JSNM

Standard of Japan Society of Newer Metals

JSNM-SI-001

Test method for low substitutional atomic carbon content of single crystal silicon by Fourier transform infrared absorption at room temperature

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List of committee members for JSNM-SI-001

This document is a translation of JSNM standard in Japanese.

In the event of any doubt arising, the original standard in Japanese is to be evidenced.

JSNM standard JSNM-SI-001

Test method for low substitutional atomic carbon content of single crystal silicon by Fourier transform infrared absorption at room temperature

1 Background

The control of impurities in silicon substrates for power semiconductor devices becomes more difficult due to stricter requirements for various device characteristics. Regarding the carbon concentration of single crystal silicon, the control of the level less than 5×10^{15} atoms/cm³ (0.1 ppma) is required which is below the measurable range of current measurement standards^{(1),(2)}. To respond this requirement, this standard has been implemented to realize the expansion of the measurable range down to 5×10^{14} atoms/cm³ (0.01 ppma) using Fourier transform infrared spectroscopy (FT-IR) at room temperature.

2 Scope

This standard provides a test method for measuring low "substitutional atomic carbon" (referred to "carbon" in the following part) concentration of single crystal silicon determined by FT-IR at room temperature. The infrared absorption by carbon in silicon single crystal appears at wavenumber of 605 cm⁻¹ (wavelength of 16.5 nm), and the absorption coefficient is proportional to the carbon concentration. In case of measuring low carbon concentration, high accuracy of absorption peak height measurement technique is required as the absorption peak becomes very small⁽³⁾. This standard is applied for measuring the carbon concentration range from 5×10^{14} atoms/cm³ (0.01 ppma) to 5×10^{15} atoms/cm³ (0.1 ppma). The test method defined by this document is applicable to process control, material study, quality assurance, and material certification.

3 Referenced standard documents

The standards listed below are considered as a part of this standard when referenced in this standard.

3.1 JEITA standard

3.1.1 JEITA EM-3503: Standard test Method for Substitutional Atomic Carbon Content of Silicon by Infrared Absorption

3.2 SEMI standard

3.2.1 SEMI MF1391: Test Method for Substitutional Atomic Carbon Content of Silicon by Infrared Absorption

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

4 Terms and definitions

4.1 Fourier transform infrared spectroscopy (FT-IR) Infrared light is collimated and directed to a twobeam interferometer (generally a Michelson interferometer), and obtain the interferogram between the two beams, one beam with a time-dependent function of optical path difference. The spectrum as a function of wavenumber is obtained

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by Fourier-transforming the interferogram. Dissimilar to dispersive infrared spectrometry which utilizes wavenumber limiting slits which determines the resolution, FT-IR measures wide range of wavenumber simultaneously and utilizes the infrared flux efficiently, resulting in high-speed measurement. FT-IR is widely applied in the field of infrared spectrometry.

4.2 Interferogram Optical interference signal as a function of optical-path difference in two-beam interferometers.

4.3 Single-beam spectrum The spectrum as a function of wavenumber, obtained by Fourier-transforming a interferogram.

4.4 Transmittance The ratio of transmitted light strength to incident light strength at a specific wavenumber.

4.5 Absorbance The common logarithm of the inverse of a transmittance. This value is a measure of light absorption of a specimen. The absorbance of the carbon peak, discussed in this standard, is proportional to the product of the carbon concentration and the specimen thickness.

4.6 Absorbance difference spectrophotometry When the concerned absorption peak is superimposed with the intrinsic absorption peak of the matrix crystal, it is difficult to measure the absorbance of the concerned impurity separately. In absorbance difference spectrophotometry, the absorbance of the concerned impurity is obtained by the difference of absorbance spectra between a test specimen and a reference specimen without the concerned impurities. Thus, the intrinsic absorption of the matrix crystal can be eliminated, and a high sensitivity measurement of the concerned impurity can be performed.

4.7 Baseline The line segment connecting two points on the difference spectrum of a test specimen and a reference specimen. The two points are on the difference spectrum at the both side of the carbon absorption peak; 615 and 580 cm⁻¹. This corresponds to the imaginary spectrum when the carbon content of the specimen and the reference specimen is equal (see Fig.1). The flatness of the difference spectrum on the baseline region affects the detection limit of this measurement method. Therefore, the following blank baseline and Si/Si baseline are defined to examine the measurement-system originated disturbance on the measurement.

4.8 Blank baseline A measure for confirming the stability of the measurement system alone. Blank baseline is defined as the common logarithm of the inverse of the ratio of two background single beam spectra, i.e., single beam spectra without specimen with a designated time interval. This value will be zero at all the wavenumber range and flat as a function of wavenumber, if the measurement system is ideally stable during the designated time span. If the system stability has any problem, this value deviates from zero and the flatness is lost.

4.9 Si/Si baseline A measure for confirming the stability of the measurement system and the repeatability of the specimen setting scheme. Prepare a stability verification specimen with a similar specification of the test specimen to be measured. First, a background single beam spectrum and the single beam spectrum of the stability verification specimen are measured and the absorbance spectrum is calculated. Second, the stability verification specimen is removed from the chamber and remounted in the chamber again, and the absorbance spectrum of the stability verification specimen is obtained again. The Si/Si baseline is defined as the difference between these two absorbance spectra. This value will be zero at wavenumber range measured, if the measurement system is ideally stable and stability verification specimen setting scheme is ideally repeatable. If the system stability and/or specimen setting scheme repeatability have any problem, this value deviates from zero and the flatness is lost.

4.10 Carbon peak Absorbance spectrum peak of carbon in absorbance difference spectrophotometry which appears around wavenumber of 605 cm⁻¹ (see Fig.1).



Figure 1 Carbon absorbance spectrum by absorbance difference spectrometry (Carbon peak)

4.11 Baseline absorbance (Ab) The absorbance of the baseline at 605 cm^{-1} corresponding to carbon peak position. This is the absorbance when there is no difference in carbon concentrations between test and reference specimens (see Fig.1).

4.12 Carbon peak absorbance (A_p) The absorbance of the test specimen at 605 cm⁻¹ corresponding to carbon peak position. (see Fig.1).

4.13 Carbon absorbance peak height The difference between carbon peak absorbance (A_p) and baseline absorbance (A_b) in carbon absorbance spectrum. (see Fig.1).

4.14 Full width at half maximum (FWHM) The absorption band width at half the difference between carbon peak absorbance (A_p) and baseline absorbance (A_b) . (see Fig.1).

5 Limitations

5.1 Reference specimen Carbon concentration of the reference specimen is desirable to be 1/3 or less of the detection limit of this test method. Therefore, the use the reference specimen with the carbon concentration of $1\sim 2\times 10^{14}$ atoms/cm³ (0.002 \sim 0.004 ppma) or less is recommended to minimize the relative error at room temperature.

