

Background Statement for SEMI Draft Document 5737A REVISION OF SEMI MF1391-1107 (Reapproved 0912) TEST METHOD FOR SUBSTITUTIONAL ATOMIC CARBON CONTENT OF SILICON BY INFRARED ABSORPTION

Notice: This background statement is not part of the balloted item. It is provided solely to assist the recipient in reaching an informed decision based on the rationale of the activity that preceded the creation of this document.

Notice: Recipients of this document are invited to submit, with their comments, notification of any relevant patented technology or copyrighted items of which they are aware and to provide supporting documentation. In this context, “patented technology” is defined as technology for which a patent has issued or has been applied for. In the latter case, only publicly available information on the contents of the patent application is to be provided.

Background

This document is a revision to the existing SEMI MF1391-1107 Test Method for Substitutional Atomic Carbon Content of Silicon by Infrared Absorption. The scope of this revision is to add a new short range for determining a better baseline for silicon with low carbon content. This revision is referred to as “Short-Baseline Method” in JEITA EM3503 and has been utilized since 1997.

The current Standard employs a range from 640 to 560 cm^{-1} . This long range has been suitable for silicon with high carbon content that produces a large absorption peak height and broad peak width. Recent silicon products contain carbon as low as $1 \times 10^{15} \text{ cm}^{-3}$ so that the absorption peak has become smaller and narrower. Thus, it has become harder to determine an accurate baseline with the current baseline range, since the background deviation within such a long range is comparable to the small absorption peak height.

Doc.5737 was submitted for Cycle 6, 2015 and was adjudicated by the EU Silicon Wafer Committee at their meeting on October 7th at MESSE DRESDEN. The ballot failed as a reject vote was submitted. The reject vote pointed out that the limitation description was not adequate for SEMI procedure guide. Int'l Test Method TF had several discussion for the limitation section. Then TF drafted the Doc. 5737A.

In detail as below,

3.2 (Before) When the carbon level of reference specimen at or greater than $2 \times 10^{15} \text{ atoms cm}^{-3}$ (0.04 ppma) would result in significant comparative error level. 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.

(After) When the carbon level of reference specimen at or greater than $2 \times 10^{15} \text{ atoms cm}^{-3}$ (0.04ppma), substitutional carbon measurement by this Test Method may result in significant comparative error level. 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.

Description of 3.4 was moved to Note 7 at 10.7.

Description of 3.7 was moved to Note 4 at 10.3.

9.2.3.1 (Before) When the measurement is made with double beam simple dispersive infrared spectrophotometers, the final thickness of test and reference slice specimens shall be equal to within $\pm 0.01 \text{ mm}$ (see §10.4).



(After) When the measurement is made with double beam simple dispersive infrared spectrophotometers, the difference of the thickness between test and reference specimens shall be within ± 0.01 mm (see §10.4).

Review and Adjudication Information

	Task Force Review	Committee Adjudication
Group:	Int'l Test Method Task Force	Silicon Wafer TC Chapter
Date:	July 2016	July, 2016
Time & Timezone:	TBD, CET	TBD, CET
Location:	NA	NA
City, State/Country:	San Francisco / NA	San Francisco / NA
Leader(s):	Peter Wagner - Consultant Dinesh Gupta - STA Ryuji Takeda - GlobalWafers Japan	
Standards Staff:		

This meeting's details are subject to change, and additional review sessions may be scheduled if necessary. Contact the task force leaders or Standards staff for confirmation.

Telephone and web information will be distributed to interested parties as the meeting date approaches. If you will not be able to attend these meetings in person but would like to participate by telephone/web, please contact Standards staff.

Check www.semi.org/standards on calendar of event for the latest meeting schedule.

LETTER BALLOT

SEMI Draft Document 5737A

REVISION OF SEMI MF1391-1107 (Reapproved 0912)

TEST METHOD FOR SUBSTITUTIONAL ATOMIC CARBON

CONTENT OF SILICON BY INFRARED ABSORPTION

Notice: Additions are indicated by underline and deletions are indicated by ~~strikethrough~~.

