RELATED INFORMATION 1
METHOD FOR ESTIMATION OF SHALLOW ETCH PIT DENSITY

NOTICE: This related information is not an official part of SEMI MF1049. It was developed as part of the development of the document. This related information was approved for publication by full letter ballot on December 4, 2003.

R1-1 Procedure

R1-1.1 Examine the wafer under magnification in the range from 200 to 1000× to distinguish between etching artifacts and shallow etch pits.

R1-1.2 Place the wafer on the microscope stage.

R1-1.3 Position the specimen so as to view the area of interest on the etched wafer surface. Choose the area to be viewed to include a high density of haze.

R1-1.4 Adjust the magnification so that up to 100 shallow etch pits are seen in the field of view. If more than 100 shallow etch pits are in the field of view at maximum magnification, report the shallow etch pit density as "Too high to count."

R1-1.5 Calculate the area of the field of view from its diameter as determined to ± 1 μm with a stage micrometer.

R1-1.6 Count and record the number of shallow etch pits in the field of view. Count as one defect, those defects that converge or overlap except when the etch pits are well defined and are individually distinguishable.

R1-1.7 Determine the estimate of the shallow etch-pit density by dividing the number of etch pits counted by the area of the field of view.

R1-1.8 If more than one area is counted, compute the average shallow etch-pit density for the wafer by dividing the sum of the shallow etch-pit densities estimated by the total number of counting positions.

R1-2 Precision and Bias

R1-2.1 *Precision* — Because this optional method is intended for use only for qualitative estimates of the area of a wafer covered by haze due to shallow etch pits and the shallow etch-pit density, no interlaboratory evaluation of this optional method has been conducted for the purposes of determining its expected repeatability or reproducibility.

R1-2.2 *Bias* — No standards exist against which the bias of this optional method can be evaluated.

NOTICE: SEMI makes no warranties or representations as to the suitability of the standards set forth herein for any particular application. The determination of the suitability of the standard is solely the responsibility of the user. Users are cautioned to refer to manufacturer' s instructions, product labels, product data sheets, and other relevant literature, respecting any materials or equipment mentioned herein. These standards are subject to change without notice.

By publication of this standard, Semiconductor Equipment and Materials International (SEMI) takes no position respecting the validity of any patent rights or copyrights asserted in connection with any items mentioned in this standard. Users of this standard are expressly advised that determination of any such patent rights or copyrights, and the risk of infringement of such rights are entirely their own responsibility.

SEMI MF1152-0305
TEST METHODS FOR DIMENSIONS OF NOTCHES ON SILICON WAFERS

These test methods were technically approved by the Global Silicon Wafer Committee and are the direct responsibility of the North American Silicon Wafer Committee. Current edition approved for publication by the North American Regional Standards Committee on November 4, 2004. Initially available at www.semi.org January 2005; to be published March 2005. Original edition published by ASTM International as ASTM F 1152-88. Last previous edition SEMI MF1152-02.

1 Purpose

1.1 Wafers must be accurately aligned in various processing equipment during integrated circuit manufacture.

1.2 A notch ground into the edge of the wafer at a specified orientation provides a positive method for such alignment. The accuracy of the critical dimensions of the notch controls the possible accuracy of the alignment.

1.3 This test method may be used for process control, quality control, and incoming or outgoing inspection.

1.4 Until an index of precision is determined based on an interlaboratory evaluation, this test method is not recommended for use in decisions between purchasers and suppliers.

2 Scope

2.1 This test method covers a nondestructive procedure to determine whether or not the dimensions, except for the blend radius, of fiducial notches on silicon wafers fall within specified limits.

2.2 This test method is specifically directed to the notch dimensions specified in SEMI M1, but with suitable modifications, the principles of this test method may be applied to any desired notch dimensions.

2.3 No test is provided for the blend radius at the apex of the notch.

2.4 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

NOTICE: This standard does not purport to address safety issues, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health guides and determine the applicability of regulatory or other limitations prior to use.

3 Limitations

3.1 Any foreign material or rough spots on the notch edge in the light path may present a distorted image which can result in the determination of incorrect dimensions.

3.2 Alignment of the notch position with respect to the center of the wafer is important in achieving an accurate determination of the notch characteristics.

3.3 Wear of grinding tools and process variations may result in notch edges which are not exactly straight and a nonunique radius at the apex of the notch. Under these conditions, great care must be taken to align the image of the notch correctly against the appropriate portions of the template.

4 Referenced Standards

4.1 *SEMI Standard*

SEMI M1 — Specifications for Polished Monocrystalline Silicon Wafers

4.2 *ANSI Standard*[1]

ANSI/ASQC Z1.4 — Sampling Procedures and Tables for Inspection by Attributes

---

1 American National Standards Institute, New York Office: 25 West 43rd Street, New York, NY 10036, USA. Telephone: 212.642.4900, Fax: 212.398.0023, Website: www.ansi.org.

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

5 Summary of Test Method

5.1 The wafer is aligned in position on an optical comparator and the image of the notch is compared with a series of templates projected on the screen of the comparator.

5.2 First, the wafer is aligned so that the sides of the image of the notch contact the image of the alignment pin for fixing the position of the wafer in use. In this case, the image of the notch bottom must lie on or below the designated line on the notch form/depth template and the image of the wafer edge must lie on or above another designated line on the template.

5.3 The wafer is then aligned so that the image of the wafer edge coincides with the wafer periphery line on the template. In this case, the image of the notch bottom must lie between maximum and minimum lines on the template.

5.4 The image of the notch sides are compared with a series of angles on the notch angle template and the angle that makes the best fit is chosen as the value of the notch angle.

6 Apparatus

6.1 *Optical Comparator* — capable of 20× and 50× magnification with a viewing screen large enough to display an area 5 by 5 mm at 20× or 2 by 2 mm at 50×.

6.2 *Fixture* — for holding the wafer to be tested. The fixture must provide means for positioning the wafer such that the plane of the surface of the wafer is perpendicular to the viewing direction and that the wafer can be rotated about its center. The horizontal and vertical motions are parallel or perpendicular to the diameter of the wafer that passes through the notch.

6.3 *Templates* — having lines which define the limits of the notch dimensions. Two templates are required.

6.3.1 The notch form and depth template has two sections that define (1) the locations of the notch bottom and wafer periphery relative to the center of the alignment pin, and (2) the location of the notch bottom relative to the wafer periphery. Separate templates are required for each wafer diameter to be tested. An example of a notch form and depth template is given in Figure 1.

6.3.2 The notch angle template contains angles from 88° to 96° in 1° increments.

6.3.3 Instructions for constructing templates are given in §9.

6.4 *Gage Block or Precision Rod* — with dimensions approximately the same as the depth of the notch and accurately known for use in establishing the magnification of the apparatus.

6.5 *Rule* — 150 mm long with scale gradations of 0.5 mm or less.

7 Sampling

7.1 Unless otherwise specified, ANSI/ASQC Z1.4 shall be used. Inspection levels shall be agreed upon between the supplier and purchaser.

8 Determination of Magnification Factor

8.1 Adjust the comparator to the desired magnification. Using the gage block or precision rod of accurately known dimensions, follow the manufacturer's instructions to establish the object-to-image magnification to three significant figures.

9 Preparation of Template

9.1 Multiply each of the chosen or specified template dimensions by the magnification factor.

9.2 Prepare on transparent material a full-scale template having the dimensions calculated in ¶9.1 with a projected image accuracy of ±0.5 mm (± 0.020 in.).

9.3 Include horizontal and vertical axes and label the lines on the notch form and depth template as shown in Figure 1.
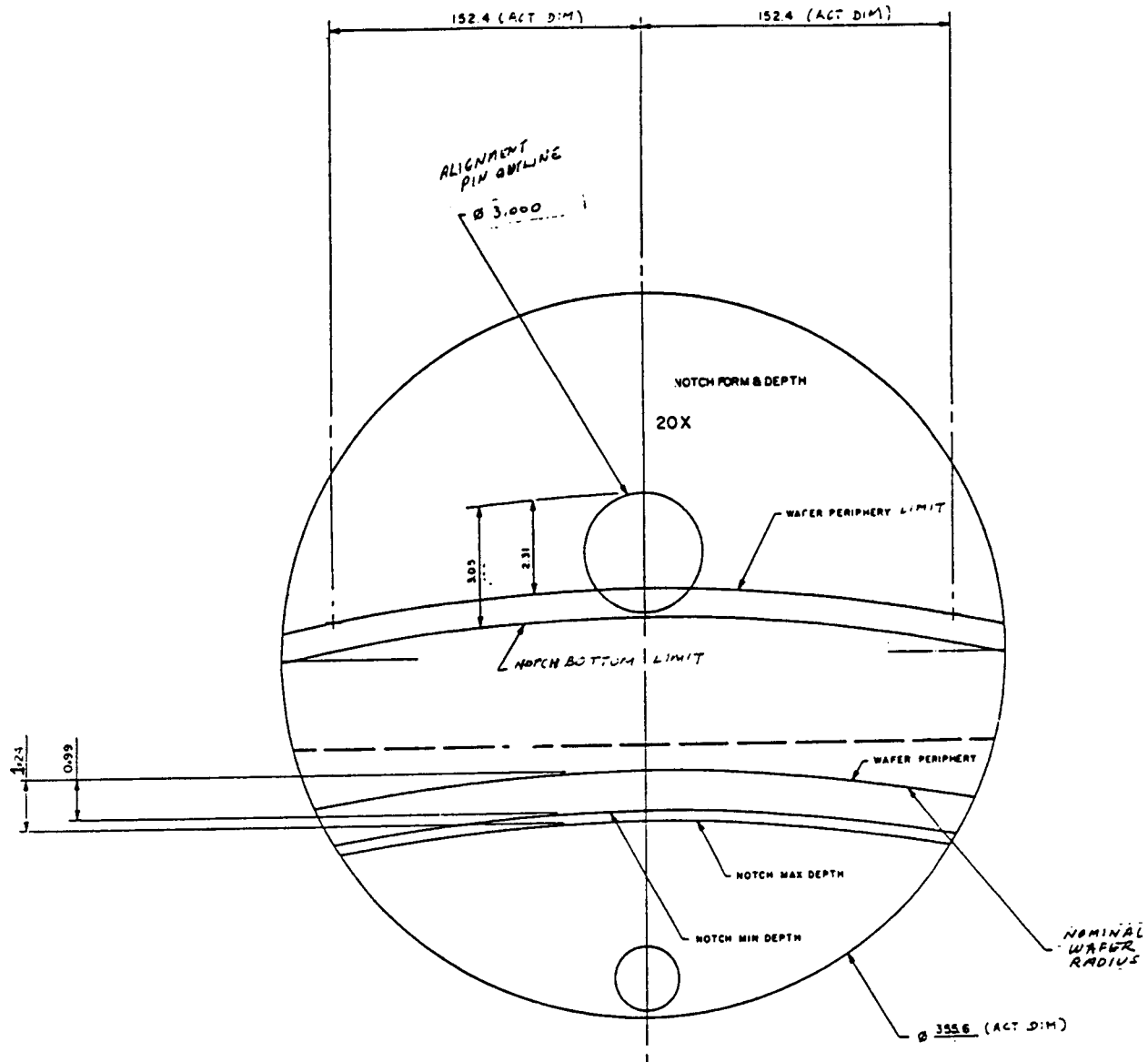
NOTE: In use the template is rotated 90° counterclockwise.
Figure 1
Example of Notch Form and Depth Template

10 Procedure

10.1 Set the magnification to 20×.

10.2 Align the fixture in the comparator so that the notch is at the nine o'clock position and the directions of horizontal and vertical motion of the fixture in the comparator are parallel with and perpendicular to, respectively, the diameter that passes through the notch.

10.3 Place the notch form and depth template on the comparator screen. Align the horizontal and vertical lines with the simulated pin outline in the nine o'clock position.

10.4 Place the first wafer to be tested in the fixture, front surface up.

10.5 Align the fixture using only table crossfeed (horizontal) control and fixture rotation so that the simulated pin outline on the template makes contact with the sides of the notch image.

10.6 Verify that the image of the notch bottom falls on or below the NOTCH BOTTOM LIMIT line. If the image of the notch bottom falls above this line, record the wafer as defective.

10.7 Verify that the image of the wafer edge falls on or above the WAFER PERIPHERY LIMIT line. If the image of the wafer edge falls above this line, record the wafer as defective.

10.8 Move the fixture to the right until the image of the wafer edge falls on the line marked WAFER PERIPHERY using only the table crossfeed control.

10.9 Verify that the notch bottom falls between the NOTCH MAX DEPTH and NOTCH MIN DEPTH lines. If the image of the notch bottom falls outside these lines, record the wafer as defective.

10.10 Repeat ¶¶10.4 through 10.9 for all remaining wafers to be tested.

10.11 Remove the notch form and depth template and replace it with the notch angle template.

10.12 Set the magnification to 50×.

10.13 Place the first wafer to be tested in the fixture, front surface up.

10.14 Align the image of the wafer notch sides with each angle on the template using the table crossfeed control and the fixture rotation. Define as the notch angle, the angle that provides the best fit to the image. If the notch angle is < 89° or > 95°, record the wafer as defective.

10.15 Repeat ¶10.14 for all remaining wafers to be tested.

10.16 On completion of the testing, return the magnification to 20×.

11 Report

11.1 Report as a minimum the following information:

11.1.1 Date of test,

11.1.2 Name of person conducting the test,

11.1.3 The lot number of other identification of the material,

11.1.4 The number of wafers in the lot,

11.1.5 The number of test wafers, and

11.1.6 The number of defective wafers.

11.2 If desired, a table of the types of defects observed may be provided.

12 Precision and Bias

12.1 An interlaboratory evaluation of this test method has not been executed, nor is one anticipated. Use of the test method for commercial transactions is not recommended unless the parties to the test establish the degree of correlation that can be obtained.

12.2 A dimension of 0.1 mm in the object plane produces a screen image of 2.0 mm at 20× and of 5.0 mm at 50×. The smallest size details of the notch contour which can be inspected by this test method are of comparable dimensions.

13 Keywords

notch; notch dimension; optical comparator; silicon; wafer

NOTICE: SEMI makes no warranties or representations as to the suitability of the standards set forth herein for any particular application. The determination of the suitability of the standard is solely the responsibility of the user. Users are cautioned to refer to manufacturer's instructions, product labels, product data sheets, and other relevant literature, respecting any materials or equipment mentioned herein. These standards are subject to change without notice.

By publication of this standard, Semiconductor Equipment and Materials International (SEMI) takes no position respecting the validity of any patent rights or copyrights asserted in connection with any items mentioned in this standard. Users of this standard are expressly advised that determination of any such patent rights or copyrights, and the risk of infringement of such rights are entirely their own responsibility.

SEMI MF1152-0305 © SEMI 2003, 2005

SEMI MF1188-0305
TEST METHOD FOR INTERSTITIAL OXYGEN CONTENT OF SILICON BY INFRARED ABSORPTION WITH SHORT BASELINE

These test methods were technically approved by the Global Silicon Wafer Committee and are the direct responsibility of the North American Silicon Wafer Committee. Current edition approved for publication by the North American Regional Standards Committee December 10, 2004 Initially available at www.semi.org January 2005; to be published March 2005. Original edition published by ASTM International as ASTM F 1188-88. Last previous edition SEMI MF1188-02.

INTRODUCTION

Prior to 2002, ASTM 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 ASTM 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.[1] 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 known standardized test methods for oxygen content of silicon, including DIN 50438, Part 1; and JEITA EM-3504; as well as the current Chinese National Standard (GB).

The long baseline method is being retained in the form published in the 1993a edition of this test method in Appendix 1. 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 affected in this manner,[2] results in improved precision of the method. Use of the shorter baseline is 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, to completely eliminate the effect of the baseline changes, the measurement equipment may be calibrated with suitable certified reference materials (CRMs) or reference materials derived from CRMs.

NIST SRM[3] 2551 oxygen-in-silicon reference material,[4,5] which is the principal CRM used for calibrating commercial infrared spectrophometers 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.

---

1 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," *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.

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

3 SRM is a registered trademark of the National Institute of Standards and Technology.

4 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.

5 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).

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 can be found in SEMI M44. It should be emphasized that these factors are at times referred to in the literature by common names 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.

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. This test method requires the use of an oxygen-free reference specimen. It is recommended that a reference material set, such as NIST SRM[3] 2551,[4] another certified reference material set for oxygen content of silicon,[6] or reference materials traceable to the CRMs, be used to calibrate the spectrophotometer in order to reduce bias.

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 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.

2.4 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 mm to 4 mm with the use of working reference materials traceable to the double-side polished CRMs.

2.5 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.

NOTICE: This standard does not purport to address safety issues, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health guides and determine the applicability of regulatory or other limitations prior to use.