5.2 Alignment and Adjustment The measurement-system alignment should be adjusted according to the system manufacturer's appropriate manuals.

5.3 2-phonon absorption The intrinsic absorption of two-phonon absorption band in silicon is located at around $630 \sim 590 \text{ cm}^{-1}$, and its absorption coefficient is around 9 cm^{-1} at room temperature⁽³⁾⁽⁴⁾. This value is 150-1500 times larger than the carbon absorption coefficient for the target carbon concentration range of $5 \times 10^{14} \sim 5 \times 10^{15}$ atoms/cm³ (0.01 \sim 0.1 ppma) of this document. Since two-phonon absorption interferes the measurement of carbon peak strength, it shall be reduced by the absorbance difference spectrophotometry.

5.4 Stability of the measurement system When applying the absorbance difference spectrophotometry, the stability of the measurement system during the test specimen and the reference specimen measurements should be guaranteed. For 2 mm test-specimen thickness, measurement system should have the following stability; the flatness of the blank and Si/Si baselines between 615 - 580 cm⁻¹ should be less than 0.0005, because the carbon absorbance corresponding to the target detection limit of 5×10^{14} atoms/cm³ (0.01 ppma) is 0.0005.

5.5 Thickness of specimen Upon applying the absorbance difference spectrophotometry, the thicknesses of the test and reference specimens should be as close as possible to remove the adverse effects unremovable by the thickness correction. Thickness of both specimens should be 2.00 ± 0.01 mm.

5.6 In-plane thickness uniformity of specimens In-plane thickness uniformity of specimens at the IR transmission area should be 0.001 mm or less. When in-plane uniformity is over 0.001 mm, the error in data analysis in the absorbance difference spectrophotometry hinders realizing the target detection limit.

Note 1: Thickness variation of 0.001 mm of specimens corresponds to the variation of the 2-phonon absorption band absorbance of 0.0004, which is equivalent to around 4×10^{14} atoms/cm³ (0.008 ppma) of carbon concentration if it is read as a carbon peak absorbance. This value is nearly equal to the detection limit of this standard; 5×10^{14} atoms/cm³ (0.01 ppma).

5.7 Detection limit The detection limit of this test method is determined by the ratio of carbon peak signal after applying the absorbance difference spectrophotometry to the noise in the absorbance spectrum. High sensitivity measurement can be expected by increasing the number of scans under the system stability condition described at section **5.4** of the standard. In case of 2 mm-thick specimen, the target carbon detection limit 5×10^{14} atoms/cm³ (0.01 ppma) corresponds to the carbon absorbance of 0.0005, and the noise level should be 0.00025 or less in the baseline range of 615 \sim 580 cm⁻¹.

5.8 Resistivity This standard applies to both p- and n-type silicon wafers of 10Ω -cm or above in resistivity to avoid the interference of free-carrier absorption.

6 Summary of method

This document standardizes the method of measuring carbon concentration of single crystal silicon by room temperature FT-IR. The summary of procedure is shown below.

- 6.1 Confirmation of the stability of the instrument alone by blank baseline measurement.
- 6.2 Confirmation of the system stability for the absorbance difference spectrophotometry by Si/Si baseline measurement.
- **6.3** Measurement of the carbon absorbance peak height in test silicon specimen.

6.4 The absorbance is calculated using carbon absorbance peak height. The carbon concentration is determined by the correlation between the carbon concentration and the carbon absorption coefficient.

7 Measurement system

7.1 FT-IR spectrometer is used. The system configuration is shown in Fig.2. Infrared light from the light source is interfered by using a two-beam interferometer, then directed on the test specimen, and the transmitted light is detected by a photodetector.

7.1.1 IR source The infrared light source. Globar lamps are usually used in FT-IR spectrometer.

7.1.2 Interferometer Instrument measuring wavelength or wavenumber by light interference. Generally, Michelson interferometer or its improved version of two-beam interferometer are used.

7.1.3 Chamber An air-tight box where the specimen is set. Preferably this box can be purged with dry nitrogen or air, or be evacuated to remove the adverse infrared absorption by water vapor, carbon dioxide, and the like in the air.

7.1.4 Detector A device to convert the light signal to electric signal. Tri-glycine sulfate detector (TGS, DTGS or DLaTGS) or mercury cadmium telluride detector (HgCdTe, MCT) etc. are available for this test method. However, when a mercury cadmium telluride detector is applied, a special care should be taken on the linearity between the incident light strength versus electric output.

7.1.5 Computer A computer is used to control the FT-IR interferometer, to perform Fourier-transformation from obtained interferogram to single beam spectrum and to display the spectra. It also calculates transmission spectrum, absorption spectrum and difference spectrum from various single beam spectra.



Figure 2 Schematic image of FT-IR

8 Preparation of specimens

8.1 Dimension and surface finish The size and the surface finish of the test and reference specimens shall be defined in the following subsections. Stability verification specimen described in section **4.9** is desirable to be prepared from a silicon crystal with a similar specification as the test specimen. Test or reference specimens can also be used as stability verification specimens.

8.1.1 Thickness of specimen The thickness of the specimen shall be 2.00 ± 0.01 mm. The thickness shall be measured with an accuracy of ± 0.0005 mm. Thickness correction factor X_S/X_R is calculated from test specimen thickness X_S and reference specimen thickness X_R .

8.1.2 Requirement for specimen shape Specimens shall be carefully shaped to satisfy the following criteria.

8.1.2.1 Thickness uniformity The thickness variation in the measurement area shall be within 0.001 mm.

8.1.2.2 Surface finish Specimens shall be both-side polished, and reference and test specimens should have the same surface condition.

8.1.2.3 Dimensions The dimensions of the specimens shall be large enough compared to the size of the probe infrared light, lest the incidental flux should bypass the test or reference specimens.

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8.2 Resistivity To avoid the free carrier absorption around the wavenumber between 640 and 560 cm⁻¹, the resistivity of specimens shall be over 10 Ω -cm for both p- and n-type silicon crystals.

8.3 Reference specimen The carbon content of the reference specimen is desirable to be below $1 \sim 2 \times 10^{14}$ atoms/cm³ (0.002~0.004 ppma). Additionally, the resistivity is recommended to be over 50 Ω ·cm.

Note 2: The best method to select a reference specimen is to prepare several candidate specimens with a same thickness from low carbon concentration ingots, and absorbance difference spectra are compared, and select the specimen with lowest absorbance at 605 cm⁻¹. It is desirable to confirm that the selected reference specimen has the low enough carbon concentration to satisfy the description at section **8.3** by SIMS (Secondary Ion Mass Spectrometer) or photoluminescence which have the lower detection limit than FT-IR.

9 Procedure

9.1 Determination of measurement conditions

9.1.1 Measurement method FT-IR spectrometers shall be used.

9.1.2 Atmosphere in chamber By gas purged atmospheres with dry nitrogen or dry air, or vacuum, the temperature and the humidity shall be kept constant in chamber.