1 Purpose

1.1 Carbon ~~may have~~ has 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 Method¹ covers the determination of substitutional carbon concentration in single crystal silicon. Because carbon ~~may~~ also resides in interstitial lattice positions when in concentrations near the solid solubility limit, the results of this Test Method ~~may~~ shall 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~~ 0.04 parts per million atomic (ppma), that is, ~~$5.2 \times 10^{15} \text{ cm}^{-3}$~~ for measurements at room temperature, and down to about 0.01 ppma, that is, $0.5 \times 10^{15} \text{ cm}^{-3}$ 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 $16.53 \text{ }\mu\text{m}$. At cryogenic temperatures (below 80 K), the absorption band peak is at 607.5 cm^{-1} or $16.46 \text{ }\mu\text{m}$.

2.3-2.4 This Test Method provides two methods/algorithms for calculating the absorption coefficient of the carbon infrared absorption band which are named the long-baseline and short-baseline algorithms. The long-baseline method/algorithm is applicable to all measurements covered herein. The short-baseline algorithm is preferably and exclusively used for carbon content above $5 \times 10^{15} \text{ atoms cm}^{-3}$ (0.1 ppma). The short-baseline method is applicable to for measuring carbon content below $5 \times 10^{15} \text{ atoms cm}^{-3}$ only at room temperature.

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; The corresponding JEITA standard is to be discontinued, and has its technical content has been merged into this Test Method as they including the addition of the "short-baseline/short-baseline method" algorithm. For historical reasons/continuity, original method is also fully retained as the "long-baseline/long-baseline method" algorithm.~~ 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.

~~2.4-2.5~~ This Test Method is applicable to ~~slices-specimens~~ of silicon with resistivity higher than 3 $\Omega\cdot\text{cm}$ for p -type and higher than 1 $\Omega\cdot\text{cm}$ for n -type. ~~Slices-Specimens~~ can be any crystallographic orientation and ~~should-shall~~ be polished on both surfaces.

~~2.5-2.6~~ This Test Method is intended to be used with infrared spectrophotometers that are equipped to operate in the region from ~~500 to 2000 cm^{-1} (20 to 5 μm)~~ ~~2000 to 500 cm^{-1} (5 to 20 μm)~~.

~~2.6-2.7~~ 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: SEMI Standards and Safety Guidelines do not purport to address all safety issues associated with their use. It is the responsibility of the users of the Documents to establish appropriate safety and health practices, and determine the applicability of regulatory or other limitations prior to use.

3 Limitations

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

3.2 ~~When the reference specimen carbon level of reference specimen is at or greater than 2×10^{15} atoms cm^{-3} (0.04ppma), the detection limit at room temperature, then room temperature substitutional carbon measurement by this Test Method may result in a significant comparative error level. 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~~For measurements at cryogenic temperatures (below ~ 80 K), ~~if the reference specimen carbon level is at or greater than 0.01 ppma then low temperature substitutional carbon measurement by this Test Method may result in a significant comparative error level is about 0.01 ppma.~~ Obtaining reference samples ~~much at or~~ 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 ~~low temperature~~ detection limit is necessarily a comparative measurement only.

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 full width at half maximum (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.

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

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 $3\ \Omega\cdot\text{cm}$ for *p*-type, or $1\ \Omega\cdot\text{cm}$ 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 and Documents

4.1 SEMI Standards and Safety Guidelines

SEMI M59 — Terminology for Silicon Technology

4.2 ASTM Standards³

ASTM E131 — Terminology Relating to Molecular Spectroscopy

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 E131.

5.2 Acronyms, terms, and symbols relating to silicon technology, including those appropriate to infrared spectroscopic analysis of impurities in silicon, are found in SEMI M59.

6 Summary of Test Method

6.1 At room temperature, test ~~slice-specimens~~ are prepared that are polished on both sides to a nominal thickness of 2 mm. At cryogenic temperatures, the thickness of the test slices can range from 2 mm up to 4 mm, due to the reduced absorption at the spectral line of interest.

6.2 A reference ~~slice-specimen of nominally the same thickness and~~ of known ~~low~~ carbon content, at or below the detection limit of the applicable measurement technique (see ~~¶~~ § 3.2), is prepared in the same manner.

