



Designation: F 1188 – 00

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.