



Standard Test Method for Interstitial Atomic Oxygen Content of Silicon by Infrared Absorption¹

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1. Scope

1.1 This test method covers the determination of the interstitial oxygen content of single crystal silicon by infrared spectroscopy.^{2, 3, 4, 5, 6, 7} This test method requires the use of an oxygen-free reference specimen and a set of calibration standards, such as those comprising NIST SRM 2551.^{8,9} It permits, but does not require, the use of a computerized spectrophotometer.

1.2 The useful range of oxygen concentration measurable by this test method is from 1×10^{16} atoms/cm³ to the maximum amount of interstitial oxygen soluble in silicon.

1.3 The oxygen concentration obtained using this test method assumes a linear relationship between the interstitial

oxygen concentration and the absorption coefficient of the 1107 cm⁻¹ band associated with interstitial oxygen in silicon.

1.4 *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:

E 1 Specification for ASTM Thermometers¹⁰

E 131 Terminology Relating to Molecular Spectroscopy¹¹

E 932 Practice for Describing and Measuring Performance of Dispersive Infrared Spectrophotometers¹¹

3. Terminology

3.1 For definitions of terms relating to absorption spectroscopy, refer to Terminology E 131.

3.2 Definitions:

3.2.1 *dispersive infrared (DIR) spectrophotometer, n*—a type of infrared spectrometer that uses at least one prism or grating as the dispersing element, in which the data are obtained as an amplitude-wavenumber (or wavelength) spectrum.

3.2.1.1 *Discussion*—Some dispersive infrared spectrometers are used in conjunction with a computer, which is used to store data. The data are then accessible for manipulation or computation, as required. These spectrometers are referred to as computer-assisted dispersive infrared spectrophotometer (CA-DIR). Dispersive infrared spectrometers that are not computer-assisted are referred to, for convenience, as simple dispersive infrared spectrometers (S-DIR).

3.2.2 *Fourier transform infrared (FT-IR) spectrophotometer, n*—a type of infrared spectrometer in which the data are obtained as an interferogram.

3.2.2.1 *Discussion*—An interferogram is a record of the modulated component of the interference signal measured by the detector as a function of retardation in the interferometer.

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 Controls.

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² ASTM Test Method F 121 (editions of 1980 through 1983, replaced by Test Method F 1188 in 1988).

³ DIN 50438, Part 1 (edition of 1973, revised to cite IOC-88 in 1995), DIN 50438, Part 1 (1995) is referred to in Tables X1.1 and X1.2.

⁴ Iizuka, T., Takasu, S., Tajima, M., Arai, T., Nozaki, T., Inoue, N., and Watanabe, M., "Determination of Conversion Factor for Infrared Measurement of Oxygen in Silicon," *Journal of the Electrochemical Society*, Vol 132, 1985, pp. 1707–1713. JEDIA standard 61-2000 (Standard Test Method for Atomic Oxygen Content of Silicon by Infrared Absorption) issued in 2000, cites IOC-88.

⁵ Old edition; cited in Reference 6. Since revised to cite IOC-88.

⁶ Baghdadi, A., Bullis, W. M., Coarkin, 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, 1989, pp. 2015–2034.

⁷ ASTM Test Method F 121 (editions of 1970 through 1979).

⁸ SRM 2551, available from the National Institute of Standards and Technology, Gaithersburg, MD 20899 USA, has been found to be suitable for this purpose.

⁹ DIN 50438 Part 1 is similar to, but more general than, this test method. It includes two methods: Method A, which is restricted to double side polished or polish-etched wafers, and Method B, which is applicable to wafers with one side polished and one side etched for wafers as thin as 0.03 cm. DIN 50438 Part 1 is intended for use with computer aided spectrophotometers, whether dispersive or FTIR. It is the responsibility of DIN Committee NMP 221 with which ASTM F-1 maintains close liaison. DIN 50438 Part 1, Determination of Impurity Content in Semiconductors by Infrared Absorption, Oxygen in Silicon, may be obtained from Beuth Verlag GmbH, Berggrafenstrasse 4-10, D-1000 Berlin 30, Germany (see also the Related Material Section of the 1993 edition of the *Annual Book of ASTM Standards*, Vol 10.05).