6.3 After verifying that the instrument is suitably set up, transmittance spectra of the sample and reference are obtained over the range from $500\text{ to }700\text{ cm}^{-1}$ ($20\text{ to }14.3\ \mu\text{m}$) ~~$700\text{ to }500\text{ cm}^{-1}$ ($14.3\text{ to }20\ \mu\text{m}$)~~ on a double-beam dispersive or single-beam Fourier Transform Infrared (FT-IR) spectrophotometer in accordance with manufacturer's instructions.

6.4 Absorbance spectra are derived from these transmittance spectra and a carbon-only spectrum is obtained as the difference between the two absorbance spectra.

³ American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken, Pennsylvania 19428-2959, USA. Telephone: 610.832.9585; Fax: 610.832.9555; <http://www.astm.org>

6.5 A baseline is calculated and/or drawn between the regions on both sides of the carbon peak on this difference-carbon-only absorbance spectrum, and absorbance values of both the peak and baseline are recorded.

6.5.1 For room temperature measurements the baseline calculation is preferably determined by the carbon content of the sample being measured.

6.5-6.5.1.1 Two regions are used, one for carbon content above 5×10^{15} atoms cm^{-3} (0.1 ppma), and one for carbon content below 5×10^{15} atoms cm^{-3} (0.1 ppma).

6.6 The absorbance peak height is taken as the difference between these two values. This peak height, corrected for sample thickness, is multiplied by a constant to calculate the substitutional carbon concentration. Two constants are used, one for measurements at room temperature (300 K), and one for measurements at cryogenic temperatures (below 80 K).

7 Apparatus

7.1 *Infrared Spectrophotometer* — Either a dispersive (computerized or non-computerized) (see Note 1) or a FT-IR spectrophotometer may be used. The resolution of the spectrophotometer must shall be at least 2 cm^{-1} at room temperature, or 1 cm^{-1} at cryogenic temperatures, over the range from 500 to 700 cm^{-1} for either dispersive or FT-IR spectrophotometers. The total operating range of the spectrophotometer shall include the range from 500 to 2000 cm^{-1} .

NOTE 1: Some dispersive infrared spectrometers are used in conjunction with a computer, which is used to store data. The data are then accessible for manipulation or computation, as required. These spectrometers are referred to as computer-assisted dispersive infrared spectrophotometers (CA-DIR). Dispersive infrared spectrometers that are not computer-assisted are referred to, for convenience, as simple dispersive infrared spectrometers (S-DIR).

7.2 *Micrometer Caliper* — Or other instrument, capable of measuring the thickness of the specimens to an accuracy of 0.005 mm.

7.3 *Equipment and Materials for Slicing and Polishing Silicon* — To a final thickness tolerance of 0.005 mm or less, and a total thickness variation of 0.01 mm or less.

7.4 *Thermometer* — Or other instrument capable of measuring the temperature of the sample chamber (for room temperature measurements) or the sample holder (for cryogenic temperature [below 80 K] measurements) to within $\pm 2^\circ\text{C}$.

7.5 *Calcium Fluoride Crystal (CaF_2)* — Cut to nominal thickness of 5 mm.

8 Sampling

8.1 Unless otherwise specified, a silicon slice specimen used for the carbon test is to be measured at the nominal slice specimen center.

9 Test and Reference Specimens

9.1 A-Use a single crystal slice specimen of about 2 mm thick ness must be used at room temperature, and a slice of 2.4 to 3.5 mm thickness is preferable at cryogenic temperatures.

9.2 Both the test and reference specimens must shall be carefully shaped to the following criteria:

9.2.1 Thickness variation over the measurement area shall be 0.005 mm or less.

9.2.2 Surface preparation shall be identical,

9.2.3 Thickness equality shall meet one of the following requirements:

9.2.3.1 When the measurement is made with double beam simple dispersive infrared spectrophotometers, the difference of the thickness between test and reference specimens shall be within ± 0.01 mm~~the final thickness of test and reference slices shall be equal to within ± 0.01 mm~~ (see ¶ 10.4).

9.2.3.2 When the measurement is made with computer assisted double-beam dispersive or single-beam FT-IR spectrophotometers, the final thickness of test and reference ~~slices-specimens~~ shall be equal to within ± 0.5 mm (see ¶ 10.5).

