NOTICE: This standard has either been superseded and replaced by a new version or discontinued. Contact ASTM International (www.astm.org) for the latest information.

Designation: F 1619 – 95 (Reapproved 2000)

Standard Test Method for Measurement of Interstitial Oxygen Content of Silicon Wafers by Infrared Absorption Spectroscopy with *p*-Polarized Radiation Incident at the Brewster Angle [1]

This standard is issued under the fixed designation F 1619; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method [2] covers determination of the absorption coefficient due to the interstitial oxygen content of commercial monocrystalline silicon wafers by means of Fourier transform infrared (FT-IR) spectroscopy. In this test method, the incident radiation is *p*-polarized and incident on the test specimen at the Brewster angle in order to minimize multiple reflections. [3]

NOTE 1—In this test method, radiation in which the electric vector is parallel to the plane of incidence is defined as *p*-polarized radiation.

NOTE 2—Committee F-1 has been advised that some aspects of this test method may be subject to a patent applied for by Toshiba Ceramics Corporation. [4] The Committee takes no position with respect to the applicability or validity of such patents, but it requests users of this test method and other interested parties to supply any information available on non-patented alternatives for use in connection with this test method.

1.2 Since the interstitial oxygen concentration is proportional to the absorption coefficient of the 1107 cm^{-1} absorption band, the interstitial oxygen content of the wafer can be derived directly using an independently determined calibration factor.

1.3 The test specimen is a single-side polished silicon wafer of the type specified in SEMI Specifications M1. The front surface of the wafer is mirror polished and the back surface may be as-cut, lapped, or etched (see 8.1.1.1).

1.4 This test method is applicable to silicon wafers with resistivity greater than 5 Ω·cm at room temperature.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

F 1188 Test Method for Interstitial Atomic Oxygen Content of Silicon by Infrared Absorption [5]

F 1241 Terminology of Silicon Technology [5]

2.2 *SEMI Standard:*

SEMI M1 Specifications for Polished Monocrystalline Silicon Wafers [6]

3. Terminology

3.1 Definitions of terms related to silicon technology are found in Terminology F 1241.

3.2 Definitions of terms related specifically to FT-IR spectroscopy are found in Test Method F 1188.

4. Summary of Test Method

4.1 The stability of the FT-IR spectrometer is established to be adequate for the measurement cycle.

4.2 The optimum angle of incidence is determined to minimize multiple internal reflection.

4.3 The transmission spectrum of an oxygen-free double-side polished float-zone wafer is recorded.

4.4 The transmission spectrum of the oxygen-containing test specimen is determined.

4.5 The negative logarithm of each of these transmission spectra is taken to determine the absorbance spectra.

4.6 The absorbance spectra are normalized by dividing by the beam path length to obtain the absorption coefficient as a function of wavenumber.

---

[1] This test method is under the jurisdiction of ASTM Committee F01 on Electronics and is the direct responsibility of Subcommittee F01.06 on Silicon Materials and Process Control.

Current edition approved Sept. 15, 1995. Published November 1995.

[2] This standard is based on draft procedures and interlaboratory tests conducted by the Silicon Wafer Committee of the SEMI Japan Standards Program and the Oxygen and Carbon Measurement Committee of the Japan Electronic Industry Development Association (JEIDA).

[3] Krishnan, K., "Precise and Rapid Measurement of Oxygen and Carbon in Silicon," *Defects in Silicon*, edited by W. M. Bullis and L. C. Kimerling, *Proceedings* Volume 83-9, The Electrochemical Society, Pennington, NJ, 1983, pp. 285–292; Shirai, H., "Determination of Oxygen Concentration in Single-Side Polished Czochralski-Grown Silicon Wavers by p-Polarized Brewster Angle Incidence Infrared Spectroscopy," *Journal of The Electrochemical Society*, Vol 138, No. 6, 1991, pp. 1784–1787; Shirai, H., "Oxygen Measurements in Acid-Etched Czochralski-Grown Silicon Wafers," *Journal of The Electrochemical Society*, Vol 139, No. 11, 1992, pp. 3272–3275.