9.1.3 Measurement range of wavenumber Measurement range shall include from 640 cm⁻¹ to 560 cm⁻¹.

9.1.4 Resolution Resolution of the measurement shall be 2 cm^{-1} .

9.1.5 Number of scans Number of scans shall be from 100 to 300.

Note 3: Improvement of the signal to noise ratio (S/N) is expected by increasing the number of scans. However, this condition should be decided considering the system stability applying the absorbance difference spectrophotometry.

9.1.6 Position of measurement Position of measurement shall be the center of the specimen, and normal incident alignment is generally employed.

Note 4: Measurement position can be decided by discussion between parties.

9.2 Confirmation of measurement-system stability All measurement in section **9.2** shall be conducted according the procedure described in section **9.1**. The stability of the measurement system without test specimen, i.e. measurement system alone is confirmed by the measurement of the blank baseline. The stability when applying the absorbance difference spectrophotometry is confirmed by the Si/Si baseline measurement with a stability confirmation specimen. Flatness of both baselines less than 0.0005 between 615 and 580 cm⁻¹ ensures the quantitative carbon concentrate-ion determination with the detection limit of 5×10^{14} atoms/cm³ (0.01 ppma).

Note 5: The frequency of stability confirmation should be determined to guarantee the instrument's stable operation.

Note 6: The results of the confirmation are recommended to be included in the test report.

9.2.1 Measurement of blank baseline

9.2.1.1 1st background single beam spectrum acquisition: Without any specimen, 1st background single beam spectrum is obtained.

9.2.1.2 2nd background single beam spectrum acquisition: A time-blank is set after measuring the 1st background single beam spectrum, then 2nd background single beam spectrum is obtained. The time-blank will be the sum of estimated time spans for test and reference specimen measurements.

9.2.1.3 Blank baseline is calculated from 1^{st} and 2^{nd} single beam spectra according to the procedure described in section **4.8**.

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9.2.1.4 Examine the flatness of the blank baseline, and confirm that the flatness is less than 0.0005 for wavenumber between 615 and 580 cm^{-1} .

Note 7: In case the flatness of the blank baseline becomes larger than 0.0005, confirm the detection limit using Table A-1 of Appendix A and proceed to next step of the measurement procedure. The confirmed detection limit is recommended to be described in the report.

9.2.2 Measurement of Si/Si baseline

9.2.2.1 1st background single beam spectrum acquisition: 1st background single beam spectrum is obtained.

9.2.2.2 1st stability verification specimen single beam spectrum acquisition: 1st single beam spectrum of

a stability verification specimen is obtained.

9.2.2.3 Remove the stability verification specimen from the chamber.

9.2.2.4 2nd background single beam spectrum acquisition: 2nd background single beam spectrum is obtained as in section 9.2.2.1.

9.2.2.5 Reload the stability verification specimen in the chamber.

9.2.2.6 2nd stability verification specimen single beam spectrum acquisition: 2nd stability verification specimen single beam spectrum is measured as in section **9.2.2.2**.

9.2.2.7 Si/Si baseline is obtained from 1^{st} and 2^{nd} stability verification specimen single beam spectra by calculating the absorbance spectra and taking the difference of them.

9.2.2.8 Confirm that the Si/Si baseline flatness is less than 0.0005 for wavenumber between 615 and 580 cm⁻¹. Figure 3 shows an example of the Si/Si baseline flatness.

Note 8: In case the flatness of the Si/Si baseline becomes larger than 0.0005, confirm the detection limit using the table of A-1 Appendix A and proceed to next step of the measurement procedure. The confirmed detection limit is recommended to be described in the report.



Figure 3 Example for flatness of Si/Si baseline

9.3 Preparation of carbon absorbance spectrum in silicon single crystal

All measurements in section 9.3 shall be conducted according the procedure described in section 9.1.

9.3.1 1st background single beam spectrum acquisition: 1st back ground single beam spectrum is obtained.

9.3.2 Test specimen single beam spectrum acquisition: Test specimen single beam spectrum is obtained.

9.3.3 2nd background single beam spectrum acquisition: 2nd back ground single beam spectrum is obtained.

9.3.4 Reference specimen single beam spectrum acquisition: Reference specimen single beam spectrum is obtained.

9.3.5 Creating absorbance spectrum: Absorbance spectrum of the test specimen is obtained from 1^{st} background and test specimen single beam spectra, and reference specimen absorbance spectrum is created from 2^{nd} background and reference specimen single beam spectra.

9.3.6 The difference spectrum of the absorbance spectrum of the test specimen and the thickness modified (the thickness correction factor multiplied) absorbance spectrum of the reference specimen, is calculated. Figure 4 shows an example of the absorbance spectrum.

Note 9: During the procedure shown in section **9.3.6**, when the effects of the 2-phonon absorption band of silicon crystal remains, the thickness correction factor of the reference specimen can be used as an adjustable parameter to obtain flat baseline around the carbon peak range of 615 to 580 cm⁻¹. This technique may be effective when the effective thicknesses of probe light incident point and the thickness measurement point are different.

9.3.7 The baseline of the difference spectrum is created.

9.3.8 Carbon peak absorbance A_p is determined.

9.3.9 Baseline absorbance A_b, i.e., absorbance of the baseline at carbon absorption peak, is determined.



Figure 4 Carbon absorbance spectra by difference spectrum method

Note 10: The measurement procedure can be shortened for the actual operation schedule (refer to Commentary R-5-4 "Measurement work plan"). The examples of the shortened operation schedule are as follows: The background single beam spectrum is commonly used by the consecutive test specimen single beam spectrum measurement and reference specimen single beam spectrum measurement. The reference single beam spectrum is used commonly with the test specimen single beam spectra measurements when multiple test specimens are measured.

8

10 Calculations (1)(2)

10.1 Absorption coefficient Calculate the absorption coefficient, α , using the formula;

$$\alpha = \frac{23.03}{X_s} \left(A_p - A_b \right) \tag{1},$$

where:

 $X_S =$ test specimen thickness (mm)

A_p= carbon peak absorbance

A_b= baseline absorbance

 $(A_p - A_b = \text{carbon absorbance peak height})$

10.2 Concentration The carbon concentration is calculated from formula in atoms/cm³ or ppma as follows:

. .

Carbon concentration = $8.2 \times 10^{16} \alpha$ atoms/cm ³						
=1.64 α ppma	(3)					

Note 11: When the correlation of carbon concentration among organizations is necessary, it is recommended to establish a calibration formula using standard specimens of 3 to 5 concentration levels.

