



Designation: F 1619 – 95 (Reapproved 2000)^{ε1}

Standard Test Method for Measurement of Interstitial Oxygen Content of Silicon Wafers by Infrared Absorption Spectroscopy with p -Polarized Radiation Incident at the Brewster Angle¹

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^{ε1} NOTE—Editorial changes were made throughout in April 2002.

1. Scope

1.1 This test method² 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 F01 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 $5\ \Omega\cdot\text{cm}$ 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 Absorption⁵

F 1241 Terminology of Silicon Technology⁵

2.2 SEMI Standard:

SEMI M1 Specifications for Polished Monocrystalline Silicon Wafers⁶

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.

⁵ Annual Book of ASTM Standards, Vol 10.05.

⁶ Available from Semiconductor Equipment and Materials International, 3081 Zanker Rd., San Jose, CA 95134 (www.semi.org).

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.

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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 and Information Technology Industries Association (JEITA), 3rd Floor Mitsui Kaijo Bldg. Annex 11, Kanda Surugadai 3-chome, Chiyoda-Ku, Tokyo, 101-0062, Japan.

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 Wafers 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.



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.

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:

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

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 $5\text{ }\Omega\cdot\text{cm}$.

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 $27 \pm 5^\circ\text{C}$ 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 $0.9\text{ }\mu\text{m}$,

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 $750\text{ }\mu\text{m}$ 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 $5\text{ }\Omega\cdot\text{cm}$.

8.2 Oxygen-Free Reference Specimen:

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

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

8.3 A second double-side polished, float-zoned wafer, $\sim 400\text{ }\mu\text{m}$ thick, for use in determining the optimum angle of incidence.

8.4 Sapphire wafer $\geq 400\text{ }\mu\text{m}$ -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 $I_{02}(\nu)$ 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 $I_{01}(\nu)/I_{02}(\nu)$ over the wavenumber range from 900 to 1300 cm^{-1} .

9.1.4.4 If the ratio $I_{01}(\nu)/I_{02}(\nu) = 1.000 \pm 0.005$ (100.0 $\pm$ 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 $I_{01}(\nu)/I_{02}(\nu)$ 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 $I_{01}(\nu)/I_{02}(\nu) = 1.000 \pm 0.005$ 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 $I_0(\nu)$ with the sample beam empty over the wavenumber range from 900 to 1300 cm^{-1} .

9.1.5.2 Then collect a spectrum $I_s(\nu)$ 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 $I_0(\nu)/I_s(\nu)$ over the wavenumber range from 900 to 1300 cm^{-1} .

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

9.1.5.5 If the ratio $I_0(\nu)/I_s(\nu) > 0.001$ (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 $I_{FZ}(\nu)$ 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 $I_{FZ}(\nu)$ can be ratioed with a background spectrum $I_0(\nu)$ 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 $I_{FZ}(\nu)$ 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 $I_{FZ}(\nu)$ over the wavenumber range from 900 to 1300 cm^{-1} .

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

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

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 $I_{CZ}(\nu)$ over the wavenumber range from 900 to 1300 cm^{-1} .

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

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

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:

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