[4] "Measuring Method of Interstitial Oxygen Content of Silicon Wafers," U.S. Patent applied for. Information concerning use of the concepts covered by this patent application and its state of issuance may be obtained from Intellectual Property Department, Toshiba Ceramics Co., Ltd., Shinjuku Nomura Building, 26-2 Nishi-Shinjuku, 1-Chome, Shinjuku-ku, Tokyo 163-05, Japan, Facsimile + 81-3-3343-8627.

[5] *Annual Book of ASTM Standards*, Vol 10.05.

[6] Available from Semiconductor Equipment and Materials International, 805 E. Middlefield Rd., Mountain View, CA 94043.

Copyright © ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, United States.

4.7 The baseline is corrected for curvature resulting from scattering from the rough back surface and the baseline value at 1107 cm^{-1} is determined.

4.8 This baseline value is subtracted from the absorbance at 1107 cm^{-1} to determine the absorption coefficient due to interstitial oxygen.

4.9 The absorption coefficient is multiplied by the appropriate calibration factor to obtain the oxygen content of the test specimen.

5. Significance and Use

5.1 Control of the oxygen content is essential for silicon wafers to be used for advanced devices and integrated circuits. It is desirable to be able to measure the oxygen content of product wafers, nondestructively and without regard for back surface finish. This test method provides a means for reducing the influence of the back surface condition on the measurement.

5.2 This test method may be used for routine process monitoring, quality control, materials acceptance, and research and development.

6. Interferences

6.1 *Multiple Reflections* are greatest for thin, double-side polished wafers with parallel front and back surfaces. In this case, the transmittance, T, is given as follows:

$$T = \frac{(1-R)^2 e^{-\alpha x}}{1 - R^2 e^{-2\alpha x}} = (1-R)^2 e^{-\alpha x}[1 + R^2 e^{-2\alpha x} + R^4 e^{-4\alpha x} + ...] \quad (1)$$

where:

R = reflectance ratio,

α = absorption coefficient in cm^{-1}, and

x = optical path length in cm (= $d/\cos\theta_r$, where d = specimen thickness, in cm, and θ_r = angle of refraction (see 10.1).

To neglect multiple reflections, the quantity $R^2 e^{-2\alpha x}$ should be less than 0.001. The reflection is suppressed for incident radiation at the Brewster angle (73.7° from the normal in silicon). However, because of the large cone angle of the incident radiation in FT-IR spectrometers with focused beam not all of the radiation is precisely at the Brewster angle. Procedures to minimize this effect are given in 9.2.

6.2 *Optical Path Length* of the transmitted beam is estimated from the central beam angle of the incident non-parallel beam flux.

6.3 *Surface Scattering*—the baseline that is due largely to surface scattering is approximated by a parabolic curve (see Appendix X1).

6.4 *Free Carrier Absorption* is minimized by requiring that the resistivity of the test and reference specimens be greater than 5 Ω·cm.

6.5 *Reference Wafer* is required in order to determine the absorption due to the silicon lattice spectrum at the wavenumber of the peak of the oxygen absorption.

6.6 *Temperature Control*—Since both oxygen and silicon lattice absorption change with temperature, the temperature inside the spectrometer chamber must be maintained at 27 ± 5°C during the measurement as required by Test Method F 1188.

6.7 *Nonlinearity* in the spectrometer and its detecting system can degrade the accuracy of the measurement.

7. Apparatus

7.1 *Single-Beam Fourier Transform Infrared Spectrometer*, as specified in Test Method F 1188, capable of collecting transmission spectra with resolution of both 4 cm^{-1} and 1 cm^{-1}.

7.2 *Polarizer*, in order that the incident beam shall be p-polarized.