---

6 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).

3 Limitations

3.1 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.

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 $27 \pm 5°C$ during the measurement.

3.3 Significant free carrier absorption occurs in *n*-type silicon with resistivity below 1 Ω·cm, and in *p*-type silicon with 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 %.

3.4 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.

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.

4 Referenced Standards

4.1 *SEMI Standards*

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

4.2 *ASTM Standards*

E 1 — Specification for ASTM Thermometers[7]

E 131 — Terminology Relating to Molecular Spectroscopy[8]

E 932 — Practice for Describing and Measuring Performance of Dispersive Infrared Spectrophotometers[8]

F 121— Test Method for Intersititial Atomic Oxygen Content of Silicon by Infrared Absorption[9]

4.3 *JEITA* (*formerly JEIDA*) *Standard*

EM-3504 (61) — Standard Test Method for Interstitial Atomic Oxygen Content of Silicon by Infrared Absorption[10]

4.4 *DIN Standard*

50438/1 — Determination of Impurity Content in Silicon by Infrared Absorption: Oxygen[11]

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 Terminology E 131.

5.2 *Definitions*

---

7 *Annual Book of ASTM Standards*, Vol 14.03, ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428. Telephone: 610-832-9500, Fax: 610-832-9555, Website: www.astm.org

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

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

10 Japan Electronics and Information Technology Industries Association, 3rd floor, Mitsui Sumitomo Kaijo Bldg. Annex, 11, Kanda-Surugadai 3-chome, Chiyoda-ku, Tokyo 101-0062, Japan, Website: www.jeita.or.jp.

11 Deutches Institut für Normung e.V., standards are available in both English and German editions from Beuth Verlag GmbH, Burggrafenstrasse 6, 10787 Berlin, Germany, Telephone: 49.30.2601-0, Fax: 49.30.2601.1263, Website: www.din.de.

5.2.1 *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.

5.2.2 *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.

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

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

5.2.3.1 *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.

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

5.2.4.1 *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.

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

5.2.5.1 *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.

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 wavenumber range from 900 cm^{-1} 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 DIR (S or CA), or FT-IR instrument, as described in ¶5.2.2 and ¶5.2.3, 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.[12]

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 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.

NOTE 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 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 Ω·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 cm^{-1} 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 cm^{-1} 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,

---

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

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 2: 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.[5]

10 Test Specimen

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 3: 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, an apparent lowering of the 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.

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 another, and choose the specimen with the lowest absorption as the oxygen-free 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 oxygen-free reference specimen.

11.1.2 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%.

11.2 Prepare separate oxygen-free reference specimens for each type of material to be tested.

12 Procedure

12.1 *Calibration of Spectrophotometer*

12.1.1 Obtain a set of certified reference materials for oxygen in silicon (see §9) and, if necessary, suitable sets of traceable reference materials with different thickness or surface condition, or both.

12.1.2 Use these reference materials to calibrate the spectrophotometer in accordance with manufacturer's instructions. The calibration results in a linear regression curve that relates the measured absorption coefficient to the interstitial oxygen content of the measured specimen (Note 1).

12.2 *Instrumental Checks* (*FT-IR Spectrophotometers*)

NOTE 4: If a DIR spectrophotometer is being used, substitute the procedure in ¶A1-2.1.2 for the procedure in ¶12.2.2.

12.2.1 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 cm^{-1} 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.

12.2.2 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 cm^{-1} 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 (Note 3).

12.2.3 Determine mid-scale linearity of the instrument by obtaining an air reference spectrum of the silicon reference specimen over the wavenumber range from 1600 cm^{-1} 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.

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

12.4 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.

12.5 Measure and record the temperature of the spectrophotometer chamber.

12.6 Determine the measurement time for the spectra by obtaining the transmittance spectrum of a high resistivity (greater than 5 Ω·cm), 0.04 cm to 0.085 cm thick double-side polished silicon slice containing between 12 ppma and 18 ppma oxygen (IOC-88) using a minimum of 64 scans. If the ratio of the net amplitude of the oxygen band, $T_b - T_p$, to the standard deviation in the transmittance spectrum is not greater than 100, increase the number of scans until that criterion is met.

12.7 Obtain the infrared transmittance spectrum with a resolution of 4 cm^{-1}, or better, at 1107 cm^{-1}, for FT-IR instruments or, for dispersive instruments, 5 cm^{-1}, or better, over (at least) the range from 900 cm^{-1} to 1300 cm^{-1}. The test and reference specimens must be positioned so that the IR beam is centered on them. On double beam 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.

12.8 Plot the transmittance spectrum over the range from 900 cm^{-1} to 1300 cm^{-1}.

12.9 Define the baseline of the oxygen-only transmittance spectrum by drawing a straight line from the transmittance at 1040 cm^{-1} to the transmittance at 1160 cm^{-1}.

12.10 Locate the wavenumber corresponding to the minimum transmittance in the region from 1102 cm^{-1} to 1112 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 ¶12.9 at W_p. Record both T_p and T_b to three significant figures.

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

13 Calculations

13.1 Calculate the peak and baseline absorption coefficients using the following equations:

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

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

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.

13.2 Calculate the net absorption coefficient, α_O, due to interstitial oxygen:

$$\alpha_O = \alpha_p - \alpha_b \qquad (3)$$

13.3 Determine the interstitial oxygen content of the silicon slice from the linear regression curve (see ¶12.1.2) that relates the measured absorption coefficient to the interstitial oxygen concentration in ppma or the interstitial oxygen volume density in cm^{-3}, as desired. This value of oxygen content is based on the IOC-88 calibration factor.

13.3.1 If reference materials are not used to calibrate the spectrophotometer, calculate the oxygen content from α_O in accordance with ¶A1-3.3.

13.4 If desired, calculate and record the interstitial oxygen content in terms of any other standardized calibration factor using the appropriate conversion factor in SEMI M44.

14 Report

14.1 Report the following information:

14.1.1 The instrument used, the operator and the date of the measurements.

14.1.2 Test method and baseline used (SEMI MF1188, Short Baseline).

14.1.3 Identification of test and reference specimens.

14.1.4 Temperature of the spectrophotometer chamber.

14.1.5 Apodization function used (FT-IR instruments).

14.1.6 Identification of all certified and working reference materials used to calibrate the spectrophotometer.

14.2 For each test and reference specimen used, report the following information:

14.2.1 Specimen thickness.

14.2.2 Location and size of the illuminated area on the specimen.

14.2.3 W_p, wavenumber of the absorption peak, in cm^{-1}.

14.2.4 Spectral full width at half maximum of the absorption peak.

14.2.5 Absorption coefficient due to interstitial oxygen, α_O, in cm^{-1}.

14.2.6 Oxygen concentration, in ppm or in atoms/cm^3.

14.2.7 The calibration factor used (IOC-88 recommended).

15 Precision

15.1 The uncertainty due to measurement variations in the certification of the NIST SRM 2551 set was found to be equal to or less than 0.17%.[5]

15.2 The certification measurements were made using a short-baseline procedure similar to that in this method except that the endpoints of the baseline were taken as the average of the values between 1030 cm^{-1} and 1050 cm^{-1} and between 1060 cm^{-1} and 1080 cm^{-1}, respectively.

15.3 This uncertainty is expected to represent the best attainable precision with this method.

16 Bias

16.1 The uncertainty in the absolute determinations of oxygen content in silicon during the Grand Round Robin resulted primarily from the uncertainty in the chemical analysis measurements and any variations in the oxygen content of the various slices used in the test. The first of these is estimated to be about 6%, and the second is estimated to be much smaller.[1] This uncertainty directly affects the assigned value of the IOC-88 calibration factor. Errors in this calibration factor cause errors in the value of the derived oxygen content. Therefore, the bias of these measurements is estimated not to exceed 6%.

17 Keywords

infrared absorption; infrared spectrophotometry; interstitial oxygen; oxygen; silicon

APPENDIX 1
ORIGINAL (LONG BASELINE) METHOD

NOTICE: The material in this appendix is an official part of SEMI MF1188. Approval was by full letter ballot procedures with publication authorized by the NA Regional Standards Committee on December 10, 2004.

A1-1 Introduction

A1-1.1 §§1 through 8, 10, and 11 of the main test method also apply to this method.

A1-1.1.1 If a dispersive spectrophotemeter is being used, disregard ¶7.2.

A1-2 Procedure

A1-2.1 *Instrumental Checks*

A1-2.1.1 Establish the 100% baseline to measure the noise level.

A1-2.1.1.1 On double beam instruments, record the transmittance spectrum with both the sample and reference beams empty.

A1-2.1.1.2 On single-beam instruments, obtain the transmittance spectrum as the ratio of two spectra taken with the sample beam empty.

A1-2.1.1.3 Plot the 100% baseline over a wavenumber range covering 900 cm^{-1} to 1300 cm^{-1}. If the baseline is not 100% ± 0.5% over the entire range, increase the measurement time until it is. If the problem persists, have the instrument repaired.

A1-2.1.2 Applies to dispersive (DIR) instruments only. Establish the 0% line. With the sample beam blocked, record the instrument zero over the range from 900 cm^{-1} 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.

A1-2.1.3 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 cm^{-1} 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.

A1-2.1.4 Determine mid-scale linearity of the instrument by obtaining an air reference spectrum of the silicon reference specimen over the wavenumber range from 1600 cm^{-1} to 2000 cm^{-1}. If the value of the transmittance is not 53.8% ± 2% over this wavenumber range, align the sample with the spectrometer in accordance with ¶8.3.

A1-2.1.5 Determine the measurement time for the spectra by obtaining the transmittance spectrum of a high resistivity (greater than 5 Ω·cm), 0.04 cm to 0.085 cm thick double-side polished silicon slice containing between 12 ppma 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_b - T_p, 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.

A1-2.2 *Surface Oxide Removal* — Immediately prior to the initial measurement in any laboratory, etch all specimens, including the reference specimen, in hydrofluoric acid to remove any surface oxide and rinse.

A1-2.3 *Specimen Thickness* — Measure the thicknesses of the test and reference specimens to within ± 0.2%, at their centers. If thickness of the reference specimen does not match the thickness of the test specimen to within ± 0.5%, obtain a reference specimen with the proper thickness.

A1-2.4 *Temperature* — Measure and record the temperature of the spectrophotometer chamber.

A1-2.5 *Infrared Transmitter Spectrum* — Obtain the spectrum with a resolution of 4 cm^{-1}, or better, at 1107 cm^{-1}, for FT-IR instruments or, for dispersive instruments, 5 cm^{-1}, or better, over (at least) the range from 900 cm^{-1} to 1300 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.

A1-2.6 Plot the transmittance spectrum over the range from 900 cm^{-1} to 1300 cm^{-1}.

A1-2.7 Define the baseline by drawing a straight line from 900 cm^{-1} to 1300 cm^{-1}. Use the average transmittance in the gions from 900 cm^{-1} to 1000 cm^{-1}, and 1200 cm^{-1} to 1300 cm^{-1}, to define the endpoints of the straight line.

A1-2.8 Locate the wavenumber corresponding to the minimum transmittance in the region from 1102 cm^{-1} to 1112 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 ¶A1-2.7 at W_p. Record both T_p and T_b to three significant figures.

A1-2.9 Determine and record the full width at half maximum (FWHM) of the peak.

A1-3 Calculations

A1-3.1 Calculate the peak and baseline absorption coefficients using the following equations:

$$\alpha_p = -\frac{1}{x}\ln\left[\frac{(0.09-e^{1.70x})+\sqrt{(0.09-e^{1.70x})^2+0.36T_p^2 e^{1.70x}}}{0.18T_p}\right] \tag{A1-1}$$

$$\alpha_b = -\frac{1}{x}\ln\left[\frac{(0.09-e^{1.70x})+\sqrt{(0.09-e^{1.70x})^2+0.36T_b^2 e^{1.70x}}}{0.18T_b}\right] \tag{A1-2}$$

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.

A1-3.2 Calculate the net absorption coefficient, α_O, due to interstitial oxygen:

$$\alpha_O = \alpha_p - \alpha_b \tag{A1-3}$$

A1-3.3 Calculate the interstitial oxygen content of the silicon slice as follows:

$$\text{Interstitial oxygen concentration, ppm atomic} = 6.28\alpha_O \tag{A1-4}$$

or

$$\text{Interstitial oxygen volume density, atoms/cm}^3 = 3.14\times10^{17}\alpha_O \tag{A1-5}$$

A1-3.4 This value of oxygen content is based on the IOC-88 calibration factor. If desired, calculate the interstitial oxygen content in terms of any other standardized calibration factor using the appropriate conversion factor in SEMI M44.

A1-4 Report

A1-4.1 Report the following information:

A1-4.1.1 The instrument used, the operator and the date of the measurements,

A1-4.1.2 Test method and baseline used (SEMI MF1188, Long Baseline),

A1-4.1.3 Identification of test and reference specimens,

A1-4.1.4 Temperature of the spectrophotometer chamber,

A1-4.1.5 Thickness of test and reference specimens,

A1-4.1.6 Location and size of the illuminated area on the specimen,

A1-4.1.7 Apodization function used (FT-IR instruments),

A1-4.1.8 Spectral full width at half maximum of the absorption peak,

A1-4.1.9 Area of specimen illuminated,

A1-4.1.10 W_p, wavenumber of the absorption peak, in cm^{-1},

A1-4.1.11 The absorption coefficient due to interstitial oxygen, α_o, in cm^{-1},

A1-4.1.12 Oxygen concentration, in ppm or in atoms/cm^3, and

A1-4.1.13 The calibration factor used (IOC-88 recommended).

A1-5 Precision

A1-5.1 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 an international experiment, in which the oxygen contents of equivalent sets of 20, 2 mm thick samples, with interstitial oxygen content ranging from 5 ppma to 30 ppma (IOC-88), 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.[1]

A1-5.2 The multilaboratory reproducibility for the same sets of test specimens was determined in the same study, with 18 participating laboratories, to be ± 3% (R1S).[1]

A1-6 Bias

A1-6.1 This experiment also included measurements of the absolute oxygen content by a variety of physical methods. The uncertainty in the absolute determinations of oxygen content in silicon during the Grand Round Robin results primarily from the uncertainty in the chemical analysis measurements and any variations in the oxygen content of the various slices used in the test. The first of these is estimated to be about 6%, and the second is estimated to be much smaller. This uncertainty directly affects the assigned value of the IOC-88 calibration factor. Errors in this calibration factor cause errors in the value of the derived oxygen content. Therefore, the bias of these measurements from these sources is estimated not to exceed 6%.

A1-6.2 There is also the possibility of bias because of interfering peaks in the infrared spectrum at the wavenumbers where the baseline is determined. The magnitude of this bias varies from sample to sample and cannot be predicted.

SEMI MF1239-0305
TEST METHOD FOR OXYGEN PRECIPITATION CHARACTERISTICS OF SILICON WAFERS BY MEASUREMENT OF INTERSTITIAL OXYGEN REDUCTION

1 Purpose

1.1 Oxide precipitates in the bulk of a silicon wafer can act as gettering sites for contamination that may be introduced during manufacture of circuits and devices. This contamination (usually metallic impurities), if not gettered, can reduce device manufacturing yields and degrade device or circuit performance. Thus, the oxygen precipitation characteristics of the silicon wafer can significantly affect both yields and performance.

1.2 Although interstitial oxygen concentration is an important factor in affecting the amount of oxygen precipitation that occurs in silicon during a specific thermal cycle, the presence of other impurities such as carbon or nitrogen, and differences in dopant type and density, thermal history, or defect properties of the crystal can also affect the precipitation characteristics. Thus, it is frequently necessary to choose particular material properties and preparation techniques to obtain the desired precipitation characteristics for a particular application.

1.3 This test method may be used to compare the oxygen reduction of two or more groups of silicon wafers. This test method is based on thermal cycles that simulate certain common device processing cycles.

1.3.1 *Cycle A*, a one-step precipitation cycle, provides an indication of the native nucleation sites present in the as-received wafers.

1.3.2 *Cycle B*, a two-step nucleation-precipitation cycle, simulates the precipitation that occurs in normal *n*-MOS device processing.

1.4 These test methods may be used to compare qualitatively the precipitation characteristics of two or more groups of wafers.

1.5 These test methods may also be used to determine the uniformity of oxygen reduction across a wafer (in conjunction with SEMI MF951) or from wafer to wafer within a lot.

2 Scope

2.1 These test methods cover complementary procedures for testing the oxygen precipitation characteristics of silicon wafers. It is assumed that the precipitation characteristics are related to the amount of interstitial oxygen lost during specified thermal cycles.

2.2 These test methods may be applied to any *n*- or *p*-type Czochralski silicon wafers of any orientation whose thickness, resistivity, and surface finish are such as to permit the oxygen concentration to be determined by infrared absorption and whose oxygen concentration is such as to produce measurable oxygen loss.