¹⁰ *Annual Book of ASTM Standards*, Vol 14.03.

¹¹ *Annual Book of ASTM Standards*, Vol 03.06.

This interferogram is then subjected to a Fourier transformation to obtain an amplitude-wavenumber (or wavelength) spectrum. Due to the complexity of the Fourier transformation, FT-IR instruments are always used in conjunction with a computer.

3.2.3 *reference spectrum, n*—the spectrum of the reference specimen.

3.2.3.1 *Discussion*—In true double-beam spectrometers, it may be obtained directly, with the reference specimen in the sample beam, and the reference beam empty. In single-beam spectrometers, it can be calculated from the ratio of a spectrum obtained with the reference specimen in the IR beam, to a background spectrum.

3.2.4 *sample spectrum, n*—the spectrum of the test specimen.

3.2.4.1 *Discussion*—In true double-beam spectrometers, it may be obtained directly, with the sample specimen in the sample beam, and the reference beam empty. In single-beam spectrometers, it can be calculated from the ratio of a spectrum obtained with the test specimen in the IR beam, to a background spectrum.

4. Summary of Test Method

4.1 The relative infrared transmittance spectrum of an oxygen-containing silicon slice, which is mirror-polished on both sides, is obtained using a reference method with an IB spectrophotometer calibrated by means of a suitable set of reference materials. The oxygen-free reference specimen is matched closely in thickness to the test specimen, so as to eliminate the effects of absorption due to silicon lattice vibrations. The absorption coefficient of the 1107 cm^{-1} oxygen-in-silicon band is then used to calculate the interstitial oxygen content of the silicon slice.

5. Significance and Use

5.1 Measurement of the intensity of the 1107 cm^{-1} oxygen-in-silicon band with an infrared spectrophotometer enables the determination of the value of the absorption coefficient and, hence, by the use of a conversion coefficient, the content of interstitial oxygen.

5.2 This test method can be used as a referee method for determining the interstitial oxygen content of silicon slices which are polished on both sides. Knowledge of the interstitial oxygen content of silicon wafers is necessary for materials acceptance and control of fabrication processes, as well as for research and development.

6. Interferences

6.1 The oxygen absorption band overlaps a silicon lattice band. The oxygen-free reference specimen must be matched within $\pm 0.5\%$ to the thickness of the test specimen in order to properly remove the effects of the silicon lattice absorption.

6.2 Since both the oxygen band and the lattice band can change with the specimen temperature, the temperature inside the spectrophotometer sample compartment must be maintained at $27 \pm 5^\circ\text{C}$ during the measurement.

6.3 Significant free carrier absorption occurs in *n*-type silicon with a resistivity below 1 $\Omega\text{-cm}$, and in *p*-type silicon

with a resistivity below 3.0 $\Omega\text{-cm}$. For test specimens below these resistivities, the reference crystal must be matched in resistivity as well as in thickness. The resistivity match must be sufficiently close so that the transmittance of the test specimen relative to the reference specimen at 1600 cm^{-1} must be $100 \pm 5\%$.

6.4 The free carrier absorption in *n*-type crystals with resistivities less than 0.1 $\Omega\text{-cm}$, or in *p*-type crystals with resistivities less than 0.5 $\Omega\text{-cm}$ reduces the available energy below that required for the satisfactory operation of most spectrophotometers.

6.5 The presence of a high concentration of oxide precipitates, that result in absorbance bands at 1230 cm^{-1} or 1073 cm^{-1} , may lead to an error in the interstitial oxygen determination.

6.6 The full width at half maximum (FWHM) of the oxygen-in-silicon band at 300 K is 32 cm^{-1} . Calculations made from spectral data having a FWHM greater than this value will be in error.