7.3 The central angle of the incident beam flux shall be adjustable between 65° and 75° from the surface normal.

7.4 *Detector* shall be large enough that the shifting of the beam by the sample (a lateral distance equal to 0.88 times the sample thickness) does not affect its sensitivity. Detector sensitivity shall be unchanged whether a sample is or is not in the measurement beam.

8. Test and Reference Specimens

8.1 *Test Specimen*:

8.1.1 A silicon wafer with chem-mechanically polished front surface and a back surface that may be as-cut, lapped or etched. The back-surface roughness shall be such that:

8.1.1.1 The rms roughness shall be less than 0.9 µm,

8.1.1.2 The transmittance through the wafer at 1107 cm^{-1} shall equal or exceed 25 %, or

8.1.1.3 The difference between the absorption coefficient at 1200 cm^{-1} and the absorption coefficient at 950 cm^{-1} shall be positive but less than 5 cm^{-1}.

8.1.2 Wafers shall have thickness in the range specified in SEMI Specifications M1 (between 500 and 750 µm for wafers with diameter from 100 to 200 mm). Measure and record as d_{CZ} the thickness of each test specimen to the nearest µm.

8.1.3 The resistivity of either n- or p-type test specimens shall be greater than 5 Ω·cm.

8.2 *Oxygen-Free Reference Specimen*:

8.2.1 A double-side polished, float-zoned silicon wafer with maximum oxygen content of 1×10^{16} atoms/cm^3(0.2 ppma) and resistivity greater than 5 Ω·cm.

8.2.2 Measure and record as d_{FZ} the thickness to the nearest µm; the thickness of the reference specimen shall be within ±20 % of that of the test specimen.

8.3 A second double-side polished, float-zoned wafer, ~400 µm thick, for use in determining the optimum angle of incidence.

8.4 Sapphire wafer ≥400 µm-thick, polished on one or both sides.

9. Procedure

9.1 *Determine Stability of FT-IR Spectrometer*:

9.1.1 Turn on the spectrometer and allow it to operate long enough to stabilize.

9.1.2 Set the resolution of the spectrometer to 4 cm^{-1}.

9.1.3 Use a minimum of 64 scans for each spectrum collection.

9.1.4 *100 % Line Check*:

9.1.4.1 Collect a background spectrum $I_{01}(\nu)$ with the

sample beam empty over the wavenumber range from 900 to 1300 cm^{-1}.

9.1.4.2 Wait a time interval, *t* minutes, long enough to make the desired measurements on the test and reference specimens, and then again collect a background spectrum $I_{02}(v)$ with the sample beam empty over the wavenumber range from 900 to 1300 cm^{-1}. The time interval *t* shall be at least 60 min.

9.1.4.3 Determine the ratio $I_{01}(v)/I_{02}(v)$ over the wavenumber range from 900 to 1300 cm^{-1}.

9.1.4.4 If the ratio $I_{01}(v)/I_{02}(v) = 1.000 \pm 0.005$ (100.0± 0.5 %) over the entire wavenumber range, the instrument is acceptable for use in any measuring sequence that requires a total elapsed time $\leq t$ minutes.

9.1.4.5 If the ratio $I_{01}(v)/I_{02}(v)$ falls outside the range 1.000 ± 0.005 in any part of the wavenumber range 900 to 1300 cm^{-1}, reduce the time interval, *t*, and repeat 9.1.4.1-9.1.4.4 until the ratio $I_{01}(v)/I_{02}(v) = 1.000 \pm 0.005$ over the entire wavenumber range.

9.1.4.6 Ensure that any sequence of measurements made using a single background spectrum is completed within the time interval *t* minutes.

9.1.5 *0 % Line Check*:

9.1.5.1 Collect a background spectrum $I_0(v)$ with the sample beam empty over the wavenumber range from 900 to 1300 cm^{-1}.

9.1.5.2 Then collect a spectrum $I_s(v)$ with the sapphire wafer (see 8.1.3) in the sample beam over the wavenumber range from 900 to 1300 cm^{-1}.