2.3 These test methods are not suitable for determining the width or characteristics of a "denuded zone," a region near the surface of a wafer that is essentially free of oxide precipitates.

2.4 Because these test methods are destructive, suitable sampling techniques must be employed.

2.5 Determination of material performance in actual device fabrication situations is beyond the scope of these methods. However, by comparing the results of these tests with actual device yields and performance, criteria for selection of specific material characteristics may be established.

2.6 The values stated in SI units are regarded as standard.

SEMI MF1239-0305 © SEMI 2003, 2005

NOTICE: This standard does not purport to address safety issues, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health guides and determine the applicability of regulatory or other limitations prior to use.

3 Limitations

3.1 All factors that affect the infrared absorption measurement (including differences in back surface condition, instrumental characteristics, and wafer resistivity) may cause errors in the determination of oxygen reduction.

3.2 If significant quantities of oxygen are outdiffused during the thermal cycles, the measured oxygen reduction may not be representative of the amount of oxygen precipitation.

3.3 If precipitate size varies from sample to sample, the variations in measured oxygen reduction may not be representative of variations in the number of oxide precipitates that are formed.

3.4 The specified thermal cycles may or may not provide adequate simulation of the cycles used in a particular device processing sequence. The results obtained in these test methods can serve as predictors of those expected in actual device processing only to the extent that the simulation is representative of the device process.

4 Referenced Standards

4.1 *SEMI Standards*

SEMI C28 — Specifications and Guidelines for Hydrofluoric Acid

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

SEMI C54 — Specifications and Guidelines for Oxygen

SEMI C59 — Specifications and Guidelines for Nitrogen

SEMI M59 — Terminology for Silicon Technology

SEMI MF951 — Test Method for Determination of Radial Interstitial Oxygen Variation Silicon Wafers

SEMI MF1188 — Test Method for Interstitial Atomic Oxygen Content of Silicon by Infrared Absorption with Short Baseline

SEMI MF1619 — Test Method for Measurement of Interstitial Oxygen Content of Silicon Wafers by Infrared Absorption Spectroscopy with *p*-Polarized Radiation Incident at the Brewster Angle

4.2 *ASTM Standard*

D 5127 — Guide for Ultra Pure Water Used in the Electronics and Semiconductor Industry [1]

4.3 *JEITA* (*formerly JEIDA*) *Standard*

EM-3504 (61) — Standard Test Method for Interstitial Atomic Oxygen Content of Silicon by Infrared Absorption[2]

4.4 *DIN Standard*

50438/1 — Determination of Impurity Content in Silicon by Infrared Absorption: Oxygen[3]

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

5 Terminology

5.1 For definitions of terms relating to silicon technology, refer to SEMI M59.

---

1 *Annual Book of ASTM Standards*, Vol 11.01, ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428. Telephone: 610-832-9500, Fax: 610-832-9555, Website: www.astm.org

2 Japan Electronics and Information Technology Industries Association, 3rd floor, Mitsui Sumitomo Kaijo Bldg. Annex, 11, Kanda-Surugadai 3-chome, Chiyoda-ku, Tokyo 101-0062, Japan, Telephone: 81.3.3518.6434, Fax: 81.3.3295.8726, Website: www.jeita.or.jp.

3 Deutches Institut für Normung e.V., standards are available in both English and German editions from Beuth Verlag GmbH, Burggrafenstrasse 6, 10787 Berlin, Germany, Telephone: 49.30.2601-0, Fax: 49.30.2601.1263, Website: www.beuth.de.

6 Summary of Test Method

6.1 A representative sample is selected from each group of wafers to be tested.

6.2 The initial value of interstitial oxygen concentration is measured by the infrared absorption method at the desired points on each wafer.

6.3 The wafers are passed through one of two simulation thermal cycles. Cycle A consists solely of a precipitation step. Cycle B consists of a nucleation step followed by a precipitation step.

6.4 After the thermal cycle, the oxide film is stripped and the final value of oxygen concentration is measured at the same points on each wafer using the same technique and instrumentation as was used to determine the initial value.

6.5 The oxygen reduction is determined for each wafer (or for each point on each wafer) tested as the difference between the initial and final values.

6.6 If all samples have the same initial oxygen concentration (within a narrow range), the average oxygen reduction for each test condition (such as, group or position on wafer) is computed, and the appropriate comparisons made.

6.7 If the samples have initial oxygen concentrations that cover a relatively wide range, a plot of oxygen reduction against initial oxygen concentration is made for each group or position. Again appropriate comparisons can be made.

7 Apparatus

7.1 *Infrared Absorption Spectrophotometer*, as specified in SEMI MF1188 or DIN 50438, Part 1.

7.2 *Resistance Heated Tube Furnace*, capable of providing temperatures in the range from 750°C to 1050°C to ±2°C over the length required to contain the load of wafers to be tested. The furnace shall be fitted with the following:

7.2.1 *Gas Manifold* — That allows dry oxygen and nitrogen to be mixed at the required ratios and flows, (see Table 1);

Table 1 Thermal Cycle Tests for Oxygen Precipitation in Silicon

Parameter	Value
Test A	1050°C for 16 h
Test B	750°C for 4 h followed by 1050°C for 16 h
Furnace Ambient	Nitrogen plus 5% dry oxygen
Gas Flow Rate	4.2 ± 0.2 L/min[#1]
Push/Pull Temperature	750°C
Push/Pull Rate	25 cm/min
Ramp Up Rate	10°C/min
Ramp Down Rate	5°C/min

[#1] For 155 mm diameter tube; for other diameters flow rate should be proportional to the cross sectional area of the tube.

7.2.2 *Quartz, Polysilicon, or Silicon Carbide Tube* — Of diameter appropriate for the wafers to be tested to isolate the wafers from external contamination;

7.2.3 *Quartz Boats* — To hold the wafers during processing;

7.2.4 *Loader* — To allow controlled insertion of the quartz boat into the hot zone, (see Table 1); and

7.2.5 *Laminar Flow Load Station* — To permit loading of the wafers without adding contamination to the surfaces.

7.3 Facilities for processing wafers through chem-mechanical polishing, or bright acid etching to provide smooth, flat surfaces.

7.4 Facilities for dipping the wafers in hydrofluoric acid prior to the oxygen determination in order to remove the surface oxide film grown during thermal cycling. Suitable protective clothing, acid disposal facilities, and ventilation shall be provided.

7.5 Facilities for cleaning and drying wafers by a standard process as customarily used in the laboratory performing the test.

7.6 *Scribe or Laser Marker* — For marking the wafers with unique identification (unless premarked wafers are available).

8 Reagents and Materials

8.1 *Hydrofluoric Acid* — In accordance with SEMI C28, Grade 1.

8.2 *Oxygen —* In accordance with grade 2.5 of SEMI C54.

8.3 *Nitrogen* — In accordance with grade 5.2 of SEMI C59.

8.4 *Deionized Water* — With purity equal to or greater than that specified for Type E-1 in ASTM Guide D 5127.

9 Hazards

9.1 The acids used in these test methods are hazardous. All precautions normally used with these chemicals should be strictly observed. Obtain and read the material safety data sheet prior to use of any chemical.

10 Selection and Preparation of Test Specimens

10.1 Choose test wafers from each group being tested in such a way as to cover the entire range of oxygen concentration found in the group. Choose at least two wafers with oxygen concentration in each 0.5 ppm (IOC-88)[4] interval in the range. For example, if the oxygen concentration range of a group is 3 ppma, at least 12 wafers from that group should be tested.

10.2 Select or prepare test wafers with thickness, resistivity, and surface finish as required by the oxygen test method being used.

10.3 Identify each test wafer individually with an alphanumeric laser marking or a hand scribed code unless pre–marked wafers are used.

10.4 Prepare the wafers in accordance with SEMI MF1188, SEMI MF1619, DIN 50 438/1, or JEITA EM-3504, as applicable.

11 Procedure

11.1 Determine the initial interstitial oxygen concentration of each wafer to be tested at the center in accordance with SEMI MF1188, SEMI MF1619, DIN 50 438/1, or JEITA EM-3504. If desired, measure the interstitial oxygen concentration at other locations on each wafer as specified by an appropriate pattern in SEMI MF951. Record the oxygen value(s), the wafer identification, and measurement locations. Record the date of the test and the instrument used in measuring the oxygen concentration. See Figure 1 for a suggested data sheet format.

11.2 Clean and dry the wafers in accordance with the usual wafer cleaning procedure employed by the laboratory performing the test.

11.3 Process the samples as soon after cleaning as possible. If the samples must be stored between cleaning and processing, store in clean covered cassettes.

11.4 Heat-treat in accordance with Cycle A or Cycle B, (see Table 1). Record the date of the heat treatment and the cycle used.

11.5 Strip the surface oxide from the wafers with hydrofluoric acid (HF), and thoroughly clean and dry them with the usual wafer cleaning procedure employed by the laboratory performing the test.

11.6 Measure the post-heat treatment interstitial oxygen concentration at each point measured before heat treatment. Use the same instrument, test method, and set up for this measurement as was used for the initial measurement. Record each final oxygen concentration on the same data sheet as was used to record initial oxygen concentration.

---

4 As defined in SEMI MF1188, SEMI MF1619, DIN 50 438/1, or JEITA EM-3504.

Date of Initial (O_i) Reading_____ Operator_____

Spectrometer Identification_____

Date of Heat Treatment_____ Operator_____

Precipitation Test_____
(TEST A OR TEST B)

Date of Final (O_i) Reading_____ Operator_____

Group Identification _____
(LOT NUMBER AND MEASUREMENT LOCATION)

Wafer Identification	Initial (O_i)	Final (O_i)	(O_i) Reduction	Average Initial (O_i)[A]	Standard Deviation[A]	Average (O_i) Reduction[A]
Average[B]		— — — — —		— — — — —	— — — — —	— — — — —
Std Dev[B]		— — — — —		— — — — —	— — — — —	— — — — —

[A] Apply only to interpretation by Method 2 (see ¶12.2.2).

[B] Average and standard deviation of initial O_i and O_i reduction apply only to interpretation by Method 1 (see ¶12.2.1).

Figure 1
Suggested Data Sheet Format

12 Calculations and Interpretation of Results

12.1 Subtract each final oxygen concentration value from the corresponding initial oxygen concentration value to determine the oxygen reduction. Record the oxygen reduction.

12.2 Interpret the results by Method 1 or Method 2 as follows:

12.2.1 *Method 1*

12.2.1.1 Use this method when the desired target oxygen concentration is already known, when each group tested has the same target oxygen concentration, and when the range of oxygen concentration values measured in each group has a range less than 4 ppma (IOC-88). This method cannot be used if the average initial oxygen concentrations of the groups tested differ by more than 0.5 ppma (IOC-88).[4]

12.2.1.2 Determine the averages and standard deviations of the initial oxygen concentration and oxygen reduction for each group of wafers tested.

12.2.1.3 If the average oxygen reduction values of the groups tested agree to within a desired amount, consider the groups equivalent.

12.2.2 *Method 2*

12.2.2.1 Use this method to de-couple oxygen content and precipitation behavior to obtain (1) a qualitative overview of the precipitation characteristics of the groups tested and (2) the important features of the characteristic precipitation curve.

12.2.2.2 Bin the oxygen reduction data for each group so that all wafers with oxygen concentration within each 0.5 ppma interval are included in the same bin.

12.2.2.3 Calculate the average of the initial oxygen concentration and the oxygen reduction for each bin in each group.

12.2.2.4 Plot the average oxygen reductions against average initial oxygen concentrations for each group tested. Use a different symbol to distinguish the data for each group. See Figures 2 through 5 for examples of such plots.
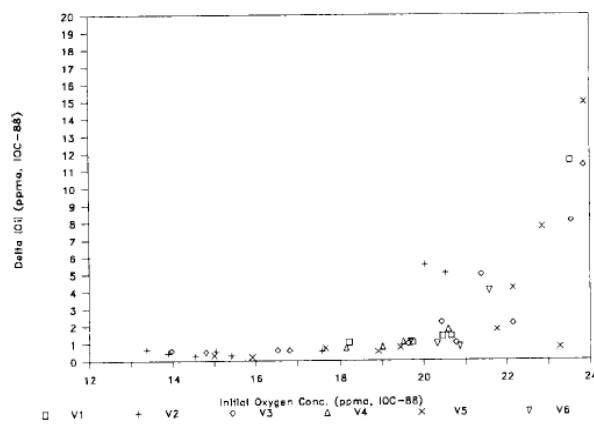
Figure 2
Oxygen Reduction in Wafers from Six Groups (V1 to V6) Following Exposure to One-Step Cycle A as Measured by Laboratory 3
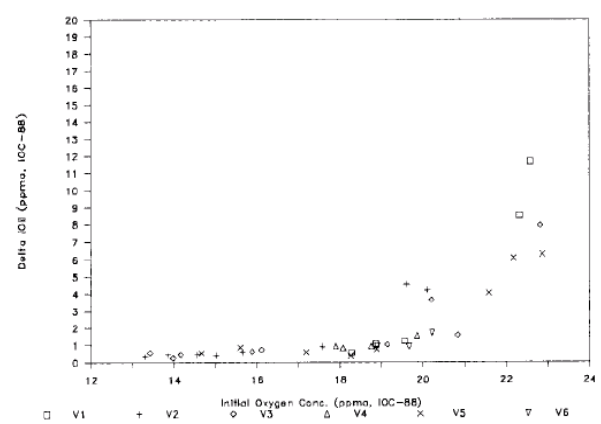
Figure 3
Oxygen Reduction in Wafers from Six Groups (V1 to V6) Following Exposure to One-Step Cycle A as Measured by Laboratory 4
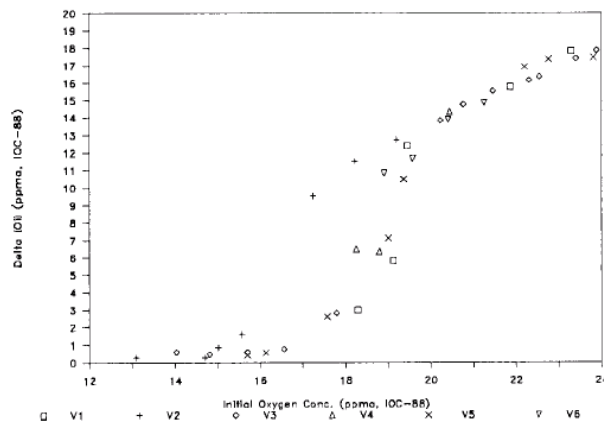
Figure 4
Oxygen Reduction in Wafers from Six Groups (V1 to V6) Following Exposure to Two-Step Cycle B as Measured by Laboratory 3
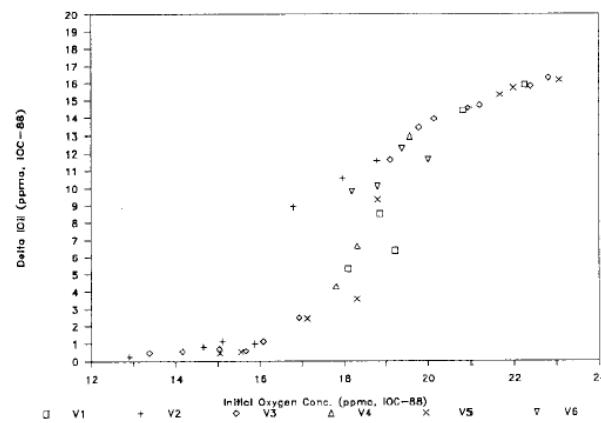

Figure 5
Oxygen Reduction in Wafers from Six Groups (V1 to V6) Following Exposure to Two-Step Cycle B as Measured by Laboratory 4

12.2.2.5 Note that the curves have three characteristic regions, as illustrated by distinct slope change in Figure 4 and Figure 5. At low initial oxygen concentration, there is essentially no oxygen reduction; at some value of initial

oxygen concentration, partial precipitation occurs (in this transition region, oxygen reduction changes rapidly with increasing initial oxygen concentration); at high initial oxygen concentration, full precipitation occurs (in this region, oxygen reduction is proportional to initial oxygen concentration).

12.2.2.6 Compare the curves obtained for each group. If the data for each group falls within a band of suitable width, consider the groups equivalent.