9.2.4 Surface area large enough such that with respect to the holders no incident radiation can bypass either the test or reference specimen.

9.3 The reference specimen ~~must-shall~~ be selected from float zone silicon with a minimal substitutional carbon concentration and a carrier concentration such that there is no measurable free carrier absorption of infrared radiation in the range from 500 to 2000 cm^{-1} .

NOTE 2: A satisfactory method of selecting usable reference specimens is to prepare polished ~~slices-specimens~~ of equal thickness from many different low-carbon silicon crystals produced by the float zone method, and then compare them to each other in an infrared radiation (IR) spectrophotometer. The selection must be carried out at cryogenic temperatures for cryogenic measurements. The specimen(s) showing the highest relative transmittance at 605 cm^{-1} at room temperature, or at 607.5 cm^{-1} at cryogenic temperatures (below 80 K), can be used as reference specimen(s).

9.4 Both test and reference specimens ~~must-shall~~ have resistivity greater than 3 $\Omega \cdot \text{cm}$ for *p*-type silicon and greater than 1 $\Omega \cdot \text{cm}$ for *n*-type silicon.

10 Procedure

10.1 Instrumental Checks

10.1.1 With reference to the transmittance spectrum, establish the 100% transmittance line to measure the stability and the noise level by one of the following methods.

10.1.1.1 For double-beam instruments, record the transmittance spectrum with both the sample and reference beams empty.

10.1.1.2 For single-beam instruments, record the transmittance spectrum as the ratio of two spectra taken with the sample beam empty. Take the two spectra at times separated by at least the time required to load samples and take two sequential spectra.

10.1.1.3 In both cases, plot the transmittance spectrum over the wavenumber range covering 500 to 700 cm^{-1} to obtain the 100% line.

10.1.1.4 If the 100% transmittance line is $100 \pm 0.5\%$ over the entire range, continue to the next step. Otherwise, adjust or repair the instrument so as to meet this criterion.

10.1.2 Establish the 0% transmittance line, (T).

10.1.2.1 For double-beam dispersive (DIR) instruments, record the transmittance spectrum between 500 and 700~~700 and 500~~ cm^{-1} with the sample beam blocked.

10.1.2.2 For FT-IR instruments, collect a background spectrum with the beam empty. Next, collect a spectrum with a CaF_2 crystal wafer, 5-mm thick, in the beam. Obtain the spectrum that is the ratio of the CaF_2 spectrum to the background spectrum, and plot the percent transmittance spectrum between 500 and 700~~700 and 500~~ cm^{-1} .

10.1.2.3 In either of these cases, the 0% transmittance ~~must~~shall not exceed $\pm 0.5\%$ over the range between ~~500 and 700~~200 to 1000 cm^{-1} .

10.1.2.4 Alternatively, for FT-IR instruments, plot the single-beam background spectrum (empty beam only) from ~~1000 to 200~~200 to 1000 cm^{-1} . The recorded energy in a region where the detector is known to give a zero response (below 300 cm^{-1} for most detectors) ~~must~~shall not be greater than 1.0% of the maximum signal in this region.^{4,5}

10.1.3 To determine the mid-scale linearity of the instrument obtain a spectrum of the silicon reference specimen over the wavenumber range from 1600 to 2000 cm^{-1} . If the value of the transmittance is not $53.8 \pm 2\%$ over this wavenumber range, align the sample 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.

NOTE 3: This angle ~~may be~~is determined by initially placing the silicon ~~slice~~specimen normal to the axis of the incoming beam, and then gradually tilting the specimen while repeatedly obtaining the transmittance spectrum above 1600 cm^{-1} . The optimum angle is typically less than 10° .

10.2 Measure and record the thicknesses of the test and reference specimens to within $\pm 0.005 \text{ mm}$, at their centers.

10.3 For room temperature measurements, measure and record the temperature of the sample chamber to $\pm 2^\circ\text{C}$. At cryogenic temperatures (below 80 K), measure and record the temperature of the sample holder.

NOTE 4: Reference and test sample specimens ~~must~~ should be as close as practically possible to the same temperature to avoid the effects of temperature on the intensity of the lattice band.