9.1.5.3 Determine the ratio $I_0(v)/I_s(v)$ over the wavenumber range from 900 to 1300 cm^{-1}.

9.1.5.4 If the ratio $I_0(v)/I_s(v) \leq 0.001$ (0.1 %) over the entire wavenumber range, the instrument is acceptable for use.

9.1.5.5 If the ratio $I_0(v)/I_s(v) > 0.001$ (0.1 %) over any part of the wavenumber range, adjust the instrument in accordance with the manufacturer's instructions and repeat the entire procedure beginning with 9.1.

9.2 *Angle of Incidence*:

9.2.1 Use one of the following two methods to determine the best angle of incidence of the *p*-polarized infrared beam.

9.2.2 *Fringe Minimum (FM) Method*:

9.2.2.1 Set the resolution of the spectrometer to 1 cm^{-1}.

9.2.2.2 Adjust the angle of the specimen holder so that the angle of incidence to a value somewhat larger than the Brewster angle, and collect a spectrum $I_{FZ}(v)$ with the thin double-side polished, float-zoned wafer (see 8.3) in the sample beam. Observe the magnitude of the interference fringes in the spectrum.

NOTE 3—If desired, the spectrum $I_{FZ}(v)$ can be ratioed with a background spectrum $I_0(v)$ collected with the sample beam empty.

9.2.2.3 Rotate the specimen holder so that angle of incidence is decreased by 1° and again collect a spectrum $I_{FZ}(v)$ with the thin double-side polished float-zoned wafer in the sample beam. Observe the magnitude of the interference fringes in the spectrum; the magnitude should decrease as the angle of incidence approaches the Brewster angle.

9.2.2.4 Repeat 9.2.2.3, decreasing the angle of incidence each time until the magnitude of the interference fringes begins to increase.

9.2.2.5 Record, to the nearest 1°, the angle of incidence for the minimum fringe magnitude as θ_{iFM}.

9.2.3 *Single Beam Maximum (SBM) Method*:

9.2.3.1 Set the resolution of the spectrometer to 4 cm^{-1}.

9.2.3.2 Adjust the angle of the specimen holder so that the angle of incidence to a value somewhat larger than the Brewster angle, and measure the intensity transmitted at 1107 cm^{-1} with the thin, double-side polished, float-zoned wafer (see 8.3) in the sample beam.

9.2.3.3 Rotate the specimen holder so that angle of incidence is decreased by 1° and again measure the intensity transmitted at 1107 cm^{-1} with the thin double-side polished float-zoned wafer in the sample beam; the intensity should increase as the angle of incidence approaches the Brewster angle.

9.2.3.4 Repeat 9.2.3.3, decreasing the angle of incidence each time until the transmitted intensity at 1107 cm^{-1} begins to decrease.

9.2.3.5 Record, to the nearest 1°, the angle of incidence for the maximum transmitted intensity at 1107 cm^{-1} as θ_{isBM}.

9.3 Collect a background spectrum I_0 over the wavenumber range from 900 to 1300 cm^{-1} with the sample beam empty. Collect this and all subsequent spectra with a minimum of 64 scans.

9.4 Place the oxygen-free reference specimen (see 8.2) in the sample beam such that the angle of incidence is θ_{iFM} or θ_{isBM} as determined in 9.2.2 or 9.2.3, respectively, and collect a spectrum $I_{FZ}(v)$ over the wavenumber range from 900 to 1300 cm^{-1}.

9.5 Determines the transmittance spectrum of the oxygen-free reference specimen as follows:

$$T_{FZ}(v) = \frac{I_{FZ}(v)}{I_0(v)} \quad (2)$$

9.6 Remove the oxygen-free reference specimen.

9.7 Place a test specimen (see 8.1) in the sample beam so that the angle of incidence is θ_i and collect a spectrum $I_{CZ}(v)$ over the wavenumber range from 900 to 1300 cm^{-1}.