11 Report

- **11.1** The instrument used, the operator, and the date of the measurements
- **11.2** The nominal temperature inside the sample chamber
- **11.3** Identification numbers of test and reference specimens
- 11.4 Thicknesses of test and reference specimens
- **11.5** Location and size of the illuminated area of the infrared probe on the test specimen
- 11.6 Full width at half maximum (FWHM) of the absorption peak
- **11.7** Wavenumber of the absorption peak, W_p in cm⁻¹
- **11.8** Absorption coefficient α due to substitutional carbon in cm⁻¹
- **11.9** Carbon concentration in atoms/cm³ or in ppma
- **11.10** The conversion factor used

11.11 Specifications of calibration specimens used (type, resistivity, carbon concentration, oxygen concentration, thickness, etc.)

11.12 Result of measurement-system stability (blank baseline, Si/Si baseline).

12 Bias and precision

Japan Society of Newer Metals (JSNM) has organized and performed round robin test (RRT) for this test method. Four organizations participated in the RRT as shown in Commentary R-5.

12.1 Comparison of Si/Si baselines obtained by each organization is shown in Fig. R-5-1. The room-temperature stability of the present measurement systems fulfilled the absorbance stability of 0.0005 in the wavenumber range of 615 \sim 580 cm⁻¹.

12.2 Under the above mentioned measurement-system stability conditions, comparisons of the carbon concentration

and reproducibility of the repetitive measurements for each organization are shown in Fig. R-5-5-3 and Table R-5-5-5. The carbon concentrations are in good agreement between each organization. In the carbon concentration range around the target detection limit of $2\sim5\times10^{14}$ atoms/cm³ (0.004 \sim 0.010 ppma), the standard deviation (σ) for each organization is $0.8\sim1.8\times10^{14}$ atoms/cm³ (0.002 \sim 0.004 ppma) and the standard deviation (σ) for three-time repetitive measurements is $0.3\sim0.4\times10^{14}$ atoms/cm³ (0.0006 \sim 0.0007 ppma). These results endorses that this test method is practically useful for the quantitative measurement of the carbon concentration with the target detection limit of 5×10^{14} atoms/cm³ (0.01 ppma).

Appendix

A-1 Relation between detection limit and baseline flatness

Table A-1 Detection limit level of carbon concentration vs. flatness of blank baseline and Si/Si baseline

Flatness	0.0005	0.0010	0.0015	0.0020		
(Absorbance)						
Level of Detection	5×10 ¹⁴ atoms/cm ³	1.0×10 ¹⁵ atoms/cm ³	1.5×10 ¹⁵ atoms/cm ³	2.0×10 ¹⁵ atoms/cm ³		
Limit	(0.01 ppma)	(0.02 ppma)	(0.03 ppma)	(0.04 ppma)		

<u>Annex</u>

(Reference)

Referenced documents

This annex describes about referenced documents, and is not a part of the standard document.

Reference

- (1) JEITA EM-3503: Standard test Method for Substitutional Atomic Carbon Content of Silicon by Infrared Absorption
- (2) SEMI MF1391: Test Method for Substitutional Atomic Carbon Content of Silicon by Infrared Absorption
- (3) R.C. Newman and J.B. Willis, J. Phys. Chem. Solids 26, p. 373 (1965)
- (4) F. A. Johnson, Proc. Phys. Soc., 73, pp. 265-272 (1959)

<u>Commentary</u>

Explanation of test method for low substitutional atomic carbon content of single crystal silicon by Fourier transform infrared absorption at room temperature

This commentary explains the provisions, descriptions and related items in the text of the standard, appendix and annex, and it is not a part of this standard document.

R-1 Background of establishment of this document

JEITA (Japan Electronic and Information Technology Industries Association: JEITA) had developed and managed the silicon material related forum standards in Japan until it finished its activity in this field in 2016, and JSNM (Japan Society of Newer Metals) succeeded the function and started to lead the standard development of silicon material industry. In January 2017, first preparatory meeting for the test method for low atomic carbon content of single crystal silicon was held, and the activity for the standardization of this test method started. This preparatory activity continued until the 6th meeting in May 2018, and was succeeded by one of the commissioned project of "Commissioned Project for Energy Conservation related Development of International Standards and Their Dissemination in FIY 2018" of METI (Ministry of Economy, Trade and Industry). The first Carbon Content Measurement Method NWG (National Working Group) meeting was held in July 2018 and 13 meetings were held until February 2021. As a result, a new JSNM Standard was expected, a new framework was set under the rules of JSNM's Standardization. The summing-up and the review were performed in FTIR Standardization WG and Silicon Standard Development Committee under Standardization Committee in January 2021, and the standard was enacted in the Standardization Committee in February 2021.

This standard describes the details of the test method to measure trace carbon content of silicon crystals for semiconductor device fabrication starting materials, and attentions to be paid during the measurement operation. The backgrounds for this standard are as follows.

So far, to improve the switching characteristics of power devices, carrier-life-time control by the impurities or defects in silicon crystals was adopted. However, the new generation device structures require longer carrier lifetime of the silicon substrates to improve the device performance. For example, carbon impurity atoms generally occupy the substitutional sites in silicon crystal, which are electrically inactive. During high energy particle irradiation in the device fabrication process, these replacement carbon atoms are repelled into the interstitial sites, and make complexes by the interactions with interstitial oxygen atoms. These complexes act as recombination centers which deteriorate carrier lifetime. Therefore, the accurate evaluation and precise control of the carbon atoms is inevitable to make good use of silicon substrates. As a test method, from the first step, the room temperature measurements are focused for measurement simplicity and versatility. The detection limits of interstitial carbon atoms by available infrared absorption methods at room temperature are,

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 2×10^{15} atoms/cm³ (0.04 ppma) by JEITA EM-3503, and 5×10^{15} atoms/cm³ (0.1 ppma) by SEMI MF1391. So far, the standard to verify and compare the silicon crystals with the carbon content less than 5×10^{15} atoms/cm³ (0.1 ppma) is not available. These days, the use of FT-IR method in infrared absorption measurement become widespread and the verification of the FT-IR as a new test method for low carbon content measurement becomes necessary. Therefore, JSNM started the activity to standardize the FT-IR method by organizing NWG.

R-2 History of standard documents for test methods of carbon content by FT-IR

The standard of carbon content in silicon single crystal by FT-IR was first established by the American Society of Testing and Materials (ASTM) as ASTM-F123 in 1974. In Japan, this ASTM standard was implemented as a JEIDA (Japan Electronic Industry Development Association, now JEITA) standard as JEIDA-56 in 1998 after a collaborated revision work by oxygen and carbon measurement expert committee during 1983 to 1992. Meanwhile, the use of FT-IR in infrared spectrometry becomes widespread and the use of FT-IR was added in the JEIDA standard. Then the information was acknowledged useful, and ASTM F-1391 including the information was established in 1993. This ASTM-F-1391 was then transferred to SEMI (Semiconductor Equipment and Material International) as SEMI MF-1391 in 2004, as ASTM transferred its function to maintain the semiconductor materials related standards to SEMI. This SEMI MF-1391 is applicable now. After the publication of JEIDA-56, it was transferred to JEITA as JEITA EM-3503 and now managed by JSNM and is available through the web site of JSNM, after the disbandment of JEITA Silicon Technologies committee in 2013.