13 Report

13.1 Report the following for each group tested:

13.1.1 Identification of group (lot number, location of measurement point on wafer, center or edge, etc.),

13.1.2 Dates of initial and final oxygen measurement and of the heat treatment and identification of operators for measurement and heat treatment,

13.1.3 Identification of infrared spectrophotometer used,

13.1.4 Table of initial and final oxygen concentrations for each wafer measured, and

13.1.5 Averages and standard deviations of the initial oxygen content and oxygen reduction for each group tested.

13.2 In addition, if Method 2 was used, report the following:

13.2.1 Table of average initial oxygen concentration and average oxygen reduction for each bin in each group, and

13.2.2 Graph of average oxygen reductions against average initial oxygen concentrations for each group.

14 Precision

14.1 Wafers from six different groups with different back surface conditions were processed in two laboratories and combined. Both Cycles A and B were used. Measurements of initial and final oxygen concentrations on all wafers tested were made at two other laboratories.

14.2 The wafers within each group did not meet the 2-wafer per 0.5 ppma interval required by these test methods.

14.3 Nevertheless, from the plots reproduced as Figures 2 through 5, both measurement laboratories (that employed different FT-IR spectrophotometers for making the oxygen determinations at the center of the wafers only) concluded that the wafers from Groups V1, V3, V4, V5, and V6 were essentially equivalent but those from Group V2 had increased precipitation in the transition region.

15 Bias

15.1 No reference standards are available for precipitated oxygen, so it is impossible to determine bias except for that of the individual oxygen measurements. Bias of the individual oxygen measurements should be determined in accordance with the procedures of the test method utilized.

16 Keywords

delta $[O_i]$; interstitial oxygen; oxygen precipitation; oxygen reduction; silicon ; $\Delta[O_i]$

SEMI MF1366-0305
TEST METHOD FOR MEASURING OXYGEN CONCENTRATION IN HEAVILY DOPED SILICON SUBSTRATES BY SECONDARY ION MASS SPECTROMETRY

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 very important to control the oxygen content of silicon crystals.

1.2 SIMS can measure the oxygen concentration in heavily-doped silicon substrates used for epitaxial silicon where the free carrier concentration obscures the infrared absorption and prevents the normal use of the infrared measurement as a characterization technique for the commercial production of silicon.

1.3 The SIMS measurement allows for the production of controlled oxygen content in heavily-doped silicon crystals.

1.4 This test method can be used for process control, research and development, and materials acceptance purposes.

2 Scope

2.1 This test method covers the determination of total oxygen concentration in the bulk of single crystal silicon substrates using secondary ion mass spectrometry (SIMS).

2.2 This test method can be used for silicon in which the dopant concentrations are less than 0.2% (1×10^{20} atoms/cm^3) for boron, antimony, arsenic, and phosphorus (see SEMI MF723). This test method is especially applicable for silicon that has resistivity between 0.0012 and 1.0 Ω·cm for p-type silicon and between 0.008 and 0.2 Ω·cm for n-type silicon (see SEMI MF43).

2.3 This test method can be used for silicon in which the oxygen content is greater than the SIMS instrumental oxygen background as measured in a float zone silicon sample, but the test method has a useful precision especially when the oxygen content is much greater (approximately 10× to 20×) than the measured oxygen background in the float zone silicon.

2.4 This test method is complementary to infrared absorption spectroscopy that can be used for the measurement of interstitial oxygen in silicon that has resistivity greater than 1.0 Ω·cm for p-type silicon and greater than 0.1 Ω·cm for n-type silicon (see SEMI MF1188). The infrared absorption measurement can be extended to between 0.02 and 0.1 Ω·cm for n-type silicon with minor changes in the measurement procedure.[1]

2.5 In principle, different sample surfaces can be used, but the precision estimate was taken from data on chemical-mechanical polished surfaces.

NOTICE: This standard does not purport to address safety issues, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health guides and determine the applicability of regulatory or other limitations prior to use.

3 Limitations

3.1 Oxygen from silicon oxide, carbon oxide, and water on the surface can interfere with the oxygen measurement.

3.2 Oxygen adsorbed from the SIMS instrument chamber to the surface can interfere with the oxygen measurement.

---

1 Hill, D. E., "Determination of Interstitial Oxygen Concentration in Low-Resistivity n-type Silicon Wafers by Infrared Absorption Measurements," *J. Electrochem. Soc.* **137**, 3926 (1990).

3.3 There are no effects upon the oxygen ion yield from the dopants for dopant densities less than 1×10^{20} atoms/cm^3.[2]

3.4 The SIMS oxygen instrumental background as measured on the float zone silicon specimen should be as low as possible and stable before the analyses are begun.

3.5 The specimen surface must be flat in the specimen holder windows so that the inclination of the specimen surface with respect to the ion collection optics is constant from specimen to specimen. Otherwise, the accuracy and precision can be degraded.

3.6 The accuracy and precision of the measurement significantly degrade as the roughness of the specimen surface increases. This degradation can be avoided by using chemical-mechanical polished surfaces.

3.7 Variability of oxygen in the calibration standards can limit the measurement precision.

3.8 Bias in the assigned oxygen of the calibration standards can introduce bias into the SIMS measured oxygen.

4 Referenced Standards

4.1 *SEMI Standards*

SEMI MF43 — Test Methods for Resistivity of Semiconductor Materials

SEMI MF723 — Practice for Conversion between Resistivity and Dopant Density for Arsenic-Doped, Boron-Doped, and Phosphorus-Doped Silicon

SEMI MF1188 — Test Method for Interstitial Atomic Oxygen Content of Silicon by Infrared Absorption

4.2 *ASTM Standard*

E 122 — Practice for Choice of Sample Size to Estimate a Measure of Quality for a Lot or Process[3]

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

5 Terminology

5.1 *Definitions*

5.1.1 *IOC-88* — calibration factor[4] for infrared absorption measurements of oxygen in silicon that gives a value equivalent to that found by the SIMS method.

5.1.2 *ion mass spectrometry* — the separation and counting of ions by their mass-to-charge ratio.

5.1.3 *primary ions* — ions created and focussed by an ion gun onto the specimen surface to sputter ionize surface atoms.

5.1.4 *secondary ions* — ions that leave the specimen surface as a result of the primary ion beam sputter ionizing the specimen surface atoms.

5.1.5 *secondary ion mass spectrometry* — mass spectrometry performed upon secondary ions from the specimen surface.

6 Summary of Test Method

6.1 SIMS is utilized to determine the bulk concentration of oxygen in single crystal silicon substrate.

---

2 Bleiler, R. J., Chu, P. K., Novak, S. W., and Wilson, R. G.," Study of Possible Matrix Effects in the Quantitative Determination of Oxygen in Heavily Doped Czochralski Silicon Crystals," *Seventh International Meeting on Secondary Ion Mass Spectrometry, SIMS VII* (John Wiley and Sons, 1990), p. 507.

3 *Annual Book of ASTM Standards*, Vol 14.02, ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428. Telephone: 610-832-9500, Fax: 610-832-9555, Website: www.astm.org.

4 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.

6.1.1 Specimens of single crystal silicon (one float-zone silicon specimen, two calibration specimens, and the test specimen) are loaded into a sample holder.

6.1.2 The holder with the specimens is baked at 100°C in air for 1 h and then transferred into the analysis chamber of the SIMS instrument.

6.1.3 A cesium primary ion beam is used to bombard each specimen. The negative secondary ions are mass analyzed.

6.1.4 The specimens are presputtered sequentially to reduce the instrumental oxygen background.

6.1.5 The specimens are then analyzed, in locations different from the presputtering locations, for oxygen and silicon in a sequential manner throughout the holder. Three measurement passes are made through the holder.

6.1.6 The ratio of the measured oxygen and silicon secondary ion intensities (O^-/Si^-) is calculated for each specimen.

6.1.7 The relative standard deviation (RSD) of the ratio is then calculated for each specimen.

6.1.8 If any specimen other than the float zone specimen has a RSD of the ratio greater than 3%, more analyses are performed.

6.1.9 The SIMS average O/Si ratios are then converted to infrared-equivalent concentrations by utilizing either the load-line calibration method[5,6] or the load factor calibration method[2] with the calibration specimens in the load.

7 Apparatus

7.1 *SIMS Instrument*, equipped with a cesium primary ion source, electron multiplier detector and Faraday cup detector, and capable of measuring negative secondary ions.

7.1.1 The SIMS instrument should be adequately prepared (that is, baked) so as to provide the lowest possible instrumental background.

7.2 *Cryopane1*, liquid nitrogen- or liquid helium-cooled, which surrounds the test specimen holder in the analysis chamber.

7.3 *Test Specimen Holder*.

7.4 *Oven*, for baking test specimen holder.

8 Sampling

8.1 Since this procedure is destructive in nature, a sampling procedure must be used to evaluate the characteristics of a group of silicon wafers. No general sampling procedure is included as part of this test method, because the most suitable sampling plan will vary considerably depending upon individual conditions. For referee purposes, a sampling plan shall be agreed upon before conducting the test. See ASTM Practice E 122 for suggested choices of sampling plans.

9 Specimen Requirements

9.1 Sample specimens must be flat and smooth on the side used for analysis.

9.2 Sample specimens must be cleaved or diced to fit within the sample specimen holder.

10 Calibration

10.1 The two calibration standards in each load must be lightly doped Czochralski silicon[7] in which the oxygen concentration is measured by infrared absorption spectroscopy (see SEMI MF1188), and the measured values of the

---

5 Goldstein, M., and Makovsky, J., "The Calibration and Reproducibility of Oxygen Concentration in Silicon Measurements Using SIMS Characterization Techniques," *Semiconductor Fabrication: Technology and Metrology, ASTM STP 990*, Dinesh C. Gupta, Ed. (ASTM, 1988) pp. 350–360.

6 Makovsky, J., Goldstein, M., and Chu, P., "Progress in the 'Load Line Calibration' Method for Quantitative Determination of [O] in Silicon by SIMS," *Seventh International Meeting on Secondary Ion Mass Spectrometry, SIMS VII* (John Wiley and Sons, 1990) p. 487.

7 Czochralski silicon is available from most silicon substrate suppliers.

two standards bracket the expected values for the test specimen (that is, one calibration standard is higher in oxygen and one is lower, compared to the expected value in the test specimen).

10.2 The calibration standards must be measured by infrared absorption to determine the concentration and homogeneity of the oxygen within the standards; each standard is assigned the averaged infrared absorption oxygen value (IOC-88) for the substrate.

10.3 Calibration standards that are included in the SIMS analyses must be taken from that portion of the wafer that provided a homogeneous measurement in the Fourier transform infrared (FT-IR) spectrophotometer analysis; this portion is typically the central portion of the wafer.

10.4 Each calibration standard specimen must be the same size and have the same polished surface as the test specimen.

10.5 The float zone specimens that are included in the SIMS analysis to measure the instrumental oxygen background must be measured by infrared absorption to determine if the oxygen concentration is low enough to measure the instrumental SIMS background. Oxygen concentrations below 0.5 ppma in the float zone specimen are normally sufficient.

10.6 Each float zone specimen must be the same size and have the same polished surface as the test specimen.

11 Procedure

11.1 *Specimen Loading*

11.1.1 Load the specimens into the SIMS sample holder, checking to see that the specimens are flat against the backs of the windows and cover the windows as much as is possible. A specimen load includes one float zone silicon specimen, two or more standard specimens, and the test specimen.

11.1.2 Bake the loaded sample holder at 100°C ± 10°C for a minimum of 1 h in air.

11.2 *Instrument Tuning*

11.2.1 Turn on the instrument in accordance with the manufacturer's instructions.

11.2.2 Fill the liquid nitrogen or helium cold trap.

11.3 *Analytical Conditions*

11.3.1 Use a cesium primary ion current and focus, which maximizes the ion count rate for an appropriate silicon isotope.

11.3.2 Typical analytical conditions are a 250 μm by 250 μm raster and 1-s integrations. Choose apertures to keep the oxygen count rate on the electron multiplier detector below 1×10^5 counts per second for the test specimen.

11.4 *Analysis of Specimen*

11.4.1 Position the specimen holder so that the sputtered crater in the specimen will form near the center of the window.

11.4.2 Center the primary ion beam and begin a SIMS profile.

11.4.3 Repeat ¶11.4.1 and ¶11.4.2 for all the specimens in the holder until all the specimens have been presputtered. This is called a presputtering round and is intended to reduce the instrumental oxygen background. No oxygen data are taken or used from these profiles.

11.4.4 Now make a second round of measurements on all the samples according ¶11.4.1 and ¶11.4.2, but in locations near the presputtered craters of the specimen. Do not make the second round of measurements in the same craters as the presputter craters.

11.4.5 At the end of each profile in the second round of craters, measure and record the $^{16}O^-$ count rate on the electron multiplier detector and the $^{30}Si^-$, or other Si isotope as appropriate, matrix ion count rate on the Faraday cup detector.

11.4.6 Repeat ¶11.4.4 and ¶11.4.5 for a third and fourth round of measurements, all in their own separate craters. Group all the analyses for each specimen near the center of the window but do not overlap. The raster area for each analysis must be the same.

11.4.7 Calculate the ratio $S(O^-/Si^-)$ of oxygen count rate to silicon count rate using the recorded secondary ion intensities at the end of each profile in the second, third, and fourth rounds of profiles, thus obtaining three ratios per specimen.

11.4.8 Calculate the average $S_{avg}(O^-/Si^-)$ of the three ratios for each specimen in the holder.

11.4.9 Compare the average ratio $S_{avg-FZ}(O^-/Si^-)$ for the float zone specimen to the average ratio $S_{avg-sp}(O^-/Si^-)$ of the other specimens. If the average ratio for the float zone specimens is not much less (approximately 10 times less) than the average ratio for the other specimens, then the precision of the measurement may be degraded. In this case, depending upon the desired precision, it may be necessary to either abort the analysis and find the cause of the high instrumental background, or to increase the number of measurements per specimen.

11.4.10 Calculate the relative standard deviation (RSD percent) of the ratio $S(O^-/Si^-)$ for each specimen, including the calibration specimen, the float zone specimen, and the test specimen.

11.4.11 Repeat the analysis of oxygen for specimens (other than float zone specimens) with a RSD percent greater than 3%.

11.4.12 Record the specimen identification, O^-/Si^- ratios, average, standard deviation, and relative standard deviation in a table and include the same for the float zone silicon specimen.

12 Calculations

12.1 *Load Line Calibration Procedure*

12.1.1 Calculate the slope *m* and intercept *b* of the load calibration line of infrared absorption oxygen values *F* versus the SIMS average O^-/Si^- ratios *S* for the standards as follows:

$$F = mS + b \tag{1}$$

where *m* and *b* are calculated from the assigned infrared absorption oxygen values F_1 and F_2 of the two calibration standards and S_1 and S_2 are the measured average ratios of the O^-/Si^- for the two calibration standards.

$$m = \frac{(F_1 - F_2)}{(S_1 - S_2)} \tag{2}$$

$$b = \frac{(F_1 S_2 - F_2 S_1)}{(S_2 - S_1)} \tag{3}$$

12.1.2 Convert the SIMS O^-/Si^- ratios S_u for each test specimen and for the float zone specimen to the infrared absorption equivalent value F_u based on the calibration line (Equation 1) and as illustrated in Figure 1.

12.2 *Load Factor Calibration Procedure*

12.2.1 Calculate the calibration load factors, $LF_1 = F_1/S_1$ and $LF_2 = F_2/S_2$, of the assigned infrared absorption oxygen *F* to the *S* ratio O^-/Si^- from the standard samples using the average ratios of the O^-/Si^- from the calibration standards.

12.2.2 Calculate the average load factor, LF_{avg}, as follows:
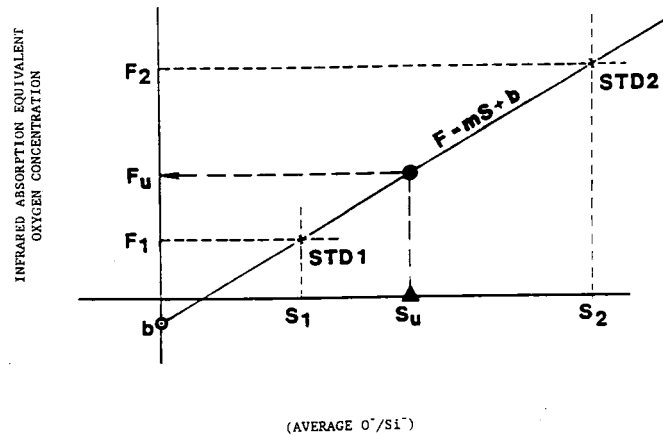
$$LF_{avg} = \frac{(LF_1 + LF_2)}{2} \tag{4}$$

Figure 1
Load Line Calibration for SIMS Bulk Oxygen Analysis

12.2.3 Convert the SIMS O⁻/Si⁻ ratios S_u for each test specimen to the infrared absorption equivalent value F_u by multiplying the S_u by the average load factor LF_{avg} derived from the calibration standards, as follows:

$$F_u = S_u \times LF_{avg} \tag{5}$$

13 Report

13.1 Report the following information:

13.1.1 The instrument used, the operator, and the date of the measurements,

13.1.2 Identification of test and standard specimens,

13.1.3 Calibration procedure used,

13.1.4 The infrared absorption equivalent oxygen values for the test specimen and the float zone silicon specimen, and

13.1.5 The relative standard deviations (RSD percent) of the oxygen values for the test specimen and the float zone silicon.