9.8 Determine the transmittance spectrum of the test specimen as follows:

$$T_{CZ}(v) = \frac{I_{CZ}(v)}{I_0(v)} \quad (3)$$

9.9 If desired, determine the transmittance spectra of additional test specimens by repeating 9.7 and 9.8. Ensure that the total elapsed time for completing all determination does not exceed *t* min (see 9.1.4).

10. Calculations

10.1 Calculate the cosine of the angle of refraction, θ_r, as follows:

$$\cos\theta_r = \frac{\sqrt{11.70 - \sin^2\theta_i}}{3.42} \quad (4)$$

where:

θ_i = angle of incidence (θ_{iFM} or θ_{iSBM}, as appropriate, see 9.2).

NOTE 4—Refer to Appendix X1 for a discussion of the numerical constants in this and subsequent equations.

10.2 Taking into account the path length increase resulting from the oblique angle of incidence, calculate the absorption spectrum of each oxygen-free reference specimen and each test specimen as follows:

$$\alpha_k(v) = \frac{\cos\theta_r}{d_k}\cdot\ln T_k(v) \quad (5)$$

where:

$\alpha(v)$ = absorption coefficient as a function of wavenumber, v, in cm $^{-1}$,
θ_r = angle of refraction (see 10.1),
d_k = measured specimen thickness, in cm,
$T_k(v)$ = specimen transmittance as a function of wavenumber, v, and
k = *CZ* or *FZ*, as appropriate.

10.3 Calculate the difference absorption spectrum as follows:

$$\Delta\alpha(v) = \alpha_{CZ}(v) - \alpha_{FZ}(v) \quad (6)$$

NOTE 5—See Appendix X2 for an alternative method of obtaining the difference absorption spectrum when a difference absorbance spectrum can be obtained internally in the infrared spectrometer.

10.4 Calculate the absorption coefficient due to interstitial oxygen at 1107 cm $^{-1}$ as follows (see Appendix X1):

$$\alpha_{Oi} = \alpha_{1107} - 0.5449\,(\alpha_{1160} - \alpha_{1040}) - \alpha_{1040} \quad (7)$$

where:

α_{Oi} = the absorption coefficient due to interstitial oxygen at 1107 cm $^{-1}$, in cm $^{-1}$,
α_{1107} = $\Delta\alpha(1107)$, the difference between the absorption coefficients of the test and reference specimens at 1107 cm $^{-1}$, in cm $^{-1}$,
α_{1040} = $\Delta\alpha(1040)$, the difference between the adsorptions coefficients of the test and reference specimens at 1040 cm $^{-1}$, in cm $^{-1}$, and
α_{1160} = $\Delta\alpha(1160)$, the difference between the absorption coefficients of the test and reference specimens at 1160 cm $^{-1}$, in cm $^{-1}$.

10.5 Perform the calculations for each test specimen measured.

10.6 Calculate the interstitial oxygen content, O_i, of each test specimen as follows:

$$(O_i)\text{, ppm atomic} = 6.28\,\alpha_{Oi} \text{ or} \quad (8)$$

$$(O_i)\text{, atoms/cm}^3 = 3.14 \times 10^{17}\,\alpha_{Oi}$$

where α_{Oi} is the absorption coefficient of interstitial oxygen at 1107 cm $^{-1}$.

NOTE 6—The calibration factor used in these relations was determined as a result of an international interlaboratory experiment.[7] The uncertainty in this calibration factor was stated to be ±0.18 ppm atomic or $\pm 9 \times 10^{15}$ atoms/cm ^{3}.