10.4 Obtain the carbon-only absorbance spectrum by one of the following methods (see Figure 1):

⁴ Chase, D. B., *Applied Spectroscopy* **38**(4), 491–494 (1984).

⁵ Hoffman, P., and Knozinger, E., *Applied Spectroscopy* **41**(8), 1303–1306, (1987).

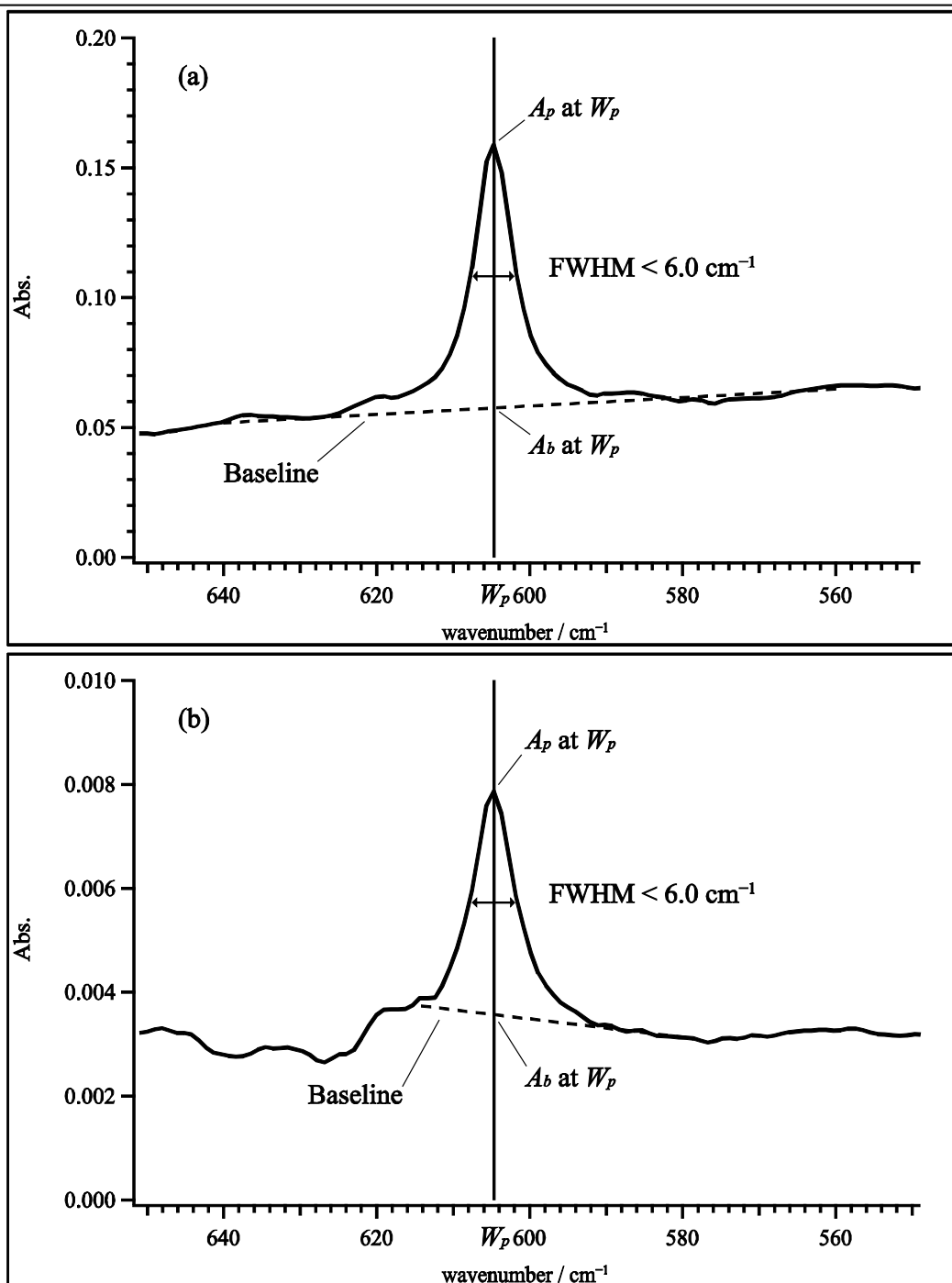


Figure 1

Carbon-Only Absorbance Spectrum, showing (a) Long-Baseline Method and (b) Short-Baseline Method

