

SEMI MF1188-1107 (Reapproved 0912) TEST METHOD FOR INTERSTITIAL OXYGEN CONTENT OF SILICON BY INFRARED ABSORPTION WITH SHORT BASELINE

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NOTICE: This Document was reapproved with minor editorial changes.

1 Purpose

1.1 The presence of oxygen can be beneficial to certain manufacturing operations by preventing the formation of process-induced defects. Oxygen is introduced into silicon wafers during the crystal growing process. Hence, it is important to control the oxygen content of silicon crystals.

1.2 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.

1.3 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.

2 Scope

2.1 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, using a short baseline drawn between 1040 and 1160 cm^{-1} to reduce the uncertainties due to perturbations in the IR absorption at the end-point regions of longer baselines that arise from effects other than absorption by interstitial oxygen.¹ Use of the short baseline results in improved precision of the method.

2.1.1 For historical reasons, the long baseline method is being retained in Appendix 1 in the form published in the 1993a edition of this Test Method. This baseline was also used in ASTM F121, the predecessor to this Test Method, and in the analysis of infrared data from the grand round robin (GRR) experiment.

2.2 This Test Method requires the use of a computerized spectrophotometer, preferably a Fourier transform infrared (FT-IR) spectrophotometer. This method is incorporated into many modern FT-IR instruments.

2.3 This Test Method requires the use of an oxygen-free reference specimen.

2.4 It is recommended that a reference material set, such as NIST SRM² 2551,^{3,4} another certified reference material set for oxygen content of silicon,⁵ or reference materials traceable to the certified reference materials (CRMs), be used to calibrate the spectrophotometer in order to reduce bias.

¹ Series, R. W., "Determination of Oxygen and Carbon in Silicon." RSRE Memorandum No. 3479 (Royal Signals & Radar Establishment, Malvern, Worcs, UK, 1982).

² SRM is a registered trademark of the National Institute of Standards and Technology (NIST) for certified reference materials prepared and distributed by NIST.

³ 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 $>3\ \Omega\cdot\text{cm}$, 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.

⁴ 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).

⁵ 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).

2.5 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.

2.6 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 double side polished CRMs.

2.7 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.

2.7.1 The recommended calibration factor to convert absorption at 1107 cm⁻¹ to oxygen content is IOC-88, which is based on the analysis of the international GRR experiment.⁶

NOTE 1: NIST SRM 2551 oxygen-in-silicon reference material, 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.

NOTICE: SEMI Standards and Safety Guidelines do not purport to address all safety issues associated with their use. It is the responsibility of the users of the Documents to establish appropriate safety and health practices, and determine the applicability of regulatory or other limitations prior to use.

3 Limitations

3.1 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.

3.2 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.

3.3 Significant free carrier absorption occurs in *n*-type silicon with resistivity below 1 $\Omega\cdot\text{cm}$, and in *p*-type silicon with resistivity below 3.0 $\Omega\cdot\text{cm}$. 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 $100 \pm 5\%$.

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

3.5 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.

3.6 Although the use of the short baseline results in a somewhat smaller net absorption coefficient for the same oxygen content than that obtained with the use of the long baseline, this change is not very large, and can be neglected in most cases. Optionally, to completely eliminate the effect of the baseline changes, the measurement equipment may be calibrated with suitable CRMs or reference materials derived from CRMs, as recommended in ¶ 2.4.

4 Referenced Standards and Documents

4.1 SEMI Standards and Safety Guidelines

SEMI C29 — Specifications and Guideline for 4.9% Hydrofluoric Acid 10:1 v/v

SEMI M44 — Guide to Conversion Factors for Interstitial Oxygen in Silicon

⁶ Baghdadi, A., Bullis, W. M., Croakin, 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." *J. Electrochem. Soc.* **136**, 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.

SEMI M59 — Terminology for Silicon Technology

4.2 *ASTM Standards*⁷

ASTM E1 — Standard Specification for ASTM Thermometers

ASTM E131 — Standard Terminology Relating to Molecular Spectroscopy

ASTM E932 — Standard Practice for Describing and Measuring Performance of Dispersive Infrared Spectrophotometers⁸

ASTM F121 — Test Method for Interstitial Atomic Oxygen Content of Silicon by Infrared Absorption (Withdrawn 1989)⁹

4.3 *JEITA (formerly JEIDA) Standard*¹⁰

EM-3504 (61) — Standard Test Method for Interstitial Atomic Oxygen Content of Silicon by Infrared Absorption

4.4 *DIN Standard*¹¹

DIN 50438-1 — Determination of Impurity Content in Silicon by Infrared Absorption: Oxygen

4.5 *Guo Biao Standard*¹²

GB/T 1557-1989 — Test Method for Interstitial Oxygen Content in Silicon Crystals by Infrared Absorption Spectroscopy (in Chinese)

NOTICE: Unless otherwise indicated, all documents cited shall be the latest published versions.

5 Terminology

5.1 For definitions of terms relating to absorption spectroscopy, refer to ASTM E131.

5.2 For acronyms, terms, and symbols relating to silicon technology, including those appropriate to infrared spectroscopic analysis of impurities in silicon, refer to SEMI M59.

6 Summary of Test Method

6.1 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.

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

6.3 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.

7 Apparatus

7.1 *Infrared Spectrophotometer* — Either a simple or computer assisted dispersive infrared (DIR) spectrophotometer (see Note 2) or a Fourier transform infrared (FT-IR) spectrophotometer 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 ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, USA; Telephone: +1.610.832.9585, Fax: +1.610.832.9555, <http://www.astm.org>

⁸ *Annual Book of ASTM Standards*, Vol. 03.06.

⁹ Withdrawn in 1989; last available edition in the 1987 edition of *Annual Book of ASTM Standards*, Vol 10.05.

¹⁰ Japanese Electronic and Information Technology Industries Association, 3rd Fl., Mitsui Sumitomo Kaijo Bldg. Annex, 11, Kanda Surugadai 3-chome, Chiyoda-ku, Tokyo 101-0062, Japan. <http://www.jeita.or.jp>

¹¹ Deutsches Institut für Normung e.V., Available from Beuth Verlag GmbH, Burggrafenstrasse 4-10, D-10787 Berlin, Germany; <http://www.din.de>

¹² China Electronic Standardization Institute, Beijing, China

NOTE 2: 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 (CA-DIR). Dispersive infrared spectrometers that are not computer-assisted are referred to, for convenience, as simple dispersive infrared spectrometers (S-DIR).

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 wave numbers.

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.¹³

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 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 $2.25\text{ }\mu\text{m}$ 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 $\pm 0.2\%$.

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.

7.8.1 **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 ASTM E932. 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 $5\text{ }\Omega\cdot\text{cm}$) 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 $1600\text{ to }2000\text{ cm}^{-1}$ 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

¹³ For a discussion of the phase correction computation, see Chase, D. B., *Applied Spectroscopy* **36** (1982): p. 240.