14 Precision

14.1 The precision was estimated for both the load line calibration and average load factor calibration procedures using 191 samples from one silicon wafer and measured in 191 different loads over a two-month period. The one standard deviation was 0.38 ppma for both methods for an oxygen level of 18 ppma. The data are given in Appendix 1.

14.2 The load line and average load line methods to quantification gave equivalent accuracy and precision in the test outlined in ¶14.1. The reason one is used versus another is more conceptual than empirical if the oxygen values for the standards bracket the oxygen values of the test specimens.

15 Keywords

FTIR; oxygen; secondary ion mass spectrometry; silicon

APPENDIX 1
ANALYSIS OF DATA FROM THE MULTI-INSTRUMENT EXPERIMENT

NOTICE: The material in this appendix is an official part of SEMI MF1366. Approval was by full letter ballot procedures with publication authorized by the NA Regional Standards Committee on December 10, 2004.

A1-1 The precision estimate was taken from data generated in one laboratory using three instruments and ten instrument operators. All the instruments were CAMECA IMS 3f or 4f SIMS instruments. The test specimens were all taken from one silicon wafer that was lightly doped and verified by infrared absorption spectroscopy to have uniform interstitial oxygen levels across the central region where the test specimens were taken. The test specimens were chemically mechanically polished on one side. The standards had oxygen levels that bracketed the expected level of oxygen of the test specimen.

A1-2 The measurements were made in 191 loads over a two-month period.

A1-3 Both the load line calibration and the average load factor calibration methods were used to convert the SIMS data to infrared absorption equivalent oxygen. The load line calibration method gave an average oxygen level of 18.64 ppma (IOC-88), a one standard deviation of 0.39 ppma, and a relative standard deviation of 2.09%. A frequency distribution is shown in Figure A1-1. The average load factor calibration method gave an average oxygen level of 18.61 ppma (IOC-88), a one standard deviation of 0.365 ppma, and a relative standard deviation of 1.97%. A frequency distribution is shown in Figure A1-2.

NOTE 1: The oxygen concentration scale in Figures A1-1 and A1-2 is given in Old ASTM units. These must be multiplied by 0.652 to get the values of oxygen concentration in IOC-88 units. This difference in scale arises from an historical basis and does not affect the conclusions reached. Note also that the oxygen values in §A1-3 have been adjusted to obtain the results in IOC-88 units.
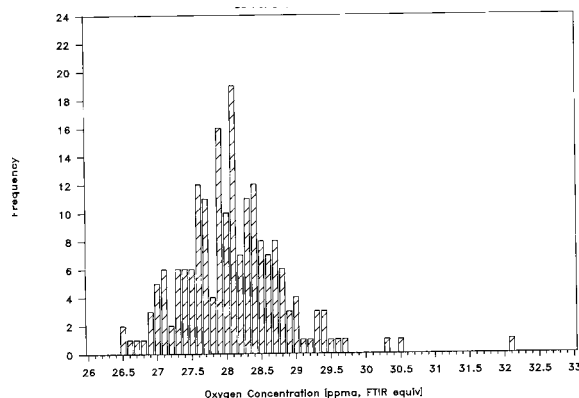

Figure A1-1
**Frequency Distribution of SIMS Measured Oxygen
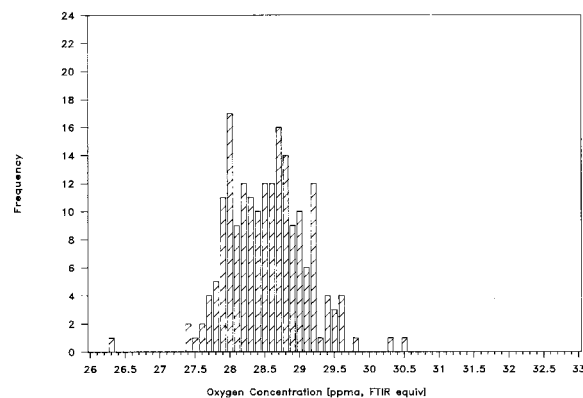Using the Load Line Calibration Method**

Figure A1-2
**Frequency Distribution of SIMS Measured Oxygen
Using the Average Load Factor Calibration Method**

SEMI MF1389-0704
TEST METHODS FOR PHOTOLUMINESCENCE ANALYSIS OF SINGLE CRYSTAL SILICON FOR III-V IMPURITIES

This guide was technically approved by the Global Silicon Wafer Committee and is the direct responsibility of the North American Silicon Wafer Committee. Current edition approved for publication by the North American Regional Standards Committee on March 14, 2004. Initially available at www.semi.org May 2004; to be published July 2004. Original edition published by ASTM International as ASTM F 1389-92. Last previous edition SEMI MF1389-00.

1 Purpose

1.1 Electronic-grade polycrystalline silicon producers and users require information regarding impurities for quality assurance as well as for research and development purposes. Polysilicon is float-zoned and a sample from the zoned rod is analyzed following these test methods to obtain impurity densities that can be related to the impurity content of the starting material (see SEMI MF1723).

1.2 Photoluminescence analysis identifies and quantifies the electrically active dopant impurities in monocrystalline silicon. These test methods address boron, phosphorus, arsenic, and aluminum, found as impurities in electronic grade silicon.

1.3 These test methods can be applied to doped and undoped float-zoned or Czochralski material.

2 Scope

2.1 These test methods cover the simultaneous determination of electrically active boron, phosphorus, arsenic, and aluminum content in low-dislocation monocrystalline silicon.

NOTE 1: These chemical species can also be determined by the low temperature infrared analysis procedure of SEMI MF1630.

2.2 These test methods can be used for samples that have dopant densities between approximately 1×10^{11} and approximately 5×10^{15} atoms/cm^3.

2.3 The concentrations obtained using these test methods are based on an empirically determined relationship of the logarithm of the concentration to the logarithm of specific luminescence line-intensity ratios.

2.4 The empirical relationship established assumes a constant sample excitation level for all measurements on a given instrument.

2.5 To accommodate differences in instrumentation, two methods are included. Test Method A refers to procedures appropriate for dispersive infrared spectrophotometers operating under the high sample excitation conditions and Test Method B refers to procedures appropriate for Fourier transform instruments operating under low excitation conditions.

2.5.1 Typical calibration curves for each test method are provided. These curves are modified for each instrument using the analysis of standard samples as reference data. Once modified, the curves for a given instrument should produce sample dopant density values that agree with other similarly operated instruments using the same test method. Data obtained using Test Method A may not agree with data obtained using Test Method B, hence values must be reported with reference to the test method used.

NOTE 2: Several different methods of photoluminescence analysis are currently in practice worldwide. These test methods address two of these, one (Test Method A) in use primarily in Japan[1] and the other (Test Method B) primarily in the United States. Recently published works[2,3] describe other approaches.

2.6 Many laboratories use photoluminescence to analyze epitaxial layers. However this application encounters many variables and the underlying physics is not fully understood; hence these test methods do not attempt to outline standard practices regarding such analysis.

NOTICE: This standard does not purport to address safety issues, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health guides and determine the applicability of regulatory or other limitations prior to use.

---

1 Test Method A is essentially equivalent to JIS H 0615, Test Method for Determination of Impurity Concentrations in Silicon Crystal by Photoluminescence Spectroscopy. This standard is available from Japanese Standard Association 1-24, Akasaka 4 Chome, Minato-ku, Tokyo 107-0000, Japan. Telephone: 81-(0)3-3583-8005; Fax: 81-(0)3-3586-2014; Website: www.jsa.or.jp.

2 Colley, P. McL., and Lightowlers, E. C., "Calibration of the Photoluminescence Technique for Measuring B, P, and Al Concentrations in Silicon in the Range 1e12 to 1e15 at/cm 3 Using Fourier Transform Spectroscopy," *Semiconductor Science and Technology* **2**, 157–166 (1987).

3 Schumacher, K. L., and Whitney, R. L., *J. Electron. Materials* 18(6), 681–687 (1989).

3 Limitations

3.1 *Variations in Excitation Intensity* — The extrinsic bound exciton (BE) and intrinsic free exciton (FE) luminescence features do not vary at the same rate with excitation intensity.[4] The FE features increase proportionally with excitation intensity, while the BE features increase proportionally at low excitation levels but become more slowly increasing at higher excitation levels generally above the electron hole droplet (EHD) onset point. Since the calculated concentrations are derived from the ratio of extrinsic features to the intrinsic feature, the ratio will be decreasing as the excitation intensity is increasing. Thus, if a sample is measured at a higher excitation level than the instrument is calibrated for, the calculated concentration will be artificially low, and vice versa.

3.2 Sample surface damage, bulk defects, or other lifetime reducers also affect the line-intensity ratios and overall luminescence intensity owing to their tendency to reduce the steady state population of excitons, thus mimicking the effects of a lower excitation intensity. Other mechanisms related to the presence of these defects may also introduce decalibrating effects. Some luminescence features are generated by defects; for example, those at 6510 and 7050 cm^{-1} are typical of thermally stressed samples. Such features can provide qualitative information about the presence of defects.[5,6]

3.3 The ratios and line widths of the silicon luminescence features vary strongly with temperature, hence variations in sample temperature must be avoided. Corrections for the effects of temperature variations are included in these test methods (see Section 7.1).

3.4 *Overlapping Spectral Features*

3.4.1 The boron $B_{TO}(BE)$ feature at 8812.6 cm^{-1} overlaps the $P_{TO}(b'_1)$ feature at 8812.7 cm^{-1} causing a direct error when calculating boron concentration. The beta-series $P_{TO}(b'_1)$ line is approximately one-tenth the intensity of the alpha series $P_{TO}(BE)$ line at 8806.6 cm^{-1}, so a subtraction based on the amount of phosphorus present can be made in the boron feature measurement. An alternative approach is to use the $B_{TO}(b_1)$ boron line, which is free from a coincident phosphorus line, provided the instrument has been calibrated for this feature.

3.4.2 The antimony $Sb_{TO}(BE)$ line falls between the boron and phosphorus transverse optical (TO) features. Since the line widths are broad relative to the line positions of these features, the presence of antimony affects the apparent intensities of boron and phosphorus TO features. The no-phonon, (NP) features for these elements could be used providing the instrument is calibrated for them.

3.4.3 In general, the broad TO phonon region features can interfere, particularly when a sample contains widely differing levels of impurities. For example, when boron greatly exceeds phosphorus, the $P_{NP}(BE)$ line must be used, and when phosphorus greatly exceeds boron, one of the approaches described in Section 3.4.1 must be used.

3.5 Stress in samples can cause NP region feature splitting and thus affect the established TO/NP line ratio for a given impurity. The concentrations calculated from such spectra would be artificially low. Peak area calculation methods reduce the measurement's sensitivity to line splitting effects.

3.6 The calibration curves presented in these test methods are traceable to resistivity measurements (see SEMI MF723), neutron transmutation doped, (NTD) reference material, and low-temperature infrared absorption spectroscopy (FT-IR) measurements (as a secondary standard) (see SEMI MF1630). Hence inaccuracies in the work that produced the calibration curves impact the accuracy of the PL results.

4 Referenced Standards

4.1 *SEMI Standards*

SEMI C28 — Specifications and Guidelines for Hydrofluoric Acid

SEMI C30 — Specifications and Guidelines for Hydrogen Peroxide

SEMI C35 — Specifications and Guideline for Nitric Acid

SEMI MF723 — Practice for Conversion Between Resistivity and Dopant Density for Boron-Doped, Phosphorus-Doped, and Arsenic-Doped Silicon

SEMI MF1630 — Test Method for Low Temperature FT-IR Analysis of Single Crystal Silicon for III-V Impurities

SEMI MF1723 — Practice for Evaluation of Polycrystalline Silicon Rods by Float-Zone Crystal Growth and Spectroscopy

---

4 Nakayama, H., Nishino, T., and Hamakawa, Y.,"Analysis of the Excitation Luminescence of Silicon for Characterization of the Content of Impurities," *Jap. J. Appl. Phys.* 19(3), 501–511, (1980).

5 Drozdov, N. A., Patrin, A. A., and Tkachev, V. D., "Recombination Radiation on Dislocations in Silicon," *Soviet Physics, J.Experimental and Theoretical Physics* 23(11), 597 1976).

6 Suezawa, M., Sasaki, Y., Nishina, Y., and Sumino, K., "Radiative Recombination on Dislocations in Silicon Crystals," *Jap. J. Appl. Phys.* 20(7), L537 (1981).

4.2 *ASTM Standard*

D 5127 — Guide for Ultra Pure Water Used in the Electronics and Semiconductor Industry [7]

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

5 Terminology

5.1 *Definitions*

5.1.1 *defect luminescence lines* — those features arising from defect structures in the silicon.

5.1.2 *electron hole droplet (EHD)* — the condensed phase (liquid) of the excitonic gas generated by photoexcitation.

5.1.2.1 *Discussion* — The exciton population is dependent upon excitation intensity and can be raised to the point where the exciton density is sufficient to allow condensation of the exciton gas into a liquid like exciton state.[8] The existence of EHD luminescence can be an indirect measurement of excitation power density on the sample.

5.1.2.2 EHD onset occurs at the point in the sample excitation intensity curve where the electron-hole-droplet begins to form.[9] The EHD luminescence includes a very broad feature underlying the TO region impurity lines which, with increasing excitation intensity, both intensifies and shifts to lower energies relative to the other silicon luminescence features.

5.1.3 *excitons* — the electron-hole pairs that give rise to the luminescence of interest upon recombination at either a free lattice site (free exciton) or an impurity atom site (bound exciton).

5.1.4 *extrinsic line* (X_{TO}(BE) or X_{NP}(BE)) — the luminescence that arises from an exciton captured by an impurity site in the crystal lattice (a bound exciton).

5.1.4.1 *Discussion* — Its energy is lower than the intrinsic emission by an amount related to the exciton binding energy of the impurity at 4.2 K. "X" is the impurity element symbol and "BE" indicates bound exciton luminescence line. Extrinsic luminescence also includes features attributed to bound multi-exciton complexes (b_1, b_2, or b_3 would indicate the first, second and third bound multi-exciton complex lines, respectively). In donor luminescence, these complexes

give rise to two series of lines in the TO region, called the alpha and beta series. The weaker beta series features are denoted by an apostrophe after the line notation (that is, $P_{TO}(b'_1)$). See Table 1 and Table 2 for line locations.

5.1.5 *intrinsic line* (I_{TO}(FE)) — the luminescence that arises from the silicon itself, with no impurity species affecting the exciton recombination.[10,11]

Table 1 Photoluminescence Line Locations (Vacuum Wavenumbers)

Silicon Free Exciton (FE) Lines:			
I_{TO} (FE) at 8848 cm^{-1}			
I_{LO} (FE) at 8860 cm^{-1}			
I_{TA} (FE) at 9166 cm^{-1}			
Major Shallow Impurity Bound Exciton (BE) Lines:			
Element	*TO region, cm^{-1}*	*NP region, cm^{-1}*	*NP/TO region BE line intensity ratio*[A]
Boron	8812.6	9281.3	0.017
(Antimony)	(8810.5)	(9280.0)	(0.010)
Phosphorus	8806.8	9275.4	1.4
Aluminum	8803.4	9271.8	0.7
Arsenic	8801.0	9269.4	2.0

NOTE 1: To convert vacuum wavenumbers to air wavenumbers:

air wavenumber = 1.00030025 × vacuum wavenumber

NOTE 2: To convert wavenumbers to electronvolts:

eV = 1.23985 × 10^{-4} × wavenumber

[A] Instrument resolution = 0.5 cm^{-1}, sample = FZ silicon.

Table 2 Photoluminescence Line Locations (Vacuum Wavenumbers) Detailed Listing for Boron and Phosphorus

Boron Features, cm^{-1}	*Phosphorus Features, cm^{-1}*
B_{NP}(BE) = 9281.3	P_{NP}(BE) = 9275.4
$B_{NP}(b_1)$ = 9263.6	$P_{NP}(b_1)$ = 9246.4
$B_{NP}(b_2)$ = 9245.9	$P_{NP}(b_2)$ = 9223.9
	$P_{NP}(b_3)$ = 9208.4
	$P_{NP}(b_4)$ = 9197.8
B_{TA}(BE) = 9130.1	P_{TA}(BE) = 9124.4
$B_{TA}(b_1)$ = 9112.4	P_{NP}(BE-2e) = 8992.8[A]
$B_{TA}(b_2)$ = 9095.2	
B_{TO}(BE)= 8812.6	$P_{TO}(b_1')$ = 8812.7[B]
$B_{TO}(b_1)$ = 8795.0	P_{TO} (BE) = 8806.8[C]

7 *Annual Book of ASTM Standards*, Vol 11.01, ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428. Telephone: 610-832-9500, Fax: 610-832-9555, Website: www.astm.org.