10.4.1 For double-beam instruments, position the sample specimen in the sample beam and the reference specimen in the reference beam and collect and plot the carbon-only absorbance spectrum over the range of at least 500 to 700 ~~700 to 500~~ cm^{-1} . Use a resolution of 2 cm^{-1} or better, at 605 cm^{-1} , at room temperature, and a resolution of 1 cm^{-1} or better, at 607.5 cm^{-1} , at cryogenic temperatures (below 80 K). Take spectra with the sample chamber in a vacuum, dry nitrogen, or dry air. Position the specimen so that the infrared beam passes through the center of the specimens.

10.4.2 For single-beam (computer-assisted) instruments, first collect a background spectrum with the beam empty, then collect sample and reference spectra sequentially under the same conditions as given in ¶ 10.4.5. Ratio the sample and reference spectra to the background and convert to absorbance. Multiply the reference absorbance spectrum by the ratio of the test specimen thickness to the reference specimen thickness and subtract this normalized absorbance from the test specimen absorbance to obtain the carbon-only spectrum.

~~NOTE 4:~~ NOTE 5: If the user has interactive arithmetic capability, subtract the reference absorbance spectrum from the sample absorbance spectrum in a ratio that provides the best flatness of the non-peak baseline between 560 and 640 cm^{-1} for carbon content above $5 \times 10^{15} \text{ atoms cm}^{-3}$ (0.1 ppma), and between 580 to 615 cm^{-1} for carbon content below $5 \times 10^{15} \text{ atoms cm}^{-3}$ (0.1 ppma). This eliminates the 2-phonon band due to silicon and gives a carbon-only spectrum.

10.5 Draw a straight baseline by one of the following methods/procedure.

10.5.1 ~~With~~ When the carbon content is unknown or estimated to be above $5 \times 10^{15} \text{ atoms cm}^{-3}$ (0.1 ppma), with regard to the plot of the carbon-only absorbance spectrum over the range from 500 to 700 cm^{-1} , define the baseline by drawing a straight line from 560 to 640 cm^{-1} . Use the average in the regions from 550 to 570 cm^{-1} , and 630 to 650 cm^{-1} , to define the endpoints of the straight line. This is the long-baseline algorithm. All cryogenic measurements shall use the long baseline method.

10.5.2 When the measurement is made at room temperature and the carbon content is estimated to be below $5 \times 10^{15} \text{ atoms cm}^{-3}$ (0.1 ppma), with regard to the plot of the carbon-only absorbance spectrum over the range from 500 to 700 cm^{-1} , define the baseline by drawing a straight line from 580 to 615 cm^{-1} . This is the short-baseline algorithm.

NOTE 6: In the short-baseline method/algorithms described in ¶ 10.5.2, it is difficult to determine the endpoints of the baseline, due to the significant background deviation compared to the maximum peak height. Use the average in the regions from 577 to 583 cm^{-1} , and 612 to 618 cm^{-1} , to define the endpoints. Alternatively, if the drawn straight line does not provide the non-peak baseline, use the absorbance values (not averaged values) at 580 and 615 cm^{-1} , respectively, for the endpoints.

~~10.5-~~

10.6 For measurements at room temperature, locate the wavenumber corresponding to the maximum absorbance in the region from 603 to 607 cm^{-1} . For measurements at cryogenic temperatures (below 80 K), locate the wavenumber corresponding to the maximum absorbance in the region from 605 to 609 cm^{-1} .

10.6.1 Record the value of that wavenumber, to four significant figures, as W_p . Record the maximum absorbance as A_p , the absorbance of the absorption peak.

10.6.2 Record the baseline absorbances, A_b , as the value of the baseline defined in ¶ 10.5 at W_p .

10.6.3 Record both A_p and A_b to three significant figures.

10.7 Determine and record the full width at half maximum (FWHM) of the peak. If the FWHM of the peak is greater than 6 cm^{-1} (room temperature) or 3 cm^{-1} (cryogenic temperatures), the spectrum was not properly obtained, and the instrument needs further adjustment, or the specimen is under stress. See § 3.4 for a list of factors that influence FWHM. Adjust the instrument in order to achieve the proper FWHM and repeat the procedure.

