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Designation: F 1188 – 02

Standard Test Method for Interstitial Atomic Oxygen Content of Silicon by Infrared Absorption with Short Baseline[1]

This standard is issued under the fixed designation F 1188; 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.

INTRODUCTION

Prior to 2002, Test Method F 1188 used a long baseline for the 1107 cm^{-1} oxygen absorption peak. This long baseline was drawn between the average transmittances in the regions from 900 to 1000 cm^{-1} and from 1200 to 1300 cm^{-1}. The same baseline was used in Test Method F 121, the predecessor to Test Method F 1188, and also in the analysis of infrared data from the Grand Round Robin (GRR) experiment.[2] The GRR experiment resulted in a calibration factor between the infrared absorption coefficient at 1107 cm^{-1} and the absolute interstitial oxygen content known as "IOC-88." This calibration factor is utilized in the current editions of all know standardized test methods for oxygen content of silicon, including DIN 50438, Part 1; and JEIDA 61; as well as the current Chinese National Standard (GB).

This test method is being retained in the form published in the 1993a edition of this test method in Annex A1. However, the long baseline is subject to uncertainties due to perturbations in the IR absorption at the end-point regions arising from effects other than absorption by interstitial oxygen. Use of a shorter baseline, drawn between 1040 and 1160 cm^{-1}, which is less afftected in this manner,[3] results in improved precision of the method. Use of the shorter baseline is being introduced into this test method to replace the long baseline method. Although the use of this baseline results in a somewhat smaller net absorption coefficient for the same oxygen content, this change is not very large, and can be neglected in most cases. Optionally, ot completely eliminate the effect of the baseline changes, the measurement equipment may be claibrated with suitable certified reference materials (CRMs) or reference materials derived from CRMs.

NIST SRM[4] 2551 oxygen-in-silicon reference material,[5,6] which is the principal CRM used for calibrating commercial infrared spectrophotometers for oxygen measurements, was certified using a subset of the GRR specimens as the absolute references. Both the GRR specimens and the SRM specimens were measured using a short baseline similar to that now incorporated in this test method. This resulted in (*1*) reduced measurement uncertainty for the SRM specimens, and (*2*) a strong metrological foundation provided by the GRR specimens. Consequently, the IOC-88 results are available to the users of the SRMs without requiring a change in the calibration factor even though the short-baseline method is used.

Despite the fact that IOC-88 has been adopted by all of the major standards developing organizations active in the semiconductor field, many older calibration factors that have been used in earlier standards issued by ASTM and other standards development organizations are still in use in the industry today. Tables showing the relations between the IOC-88 interstitial oxygen calibration factor and other standardized calibration factors that have been used in the silicon semiconductor industry are included as Appendix X1. It should be emphasized that these factors are at times referred to in the literature by common names (as listed in the first column of the tables in Appendix X1) and at other times by the designation of the standard where they were used. Furthermore, in the jargon of the industry, interstitial oxygen content is frequently described as being determined in accordance with a particular standardized method whereas, in actual fact, only the calibration factor is taken from the standard while the measurement itself is made by whatever method is employed within the instrumentation used.

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1. Scope

1.1 This test method covers the determination of the interstitial oxygen content of single crystal silicon by measurement of an infrared absorption band at room temperature. This test method requires the use of an oxygen-free reference specimen. It is recommended that a reference material, such as NIST SRM[4] 2551,[5] another certified reference material for oxygen content of silicon,[7] or reference materials traceable to the CRMs, be used to calibrate the spectrophotometer in order to reduce bias.

1.2 This test method requires the use of a computerized spectrophotometer, preferably an FT-IR spectrophotometer. This method is incorporated into many modern FT-IR instruments.

1.3 The useful range of oxygen concentration measurable by this test method is from 1×10^{16} atoms/cm^3 to the maximum amount of interstitial oxygen soluble in silicon.

1.4 If the spectrophotometer is calibrated using 2-mm thick double-side polished CRMs, this test method is suitable for use only with 2-mm thick, double-side polished test specimens. It can be extended to the measurement of test specimens polished on one or both sides with thickness in the range 0.4 to 4 mm with the use of working reference materials traceable to the CRMs.