R-3 Discussion at NWG (National Working Group)

The 1st RRT (Round Robin Test) was planned and by NWG, and the samples were supplied by all the silicon vendors participated in the NWG. The prepared samples were confirmed to have low carbon concentration of the target concentration of this standard; less than 2×10^{15} atoms/cm³ (0.04 ppma). The results of the 1st RRT reconfirmed the importance of carefully extracting the weak carbon signals from strong signals due to silicon lattice vibration. (See figure R-3-1).

The carbon peak at 605 cm⁻¹ is buried in 2-phonon absorption band peaked at 610 cm⁻¹. Figure R-3-1 shows the all the absorption spectra obtained at 1st RRT. The orders of the carbon concentration of the RRT samples obtained by each organization using one measurement system was revealed to be nearly the same, and the possibility of adopting the room temperature FT-IR method was confirmed.



Figure R-3-1 Example for 1st Round Robin test Result

R-4 Technical concern for applying room temperature FT-IR

Technical problems discussed by NWG on the 1st RRT results are summarized here.

R-4-1 Thickness nonuniformity of specimens

The effects of the thickens nonuniformity on the carbon absorption peak height are discussed.

Consider a sample of nominal thickness X with the thickness variation of Δ . A simple consideration leads to the error in the order of $A \times (\Delta X)$, as the absorbance is proportional to the sample thickness. However, in actual, the thinness nonuniformity has much larger effects on the absorbance created by the 2-phonon absorbance band subtraction process in absorbance difference spectrophotometry. The order of the effects is discussed below.

For simplicity, assume the thickness of a test sample and a reference sample to be the same thickness X, and only the test sample has the thickness nonuniformity of Δ , the carbon absorbances of the test sample and the reference sample at carbon absorbance peak position are described as below. (All thicknesses are in mm.)

$$A_{pS} = \frac{\alpha_c + \alpha_B}{23.03} (X + \Delta)$$
 (Eq. R-4-1)
 $A_{pR} = \frac{\alpha_B}{23.03} (X)$ (Eq. R-4-2)

Here, $\alpha_{\rm C}$ and $\alpha_{\rm B}$ are absorbance coefficient of carbon peak and 2-phonon absorbance band. The carbon peak of the reference sample is assumed to be negligibly small. The carbon absorbance peak height $(A_p - A_b)$ as defined at section **4.12** can be calculated below as $(A_{pS} - A_{pR})$ for difference spectrum between test and reference sample.

$$A_p - A_b = A_{pS} - A_{pR}$$

$$= \frac{\alpha_{C} + \alpha_{B}}{23.03} (X + \Delta) - \frac{\alpha_{B}}{23.03} (X)$$
$$= \frac{\alpha_{C}}{23.03} (X) + \frac{\alpha_{C} + \alpha_{B}}{23.03} (\Delta)$$
(Eq. R-4-3)

Here, the first term of the equation R-4-3 is the carbon absorbance peak height for the sample with nominal thickness, and the same as the equation described at section **10**. The second term is the error generated by nonuniformity of the test sample thickness. Since the 2-phonon absorbance coefficient α_B is 150 to 1500 times larger than the carbon absorbance coefficient α_c , as shown in section **5.3**, the error caused by the non-uniformity of the sample thickness can be estimated practically to be $\alpha_B \Delta / 23.03$. The carbon peak height, i.e., the first term, for the target detection limit of 5×10^{14} atoms/cm³ (0.01 ppma) of this standard can be calculated to be 0.0005 from the description in **10.1** and **10.2**. Therefore, to realize the target detection limit, the error, i.e., the second term should fulfill the following inequality.

$$\frac{\alpha_B \Delta}{23.03} < 0.0005$$
 (Eq. R-4-4)

When absorbance coefficient of 2-phonon absorption band of $\alpha_B = 9 \text{ cm}^{-1}$ is substituted in equation R-4-4, Δ needs to be smaller than 0.0013 mm. This is the reason for the statement, "the uniformity of measurement area within 0.001 mm" in the standard section **5.6** and **8.1.1**.

For reference, current JEITA, SEMI (ASTM) standards describe thickness tolerance of ± 0.01 mm for a 2 mmthick specimen. According to the discussion above, the error of 4×10^{15} atoms/cm³ in carbon concentration is derived for this thickness variation of 0.01 mm. Contrast to above, if the thickness uniformity of 0.001 mm is fulfilled, the error becomes around 4×10^{14} atoms / cm³ (0.008 ppma), which realizes the target detection limit of this standard. Consequently, this standard adopted the nominal specimen thickness of 2.00 ± 0.01 mm with the thickness uniformity of less than 0.001 mm within the measurement area. Actually, the uniformities of thickness within the measurement area of all the 1st RRT specimens were measured to be within 0.001 mm (see table R-4-1), and NGW confirmed that the sample thickness uniformity is available by present sample preparation techniques.

Table R-4-1 The evaluation of thickness uniformity for 1stRRT samples (unit: mm)

r	1									
Measurement					1st RRI	Sample				
Time	CZ1	CZ2	CZ3	CZ4	CZ5	CZ6	FZ1	FZ2	FZ3	FZ4
1	2.004	2.023	1.925	1.999	1.996	2.007	1.999	2.003	1.993	1.995
2	2.004	2.023	1.925	2.000	1.996	2.008	1.998	2.003	1.994	1.995
3	2.003	2.023	1.925	2.000	1.997	2.009	1.998	2.004	1.994	1.996
4	2.002	2.024	1.925	1.999	1.997	2.008	1.999	2.004	1.994	1.995
5	2.003	2.023	1.925	2.000	1.996	2.008	2.000	2.004	1.994	1.995
6	2.003	2.024	1.925	2.000	1.997	2.009	1.999	2.003	1.994	1.995
7	2.004	2.025	1.924	2.001	1.997	2.009	1.999	2.004	1.994	1.995
8	2.005	2.024	1.925	1.999	1.999	2.007	2.000	2.004	1.994	1.996
9	2.004	2.023	1.925	1.999	1.996	2.007	2.001	2.004	1.994	1.995
10	2.003	2.024	1.925	1.999	1.997	2.007	2.001	2.004	1.994	1.996
11	2.003	2.024	1.925	2.001	1.996	2.008	1.999	2.004	1.994	1.995
12	2.004	2.024	1.924	2.000	1.997	2.009	1.999	2.004	1.995	1.995
13	2.003	2.023	1.925	2.001	1.997	2.009	2.000	2.004	1.994	1.995
14	2.003	2.024	1.925	2.001	1.996	2.007	1.999	2.004	1.994	1.995
Average	2.003	2.024	1.925	2.000	1.997	2.008	1.999	2.004	1.994	1.995
STD	0.0008	0.0006	0.0004	0.0008	0.0008	0.0009	0.0009	0.0004	0.0004	0.0004

R-4-2 Effects of free carriers

The effects of free-carrier absorption are estimated from the sample resistivity. Therefore, the samples were prepared considering the range of resistivity on the 2^{nd} RRT (see R-5).