~~10.7-~~

10.8 Record the apodization function and the number of zero-fills used (FT-IR instruments) or the spectral slit width for dispersive infrared (DIR) instruments.

11 Calculation

11.1 Calculate the absorption coefficient, α , using the expression:

$$\alpha = \frac{23.03}{X} (A_p - A_b) \quad (1)$$

where:

- X = test specimen thickness, mm,
 A_p = the peak value of the carbon-only absorbance spectrum, and
 A_b = the baseline value of the carbon-only absorbance spectrum.

~~NOTE 5-~~NOTE 7: This equation does not include a correction factor for multiple reflections. Such a correction factor is not necessary, due to the very strong lattice absorption at 610 cm^{-1} .

11.2 For measurements at room temperature, calculate the content of substitutional carbon in atoms cm^{-3} or ppma as follows:⁶

$$\begin{aligned} \text{Carbon Content} &= 8.2 \times 10^{16} \alpha \text{ atoms cm}^{-3} \\ &= 1.64 \alpha \text{ ppma} \end{aligned} \quad (2)$$

11.2.1 If the ~~long-baseline~~long-baseline methodalgorithm has been used as in ¶ 10.5.1 and the calculated carbon content is below $5 \times 10^{15} \text{ atoms cm}^{-3}$ (0.1 ppma), the ~~short-baseline~~short-baseline methodalgorithm shall be used to draw a practical baseline. Use the ~~short-baseline~~short-baseline method as in ¶ 10.5.2 followed by the rest of the procedure to record the absorbances to recalculate the carbon content.

11.3 For measurements at cryogenic temperature, use the long-baseline algorithm and calculate the content of substitutional carbon in atoms cm^{-3} or ppma as follows:

$$\begin{aligned} \text{Carbon Content} &= 3.7 \times 10^{16} \alpha \text{ atoms cm}^{-3} \\ &= 0.74 \alpha \text{ ppma} \end{aligned} \quad (3)$$

~~NOTE 6-~~NOTE 8: The calibration factor used in this test method was determined as result of a study carried out in Japan by the Silicon Technologies Committee of the Japanese Electronic Industries Development Association (JEIDA), now JEITA,¹ in the mid-1980's. Further data analysis to eliminate recognized systematic error was carried out by the ASTM subcommittee assigned for this effort. The factor resulting from this study has an uncertainty of $\pm 0.4 \times 10^{16} \text{ atoms cm}^{-2}$ or $\pm 0.08 \text{ ppma-cm}$ for the room temperature measurements. The calibration factor for cryogenic measurements was calculated using the ratio given in Kolbesen and Mladcnović.⁷ The uncertainty of the cryogenic temperature factor is therefore $\pm 0.2 \times 10^{16} \text{ atoms cm}^{-2}$ or $\pm 0.04 \text{ ppma-cm}$.

12 Report

12.1 Report the following information:

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

12.1.2 Identification of test and reference specimens,

12.1.3 For room temperature measurements, the nominal temperature of the sample chamber. For cryogenic measurements, the nominal temperature of the sample holder,

⁶ Inoue, N., Stso. T. Nozaki, T., Endo, K., and Mizauma, K., "High Reliability Infrared Measurement of Oxygen and Carbon in Silicon," in *Emerging Semiconductor Technology*, ASTM STP 960, D. C. Gupta and P. H. Langer, eds., (ASTM, Philadelphia, 1987), pp. 365-377.

⁷ Kolbesen, B. O., and Mladcnović, T., *Kristall und Technik* 15(1), K1-K3 (1980).