1.5 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.

1.6 *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 Thermometers[8]
E 131 Terminology Relating to Molecular Spectroscopy[9]
E 932 Practice for Describing and Measuring Performance of Dispersive Infrared Spectrophotometers[9]
F 121 Test Method for Intersititial Atomic Oxygen Content of Silicon by Infrared Reflectance[10]
2.2 *Other Standards:*
DIN 50438, Part 1 Determination of Impurity Content in Semiconductors by Infrared Absorption: Oxygen in Silicon[11]
JEIDA 61 Standard Test Method for Interstitial Atomic Oxygen Content of Silicon by Infrared Absorption[12]
SEMI C29 Specifications and Guideline for 4.9 % Hydrofluoric Acid 10:1 v/v[13]

3. Terminology

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

3.2 *Definitions:*

3.2.1 *certified reference material (CRM)*, *n*—a reference material, one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation issued by a certifying body.

3.2.2 *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.

3.2.2.1 *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 spectrophotometers

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[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] 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, pp 2015–2034 (1989); Baghdadi, A., Scace, R. I., and Walters, E. J., "*Semiconductor Measurement Technology:* Database for and Statistical Analysis of the Interlaboratory Determination of the Calibration Factor for the Measurement of the Interstitial Oxygen Content of Silicon by Infrared Absorption," NIST Special Publication 400-82, July 1989.

[3] Series, R. W., "Determination of Oxygen and Carbon in Silicon," RSRE Memorandum No. 3479 (Royal Signals & Radar Establishment, Malvern, Worcs, UK, 1982).

[4] SRM is a registered trademark of the National Institute of Standards and Technology.

[5] Available as a set of four 25-mm square, 2-mm thick, silicon specimens, mirror polished on both sides. One of the specimens is a high resistivity float zoned specimen with negligible oxygen content. The other three specimens were cut from *n*-type crystals grown by a modified Czochralski process. Their room temperature resistivity is >3 Ω·cm, and they have nominal oxygen content of 17, 23, and 26 parts per million atomic (IOC-88). The specimens are mounted on aluminum disks for ease of use in production spectrophotometers.

[6] Details of the certification of this SRM are given in Rennex, B. G., "*Standard Reference Materials:* Certification of a Standard Reference Material for the Determination of Interstitial Oxygen Concentration in Semiconductor Silicon by Infrared Spectrophotometry," NIST Spec. Publ. 260-121 (Aug. 1994).

[7] In addition to NIST SRM 2551 (Office of Standard Reference Materials, National Institute of Standards and Technology, 100 Bureau Drive, Gaithersburg, MD 20899-2322), certified reference materials for measurements of the interstitial oxygen content of silicon can be obtained from JEITA (see footnote 12) and from BCR (CRM 368 and CRM 369, from the Community Bureau of Reference, Commission of the European Communities, rue de la Loi, 200, B-1040 Brussels, Belgium).

[8] *Annual Book of ASTM Standards*, Vol 14.03.

[9] *Annual Book of ASTM Standards*, Vol 03.06.

[10] Withdrawn in 1988; last available edition in the 1987 edition of *Annual Book of ASTM Standards*, Vol 10.05.

[11] 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 as thin as 0.03 cm with one side polished and one side etched. 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 F01 maintains close liaison. Both English and German editions of DIN 50438 Part 1 may be obtained from Beuth Verlag GmbH, Berggrafenstrasse 4-10, D-10787 Berlin, Germany, website: www.din.de.

[12] Available from Japanese Electronic and Information Technology Industries Association, Third floor, Mitsui Sumitomo Kaijo Bldg. Annex 11, Kanda Surugadai 3-chome, Chiyoda-ku, Tokyo 101-0062, Japan, website: www.jeita.or.jp.

[13] Available from Semiconductor Equipment and Materials International, 3081 Zanker Road, San Jose, CA 95134, website: www.semi.org.

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(CA-DIR). Dispersive infrared spectrometers that are not computer-assisted are referred to, for convenience, as simple dispersive infrared spectrometers (S-DIR).

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

3.2.3.1 *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. 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.

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

3.2.4.1 *Discussion*—In true double-beam spectrometers, the reference spectrum 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.