R-4-3 Determination of baselines

There are 2 kinds of baselines generally used by SEMI standard and JEITA standard. So called long baseline applied by SEMI MF-1391 is determined in between 640 cm⁻¹ and 560 cm⁻¹ and so-called short baseline applied by JEITA EM-3503 determined in between 615cm⁻¹ and 580 cm⁻¹. The NWG adopted the short baseline to minimize the measurement error in this test method.

R-4-4 Stability of the measurement system

The carbon absorption peak height corresponding to the target detection limit of carbon concentration, 5×10^{14} atoms/cm³ (0.01 ppma), is calculated to be 0.0005 from the discussion in **10.1** and **10.2**, and is as small as the baseline stability of conventional measurement system. In order to realize the measurement of carbon absorption peak height as low as 0.0005, the blank baseline flatness, which is an index for the stability of the system alone, and the Si/Si baseline flatness, which is an index for the system applying the absorbance difference spectrophotometry, needs to be less than 0.0005 between 615 and 580 cm⁻¹.

Present JEITA/SEMI/(ASTM) standards have no requirement on the blank and the Si/Si baseline flatness. 1st RRT operated by the NWG did not check the blank and the Si/Si baseline flatness, and dispersion of measurement data among organizations were large. In the 2nd RRT, the measurements were performed after the blank baseline flatness and the Si/Si baseline flatness were checked according to the procedure written in section **9.2** of this standard, and sufficient flatness was found to be obtained. As a result, a good agreement of the measurement data was reported from each organization as shown in Table R-5. Accordingly, it was confirmed that the blank baseline flatness and the Si/Si baseline flatness are very important indexes to determine the detection limit of this measurement method.

In case that the blank and the Si/Si baseline flatness measured are over 0.0005, the detection limit of the concerned measurement system can be referred to Table A-1 of **Appendix A-1**.

R-5 The report of 2nd Round Robin Test

After the 1st RRT, technical problems of the 1st RRT were reviewed and countermeasures were considered. The effectiveness of the countermeasures was confirmed in the 2nd RRT. Also, a new arrangement of the measurement procedure was devised to shorten the measuring time. Details are described below.

R-5-1 Implementing RRT

Special cares were taken for sample preparation before starting 2^{nd} RRT. Samples were made to fulfill the thickness condition described in **8.1.1** and the thickness uniformity condition described in **8.1.2**. Samples in respective sets (venders) were obtained from several appropriate positions from an ingot to have an appropriate variation of carbon concentration. Thus, a good set of samples with similar resistivity was obtained. Reference samples with the carbon concentration of 1 to 2×10^{14} atoms/cm³ (0.002 to 0.004 ppma) were prepared separately.

R-5-2 Test Specimens

The specifications for the prepared specimens for the 2nd RRT are described below (see table R-5-1). Crystal growth methods for specimens were MCZ (Magnetic-field applied Czochralski) or FZ (Floating Zone) method. Electrical conduction type was n-type, and resistivity ranged from $10 \Omega \cdot \text{cm}$ to non-doped (around $1000 \Omega \cdot \text{cm}$). Estimated carbon concentrations were 3×10^{14} to 5×10^{15} atoms/cm³ (0.006 to 0.1 ppma). As the reference specimen, not listed in the table, non-doped n-type (over $500 \Omega \cdot \text{cm}$) silicon single crystal was prepared. All specimens were processed to satisfy the target thickness of 2.00 ± 0.01 mm and thickness uniformity of less than 0.001 mm, and were double-surface polished.

		0		Expected	Expected Carbon Concentration				
Vvater Vender	Sample No.	Method	Туре	Resistivity (Ω⋅cm)	[atoms/cm ³]	ppma			
а	1~4	MCZ	n	10	3x10 ¹⁴ to 1x10 ¹⁵	0.006 to 0.02			
b	5~7	MCZ	n	Non-doped	3x10 ¹⁴ to 5x10 ¹⁵	0.006 to 0.1			
с	8~10	FZ	n	60	-	-			

Table R-5-1 The list of 2nd RRT specimens

R-5-3 Measurement conditions

The measurement-condition requirements of the test method are listed in section **9.1**. An example of the measurement conditions employed in the 2nd RRT is listed in Table R-5-2. These conditions could not be fulfilled according to the specifications of the particular measurement system; therefore, some different conditions were applied in some organization. To minimize the noise, larger number of scans than the conventional measurements were recommended; however, number of scans between 100 to 300 was recommended, considering the system stability. The measurements were performed at the center point of the sample. The conditions of Fourier transformation of apodization functions and zero-filling were not specified, as they scarcely had effects on the results of this test method as far as the same conditions were used in a series of measurements.

	Parameter	Setting / Comment
1	Method	Normal incident
2	Sampling position	Center of specimen
3	Detector	TGS
4	Atmosphere in sample chamber	Nitrogen purge
5	Wavenumber range	4000-400 cm ⁻¹
6	Resolution	2 cm ⁻¹
7	Scan time	100 to 300
8	Measurement portion	Center position
		Single-beam spectrum of air (Background)
9	Data	Single-beam spectrum of test specimen
		Single-beam spectrum of reference specimen

Table R-5-2 Example for measurement conditions

R-5-4 Measurement plan

Basic measurement plan consists of measuring single beam spectra of background measurement (air), reference and test specimens consecutively. In 2^{nd} RRT, the procedure described in section **9** of this standard was performed with the

time-saving plan as shown in Table R-5-3. Single beam spectra of background and reference specimen were shared by preceding and following test specimens, and number of measurements was saved. The table shows a plan for measuring 4 test samples (S1, S2, S3, S4) and a reference specimen. Firstly, background (air) measurement was shared by S1 and reference measurement in Flow 1 to 3. Similarly, background (air) measurements were shared by reference and S2 in Flow 3 to 5, S3 and reference in Flow 6 to 8, reference and S4 in Flow 8 to 10, respectively. Furthermore, reference specimen measurements were shared by S1 and S2 in Flow 1 to 5, and by S3 and S4 in Flow 6 to 10, respectively.