7. Apparatus

7.1 *Infrared spectrophotometer*, either a S-DIR, CA-DIR or FT-IR instrument, as described in 3.2.1 and 3.2.2 may be used. It must be possible to set the resolution of the spectrophotometer to 4 cm^{-1} , or better, for Fourier transform infrared spectrophotometers, and to 5 cm^{-1} , or better, for dispersive spectrophotometers.

7.2 The three following paragraphs apply only to FT-IR spectrophotometer:

7.2.1 *Zero Filling*—When an FT-IR instrument collects an unsymmetrical interferogram, an additional set of points whose values are all zero shall be added to the end of the collected interferogram such that the total number of points for performing the Fourier transform is double the number of data points originally collected.

7.2.2 *Undersampling*—The data collection method shall produce interferograms which, when zero-filled and Fourier transformed, produce a spectrum containing at least two data points per resolution increment. For example, after transformation, a spectrum obtained at 4 cm^{-1} resolution shall contain at least one data point every two wavenumbers.

7.2.3 *Phase Correction*—The phase correction routine used during Fourier transformation shall use at least 200 points on both sides of the point of zero retardation in order to produce a phase array that can be used to eliminate phase errors.¹²

7.3 *Specimen Holders of Appropriate Size*—If the test specimen is small, it must be mounted in a holder that has an opening small enough to prevent any of the infrared beam from bypassing the specimen. The specimens shall be held normal, or nearly normal, to the axis of the incident infrared beam (see 8.3).

7.4 *Equipment and Materials*, for slicing and polishing crystals to a thickness similarity of 0.5 % or less and a surface flatness equal to one fourth the wavelength at the maximum absorption of the impurity band under study.

¹² For a discussion of the phase correction computation, see Chase, D. B., *Applied Spectroscopy*, Vol 36, 1982, p. 240.

7.5 *Micrometer Caliper*, or other instrument suitable for the measurement of the thickness of the specimens to a tolerance of $\pm 0.2\%$.

7.6 *Thermocouple-Millivolt Potentiometer*, or other system suitable for measurements of the specimen temperature during test.

8. Testing of the Apparatus

8.1 Evaluate the performance of S-DIR spectrometers according to Instrument Operation Section, and Nature of Test Sections of Practice E 932. Follow the appropriate paragraphs of these sections to evaluate the performance of CA-DIR instruments.

8.2 Verify a proper purge condition for the specimen chamber by monitoring water vapor or carbon dioxide absorption bands. The water vapor line is monitored at 1521 cm^{-1} and the carbon dioxide line at 667 cm^{-1} . The instrument shall be sufficiently well purged or evacuated that the transmittance at these locations is between 98 and 102 %.

8.3 Under certain conditions, the spectrophotometer may have a nonlinear response, or be plagued by undesirable extraneous reflections between the specimen surfaces and the spectrometer components. Place a flat, double-side polished and high resistivity (greater than $5\Omega\cdot\text{cm}$) silicon slice in the instrument. The effective transmittance of the silicon slice, due to reflective losses at the silicon surfaces, should be $53.8 \pm 2\%$ in the 1600 to 2000 cm^{-1} region. In some instruments, this silicon slice may have to be placed at a small angle to the axis of the incoming IR beam, in order to minimize undesirable reflections between the silicon surfaces, and the spectrometer components. This angle may be determined by initially placing the silicon slice normal to the axis of the incoming beam, and then gradually tilting the sample while repeatedly obtaining the transmittance spectrum of the slice above 1600 cm^{-1} . The optimum angle is reached when a flat baseline as close as possible to $53.8 \pm 2\%$ is obtained from 1600 to 4000 cm^{-1} . This optimum angle is typically less than 10° .

9. Test Specimen

9.1 Specimens ranging in thickness from 0.4 to 4 mm can be measured by this method.

9.2 Choose test and reference specimens that are as homogeneous as possible, so that the oxygen content measured is a fair representation of the oxygen content of the whole crystal. This is particularly important for dispersive spectrophotometers, since in many such instruments the illuminated area of the sample varies during the scan. For specimens with a large lateral inhomogeneity, this may result in the appearance of undesirable instrumental artifacts in the spectra.