8 Nakashima, H., and Shiraki, Y., "Role of Shallow Impurities and Lattice Defects in Nucleation of Electron-Hole Droplets in Si," *Solid State Comm.* 40, 195–197 (1981).

9 Hammond, R. B., and Silver, R. N., "Onsets of the Electron-Hole Droplet Luminescence in Si," *Phys. Rev. Lett.* 42(8), 523–526 (1979).

10 Tajima, M., "Determination of Boron and Phosphorus Concentration in Silicon by Photoluminescence Analysis," *Appl. Phys. Lett.* 32(11), 719 (1978).

11 Tajima, M., "Quantitative Impurity Analysis in Si by the Photoluminescence Technique," edited by J. Nishizawa, *Japan Annual Reviews in Electronics, Computers, and Telecommunications—Semiconductor Technology* (OHM—North Holland, 1982).

Boron Features, cm^{-1}	Phosphorus Features, cm^{-1}
$B_{TO}(b_2) = 8777.5$	$P_{TO}(b_2') = 8790.4^B$
$B_{TO}(b_3) = 8763.2$	$P_{TO}(b_1) = 8778.0^C$
$B_{TO}(b_4) = 8752.1$	$P_{TO}(b_3') = 8771.3^B$
$B_{TO}(b_5) = 8742.6$	$P_{TO}(b_2) = 8756.0^C$
	$P_{TO}(b_4') = 8756.2^B$
	$P_{TO}(b_5') = 8745.4^B$
	$P_{TO}(b_3) = 8740.2^C$

[A] The two-electron transition is represented by 2e (see Ref (**3**)).

[B] Beta series transition.

[C] Alpha series transition.

5.1.5.1 *Discussion* — "I" indicates intrinsic silicon emission, "TO" indicates the transverse optical phonon associated with the transition, and "FE" refers to the free exciton recombination responsible for the emission (see Table 1 for line locations).

5.1.6 *phonon* — a quantum of lattice vibrational energy, as a photon is a quantum of electromagnetic energy.

5.1.6.1 *Discussion* — The recombination of excitons in silicon requires a momentum conserving mechanism owing to the indirect band gap of the crystal. Phonons provide such a mechanism. The principal phonon types of interest in silicon luminescence are the transverse acoustic (TA), transverse optical (TO), and longitudinal optical (LO) phonons. These test methods address the use of features associated with the TO phonon as well as those not including phonon emission in their momentum-conserving processes. These latter features are designated "NP" or no-phonon features.

6 Summary of Test Method

6.1 A sample of monocrystalline silicon is cooled to 4.2 K and photoexcited with greater-than-bandgap energy light at one of two intensities listed, depending upon the type of instrument used. The resulting luminescence is collected and recorded. Spectral features corresponding to intrinsic silicon and extrinsic impurity emissions are measured and related to calibration curves to yield dopant density.

7 Apparatus

7.1 *Cryostat* — To maintain sample temperature at 4.2 K. Either open-cycle liquid helium immersion or exchange gas cryostats, or closed-cycle refrigeration systems may be used. The bath immersion type cryostat is recommended for higher confidence in the temperature stability of the sample. In both the exchange gas and closed-cycle systems, careful attention must be paid to thermal sinking and accurate sample temperature measurement (see Section 3.3).

7.2 *Sample Holder* — Which does not cause excessive strain on the ample through spring forces or other means, so as to avoid line splitting associated with crystal stresses.

7.3 *Laser Excitation Source* — Capable of generating electron hole pairs in the silicon crystal. An argon-ion laser operated at 514.5 nm is used. To obtain accurate measurements, laser light intensity must be controllable and stable.

7.4 *Infrared Spectrophotometer* — Equipped with a detector and optics suitable for use between 8750 and 9300 cm^{-1}, and capable of at least 0.5 cm^{-1} resolution at 9300 cm^{-1}.

8 Reagents

8.1 *Purity of Water* — Reference to water shall be understood to mean Type E-1 or better water as described in ASTM Guide D 5127.

8.2 *Nitric Acid* (HNO_3), 65%, in accordance with Grade 2 of SEMI C35.

8.3 *Hydrofluoric Acid* (HF), 48%, in accordance with Grade 2 of SEMI C28.

8.4 *Hydrogen Peroxide* (H_2O_2), 30%, in accordance with Grade 2 of SEMI C30.

8.5 *Acid Cleaning Mixture*, (1:1:1:25) HNO_3:HF:H_2O_2:H_2O.

8.6 *Hazards* — Chemicals used in this procedure are potentially harmful and must be handled with the utmost care at all times.

9 Sample Preparation

9.1 Perform either the procedure in Section 9.1.1 or the procedure in Section 9.1.2 to remove all work damage and surface contamination on all samples except as-received chemical-mechanically polished wafers, which do not need further preparation.

9.1.1 Etch sample using a suitable etchant (for example, the acid cleaning mixture of Section 8.5 or CP-5 hydrofluoric-nitric-acetic acid etchant).

9.1.2 Polish the surface of the sample with a suitable chemical-mechanical polishing compound.

9.2 Luminescence efficiency has been observed to decrease after etching, thus introduction of the sample into the cryostat within a few hours of etching is recommended. Chemical-mechanically polished samples appear to be more stable.[2]

10 Instrument Calibration

10.1 Measurement of silicon standards need only be performed once unless instrument characteristics have drifted or hardware changes have been made. The regular use of secondary standard samples, herein referred to as audit samples, provides a mechanism for instrument calibration verification and generates long-term instrument performance statistics. At least four standard samples should be used for each impurity calibration curve adjustment.

10.1.1 Obtain samples with known impurity densities and collect luminescence spectra at the appropriate excitation intensity listed in Section 11.1 using both standard and high resolution as listed in Section 11.2.2.

10.1.2 Find the peak intensities as described in Section 12 and find the ratios of appropriate spectral lines as described in Section 13.1.

10.1.3 Adjust calibration data of Figure 1 or Figure 2, corresponding to Test Method A or Test Method B, respectively, by translating the curves such that they intersect points corresponding to PL data of the known samples. Make no change to the slope of the curves because the slopes are a function of the exciton recombination dynamics only and are independent of a given instrument's optical response.

10.1.4 These curves become the given instrument's calibration curves for the analysis conditions used.

10.2 Determine the short-term one-sigma precision of the instrument.

NOTE 3: This procedure needs to be performed only occasionally unless instrument characteristics have drifted or hardware changes have been made.

10.2.1 Analyze a sample with impurity concentrations similar to typical samples analyzed by the instrument in normal operation nine times.

10.2.2 Find the peak intensity as described in Section 12 to obtain dopant densities.

10.2.3 Calculate the open-sigma standard deviation for all impurities present.

11 Procedure

11.1 *Excitation Conditions*

11.1.1 *Test Method A—High Excitation Intensity Conditions —* Set the output of the argon ion laser to 300 mW of 514.5 nm light. Pass the beam through an infrared cutoff filter and a chopper with a 50% duty cycle. Beam can be reflected with 2 to 3 mirrors with a nominal 1-m distance between sample and laser. Pass the unfocused beam through the cryostat windows. Set the beam diameter to 2.5 mm.
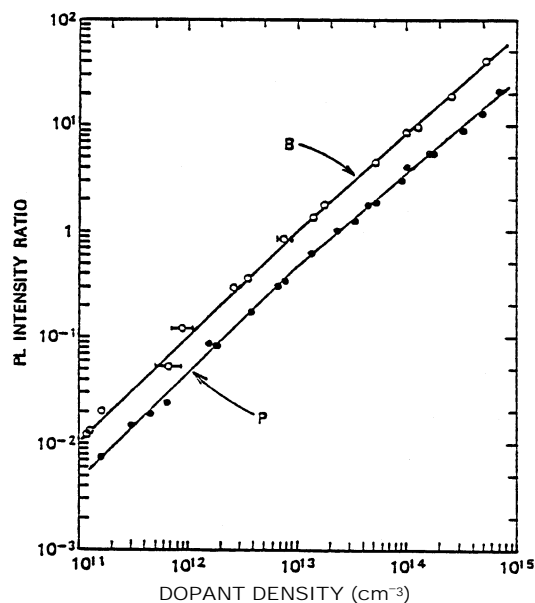
NOTE: The PL intensity ratio of $P_{TO}(BE)/I_{TO}(FE)$ and $B_{TO}(BE)/I_{TO}(FE)$ are plotted against the P and B dopant densities, respectively.[11]

Figure 1
High Excitation Condition Impurity Calibration Curves for Boron and Phosphorus (Test Method A)
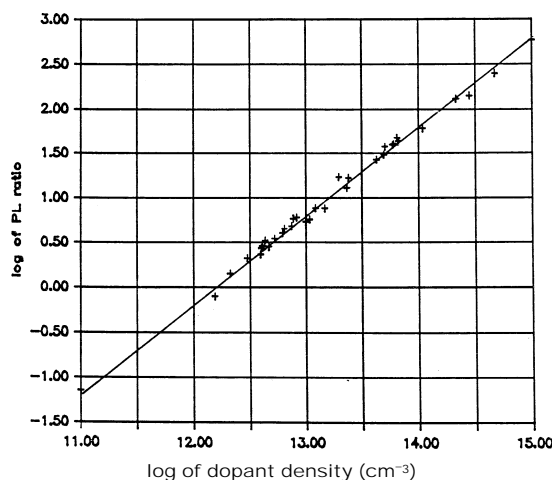
NOTE: Slope = 1.0.
Figure 2
Low Excitation Condition Generic Impurity Calibration Curve (Test Method B)

11.1.2 *Test Method B—Low Excitation Intensity Conditions —* Set laser intensity such that the luminescence is as close to the EHD onset point as practical from signal-to-noise considerations. Collect luminescence spectra of the sample used in Section 10.2 at several different laser beam intensities, extending from well above to as far below the EHD

onset point as permissible from signal-to-noise considerations. Plot the EHD luminescence intensity at 8720 cm^{-1} versus the laser intensity used on linear axes. The EHD onset power can be determined by extrapolating the nonzero EHD points down to zero intensity using a straight line.[9]

11.2 *Spectrometer Parameters*

11.2.1 *Spectral Ranges Required (*minimum*)*

TO region—8757–8889 cm^{-1} (1142–1125 nm)

NP region—9242–9294 cm^{-1} (1082–1076 nm)

11.2.2 *Resolution*

Standard resolution = 2 cm^{-1} (or 0.2 nm)

High resolution = 0.5 cm^{-1} (or 0.05 nm)

Use standard resolution when calculation of aluminum or arsenic concentrations is not desired or not necessary. Otherwise, use high resolution.

11.3 *Audit Specimen Analysis* — Keep one set of specimens as secondary standards to periodically check instrument performance. Make sure that at least one of these samples reflects typical material properties for the laboratory concerned. Analyze at least one of these samples with each batch of unknown samples to confirm reproducibility of analysis conditions from run to run.

11.3.1 Collect luminescence spectra of audit specimens before unknown samples, preferably in the same sample holder batch using parameters listed in Sections 11.1 and 11.2.2. Reduce spectral data as described in Section 12.

11.3.2 If the audit specimen results fall outside the acceptable control limits established for an instrument, the laser power may be adjusted to compensate for drift in the optical transfer efficiency of the sample excitation subsystem. If the source of drift cannot be ascribed to this simple mechanism, thoroughly investigate and correct the problem. Instrument recalibration may be required.
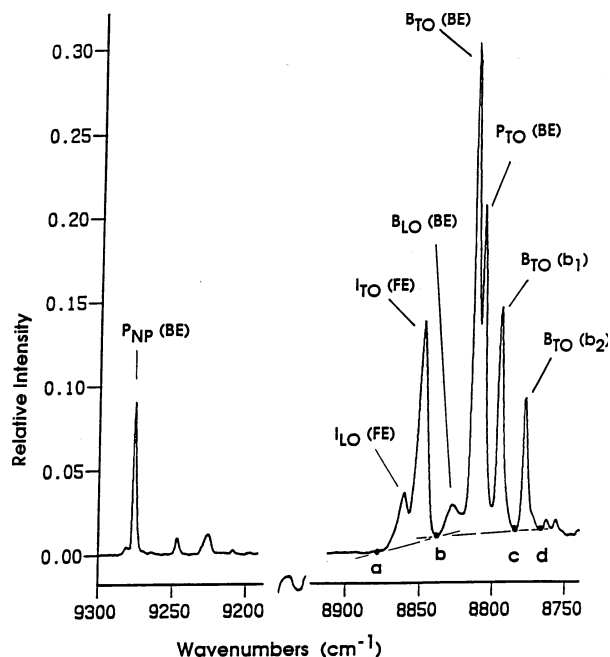

Figure 3
Baseline Drawing Example

11.4 Collect luminescence spectra of unknown samples with spectrometer parameters as listed in Section 11.2. Find the peak intensity as described in Section 12.
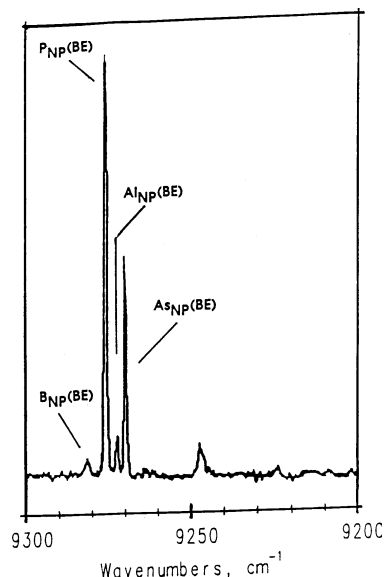

Figure 4
High-Resolution (0.5 cm^{-1}) Spectrum of NP (No-phonon) Region

12 Peak Intensity Determination

12.1 Use the baseline drawing method depicted in Figure 3. For the I_{TO}(FE) line, draw the baseline between (*a*), the point at 8880 cm^{-1}, and (*b*), the point

at the minimum near 8840 cm^{-1}. For the TO region impurity lines, draw a line from (*b*) to (*c*) or (*d*): the minimum near 8785 cm^{-1} or 8768 cm^{-1}, whichever is lower.

12.2 Measure peak heights or areas. Use TO lines for B and P. Use NP lines for Al and As. If the $P_{TO}(BE)$ is not usable, also use NP lines for P.[12] See Figure 4 for identification of bands in the NP region.

12.3 Correct for the $P_{TO}(b_1')$ line underlying the $B_{TO}(BE)$ line by subtracting one-tenth of the intensity of the $P_{TO}(BE)$ line from the $B_{TO}(BE)$ line intensity.

13 Calculation

13.1 Calculate the ratio of the extrinsic line intensity to the $I_{TO}(FE)$ line intensity for each impurity present.

13.2 Using the adjusted calibration curves obtained in Section 10.1.3 find the point on the calibration curve corresponding to the ratio (or log of the ratio, as appropriate) obtained in Section 13.1 by graphical or numerical methods.

13.3 Record the corresponding value of dopant density in atoms/cm^3.

14 Report

14.1 Report the following each time the instrument is calibrated using primary or secondary standard samples:

14.1.1 Standard sample ratios obtained in Section 10.1.2, and

14.1.2 Square Root of Mean Square, (RMS) precision data obtained in Section 10.2.3

14.2 Report the following each time a load of samples is run:

14.2.1 Audit specimen dopant density results,

14.2.2 Unknown ample dopant density results,

14.2.3 Sample lot number, number of samples, operator name, and date of analysis, and

14.2.4 Test Method used (A or B).

15 Precision

15.1 A single laboratory investigation of the precision of measurement of phosphorus and boron by photoluminescence was conducted on a Fourier transform instrument. Two samples were analyzed once a day over a period of several weeks (45 measurement values were taken on each sample). The first sample contained a nominal 70 ppta phosphorus and a nominal 30 ppta boron; and the second sample contained a nominal 20 ppta phosphorus and a nominal 15 ppta boron. From this multiple-day, two-operator, two-sample investigation, values of single laboratory standard deviation and relative standard deviation based on the average measured value were obtained and are tabulated in Table 3.

Table 3 Standard Deviation Values for a Single Laboratory Study of Variation of Photoluminescence Measurements of Phosphorus and Boron

Sample	*Dopant*	*1s, ppta*	*R1s, percent*
1	Phosphorus	2.43	3.4
1	Boron	1.69	6.2
2	Phosphorus	1.13	5.6
2	Boron	1.00	7.0

16 Bias

16.1 The bias of this test method cannot be evaluated because there are no available reference standards suitable for evaluating bias.