11. Report

11.1 Report the following information:

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

11.1.2 Identification of reference and test specimens,

11.1.3 Thickness of reference and test specimens,

11.1.4 Apodization function used,

11.1.5 Angle of incidence (θ_i) employed and method (*FM*, see 9.2.2, or *SBM*, see 9.2.3) by which it was established,

11.1.6 For each test specimen:

11.1.6.1 The absorption coefficient due to interstitial oxygen, α_{Oi}, and

11.1.6.2 Oxygen content, in ppm atomic or atoms/cm ^{3}.

11.2 Refer to the calibration factor used as *IOC*-88.

12. Precision and Bias

12.1 *Precision*—An interlaboratory evaluation by the SEMI Japan Silicon Wafer Committee (see Appendix X3) was carried out in which 13 laboratories each reported a single measurement on 15 single side polished and 15 double side polished silicon wafers. There were two sets of nominally similar test specimens, but different results were obtained on each set. The pooled results suggest that the reproducibility of this test method, when applied to typical single-side polished silicon wafers, lies in the range from about 0.3 cm $^{-1}$ to about 1.1 cm $^{-1}$, equivalent to variations in oxygen content of about 1.7 to about 7 ppm atomic (*IOC*-88). The results also show that the reproducibility of measurements on double side polished, 2-mm slices is usually less than about 0.3 cm $^{-1}$, equivalent to about 1.7 ppm atomic (*IOC*-88).

12.2 *Bias*—The results of measurements on double-side polished, 2-mm slices are taken as yielding the correct value for oxygen content. The difference between the mean absorption coefficient determined on the single side polished wafers and that determined on the double side polished slices was typically less than 0.1 cm $^{-1}$. However, individual values ranged from – 0.2 to + 0.7 cm $^{-1}$, equivalent to differences in measured oxygen content as much as about 4.4 ppm atomic (*IOC*-88).

13. Keywords

13.1 Brewster angle; infrared absorption; interstitial oxygen; oxygen; silicon

[7] Baghdadi, A., Bullis, W. M., Croarkin, M. C., Li Yue-zhen, Scace, R. I., Series, R. W., Stallhofer, P., and Watanabe, M., "Interlaboratory Determination of the Calibration Factor for the Measurement of the Interstitial Oxygen Content of Silicon by Infrared Absorption," *Journal of The Electrochemical Society*, Vol 136, No. 7, 1989, pp. 2015–2024.

APPENDIXES

(Nonmandatory Information)

X1. NUMERICAL CONSTANTS

X1.1 The numerical constants given in the equation in 10.1 are lumped constants. This provides details as to the composition of these lumped constants and the values of the individual constants used in deriving them.

X1.1.1 The constant, 11.70, in the numerator of this equation is the relative dielectric constant of silicon, K_{Si}.

X1.1.2 The constant, 3.42, in the denominator of this equation is the index of refraction for silicon, n.

X1.1.3 Note that $K_{Si} = n^2$.

X1.2 In deriving the equation in 10.4, it is assumed that the curved baseline is due to scattering from the back surface of the wafer and that this scattering can be represented by an effective absorption coefficient, α_{SU}, that is given as follows:

$$\alpha_{SU} = av^2 + c \tag{X1.1}$$

where a and c are constants that are determined from the absorption at 1160 and 1040 cm $^{-1}$ where the there is no absorption due to interstitial oxygen.

X1.2.1 Thus, since $\alpha_{1160} = 1160^2 \cdot a + c$ and $\alpha_{1040} = 1040^2 \cdot a + c$,

$$\alpha = \frac{(\alpha_{1160} - \alpha_{1040})}{(1160^2 - 1040^2)} = 3.7879 \times 10^{-6} (\alpha_{1160} - \alpha_{1040}) \tag{X1.2}$$

and

$$c = \alpha_{1040} - a \cdot 1040^2 = \alpha_{1040} - 4.0970 (\alpha_{1160} - \alpha_{1040}) \tag{X1.3}$$

X1.2.2 Therefore,

$$\alpha_{SU}(v) = 3.7879 \times 10^{-6} (\alpha_{1160} - \alpha_{1040}) v^2 + \alpha_{1040} - 4.0970 (\alpha_{1160} - \alpha_{1040}) \tag{X1.4}$$

X1.2.3 At $v = 1107$ cm $^{-1}$,

$$\alpha_{SU}(1107) = \alpha_{1040} + (4.6419 - 4.0970)(\alpha_{1160} - \alpha_{1040}) = \alpha_{1040} + 0.5449 (\alpha_{1160} - \alpha_{1040}) \tag{X1.5}$$

X1.2.4 The equation for α_{Oi} follows directly since

$$\Delta\alpha(v) = \alpha_{CZ}(v) - \alpha_{FZ}(v) = \alpha_{Oi}(v) + \alpha_{SU}(v). \tag{X1.6}$$

X1.3 The numerical constants given in the equations in 10.6 are the calibration factors for oxygen in silicon (see Note 6).