9.2.1 Prepare a slice of the crystal to be tested so as to obtain two optically flat surfaces parallel to 5 min of arc or less, as measured with a micrometer caliper or other suitable instrument. The surfaces of the specimen must be as free as possible of surface films.

NOTE 1—When the specimen faces are parallel and well-polished, and the data are being obtained at a sufficiently high resolution, interference may occur between light rays reflecting from the front and back surfaces of the specimen. The contrast of the interference fringes depends upon the parallelism of the specimen surfaces, and the fringe spacing depends on

the optical thickness of the specimen. These fringes can obscure a weak spectral line and prevent accurate measurement of the baseline. To prevent obscuration by these interference fringes, nonparallel specimen surfaces may be a necessity.

NOTE 2—However, the use of specimens with nonparallel surfaces can also result in photometric errors. If a material has a high refractive index, any nonparallelism of the specimen can displace the spectrometer beam relative to the active area of the detector. The same effect can occur even with a thin specimen in a cryogenic system if the specimen is not cemented properly or the holder plate twists. Thus, a lowered transmission occurs. Improper positioning or nonparallelism of the specimen can be checked by rotating the specimen to determine whether the transmission level stays constant. Any variation is a possible indication of problems with the specimen positioning or preparation.

9.3 Since a difference technique is used in this test method, prepare a reference specimen of the same type of material as the sample, but chosen to be free of oxygen (see 9.3.1). The reference crystal must be prepared to the same tolerances as the test specimen. The thickness of the reference specimen shall be equal to that of the test specimen to within $\pm 0.5\%$.

9.3.1 Choose the reference specimen from slices taken from five to ten different crystals that are thought to be free of oxygen. Compare these slices with one another and choose the specimen with the lowest absorption as the reference specimen. If no absorption is seen for any of the specimens, then the assumption can be made that all specimens contain less than the limit of detection of oxygen and any of the specimens can be used as the reference specimen.

10. Procedure

10.1 Calibration of Spectrophotometer:

10.1.1 Obtain a set of suitable certified reference materials for oxygen in silicon.¹²

10.1.2 Use these reference materials to calibrate the spectrophotometer in accordance with manufacturer's instructions.

NOTE 3—A practice for calibrating infrared spectrophotometers for measuring oxygen in silicon is being developed by Subcommittee F01.06 of ASTM Committee F-1 on Electronics. Some, but not complete, guidance for this calibration is provided in the NIST SRM 2551 Report.

10.2 Instrumental Checks:

10.2.1 Establish the 100 % baseline to measure the noise level: On double beam instruments, record the transmittance spectrum with both the sample and reference beams empty. On single-beam instruments, obtain the transmittance spectrum as the ratio of two spectra taken with the sample beam empty. Plot the 100 % baseline over a wavenumber range covering 900 to 1300 cm^{-1} . If the baseline is not $100 \pm 0.5\%$ over the entire range, increase the measurement time until it does. If the problem persists, have the instrument repaired.

10.2.2 Applies to dispersive (DIR) instruments only. Establish the 0 % line. With the sample beam blocked, record the instrument zero over the range from 900 to 1300 cm^{-1} . If a significant non-zero signal is recorded in that range, check the instrument for stray light reaching the detector. If the problem persists, have the instrument repaired.

10.2.3 Applies to Fourier transform (FT-IR) instruments only. Record the throughput characteristics of the spectrophotometer by plotting a single-beam spectrum, obtained with the sample beam empty, over the wavenumber range from 450 to 4000 cm^{-1} . Use such a spectrum, recorded after the instrument

has been properly aligned according to factory specifications as a reference to evaluate the instrument's performance. Whenever the spectrum obtained deviates significantly from the instrument's reference spectrum, realign the instrument.

10.2.4 Determine mid-scale linearity of the instrument by obtaining an air reference spectrum of the silicon reference specimen over the wavenumber range from 1600 to 2000 cm^{-1} . If the value of the transmittance is not $53.8 \pm 2\%$ over this wavenumber range, align the sample with the spectrometer in accordance with 8.3.