17 Keywords

17.1 aluminum; arsenic; boron; dopant; impurities; impurity analysis; phosphorus; photoluminescence; silicon.

---

12 Tajima, M., Masui, T., Itoh, D., and Nishino, T.," Calibration of the Photoluminescence Method for Determining As and Al Concentrations in Si," *Journal of Electrochemical Society*, Vol 137, 1990, pp. 3544–3551.

NOTICE: SEMI makes no warranties or representations as to the suitability of the standards set forth herein for any particular application. The determination of the suitability of the standard is solely the responsibility of the user. Users are cautioned to refer to manufacturer's instructions, product labels, product data sheets, and other relevant literature, respecting any materials or equipment mentioned herein. These standards are subject to change without notice.

By publication of this standard, Semiconductor Equipment and Materials International (SEMI) takes no position respecting the validity of any patent rights or copyrights asserted in connection with any items mentioned in this standard. Users of this standard are expressly advised that determination of any such patent rights or copyrights, and the risk of infringement of such rights are entirely their own responsibility.

SEMI MF1390-1104
TEST METHOD FOR MEASURING WARP ON SILICON WAFERS BY AUTOMATED NON-CONTACT SCANNING

1 Purpose

1.1 Warp can significantly affect the yield of semiconductor device processing.

1.2 Knowledge of this characteristic can help the producer and consumer determine if the dimensional characteristics of a specimen wafer satisfy given geometrical requirements.

1.3 Changes in wafer warp during processing can adversely affect subsequent handling and processing steps. These changes can also provide an important process monitoring function.

1.4 This test method is suitable for measuring the warp of wafers used in semiconductor device processing in the as-sliced, lapped, etched, polished, epitaxial or other layer condition and for monitoring thermal and mechanical effects on the warp of wafers during device processing.

2 Scope

2.1 This test method covers a non-contacting, nondestructive procedure to determine the warp of clean, dry semiconductor wafers.

2.2 This test method employs a two-probe system that examines both external surfaces of the wafer simultaneously.

2.3 The test method is applicable to wafers 50 mm or larger in diameter, and approximately 100 μm and larger in thickness, independent of thickness variation and surface finish, and of gravitationally induced wafer distortion.

2.4 This test method is not intended to measure the flatness of either exposed silicon surface. Warp is a measure of the distortion of the median surface of the wafer.

2.5 This test method measures warp of a wafer corrected for mechanical forces applied during the test. Therefore, the procedure described gives the unconstrained value of warp.

NOTE 1: This warp is indicated by the acronym "GMLYMER" in Appendix 2, Shape Decision Tree, of SEMI M1.

NOTE 2: SEMI MF657 measures median surface warp using a three-point back-surface reference plane. The back-surface reference results in thickness variation being included in the recorded warp value. The use (in this test method) of a median surface reference plane eliminates this effect. The use (in this test method) of a least-squares fit reference plane reduces the variability introduced in three-point plane calculations by choice of reference point location. The use (in this test method) of special calibration or compensating techniques minimizes the effects of gravity-induced distortion of the wafer.

2.6 This test method includes several methods for canceling gravity-induced deflection which could otherwise alter the shape of the wafer.[1]

NOTE 3: One of these methods, the Representative Wafer Inversion Method, is covered by a patent held by ADE Corporation, 80 Wilson Way, Westwood, MA 02090-1806.

NOTICE: This standard does not purport to address safety issues, 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 or other limitations prior to use.

3 Limitations

3.1 Any relative motion along the probe measuring axis between the probes and the wafer holding device during scanning will produce error in the measurement data. Vibration of the test specimen relative to the probe-measuring axis will introduce error. Such errors are minimized by system signature analysis and correction algorithms. Internal system monitoring may also be used to correct non-repetitive and repetitive system mechanical translations. Failure to provide such corrections may cause errors.

3.2 If a measured wafer differs substantially in diameter, thickness, fiducials, or crystal orientation from that used for the gravitational compensation procedure, the results may be incorrect. Estimates of the errors in gravity induced deflection for differences

---

1 Poduje, N., "Eliminating Gravitational Effect in Wafer Shape Measurements," *NIST/ASTM/SEMI/SEMATECH Technology Conference, Dallas, TX.* Technology for Advanced Materials/Process Characterization, February 1, 1990.

in diameter and thickness are shown in Related Information 1. If the crystal orientation of the sample to be measured differs from the crystal orientation of the gravity-compensation wafer, then the measured warp value may differ from the actual warp value by up to 15%. Error tables for fiducial variation have not been generated.

3.3 Different methods for implementing gravitational compensation may give different results. Varying levels of completeness of implementing a method may also give different results.

3.4 Mechanical variations in wafer holding devices between systems may introduce measurement differences. This test method allows the use of a variety of wafer holding devices (see Section 7.1.4.1); results obtained with different geometrical configurations of wafer holding device on the same test samples may differ.

3.5 Most equipment systems capable of this measurement have a definite range of wafer thickness combined with warp (dynamic range) that can be accommodated without readjustment. If the sample moves outside this dynamic range during either calibration or measurement, results may be in error. An over-range signal can be used to alert the operator and measurement data examiners to this event.

3.6 The quantity of data points and their spacing may affect the measurement results. This test method does not specify the data point spacing (see Section 7.1.4.2); results obtained with different data point spacings on the same test samples may differ.

4 Referenced Standards

4.1 *SEMI Standards*

SEMI M1 — Specifications for Polished Monocrystalline Silicon Wafers

SEMI MF657 — Test Method for Measuring Warp and Total Thickness Variation on Silicon Wafers by Noncontact Scanning

SEMI MF1241 — Terminology of Silicon Technology

SEMI MF1530 — Test Method for Measuring Flatness, Thickness, and Thickness Variation on Silicon Wafers by Automated Non-contact Scanning

4.2 *ASTM Standard*

E 691 — Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[2]

---

2 Available from ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428, Tel: 610-832-9500, Fax: 610-832-9555, Website http://www.astm.org. Appears in Volume 14.02 of *Annual Book of ASTM Standards.*

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

5 Terminology

5.1 *Definitions*

5.1.1 *mechanical signature, of an instrument* — that component of a measurement that is introduced by the instrument and that is systematic, repeatable, and quantifiable.

5.1.2 *median surface, of a semiconductor wafer* — the locus of points equidistant from the front and back surfaces of the wafer.

5.1.3 *reference plane, of a semiconductor wafer* — a plane from which deviations of a specified surface of the wafer are measured.

5.1.4 *reference plane deviation (RPD)* — the distance from a point on a reference plane to the corresponding point on a wafer surface.

5.1.4.1 *Discussion* — A dome-shaped wafer is considered to have positive RPD at its center; a bowl-shaped wafer is considered to have negative RPD at its center.

5.1.5 *thickness, of a semiconductor wafer* — the distance through the wafer between corresponding points on the front and back surfaces.

5.1.6 *warp, of a semiconductor wafer* — the algebraic difference between the most positive and the most negative deviations of the median surface of a wafer that is not chucked from a reference plane that is a least squares fit to the median surface.

5.1.6.1 *Discussion* — Although warp may be caused by unequal stresses on the two exposed surfaces of the wafer, it cannot be determined from measurements on a single exposed surface. The median surface may contain regions with upward or downward curvature or both; under some conditions the median surface may be flat. In all cases, warp is a zero or positive quantity. Stylized examples of visualization of warp are shown in SEMI MF657.

5.2 Definitions of other terms related to silicon material technology can be found in SEMI MF1241.

6 Summary of Test Method

6.1 A calibration procedure is performed to set the instrument's scale factor and other constants. If the representative wafer inversion method is used for gravity correction, the calibration procedure also determines the mechanical signature of the instrument and the effect of gravity on the wafer.

6.2 The wafer is supported by a small-area chuck with front surface up.

6.3 Both external surfaces are simultaneously scanned along a prescribed pattern by an opposed pair of probes to obtain a set of values of the distances between each surface and the nearest probe. In each case, both members of the pair of distances is taken at the same value of the x and y coordinates.

6.4 The paired displacement values are used to construct the median surface.

6.5 A correction for gravity effects on the median surface is made either by subtracting a gravity correction obtained (1) from measurements on a representative wafer or (2) from theoretical considerations or by repeating the scan with the wafer inverted.

6.6 A least-squares reference plane is constructed from the corrected median surface.

6.7 The reference plane deviation (RPD) is calculated at each measured pair of points.

6.8 Warp is reported as the algebraic difference between the most positive RPD and the most negative RPD.

7 Apparatus

7.1 *Measuring Equipment*, consisting of wafer holding device, multiple-axis transport mechanism, probe assembly with indicator, and system controller/computer, including data processor and suitable software.

7.1.1 The equipment shall be direct reading with all necessary calculations performed internally and automatically as outlined in Section 11.2.

7.1.2 The equipment shall be equipped with an over-range signal.

7.1.3 Instrument data reporting resolution shall be 100 nm or smaller.

7.1.4 The measuring equipment contains the following subsystems:

7.1.4.1 Wafer-holding device, for example a chuck whose face is perpendicular to the measurement axis, and on which the wafer is placed for the measurement scan. The nature and size of the wafer holding device shall be agreed upon between the parties to the test.

7.1.4.2 Multiple-axis transport mechanism, which provides a means for moving the wafer-holding device, or the probe assembly, perpendicularly to the measurement axis in a controlled fashion in several directions. This motion must permit data gathering

over a prescribed scan pattern covering the entire fixed quality area. Data point spacing to be used shall be agreed upon between the parties to the test.
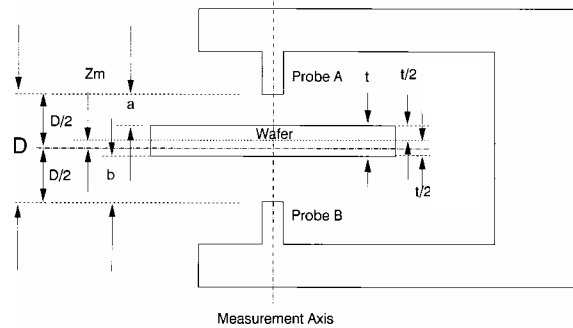

Figure 1
Schematic View of Wafer, Probes, and Fixture

7.1.4.3 Probe assembly with paired non-contacting displacement-sensing probes, probe supports, and indicator unit (see Figure 1).

7.1.4.3.1 The probes shall be capable of independent measurement of the distances a and b between the probed site on each surface of the sample wafer and the nearest probe surface.

7.1.4.3.2 The probes shall be mounted above and below the wafer in a manner so that the probed site on one surface of the wafer is opposite the probed site on the other.

7.1.4.3.3 The common axis of these probes is the measurement axis.

7.1.4.3.4 The probe separation D shall be kept constant during calibration and measurement.

7.1.4.3.5 Displacement resolution shall be 100 nm or smaller.

7.1.4.3.6 The probe sensor size shall be 4 mm by 4 mm, or other value to be agreed upon between the parties to the test.

7.1.4.3.7 Measuring equipment employing either the Representative Wafer Inversion Method or the Sample Wafer Inversion Method for gravity compensation must provide precise positioning in both measurement orientations so that measurements are taken at identical locations for each orientation of the sample.

8 Materials

8.1 *Set-up Masters* — suitable to accomplish calibration and standardization as recommended by the equipment manufacturer.

8.2 *Reference Wafer* — with a reference warp value ≤ 20 μm that is used to determine the level of agreement

between the warp value obtained by the measuring equipment under test and the reference warp value (see Section 9).

8.3 *Representative Wafer* — required only if the Representative Wafer Inversion Method is used for the gravity correction. A representative wafer shall be identical in nominal diameter, nominal thickness, fiducials, composition and crystalline orientation to those being measured. Its warp need not be known.

9 Suitability of Measuring Equipment

9.1 Determine the suitability of the measuring equipment with the use of a reference wafer and its reference warp value in accordance with the procedures of Sub-section 9.2, or by performance of a statistically-based instrument repeatability study to ascertain whether the equipment is operating within the manufacturer's stated specification for repeatability.

9.1.1 The reference warp value is the average of a number of values obtained for that wafer over a number of "passes" (repeat measurements). The reference wafer is measured on the measuring equipment under test and its reference warp value is compared against the measured warp value. The acceptable level of the agreement between the reference and measured warp values is to be agreed upon by the parties to the test.

9.2 *Procedure*

9.2.1 Select a reference wafer of appropriate criteria, together with its associated reference warp value.

9.2.2 Measure the reference wafer on the measuring equipment under test to obtain a sample warp value.

9.2.3 Subtract the two warp values to obtain the difference:

$$\Delta_{warp} = Warp_{reference} - Warp_{sample} \qquad (1)$$

9.2.4 The metric to be used to determine acceptability is difference, Δ_{warp}. Accept the measuring equipment as suitable for use if this difference is less than a value that is agreed upon between the parties to the test.

NOTE 4: If the measuring equipment is to be used to measure other parameters, such as flatness and thickness variation in addition to warp, the reference and sample warp values may be included in the reference and sample data sets specified in SEMI MF1530, but this is not necessary if only warp measurements are to be made.

10 Sampling

10.1 This test method is nondestructive and may be used on either 100% of the wafers in a lot or on a sampling basis.

10.1.1 If samples are to be taken, procedures for selecting the sample from each lot of wafers to be tested shall be agreed upon between the parties to the test, as shall the definition of what constitutes a lot.

11 Calibration and Standardization

11.1 Calibrate the measuring equipment in accordance with the manufacturer's instructions.

11.2 When using the Representative Wafer Inversion Method for correcting the gravity-induced deflection, determine $z_{gravity}$, the deflection due to gravity and machine effects on the representative wafer, in accordance with Section 12 and Section 13 through paragraph 13.7.

12 Procedure

12.1 Prepare the apparatus for measurement of wafers, including selection of diameter, peripheral fiducials, scan area and data display/output functions. Also select the gravitational correction method from one of the following:

- Reference Wafer Inversion Method (see Note 3),

- Sample Wafer Inversion Method, or

- Theoretical Modeling Method.

12.2 Select the fixed quality area (FQA) by specifying the nominal edge exclusion (*EE*).

12.3 Introduce the test specimen into the measurement mechanism with the front surface upward and initiate the measurement sequence to determine and record the distances between each probe and the nearest wafer surface in pairs, *a* and *b*, at each measurement position. Proceed directly to Section 13 unless (1) the Sample Wafer Inversion Method is being used to correct for effects of distortion due to gravity or (2) a representative wafer is being measured to obtain the gravity correction for use in the Representative Wafer Inversion Method (see Note 3).

12.4 Repeat Section 12.2 with the wafer inverted (front surface downward).

13 Calculations

13.1 The following calculations are performed automatically within the instrument. An outline of the calculation structures is provided here to indicate the nature of the procedure.

13.2 Determine the displacements (distances) between each probe and the nearest surface of the wafer (in pairs) at intervals along the scan pattern.

NOTE 5: From Figure 1, note that the distance between Probe *A* and the nearest surface of the wafer is displacement value *a* and the distance between Probe *B* and the nearest surface of the wafer is displacement value *b*.

13.3 Set the origin of the *z*-axis at the midpoint between the two probes, *A* and *B*.

13.4 Find the distance, z_m, of the median surface from the *z*-axis origin at each point. From Figure 1,

$$z_m = \frac{D}{2} - a - \frac{t}{2} \qquad (2)$$

and

$$z_m = -\frac{D}{2} + b + \frac{t}{2} \qquad (3)$$

Therefore,

$$\frac{D}{2} - a - \frac{t}{2} - \frac{D}{2} + b + \frac{t}{2} = 2z_m \qquad (4)$$

and

$$z_m = \frac{b - a}{2} \qquad (5)$$

where

D	=	distance between Probes *A* and *B*,
a	=	distance between Probe *A* and the nearest (top) wafer surface,
b	=	distance between Probe *B* and the nearest (bottom) wafer surface, and
t	=	wafer thickness.

13.5 For measurements in the normal orientation (front surface up), call the position of the median surface z_{nor}.

13.6 For measurements in the inverse orientation (back surface up), call the position of the median surface z_{inv}.

13.7 For measurements on representative wafers or on sample wafers when the Sample Wafer Inversion Method is being used, determine the gravitational correction to the median surface as follows:

$$z_{gravity} = \frac{z_{nor} + z_{inv}}{2} \qquad (6)$$

NOTE 6: This cancels the effect of the representative wafer's shape while retaining the effect of gravity.

13.8 Determine the gravity compensated median surface as follows:

13.8.1 *Representative Wafer Inversion Method* — Subtract $z_{gravity}$ from z_{nor} to produce z_{com} at each measurement point.

NOTE 7: The Representative Wafer Inversion Method deals not only with first-order gravitational effects, but also with other effects that may influence the measured value, such as wafer-periphery effects, some machine-specific signature, etc.