X2. ALTERNATIVE METHOD FOR DETERMINING DIFFERENCE ABSORPTION SPECTRUM

X2.1 This appendix describes an alternative method for determining the difference absorption spectrum in lieu of the calibration in 10.2 and 10.3.

X2.2 First, determine the difference absorbance spectrum within the infrared spectrometer as follows:

$$\Delta A(v) = A_{CZ}(v) - \frac{d_{CZ}}{\cos\theta_r d_{FZ}} A_{FZ}(v) \tag{X2.1}$$

where:

$A_k(v)$ = $-\log T_k(v)$,
k = CZ or FZ, as appropriate, and
the other symbols are defined in 10.2.

X2.3 Then, determine the difference absorption spectrum as follows:

$$\Delta\alpha(v) = \frac{2.3026\cos\theta_r}{d_{CZ}} \Delta A(v) \tag{X2.2}$$

where:

$\Delta A(v)$ = the difference absorbance as a function of wavenumber as found in X2.2, and
the other symbols are defined in 10.2.

X3. RESULTS OF INTERLABORATORY EVALUATION

X3.1 *Outline of Experiment:*

X3.1.1 The SEMI Japan Silicon Wafer Committee has conducted an interlaboratory evaluation of this test method. Thirteen laboratories measured fifteen single-side polished wafers, nominally 625 μm-thick, from five different suppliers together with fifteen double-side polished slices, nominally 2 mm-thick. Corresponding slices and wafers were cut from the same region of a 125-mm diameter crystal. Two groups of samples were used. The samples in one group, circulated to seven laboratories, were cut down to a diameter of 100 mm so that they would fit into the spectrometers used by these laboratories. The samples in the other group, circulated to six laboratories, remained at a diameter of 125 mm.

X3.1.2 Each laboratory reported a single measurement of the difference absorption spectrum of both the 2-mm double-side polished slice (determined in accordance with Test

Method F 1188) and the 625-μm single-side polished wafer (determined in accordance with this test method). Consequently, only an estimate of the interlaboratory reproducibility of the measurement could be made; no estimate of intralaboratory repeatability is possible from the data set supplied.

X3.1.3 One laboratory in the 100-mm group reported clearly erroneous values of absorption and one laboratory in the 125-mm group failed to provide data for the baseline required by this test method; data from both these laboratories were excluded from the analysis. In addition, one wafer in each group of samples was broken; no data from this wafer were included in the analysis. Thus the estimate of precision of this test method is based on data from 14 100-mm sample sets measured by six laboratories and data from 14 125-mm sample sets measured by five laboratories.

X3.2 *Reproducibility:*

X3.2.1 *Variability of Measurements on Single-Side Polished Wafers*—For reasons that have not been determined, the two data sets yielded different estimates of measurement reproducibility. The sample standard deviations, $s_{100,w}$, obtained from the 100-mm data set ranged from 0.106 cm $^{-1}$ to 0.406 cm $^{-1}$, generally increasing with mean absorption coefficient, $\alpha_{100,w}$, as follows:

$$s_{100,w} = 0.0925\alpha_{100,w} + 0.135 \quad \text{(X3.1)}$$

X3.2.1.1 On the other hand, the sample standard deviations, $s_{125,w}$, obtained from the 125-mm data set ranged from 0.072 cm $^{-1}$ to 0.107 cm $^{-1}$, generally independent of the mean absorption coefficient, $\alpha_{125,w}$. The small dependence on $\alpha_{125,w}$ was as follows:

