

SEMI MF1391-0704

TEST METHOD FOR SUBSTITUTIONAL ATOMIC CARBON CONTENT OF SILICON BY INFRARED ABSORPTION

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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 reference Test Method¹ 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, $5 \times 10^{15} \text{ cm}^{-3}$ 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 $16.46 \text{ }\mu\text{m}$.
- 2.4 This Test Method is applicable to slices of silicon with resistivity higher than $3 \text{ }\Omega\cdot\text{cm}$ for *p*-type and higher than $1 \text{ }\Omega\cdot\text{cm}$ 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 $20 \text{ }\mu\text{m}$).
- 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: 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.

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 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/cm³ (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.

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.

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 and Documents

4.1 SEMI Standards and Safety Guidelines

SEMI MF1241 — Terminology of Silicon Technology

4.2 ASTM Standard³

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.

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

³ ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, USA; Telephone: +1.610.832.9585, Fax: +1.610.832.9555, <http://www.astm.org>

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.

5.3.5 *full width at half maximum (FWHM)* — the width of the absorbance band at half its magnitude as measured from the baseline.

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

5.3.6.1 *Discussion* — For true double-beam instruments, it is obtained directly with the reference specimen in the sample beam and the reference (second) beam empty. For FT-IR and other single-beam instruments, it is the result of ratioing the single-beam spectrum of the reference specimen to the background spectrum (see ¶ 5.3.1).

5.3.7 *sample spectrum, n* — the spectrum obtained when the sample specimen is placed in the infrared beam.

5.3.7.1 *Discussion* — For true double-beam instruments, it is obtained directly with the sample in the sample beam and the reference (second) beam empty. For FT-IR and other single-beam instruments, it is the result of ratioing the single-beam spectrum of the sample to the background spectrum (see ¶ 5.3.1).

6 Summary of Test Method

6.1 At room temperature, test slices are prepared that are polished on both sides to a nominal thickness of 2.0 mm. At cryogenic temperatures, the thickness of the test slices can range from 2.0 to 4.0 mm.

6.2 A reference slice of known low carbon content (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 700 to 500 cm^{-1} (14.3 to 20.0 μm) 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.

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

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) or a FT-IR spectrophotometer may be used. The resolution of the spectrophotometer must 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} .