12.1.4 Thickness of test and reference specimen,

12.1.5 Location and size of the illuminated area on the specimen,

12.1.6 For FT-IR instruments: (a) the apodization function used, (b) the amount of zero-filling, and (c) the number of data/cm⁻¹. For dispersive instruments: (a) the scan time/cm⁻¹, (b) the spectral slit width, and (c) the resolution at 600 cm⁻¹,

~~12.1.6~~ 12.1.7 Baseline ~~method~~ algorithm used: ~~long-baseline~~ long-baseline or ~~short-baseline~~ short-baseline,

~~12.1.7~~ 12.1.8 Spectral full width at half maximum of the absorption peak,

~~12.1.8~~ 12.1.9 W_p , wavenumber in cm⁻¹ of the absorption peak,

~~12.1.9~~ 12.1.10 The absorption coefficient due to substitutional carbon, α , in cm⁻¹,

~~12.1.10~~ 12.1.11 Carbon concentration, in ppma or in ~~atoms/cm³~~ atoms cm⁻³, and

~~12.1.11~~ 12.1.12 The conversion coefficient used.

13 Precision

13.1 A round robin was carried out by JEIDA (now JEITA¹) ~~onusing the long-baseline~~ method algorithm. The results of this 21-laboratory study are shown in Table 1. Figure 2 shows that the sample standard deviation increases with concentration value.

13.2 A single-laboratory investigation of the precision of carbon measurement at cryogenic temperature with a Fourier transform spectrophotometer was conducted using a single sample analyzed once a day over a 30-day period (30 measurement values). From this single-laboratory, multiple-operator, multiple-day, single-sample investigation of precision of carbon measurement, the precision was found to be 0.02 ppma (2 standard deviations). Nominal carbon content of the sample was 0.16 ppma.

14 Bias

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

15 Keywords

15.1 carbon; infrared absorption; infrared spectroscopy; silicon; single crystal silicon

Table 1 Results of JEIDA Round Robin Results on Long Baseline Long-baseline Method Algorithm

<i>Sample Number</i>	<i>Number of Measurements</i>	<i>Mean Value of Concentration of Substitutional Carbon (ppma)</i>	<i>Sample Standard Deviation (ppma)</i>
1	21	2.79	0.204
2	21	3.89	0.342
3	21	0.23	0.059
4	21	0.35	0.050
5	21	0.74	0.100
6	21	0.93	0.081
7	21	1.03	0.079
8	21	1.25	0.168
9	21	0.41	0.083
10	21	0.84	0.123
11	20	0.80	0.081
12	20	0.94	0.135
13	20	0.99	0.095
14	20	0.30	0.097
15	20	0.46	0.084
16	20	0.71	0.070
17	20	1.07	0.108
18	20	1.46	0.145
19	20	3.41	0.276
20	20	0.31	0.051
21	20	0.45	0.073
22	20	0.51	0.099
23	20	0.74	0.116
24	20	0.16	0.055
25	20	0.62	0.100
26	20	0.87	0.131

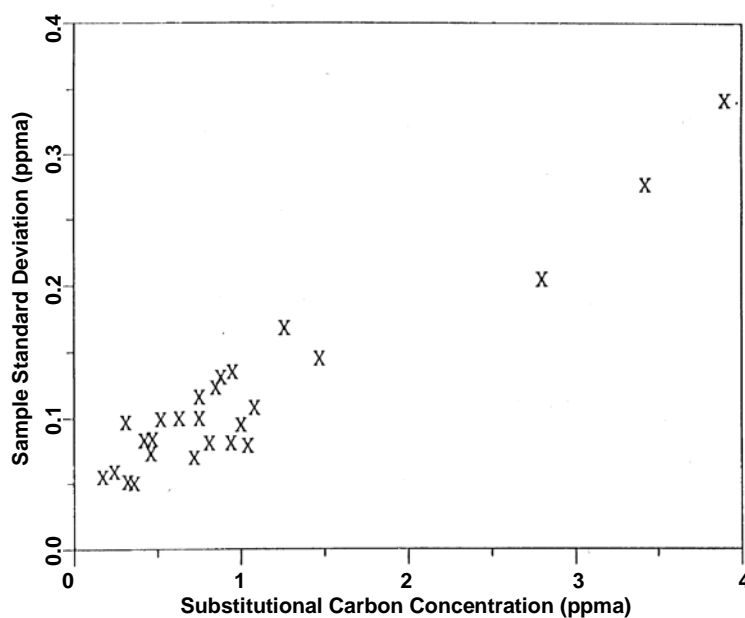


Figure 2
Round Robin Results on Long Baseline Long-baseline Method

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