10.3 Immediately prior to the initial measurement in any laboratory, etch all specimens, including the reference specimen, in hydrofluoric acid to remove any surface oxide.

10.4 Measure the thicknesses of the test and reference specimens to within $\pm 0.2\%$, at their centers. If thickness of the reference specimen does not match the thickness of the test specimen to within $\pm 0.5\%$, obtain a reference specimen with the proper thickness.

10.5 Measure and record the temperature of the spectrophotometer chamber.

10.6 Determine the measurement time for the spectra by obtaining the transmittance spectrum of a high resistivity (greater than $5\ \Omega\text{-cm}$), 0.04 to 0.065-cm thick double-side polished silicon slice containing between 12 and 18 ppma oxygen (IOC-88)) using a minimum of 64 scans for FT-IR instruments, or, for dispersive instruments, a speed such that the full peak height is recorded. If the ratio of the net amplitude of the oxygen band, $T_{base} - T_{peak}$, to the standard deviation in the transmittance spectrum is not greater than 100, increase the number of scans (FT-IR) or reduce the scan speed (DIR) until that criterion is met.

10.7 Infrared Transmittance Spectra:

10.7.1 Obtain the spectrum with a resolution, at $1107\ \text{cm}^{-1}$, of $4\ \text{cm}^{-1}$, or better, for the FT-IR instruments and $5\ \text{cm}^{-1}$, or better, for the dispersive instruments over (at least) the range from 900 to $1300\ \text{cm}^{-1}$. The test and reference specimens must be positioned so that the IR beam is centered on them. On double beam dispersive instruments, obtain the transmittance spectrum with the oxygen-free reference specimen in the reference beam, and the test specimen in the sample beam. On single beam instruments, compute the transmittance spectrum as the ratio of the emission spectrum of the test specimen to the emission spectrum of the reference specimen.

10.8 Plot the transmittance spectrum over the range from 900 to $1300\ \text{cm}^{-1}$.

10.9 Define the baseline by drawing a straight line from 900 to $1300\ \text{cm}^{-1}$. Use the average transmittance in the regions from 900 to $1000\ \text{cm}^{-1}$, and 1200 to $1300\ \text{cm}^{-1}$, to define the endpoints of the straight line.

10.10 Locate the wavenumber corresponding to the minimum transmittance in the region from 1102 to $1112\ \text{cm}^{-1}$. Record the value of that wavenumber, to five significant figures, as W_p . Record the minimum transmittance as T_p , the transmittance at the absorption peak. Record the baseline transmittance, T_b , as the value of the baseline defined in 10.9 at W_p . Record both T_p and T_b to three significant figures.

10.11 Determine and record the full width at half maximum (FWHM) of the peak.

11. Calculation

11.1 Calculate the peak and baseline absorption coefficients using the following equations:²

$$\alpha_p = -\frac{1}{x} \ln \left[\frac{(0.09 - e^{1.70x}) + \sqrt{(0.09 - e^{1.70x})^2 + 0.36T_p^2 e^{1.70x}}}{0.18T_p} \right] \quad (1)$$

$$\alpha_b = -\frac{1}{x} \ln \left[\frac{(0.09 - e^{1.70x}) + \sqrt{(0.09 - e^{1.70x})^2 + 0.36T_b^2 e^{1.70x}}}{0.18T_b} \right] \quad (2)$$

where:

- α_p = peak absorption coefficient, cm^{-1} ,
- α_b = baseline absorption coefficient, cm^{-1} ,
- x = thickness, cm,
- T_p = peak transmittance, and
- T_b = baseline transmittance.

11.2 Calculate the net absorption coefficient due to interstitial oxygen α_o :

$$\alpha_o = \alpha_p - \alpha_b \quad (3)$$

where:

- α_o = interstitial oxygen.