Comparing the devised time-saving procedure shown in Table R-5-3 and the basic procedure described in section 9.3 of the standard, the basic procedure requires 4 single beam measurements for one test sample (air, test, air, and reference). Therefore, in case of 4 test samples, total of 16 measurements are necessary. In contrast to this basic procedure, the time-saving procedure requires 3 single beam measurements for one test sample (test, air, and reference), where background measurements (air) are shared by the test specimen and the reference. Consequently, in case of 4 test specimens, only 12 measurements are necessary. Furthermore, if the reference measurements are shared as shown in Table R-5-3, 10 single beam measurements give the equivalent information. Thus, sharing background and reference single beam measurements gives an effective measurement plan when measuring multiple test specimens.



	1		2		3		4		5		6		7		8		9		10				Re	prod	lucit	oility	' che	eck			
WORK FIOW	S1	Ŷ	Air	î	Ref	ħ	Air	ħ	S2	î	S3	ħ	Air	Ŷ	Ref	î	Air	ħ	S4	î	Ref	ħ	Air	¢	S4	Ŷ	Ref	ħ	Air	ħ	S4
S1 Data	☆	⇒	☆	ţ	★																										
S2 Data					★	t	☆	t	★																						
S3 Data											★	ſ	★	\$	☆																
S4 Data															☆	f	☆	ſ	☆	t	☆	ſ	☆	î	☆	t	★	t	≯	ſ	☆

R-5-5 Results and Discussions

The 2nd RRT results are described below.

R-5-5-1 Assessment of the stability of the system

In case of applying the absorbance difference spectrophotometry, stability of the measurement system is inevitable. An example of Si/Si baseline measurements described at section **9.2.2** of the standard is shown in figure R-5-1. In the 2nd RRT, the requirement that the Si/Si baseline flatness should be less than 0.0005 was mostly fulfilled by measurement systems of the participated organizations. As descried at **R-4-4**, this requirement of the Si/Si baseline flatness less than 0.0005 shall be secured for stable separation and measurement of the carbon peak corresponding to the target detection limit of 5×10^{14} atoms/cm³ (0.01 ppma). Therefore, the measurement systems used for this test method should satisfy the stability described above.



Figure R-5-1 The comparison of Si/Si baseline flatness for each organization

R-5-5-2 Absorbance difference spectrophotometry

Examples of absorbance difference spectra for carbon concentration range of this test method are shown in figure R-5-2 (samples 5,6,7). The carbon concentration determined by SIMS using the adjacent crystals are 2.1×10^{15} atoms/cm³ (0.042 ppma) for sample 5, 5.3×10^{14} atoms/cm³ (0.011 ppma) for sample 6 and 3.6×10^{14} atoms/cm³ (0.0072 ppma) for sample 7. For each sample, clear carbon peak at 605 cm⁻¹ is identified in the absorbance difference spectrum. It should be noted that even in sample 7 which has the carbon concentration less than the detection limit of this standard, the carbon peak is still recognized.



Figure R-5-2 Example of absorbance difference spectrum for each concentration ranges

R-5-5-3 Comparison of carbon concentration reported by participant organizations

The carbon concentrations were calculated from the carbon peaks of the absorbance difference spectra according to chapter **10** of this standard, and the values of respective participant organizations were compared. For each test specimen, absorption coefficient and carbon concentration with their averages and standard deviations (σ) are listed in Table R-5-4. The carbon concentrations for each organization are plotted against their averages in Fig. R-5-3. The order of the carbon concentration of the samples is adequate in respective organizations as shown in Fig. R-5-3. The standard deviations (σ) for samples 2, 3, 4, 6 and 7 with low carbon concentration near the detection limit of this test method are $0.84 \sim 1.88 \times 10^{14}$ atoms/cm³ (0.0017 \sim 0.0036 ppma). This ensures that the quantitative measurement of carbon concentration down to 5×10^{14} atoms/cm³ (0.01 ppma), target detection limit of this test method, is possible.



Figure R-5-3 Total average and Data for each organization

Table R-5-4 Each organization of absorption coefficient and carbon concentration	
(Upper: Absorption, Mid: carbon concentration atoms/cm ³ and Lower: carbon concentration pp	oma)

Manadan	0		Organ	ization		A	_
vender	Sample	Α	В	С	D	Average	σ
	1	0.012	0.012	0.010	-	0.011	0.0011
	2	0.007	0.008	0.004	-	0.006	0.0022
а	3	0.004	0.007	0.003	-	0.005	0.0016
	4	0.002	0.005	0.003	-	0.003	0.0015
	5	0.031	0.033	0.032	0.018	0.029	0.0073
b	6	0.006	0.007	0.005	0.005	0.006	0.0010
	7	0.003	0.004	0.003	0.001	0.003	0.0012
	8	-	0.019	0.015	0.013	0.016	0.0031
с	9	-	0.017	0.016	0.013	0.015	0.0021
	10	-	0.013	0.010	0.007	0.010	0.0034
Manalan	Comula		Organ	ization		A	_
vender	Sample	Α	В	С	D	Average	0
	1	9.9E+14	9.9E+14	8.3E+14	-	9.4E+14	9E+13
_	2	5.7E+14	6.6E+14	3.1E+14	-	5.1E+14	2E+14
а	3	3.5E+14	5.3E+14	2.9E+14	-	3.9E+14	1E+14
	4	1.7E+14	4.1E+14	2.2E+14	-	2.7E+14	1E+14
	5	2.6E+15	2.7E+15	2.6E+15	1.5E+15	2.4E+15	6E+14
b	6	5.2E+14	5.7E+14	3.9E+14	4.2E+14	4.7E+14	8E+13
	7	2.7E+14	3.4E+14	2.8E+14	1.2E+14	2.5E+14	1E+14
	8	-	1.6E+15	1.2E+15	1.1E+15	1.3E+15	3E+14
с	9	-	1.4E+15	1.3E+15	1.1E+15	1.3E+15	2E+14
	10	-	1.1E+15	8.0E+14	5.4E+14	8.1E+14	3E+14
Manalan	Comula		Organ	ization		A	_
vender	Sample	Α	В	С	D	Average	σ
	1	0.020	0.020	0.017	-	0.019	0.0018
_	2	0.011	0.013	0.006	-	0.010	0.0036
а	3	0.007	0.011	0.006	-	0.008	0.0026
	4	0.003	0.008	0.004	-	0.005	0.0025
	5	0.052	0.055	0.053	0.029	0.047	0.0120
b	6	0.010	0.011	0.008	0.008	0.009	0.0017
	7	0.005	0.007	0.006	0.002	0.005	0.0019
	8	-	0.031	0.025	0.022	0.026	0.0050
с	9	-	0.028	0.026	0.022	0.025	0.0034
	10	-	0.022	0.016	0.011	0.016	0.0056

R-5-5-4 Comparison of carbon concentration using calibration by standard specimens