$$s_{125,w} = -0.0078\alpha_{125,w} + 0.1098 \quad \text{(X3.2)}$$

X3.2.1.2 If both data sets were pooled, the sample standard deviations, s_w, ranged from 0.098 cm $^{-1}$ to 0.293 cm $^{-1}$, generally increasing with mean absorption coefficient, α_w, as follows:

$$s_w = 0.0588\alpha_w + 0.0393 \quad \text{(X3.3)}$$

X3.2.1.3 These results suggest that the reproducibility that can be obtained with the use of this test method lies in the range from about 0.3 cm $^{-1}$ to about 1.1 cm $^{-1}$, equivalent to variations in oxygen content of about 1.7 to about 7 ppm atomic (IOC-88). Measurements on the 125-mm data set yielded results consistently at the lower end of this range, suggesting that the intrinsic capability of this test method is barely adequate for controlling to current oxygen content specifications which have a range of ±2 ppm.

X3.2.2 *Variability of Measurements on Double-Side Polished Wafers*—As part of the experiment, the variability of the measurements on the double-side polished, 2-mm thick slices, made in accordances with Test Method F 1188, was also determined. This variability was generally less than that obtained on the single-side polished wafers measured in accordance with this test method. Again, the behavior of the two data sets differed.

X3.2.2.1 The 100-mm data set yielded as follows:

$$s_{100,s} = 0.0464\alpha_{100,s} - 0.0119 \quad \text{(X3.4)}$$

where:

$s_{100,s}$ = the sample standard deviation of the measured absorption coefficient of the double-side polished, 2-mm slices in the 100-mm data set, in cm $^{-1}$, and

$\alpha_{100,s}$ = the mean absorption coefficient of the double-side polished, 2-mm slices in the 100-mm data set.

X3.2.2.2 The 125-mm data set yielded slightly smaller values, with a less pronounced dependence on the mean absorption coefficient as follows:

$$s_{125,s} = 0.0166\alpha_{125,s} + 0.0356 \quad \text{(X3.5)}$$

where:

$s_{125,s}$ = the sample standard deviation of the measured absorption coefficient of the double-side polished, 2-mm slices in the 125-mm data set, in cm $^{-1}$, and

$\alpha_{125,s}$ = the mean absorption coefficient of the double-side polished, 2-mm slices in the 125-mm data set.

X3.2.2.3 If both data sets were pooled, the sample standard deviations, s_s, ranged from 0.098 cm $^{-1}$ to 0.293 cm $^{-1}$, generally increasing with mean absorption coefficient, α_s, as follows:

$$s_s = 0.0310\alpha_s + 0.0117 \quad \text{(X3.6)}$$

These results suggest that the reproducibility of the measurements on double-side polished, 2-mm slices is usually less than about 0.3 cm $^{-1}$, equivalent to about 1.7 ppm atomic (IOC-88).

X3.3 *Bias:*

X3.3.1 The relationship between the average values of absorption coefficient due to interstitial oxygen obtained from the 100-mm data set was as follows:

$$\alpha_{100,w} = 1.0312\alpha_{100,s} - 0.0291 \quad \text{(X3.7)}$$

where the symbols have the same meaning as in the previous section. Similarly, the 125-mm data set yielded the following relation:

$$\alpha_{125,w} = 1.0076\alpha_{125,s} + 0.0092 \quad \text{(X3.8)}$$

and the pooled data sets yielded the following relation:

$$\alpha_w = 1.0204\alpha_s - 0.0127 \quad \text{(X3.9)}$$

X3.3.2 The difference between the mean absorption coefficient determined on the single-side polished wafers and that determined on the double-side polished slices was typically less than 0.1 cm $^{-1}$. However, individual values ranged from − 0.2 to + 0.7 cm $^{-1}$, equivalent to differences in measured oxygen content as much as about 4.4 ppm atomic (IOC-88).

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org).