11.3 Calculate the interstitial oxygen content of the silicon slice as follows:

$$\text{Interstitial oxygen concentration, ppm atomic} = 6.28\alpha_o$$

$$\text{Interstitial oxygen concentration, atoms/cm}^3 = 3.14 \times 10^{17} \alpha_o \quad (4)$$

NOTE 4—The calibration factor used in this test method was determined as a result of an international interlaboratory experiment. Refer to this calibration factor as IOC-88.⁶ The uncertainty in the calibration factor was stated as $\pm 9 \times 10^{15}$ atoms/ cm^3 or ± 0.18 ppm atomic.⁶

12. Report

12.1 Report the following information:

- 12.1.1 The instrument used, the operator and the date of the measurements.
- 12.1.2 Identification of test and reference specimens.
- 12.1.3 Temperature of the spectrophotometer chamber.
- 12.1.4 Thickness of test and reference specimens.
- 12.1.5 Location and size of the illuminated area on the specimen.
- 12.1.6 Apodization function used (FT-IR instruments).
- 12.1.7 Spectral full width at half maximum of the absorption peak.
- 12.1.8 Area of specimen illuminated.
- 12.1.9 W_p , wavenumber of the absorption peak.
- 12.1.10 The absorption coefficient due to interstitial oxygen, α_o , in cm^{-1} .
- 12.1.11 Oxygen concentration, in ppm or in atoms/ cm^3 .
- 12.2 Refer to the calibration factor used as IOC-88.

13. Precision

13.1 The precision of this test method depends upon the thickness of the test specimen and its oxygen content. The single instrument repeatability of this test method was studied in a recent round-robin experiment, in which the oxygen contents of equivalent sets of 20 2-mm thick samples, with

oxygen contents ranging from 5 to 30 ppma, were measured by 18 different laboratories. The single instrument repeatability of this test method, pooled over all 20 specimens in the test set, ranged from 0.4 to 1.2 % (R1S) for the 18 laboratories in the study.⁶

13.2 The multilaboratory reproducibility for the same sets of test specimens was determined in the same study, with 18 participating laboratories, to be ± 3 % (R1S).⁶

14. Keywords

14.1 infrared absorption; interstitial oxygen; oxygen; silicon

APPENDIX

(Nonmandatory Information)

X1. CONVERSIONS AMONG STANDARDIZED CALIBRATION FACTORS

X1.1 Over the years a number of calibration factors used to calculate the interstitial oxygen content of silicon from the peak room-temperature infrared absorption at 1107 cm^{-1} have been standardized by a number of standards developing organizations. All such standards have since been revised to use the IOC-88 calibration factor that more correctly relates the true oxygen content of silicon to the absorption peak. Nevertheless, many of the old calibration factors remain in common use throughout the industry. The following tables are provided to facilitate conversions between these obsolete, but still used, factors.

X1.2 Table X1.1 gives the calibration factors to relate peak absorption coefficient to interstitial oxygen content in both parts per million atomic (ppma) and atoms/cm³ for various standards that have been replaced by newer revisions.

X1.3 Table X1.2 gives conversion factors to relate these factors to one another.

TABLE X1.1 Calibration Factors

Calibration Factor ^A	Value to Obtain Oxygen Content in ppma	Value to Obtain Oxygen Content in atoms/cm ³
New ASTM ^{2,3}	4.90	2.45×10^{17}
JEIDA ⁴	6.10	3.05×10^{17}
Guo Biao ⁵	6.20	3.10×10^{17}
DIN ² or IOC-88 ⁶	6.28	3.14×10^{17}
Old ASTM ⁷	9.63	4.815×10^{17}

^ASee Corresponding footnotes in the text.

TABLE X1.2 Conversion Factors

To Convert from	To New ASTM or DIN	To JEIDA	To Guo Biao	To IOC-88	To Old ASTM
New ASTM	1	1.245	1.265	1.282	1.965
JEIDA	0.803	1	1.016	1.030	1.579
Guo Biao	0.790	0.984	1	1.013	1.553
DIN ² or IOC-88	0.780	0.971	0.987	1	1.533
Old ASTM	0.509	0.633	0.644	0.652	1

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