Calibration using standard specimens is not included in this standard, and information given here is for reference only. Systematic dispersion was recognized among organizations in Table R-5-4 and Fig. R-5-3. To minimize this dispersion, a correction using a calibration formula defined by the carbon concentrations for 3 selected samples with linear approximation was performed as advised in **note 11** of the standard (Table R-5-5 and Fig. R-5-4). The 3 samples used as standard samples were samples 5, 6 and 7 with carbon concentration of 2.1×10^{15} atoms/cm³ (0.042 ppma), 5.3×10^{14} atoms/cm³ (0.0011 ppma) and 3.6×10^{14} atoms/cm³ (0.007 ppma) determined by SIMS, respectively. The variation among organizations of the calibrated carbon concentration for the samples 1-4 and 8-9 which were not used for the standard samples became smaller in Fig. R-5-4 than that in Fig. R-5-3. The standard deviations (σ) of the carbon concentrations of samples 2, 3, and 4 with the carbon concentration near the detection limit, become $3 \sim 6 \times 10^{13}$ atoms/cm³ (0.0006~ 0.0012 ppma) as shown in Table R-5-5. This value with the present calibration is smaller than that of without the calibration, $0.84 \sim 1.88 \times 10^{14}$ atoms/cm³ (0.0017~0.0036 ppma). This reveals the effectiveness of the use of standard samples and a calibration formula. It should be noted that calibrated data can contain errors originated by the calibration

method itself, and caution is necessary for its application.



Figure R-5-4 Each organization value for average

Vandan	Comula		Organ		A	_		
vender	Sample	Α	В	С	D	Average	σ	
	1	8.8E+14	7.9E+14	8.1E+14	-	8.3E+14	5E+13	
	2	5.6E+14	5.6E+14	4.3E+14	-	5.2E+14	7E+13	
а	3	3.9E+14	4.7E+14	4.2E+14	-	4.3E+14	4E+13	
	4	2.6E+14	3.8E+14	3.7E+14	-	3.4E+14	6E+13	
	5	2.1E+15	2.1E+15	2.1E+15	2.0E+15	2.1E+15	4E+13	
b	6	5.2E+14	4.9E+14	4.9E+14	6.1E+14	5.3E+14	6E+13	
	7	3.4E+14	3.3E+14	4.1E+14	2.0E+14	3.2E+14	9E+13	
	8	-	1.2E+15	1.1E+15	1.5E+15	1.3E+15	2E+14	
с	9	-	1.1E+15	1.1E+15	1.5E+15	1.2E+15	2E+14	
	10	-	8.8E+14	7.8E+14	7.7E+14	8.1E+14	6E+13	
Vandar	Samula		Organ	Average	-			
vender	Sample	Α	В	С	D	Average	σ	
	1	0.018	0.016	0.016	-	0.017	0.0010	
	2	0.011	0.011	0.009	-	0.010	0.0015	
a	3	0.008	0.009	0.008	-	0.009	0.0008	
	4	0.005	0.008	0.007	-	0.007	0.0013	
	5	0.042	0.041	0.042	0.040	0.041	0.0008	
b	6	0.010	0.010	0.010	0.012	0.011	0.0012	
	7	0.007	0.007	0.008	0.004	0.006	0.0018	
	8	-	0.024	0.022	0.030	0.026	0.0041	
с	9	-	0.022	0.023	0.030	0.025	0.0044	
	10	-	0.018	0.016	0.015	0.016	0.0012	

Table R-5-5 Carbon concentrations for each organization (Upper: unit atoms/cm³, Lower: unit ppma)

R-5-5-5 Evaluation of measurement reproducibility

The reproducibility test was performed on sample 3 which has the carbon concentration near the target carbon concentration of this test method. The measurement was performed three times on the sample and the reproducibility of

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the obtained data was examined, as shown in Table R-5-6 listing absorption coefficient and carbon concentration. The standard deviation (σ) of the carbon concentration is $3 \sim 4 \times 10^{13}$ atoms/cm³ (0.0005 \sim 0.0007 ppma) and it is small enough to realize the target detection limit of 5×10^{14} atoms/cm³ (0.01 ppma).

Organization A		Deals haimht	Absorption	Carbon concentration					
Organiza	tion A	Peak neight	coefficient	atoms/cm ³	ppma				
	1	0.00049	0.0056	4.6E+14	0.0092				
Specimen	2	0.00049	0.0057	4.7E+14	0.0093				
	3	0.00044	0.0051	4.2E+14	0.0083				
		Average		4.5E+14	0.0089				
		σ		2.7E+13	0.0006				
Ommonia	L'an D	Deels heimht	Absorption	Carbon cor	ncentration				
Organiza	tion B	Peak neight	coefficient	atoms/cm ³	ppma				
	1	0.00067	0.0077	6.3E+14	0.013				
Specimen	2	0.00070	0.0080	6.6E+14	0.013				
	3	0.00063	0.0073	6.0E+14	0.012				
		Average		6.3E+14	0.013				
		σ		2.9E+13	0.0006				
. .			Absorption	Carbon cor	ncentration				
Organiza	tion C	Peak height	coefficient	atoms/cm ³	ppma				
	1	0.00033	0.0038	3.1E+14	0.0063				
Specimen	2	0.00040	0.0046	3.8E+14	0.0076				
	3	0.00033	0.0038	3.1E+14	0.0063				
		Average	3.4E+14	0.0067					

3.6E+13

0.0007

 Table R-5-6
 The result of repeated measurement using identical specimen for each organization

 (Upper: Organization A, Mid: Organization B and Lower: Organization C)

R-5-6 Summary

In the 2nd RRT, using the measurement systems owned by respective participants, the variation and reproducibility of the carbon concentration were examined. To realize the target detection limit of 5×10^{14} atoms/cm³ (0.01 ppma), the necessary condition of Si/Si baseline flatness to be less than 0.0005 was fulfilled and the carbon peak was clearly recognized for samples near the target detection limit by each organization. Consequently, the carbon concentrations agreed well among organizations, and the standard deviation (σ) around the target detection limit of $2 \sim 5 \times 10^{14}$ atoms/cm³ (0.004~0.010 ppma) was $0.8 \sim 1.8 \times 10^{14}$ atoms/cm³ (0.002 ~ 0.004 ppma). Also, the three-time reproducibility of measurements around the same carbon concentration range gave the standard deviation (σ) of $3 \sim 4 \times 10^{13}$ atoms/cm³ (0.0005~0.0007 ppma).

σ

These results guarantee that the quantitative measurements with target detection limit of 5×10^{14} atoms/cm³ (0.01 ppma) with this standard is possible. When standard samples with accurate carbon concentrations are available, the variation among organizations can be reduced according to the procedure given in the **Note 11** of the standard and **R-5-5-4** of this Commentary.

R-5-7 Future Plan

The remaining problems are concerned with the fabrication and selection of the reference sample and the standard samples, and how to handle them. The carbon concentration of the reference specimens gives rise to the basic errors when

absorbance difference spectrophotometry is applied. The reference sample should be fabricated or selected with the carbon concentration as low as possible according to the procedure described in **note 2** of the standard.

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