

SEMI MF1391-93 (Reapproved 2000)

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

This Standard was originally published by ASTM International as ASTM F1391-92. It was formally approved by ASTM balloting procedures and adhered to ASTM patent requirements. Though ownership of this Standard has been transferred to SEMI, it has not been formally approved by SEMI balloting procedures and does not adhere either to SEMI Standards Regulations dealing with patents or to SEMI Standards Style Manual. Available at www.semi.org November 2003. Last published by ASTM International as ASTM F1391-93 (Reapproved 2000).

INTRODUCTION

This test method is a replacement for Test Method F123. This current version uses a new coefficient for the conversion between the absorption-coefficient, carbon peak-height, and the substitutional carbon content. Also, the curve fitting is done in the carbon-only absorbance spectrum, and it incorporates an improved method of determining the baseline.

1. Scope

1.1 This referee 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.

1.2 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).

1.3 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 $16.46 \text{ }\mu\text{m}$.

1.4 This test method is applicable to slices of silicon with resistivity higher than $3 \text{ }\Omega\text{-cm}$ for *p*-type and higher than $1 \text{ }\Omega\text{-cm}$ for *n*-type. Slices can be any crystallographic orientation and should be polished on both surfaces.

1.5 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 $20 \text{ }\mu\text{m}$).

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

1.7 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

E131 Terminology Relating to Molecular Spectroscopy²

F1241 Terminology of Silicon Technology³

3. Terminology

3.1 Definitions:

¹ DIN 50438-2 is also a method for measuring the substitutional carbon content of silicon. It differs in some aspects, including different conversion coefficients, from this proposed test method (see "Related Materials Section," Vol 10.05). It is the responsibility of DIN Committee NMP 221, with which Committee F-1 maintains close technical liaison. DIN 50438/2, Testing of Inorganic Semiconductor Materials: Determination of the Impurity Content in Silicon by Means of Infrared Absorption; Carbon, is available from Beuth Verlag GmbH, Burggrafenstrasse 4-10, D-1000 Berlin 30, Federal Republic of Germany.

² Annual Book of ASTM Standards, Vol 03.06, ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428. Telephone: 610-832-9500, Fax: 610-832-9555, Website: www.astm.org

³ Annual Book of ASTM Standards, Vol 10.05.

3.1.1 General definitions for terms related to infrared absorption spectroscopy are found in Terminology E131.

3.1.2 Definitions for terms related to silicon materials technology are found in Terminology F1241.

3.2 Definitions of Terms Specific to This Standard:

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

3.2.2 *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 (see Fig. 1).

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

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

3.2.4.1 *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.

3.2.5 *FWHM*—acronym for full width at half maximum, the width of the absorbance band at half its magnitude as measured from the baseline.

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

3.2.6.1 *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 obtained in 3.2.1.

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

3.2.7.1 *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 obtained in 3.2.1.

4. Summary of Test Method

4.1 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 test slices' thicknesses can range from 2.0 to 4.0 mm.

4.2 A reference slice of known low carbon content is prepared in the same manner.

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

4.4 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. The absorbance peak height is 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).

5. Significance and Use

5.1 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.

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

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

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

6. Interferences

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

6.2 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.

6.3 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.

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

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

6.6 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.⁴ This broad band peaks close to the wavelength of the carbon-in-silicon band and so presents a problem in measuring the intensity of the carbon band.

6.7 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.

6.8 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.

6.9 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.

6.10 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 will reduce these interference fringes.

7. Apparatus

7.1 *Infrared Spectrophotometer*, either a dispersive (computerized or non-computerized) or a Fourier transform (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 Fourier transform infrared spectrophotometers. The total operating range of the spectrophotometer shall include the range from 500 to 2000 cm⁻¹.

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 ±2°C.

7.5 *Calcium Fluoride Crystal (CaF₂)*, cut to nominal thickness of 5 mm.

8. Sampling

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

⁴ Johnson, F. A., *Proceedings of the Physics Society*, Vol 73, 1959, pp. 265–272.