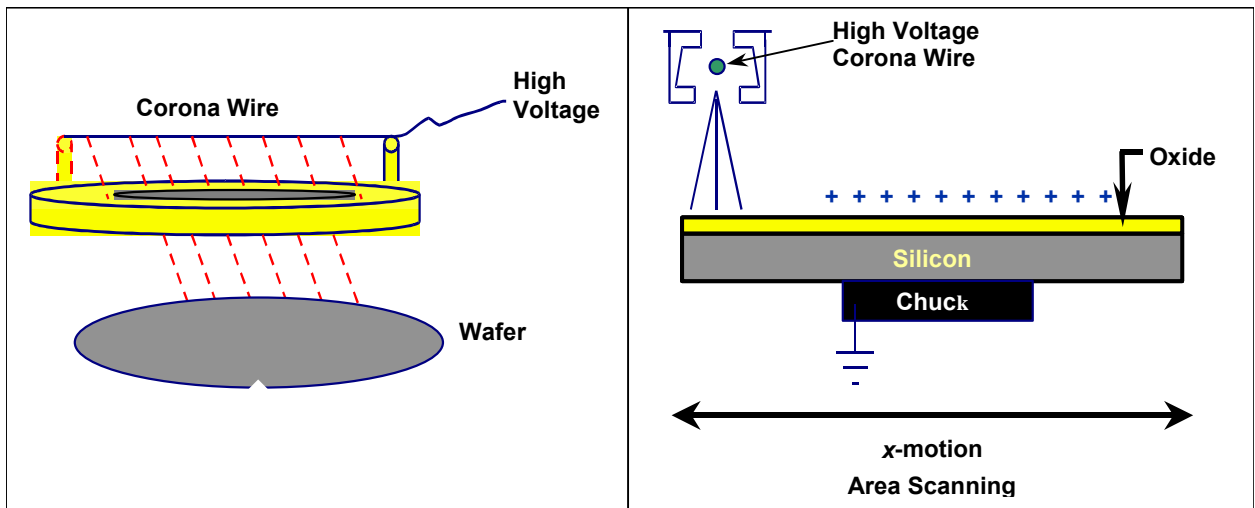


Figure R1-2
Wire Corona – Method B and C



NOTICE: SEMI makes no warranties or representations as to the suitability of the standards set forth herein for any particular application. The determination of the suitability of the standard is solely the responsibility of the user. Users are cautioned to refer to manufacturer's instructions, product labels, product data sheets, and other relevant literature, respecting any materials or equipment mentioned herein. These standards are subject to change without notice.

By publication of this standard, Semiconductor Equipment and Materials International (SEMI) takes no position respecting the validity of any patent rights or copyrights asserted in connection with any items mentioned in this standard. Users of this standard are expressly advised that determination of any such patent rights or copyrights, and the risk of infringement of such rights are entirely their own responsibility.

SEMI INTERNATIONAL STANDARDS



TRACEABILITY

Semiconductor Equipment and Materials International

SEMI T1-95 (Reapproved 0303) SPECIFICATION FOR BACK SURFACE BAR CODE MARKING OF SILICON WAFERS

This specification was technically approved by the Global Traceability Committee and is the direct responsibility of the North American Traceability Committee. Current edition approved by the North American Regional Standards Committee on October 25, 2002. Initially available at www.semi.org December 2002; to be published March 2003. Originally published in 1993; previously published in 1995.

NOTICE: The user's attention is called to the possibility that compliance with this specification may require use of an invention covered by patent rights. By publication of this specification, SEMI takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this specification. Users of this specification are expressly advised that determination of any such patent rights, and the risk of the infringement of such rights, are entirely their own responsibility.

1 Scope

1.1 This specification defines the content and location of a back surface bar code marking for silicon wafers. The code is variable in length from 7 to 18 characters which are user-definable. A character position is held constant for the checksum character, but all else is variable. No other information is encrypted into the code. This allows the performance requirements of related bar code reading equipment to be simplified. In addition, provision is made for an optional alphanumeric marking of the size specified in SEMI M12 to enable operator identification of wafers. This alphanumeric code is not intended to be machine readable.

NOTE 1: The example as described in Table 1 and Figure 1 pertains to a hard mark applied by the silicon wafer manufacturer.

1.2 This specification applies to flatted or notched silicon wafers of nominal diameter 125 mm and larger, as specified in SEMI M1.

1.3 The bar code can be read bi-directionally without impairing its decodability.

NOTICE: This standard does not purport to address safety issues, if any, associated with its use. It is the responsibility of the users of this standard to establish appropriate safety and health practices and determine the applicability of regulatory or other limitations prior to use.

2 Referenced Standards

2.1 SEMI Standards

SEMI M1 — Specifications for Polished Monocrystalline Silicon Wafers

SEMI M12 — Specification for Serial Alphanumeric Marking of the Front Surface of Wafers

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

3 Terminology

3.1 Definitions

3.1.1 *background* — of a bar code symbol — the uniform lighter or more reflective region, including quiet zones, that provides contrast for the darker bars.

3.1.2 *bar* — in a bar code symbol — the darker or less reflective element of the symbol.

3.1.3 *bar code density* — the number of characters, exclusive of quiet zones, that can be represented in a linear unit of measure.

NOTE 2: Bar code density can be expressed in characters per inch (cpi) or characters per millimeter (cpmm).

3.1.4 *bar code symbol* — an array of rectangular bars and spaces that are arranged in a predetermined pattern following specific rules to represent elements of data that are referred to as characters.

NOTE 3: The symbol is comprised of message characters and start/stop characters, check characters, and quiet zones as required by a particular symbology to form a complete scannable entity.

3.1.5 *bar code character* — a single group of bars and spaces which represent an individual number, letter, punctuation mark, or other symbol.

3.1.6 *bar height* — of a bar code symbol — the bar dimension perpendicular to the bar width.

3.1.7 *bar width* — of a bar code symbol — the perpendicular distance across a bar between the points on opposite edges that have a reflectance that is midway between the background and bar reflectance.