13.8.2 *Sample Wafer Inversion Method* — Subtract $z_{gravity}$ from z_{nor} to produce z_{com} at each measurement point. Note that this is equivalent to taking the difference between the normal and inverted measurement values at each point:

$$z_{com} = z_{nor} - z_{gravity} = z_{nor} - \frac{z_{nor} + z_{inv}}{2} = \frac{z_{nor} - z_{inv}}{2} \qquad (7)$$

13.8.3 *Theoretical Modeling Method* — Apply gravitational correction developed from a theoretical model. Although a rigorous model is not known to exist, approximate corrections have been calculated[3] (see Related Information 1).

13.9 Construct a reference plane that is a least-squares fit to the gravity-compensated median surface data at all the points of the of the scan pattern. The reference plane is of the form:

$$z_{ref} = a_R x + b_R y + c_R \qquad (8)$$

where a_R, b_R, and c_R are constants selected so that

$$\sum_{x,y} [z_{com}(x, y) - (a_R x + b_R y + c_R)]^2 \qquad (9)$$

is minimized over the FQA (see Section 12.2).

13.10 Subtract the *z*-value of the reference plane (z_{ref}) from the compensated *z*-value, z_{com}, at all the points of the scan pattern to yield the reference plane deviation (RPD) at each point:

$$RPD = z_{com} - z_{ref} \qquad (10)$$

13.11 Calculate the warp of the wafer as the difference between the maximum (most positive) and minimum (most negative) RPD:

$$\text{warp} = RPD_{max} - RPD_{min} \qquad (11)$$

13.12 Record the calculated warp value.

13.13 For referee or other measurements where the wafer is measured more than once, calculate the maximum, minimum, sample standard deviation, average, and range of all measurements on the sample.

---

3 Application Note: "Gravitational Sag in Silicon Wafers," ADE Corporation, 80 Wilson Way, Westwood, MA 02090-1806, Nov. 20, 1991.

13.13.1 Record sample standard deviation and other statistical parameters as agreed upon between the parties to the test.

14 Report

14.1 Report the following information:

14.1.1 Date, time, and temperature of test,

14.1.2 Identification of operator,

14.1.3 Location (laboratory) of test,

14.1.4 Identification of measuring instruments, including wafer-holding device diameter, data point spacing, sensor size, and gravitation-correction method,

14.1.5 Lot identification, including nominal diameter, nominal center-point thickness, and nominal edge exclusion (*EE*) specified.

14.1.6 Description of sampling plan, if any, and

14.1.7 Warp of each wafer measured.

14.2 For referee tests also include in the report the standard deviation of each set of wafer measurements and such other statistical parameters as have been agreed to by the parties to the test.

15 Precision

15.1 Twenty-three 200 mm diameter, single-side polished silicon wafers were employed in a round-robin experiment. These wafers represented three different manufacturing processes. All three subsets were bare on the front surface. Two subsets were bare on the back surface and one subset had oxide on the back surface.

15.2 Eight laboratories measured warp. Warp value ranges for these three subsets are shown in Table 1. Each of the twenty-three wafers was measured three times in succession (in three cassette-to-cassette "passes"), on a single day on automatic measurement systems in accordance with this test method.

15.3 All measurement data were acquired with 3 mm nominal edge exclusion.

15.4 The number of laboratories, samples, and determinations in this study met the minimum requirements for determining precision prescribed in ASTM Practice E 691.

15.5 The ranges of 95% confidence interval, within which two measurements are considered statistically to be the same, for within-laboratory repeatability (*r*) and between-laboratory reproducibility (*R*) are shown in Table 1.

15.6 Figure 2 contains plots of the 95% confidence intervals for repeatability (*r*) and reproducibility (*R*) against mean value of warp.

15.7 For more details, refer to the Research Report.[4]

Table 1 Summary Warp Measurement Statistics

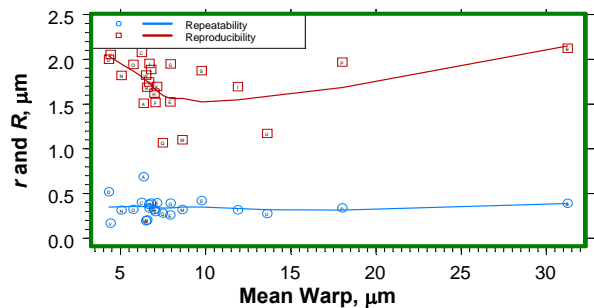| Parameter		Warp
Mean, μm	Smallest	4.30
	Largest	31.25
r, μm	Smallest	0.184
	Largest	0.695
R, μm	Smallest	1.112
	Largest	2.097

Figure 2
Warp Repeatability & Reproducibility

16 Bias

16.1 *Bias* — No standards exist against which the bias of this test method can be evaluated.

17 Keywords

non-contact measurement; semiconductor; shape; silicon; wafers; warp

---

4   Available on request from SEMI Headquarters, Publications Department, San Jose, CA, Fax: 408-943-7015. Request International Standards Research Report F01-1016, ASTM Interlaboratory Round Robin Experiment on Measuring Warp on Silicon Wafers by Automated Noncontact Scanning and Measuring Flatness, Thickness and Thickness Variation of Silicon Wafers by Automated Noncontact Scanning.

RELATED INFORMATION 1
MEASUREMENT ERRORS DUE TO DIFFERENCES IN DIAMETER AND THICKNESS BETWEEN A REPRESENTATIVE WAFER AND A WAFER UNDER TEST

NOTICE: This related information is not an official part of SEMI MF1390.   This related information was approved for publication by full letter ballot on April 22, 2004.

R1-1 The sag, or deflection induced by gravity at the edge of a wafer supported at its center, in μm, has been estimated[3] as:

$$S = \frac{(3\times10^8)kgdD^4}{32Et^2} = \frac{KD^4}{t^2} \qquad \text{(R1-1)}$$

where:

S	=	deflection , in μm,
k	=	geometrical constant (=0.5854),
g	=	gravitational constant (980 cm/s^2),
d	=	density of silicon (2.329 g/cm^3),
E	=	Young's modulus (~1.6×10^{12} dyne/cm^2,
D	=	nominal wafer diameter, in mm, and
t	=	nominal wafer thickness, in μm.

K, the constant of proportionality is therefore equal to 7.83×10^{-3} μm^3/mm^4. Table R1-1 gives estimated values of sag in micrometers for 100 mm through 300 mm diameter wafers with thickness and diameter as specified in SEMI M1.

Table R1-1. Estimated Sag, in μm, of Wafers of Nominal Diameter and Thickness

Diameter, mm	*Thickness, μm*	*SEMI M1 Reference*	*Estimated Sag, μm*
300	775	SEMI M1.15	105.6
200	725	SEMI M1.9	23.8
150	675	SEMI M1.8	8.7
150	625	SEMI M1.13	10.1
125	625	SEMI M1.7	4.9
100	525	SEMI M1.5	2.8

R1-2 For small variations about the nominal values of diameter and thickness, the relative change of the gravity effect is 4 times the relative change of the diameter and −2 times the relative change of thickness:

$$\frac{\partial S}{\partial d} = 4\frac{Kd^3}{t^2} \qquad \text{(R1-2)}$$

and

$$\frac{\partial S}{\partial t} = -2\frac{Kd^4}{t^3} \qquad \text{(R1-3)}$$

Therefore the relative changes are as follows:

$$\frac{\partial S}{S} = 4\frac{\partial d}{d} \qquad \text{(R1-4)}$$

and

$$\frac{\partial S}{S} = -2\frac{\partial t}{t} \qquad \text{(R1-5)}$$

R1-3 Table R1-2 gives examples of worst-case gravity effect errors (in micrometers), for 100 mm through 300 mm diameter wafers with thickness and diameter tolerances as specified in SEMI M1.

Table R1-2 Examples of Gravity Effect Errors

300 mm Diameter Wafers			
	Actual Diameter, mm		
	299.8	*300.0*	*300.2*
	Gravity Effect Errors, μm		
Actual Thickness, μm 755	3.90	5.67	5.96
775	−1.68	0.00	0.28
795	−6.84	−5.25	−4.98

200 mm Diameter Wafers			
	Actual Diameter, mm		
	199.8	*300.0*	*200.2*
	Gravity Effect Errors, μm		
Actual Thickness, μm 705	0.77	1.37	1.47
725	−0.57	0.00	0.10
745	−0.52	−1.26	−1.17

150 mm Diameter Wafers			
	Actual Diameter, mm		
	149.8	*150.0*	*150.2*
	Gravity Effect Errors, μm		
Actual Thickness, μm 655	0.49	0.54	0.59
675	−0.05	0.00	0.05
695	−0.54	−0.49	−0.45

150 mm Diameter Wafers			
	Actual Diameter, mm		
	149.8	*150.0*	*150.2*
	Gravity Effect Errors, μm		
Actual Thickness, μm 610	−0.45	0.51	0.56
625	−0.05	0.00	0.05
640	−0.52	−0.47	−0.42

125 mm Diameter Wafers			
	Actual Diameter, mm		
	124.5	*125.0*	*125.5*
	Gravity Effect Errors, μm		
Actual Thickness, μm 605	0.25	0.33	0.41
625	−0.08	0.00	0.08
645	−0.37	−0.30	−0.22

100 mm Diameter Wafers			
	Actual Diameter, mm		
	99.5	*100.0*	*100.5*
	Gravity Effect Errors, μm		
Actual Thickness, μm 505	0.17	0.23	0.29
525	−0.06	0.00	0.06
545	−0.26	−0.20	−0.15

SEMI MF1391-0704
TEST METHOD FOR SUBSTITUTIONAL ATOMIC CARBON CONTENT OF SILICON BY INFRARED ABSORPTION

1 Purpose

1.1 Carbon may have an important role in defect formation processes. Some laboratories have attributed carbon as being involved in the formation of swirl. Carbon has also been shown to serve as a nucleation center for the precipitation of oxygen.

1.2 Although electrically inactive, substitutional carbon causes stress that can be observed by X-ray topography.

1.3 Direct effects on the reverse bias characteristics of power devices and annealing problems in neutron transmutation doped silicon have been associated with carbon.

1.4 This test method has applicability in production control, materials research, quality assurance, and materials acceptance.

2 Scope

2.1 This referee test method[1] covers the determination of substitutional carbon concentration in single crystal silicon. Because carbon may also reside in interstitial lattice positions when in concentrations near the solid solubility limit, the results of this test method may not be a measure of the total carbon concentration at such concentrations.

2.2 The useful range of carbon concentration measurable by this test method is from the maximum amount of substitutional carbon soluble in silicon down to about 0.1 parts per million atomic (ppma), that is, 5×10^{15} cm^{-3} for measurements at room temperature,

and down to about 0.01 ppma, that is, 0.5×10^{15} cm^{-3} at cryogenic temperatures (below 80 K).

2.3 This test method utilizes the relationship between carbon concentration and the absorption coefficient of the infrared absorption band associated with substitutional carbon in silicon. At room temperatures (about 300 K), the absorption band peak is at 605 cm^{-1} or 16.53 μm. At cryogenic temperatures (below 80 K), the absorption band peak is at 607.5 cm^{-1} or 16.46 μm.

2.4 This test method is applicable to slices of silicon with resistivity higher than 3 Ω·cm for p-type and higher than 1 Ω·cm for n-type. Slices can be any crystallographic orientation and should be polished on both surfaces.

2.5 This test method is intended to be used with infrared spectrophotometers that are equipped to operate in the region from 2000 to 500 cm^{-1} (5 to 20 μm).

2.6 This test method provides procedure and calculation sections for the cases where thickness values of test and reference specimens are both closely and not closely matched.

NOTICE: This standard does not purport to address safety issues, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health guides and determine the applicability of regulatory or other limitations prior to use.

3 Limitations

3.1 Stray light that reaches the detector tends to reduce the calculated absorbance value and thereby reduces the reported carbon concentration.

3.2 The carbon level of the reference slice should be less than 2×10^{15} atoms/cm^3 (0.04 ppma) to minimize the comparative error at room temperature. The detection limit at cryogenic temperatures (below 80 K) is about 0.01 ppma. Obtaining reference samples much below 0.01 ppma in carbon content may prove to be difficult. Therefore the measurement of very low carbon content silicon near the 0.01 ppma detection limit is necessarily a comparative measurement only.

---

1 This test method was developed in cooperation with the Silicon Technologies Committee of the Japan Electronics and Information Technology Industries Association (JEITA). It is essentially equivalent to JEITA EM-9503, Standard Test Method for Substitutional Atomic Carbon Content of Silicon by Infrared Absorption, which is available from JEITA, 3rd floor, Mitsui Sumitomo Kaijo Bldg. Annex, 11, Kanda-Surugadai 3-chome, Chiyoda-ku, Tokyo 101-0062, Japan, Web site: www.jeita.or.jp. DIN 50438/2, Testing of Inorganic Semiconductor Materials: Determination of the Impurity Content in Silicon by Means of Infrared Absorption; Carbon, is also a method for measuring the substitutional carbon content of silicon. It differs in some aspects, including different conversion coefficients, from this test method. It is available from Beuth Verlag GmbH, Burggrafenstrasse 4-10, D-1000 Berlin 30, Germany.

3.3 Spectrophotometer technique is critical to a successful carbon determination. The manufacturer's instrument instruction manuals should be studied to familiarize the operator with the proper use of the spectrometer. Since the transmittance at the carbon peak can be very low, while the transmittance at the baseline regions is about 40%, extremely good photometric linearity is critical. Wavenumber precision is also critical because the carbon peak lies on the shoulder of a very intense lattice absorption band.

3.4 The FWHM of the carbon absorption band at room temperature must be less than 6 cm^{-1} for acceptable measurements. At cryogenic temperatures (below 80 K), it must be less than 3 cm^{-1} for acceptable measurements. Excessive width may be due to improper thickness matching, to stress, or to the use of a low resolution setting of the instrument. In dispersive instruments, excessive widths can also result from incorrect instrument balance setting or too fast a scan speed. In Fourier transform instruments, excessive widths can result from the use of a source-defining aperture that is too wide.

3.5 Specimens that do not exceed the instrument beam size cause error. Use of apertures at the sample, or preferably beam condensers to reduce the beam size at the sample, can correct this problem.

3.6 The main two-phonon lattice band of silicon, at about 610 cm^{-1} (16 μm), is very intense; the absorption coefficient for this band is about 9 cm^{-1} at room temperature, and about 5 cm^{-1} at 78 K.[2] This broad band peak is close to the wavelength of the carbon-in-silicon band and so presents a problem in measuring the intensity of the carbon band.

3.7 Reference and test slices must be as close as practically possible to the same temperature to avoid the effects of temperature on the intensity of the lattice band.

3.8 The minimum detection level of this test method is limited by the signal-to-noise ratio of the spectrum. Thus attaining the highest possible sensitivity by this test method requires long measurement times and stable spectrophotometers.

3.9 Free carrier absorption in silicon specimens with resistivities less than 3 Ω·cm for *p*-type, or 1 Ω·cm for *n*-type, reduces the available energy below the level required for satisfactory operation of most spectrophotometers.

3.10 For samples at cryogenic temperatures (below 80 K), plane parallel, polished surfaces may cause interference fringes on the spectrum. Increasing the sample thickness or inducing a wedge (non-flatness) in the sample reduces these interference fringes.

4 Referenced Standards

4.1 *SEMI Standard*

SEMI MF1241 — Terminology of Silicon Technology

4.2 *ASTM Standard*

E 131 — Terminology Relating to Molecular Spectroscopy[3]

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

5 Terminology

5.1 General definitions for terms related to infrared absorption spectroscopy are found in ASTM Terminology E 131.

5.2 Definitions for terms related to silicon materials technology are found in SEMI MF1241.

5.3 *Definitions*

5.3.1 *background spectrum*, *n* — in FT-IR instruments, the single-beam spectrum obtained without a specimen in the infrared light path that is usually obtained with only nitrogen, dry air, or a vacuum in the beam.

5.3.2 *baseline*, *n* — a straight line interpolation between points on either side of the carbon peak of the absorbance spectrum, drawn to represent the spectrum that would have been obtained in the absence of the impurity.

5.3.3 *baseline absorbance*, *n* — the value of the baseline at the wavenumber corresponding to the carbon peak that is used for evaluating the absorbance peak height.

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

5.3.4.1 *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. FT-IR instruments are always used in conjunction with a computer to control the interferometer, collect and manipulate the data, and for spectral output.

---

2 Johnson, F. A., *Proc. Physics Society* 73, 265–272 (1959).

3 *Annual Book of ASTM Standards*, Vol 03.06, ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428. Telephone: 610-832-9500, Fax: 610-832-9555, Website: www.astm.org