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

3.2.5.1 *Discussion*—In true double-beam spectrometers, the sample spectrum 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 is obtained using a reference method with a calibrated IR spectrophotometer, preferably one 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.

4.2 After conducting selected instrumental checks, the infrared transmittance spectrum is measured over the wavenumber range from 900 to 1300 cm^{-1}.

4.3 The measured absorption coefficient of the 1107 cm^{-1} oxygen-in-silicon band is then used to determine 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 calibration factor or linear regression curve, 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. 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 ±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 ± 5°C during the measurement.

6.3 Significant free carrier absorption occurs in *n*-type silicon with a resistivity below 1 Ω·cm, and in *p*-type silicon with a resistivity below 3.0 Ω·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 ± 5 %.

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

6.5 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 may be in error.

7. Apparatus

7.1 *Infrared Spectrophotometer*, either a DIR (S or CA), or FT-IR instrument, as described in 3.2.2 and 3.2.3, respectively, may be used. It must be possible to set the resolution of the spectrophotometer to 4 cm^{-1}, or better, for FT-IR spectrophotometers, and to 5 cm^{-1}, or better, for DIR spectrophotometers. Use of an FT-IR spectrophotometer is strongly recommended.

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

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.[14]

7.3 *Specimen Holders of Appropriate Size*—If the specimen being measured is small, it must be mounted in a holder that has an opening small enough to prevent any of the infrared

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

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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 of 2.25 µm or better (one fourth the wavelength at the maximum absorption of the interstitial oxygen impurity band).

7.5 *Acid Fume Hood and Suitable Protective Gear*, for use when stripping oxide films from specimens for measurement.

7.6 *Micrometer Caliper*, or other instrument suitable for the measurement of the thickness of the specimens to a tolerance of ±0.2 %.

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

7.8 *Hydrofluoric Acid, 4.9 % (10:1 v/v)*, in accordance with Grade 2 of SEMI C29. (**Warning—**The acids used in this test method are hazardous. All precautions normally used with these chemicals should be strictly observed. Obtain and read the Material Safety Data Sheet prior to the use of any chemical.)

8. Testing of the Apparatus

8.1 Evaluate the performance of S-DIR spectrometers according to Instrument Operation 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 5Ω·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 ± 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 ± 2 % is obtained from 1600 to 4000 cm^{-1}. This optimum angle is typically less than 10°.

9. Calibration Specimens

9.1 Obtain a set of certified reference materials (CRMs) for interstitial oxygen in silicon with the following characteristics:

9.1.1 Each specimen in the set is double-side polished and approximately 2 mm thick,

9.1.2 The set covers the oxygen range of interest with three or more samples,

9.1.3 The stated oxygen content is referenced to the IOC-88 calibration factor,[2] and

9.1.4 The set includes an additional oxygen-free reference specimen.

9.2 To extend the test method to measurement of test specimens with thickness other than 2 mm or with only a single polished surface, suitable sets of reference materials must be used to calibrate the instrument for use with specimens of similar thickness and surface conditions.

9.2.1 Sufficient sets of reference materials must be utilized to provide traceability from the reference materials with thickness and surface conditions of the specimens to be tested to the set of CRMs.

NOTE 1—A practice for calibrating infrared spectrophotometers for measuring oxygen in silicon using reference materials of various thickness and surface condition is under development. In the interim, some, but not complete, guidance for this calibration can be found in the NIST SRM 2551 Report.[6]

10. Test Specimens

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

10.1.1 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 2—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. However, the use of specimens with nonparallel surfaces can also result in photometric errors. Because silicon has a high refractive index, any nonparallelism of the specimen can displace the spectrometer beam relative to the active area of the detector. 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.

11. Reference Specimens

11.1 Because a difference technique is used in this test method, prepare an oxygen-free reference specimen of the same type of material as the sample (see 11.1.1). Prepare the oxygen-free reference specimen to the same tolerances as the test specimen with a thickness of equal to that of the test specimen to within ±0.5 %.

11.1.1 Choose the oxygen-free reference specimen from slices taken from five to ten different silicon crystals that are thought to be free of oxygen. Compare these slices with one

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