SEMI MF1619-1107 (Reapproved 1023)
TEST METHOD FOR MEASUREMENT OF INTERSTITIAL OXYGEN CONTENT OF SILICON WAFERS BY INFRARED ABSORPTION SPECTROSCOPY WITH p-POLARIZED RADIATION INCIDENT AT THE BREWSTER ANGLE

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1 Purpose

1.1 Control of the oxygen content is essential for silicon wafers to be used for advanced devices and integrated circuits. It is desirable to be able to measure the oxygen content of product wafers, nondestructively and without regard for back surface finish. This Test Method provides a means for reducing the influence of the back surface condition on the measurement.

1.2 This Test Method may be used for routine process monitoring, quality control, materials acceptance, and research and development.

2 Scope

2.1 This Test Method[1] covers determination of the absorption coefficient due to the interstitial oxygen content of commercial monocrystalline silicon wafers by means of Fourier transform infrared (FT-IR) spectroscopy. In this Test Method, the incident radiation is *p*-polarized and incident on the test specimen at the Brewster angle in order to minimize multiple reflections.[2]

NOTE 1: Radiation in which the electric vector is parallel to the plane of incidence is defined as *p*-polarized radiation.

2.2 Since the interstitial oxygen concentration is proportional to the absorption coefficient of the 1107 cm^{-1} absorption band, the interstitial oxygen content of the wafer can be derived directly using an independently determined calibration factor.

2.3 The test specimen is a single-side polished silicon wafer of the type specified in SEMI M1. The front surface of the wafer is mirror polished and the back surface may be as-cut, lapped, or etched with rms roughness less than 0.9 µm (refer to ¶ 8.1.1.1).

2.4 This Test Method is applicable to silicon wafers with resistivity greater than 5 Ω·cm at room temperature.

[1] This Standard is based on draft procedures and interlaboratory tests conducted jointly by the Silicon Wafer Committee of the SEMI Japan Standards Program and the Oxygen and Carbon Measurement Committee of the Japan Electronic and Information Technology Industries Association (JEITA), 3rd Floor Mitsui Kaijo Bldg. Annex 11, Kanda Surugadai 3-chome, Chiyoda-Ku, Tokyo, 101-0062, Japan.

[2] Krishnan, K., "Precise and Rapid Measurement of Oxygen and Carbon in Silicon." *Defects in Silicon*, edited by W. M. Bullis and L. C. Kimerling, *Proceedings* Vol. 83-9, The Electrochemical Society, Pennington, NJ (1983): pp. 285–292; Shirai, H., "Determination of Oxygen Concentration in Single-Side Polished Czochralski-Grown Silicon Wafers by *p*-Polarized Brewster Angle Incidence Infrared Spectroscopy." *J. Electrochem. Soc.*, 138 (1991): pp. 1784–1787; Shirai, H., "Oxygen Measurements in Acid-Etched Czochralski-Grown Silicon Wafers." *J. Electrochem. Soc.*, 139 (1992): pp. 3272–3275.

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3 Limitations

3.1 Multiple reflections are greatest for thin, double-side polished wafers with parallel front and back surfaces. Such wafers are excluded from the test procedure, but they are used in establishing the optimum angle of incidence. For such wafers, the transmittance, T, is given as follows:

$$T = \frac{(1-R)^2 e^{-\alpha x}}{1-R^2 e^{-2\alpha x}} = (1-R)^2 e^{-\alpha x}[1 + R^2 e^{-2\alpha x} + R^4 e^{-4\alpha x} + \ldots] \quad (1)$$

where:

R = reflectance ratio,

α = absorption coefficient in cm^{-1},

x = optical path length in cm (= dcosθr),

d = specimen thickness, in cm, and

θr = angle of refraction (¶ 10.2).

To neglect multiple reflections, the quantity $R^2e^{-\alpha x}$ should be less than 0.001. The reflection is suppressed for incident radiation at the Brewster angle (73.7° from the normal in silicon). However, because of the large cone angle of the incident radiation in FT-IR spectrometers with focused beam not all of the radiation is precisely at the Brewster angle. Procedures to minimize this effect are given in ¶ 9.2.

3.2 The optical path length of the transmitted beam is not determined directly, but is estimated from the central beam angle of the incident non-parallel beam flux.

3.3 The baseline is assumed to be due largely to surface scattering and is approximated by a parabolic curve (refer to ¶¶ 10.4 and Related Information 1, § R1-1.2).

3.4 Absorption due to free carriers can interfere with the measurement, but it is minimized by requiring that the resistivity of the test and reference specimens be greater than 5 Ω·cm.

3.5 Silicon has absorption due to the lattice spectrum at the wave number of the peak of the oxygen absorption. A reference wafer is required in order to determine the value of this absorption so that a correction for it can be made.

3.6 Since both oxygen and silicon lattice absorption change with temperature, the temperature inside the spectrometer chamber must be maintained at 27 C ± 5°C during the measurement as required by SEMI MF1188.

3.7 Nonlinearity in the spectrometer and its detecting system can degrade the accuracy of the measurement.

4 Referenced Standards and Documents

4.1 *SEMI Standards and Safety Guidelines*

SEMI M1 — Specification for Polished Single Crystal Silicon Wafers

SEMI M59 — Terminology for Silicon Technology

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

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

5 Terminology

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

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6 Summary of Test Method

6.1 The stability of the FT-IR spectrometer is established to be adequate for the measurement cycle.

6.2 The optimum angle of incidence is determined to minimize multiple internal reflection.

6.3 The transmission spectrum of an oxygen-free double-side polished float-zone reference wafer is recorded.

6.4 The transmission spectrum of one or more oxygen-containing test specimens is determined.

6.5 The negative logarithm of each of these transmission spectra is taken to determine the absorbance spectrum for each reference wafer and test specimen.

6.6 The absorbance spectra are normalized by dividing by the beam path length to obtain the absorption coefficient as a function of wave number.

6.7 The baseline is corrected for curvature resulting from scattering from the rough back surface and the baseline value at 1107 cm^{-1} is determined.

6.8 This baseline value is subtracted from the absorbance at 1107 cm^{-1} to determine the absorption coefficient due to interstitial oxygen.

6.9 The absorption coefficient is multiplied by the appropriate calibration factor to obtain the oxygen content of the test specimen.

7 Apparatus

7.1 *Single-Beam FT-IR Spectrometer* — As specified in SEMI MF1188, capable of collecting transmission spectra with resolution of both 4 cm^{-1} and 1 cm^{-1}.

7.2 *Polarizer* — In order that the incident beam shall be *p*-polarized.

7.3 The central angle of the incident beam flux shall be adjustable between 65° and 75° from the surface normal.

7.4 *Detector* — Shall be large enough that the shifting of the beam by the sample (a lateral distance equal to 0.88 times the sample thickness) does not affect its sensitivity. Detector sensitivity shall be unchanged whether a sample is or is not in the measurement beam.

8 Test and Reference Specimens

8.1 *Test Specimen*

8.1.1 A silicon wafer with chem-mechanically polished front surface and a back surface that may be as-cut, lapped, or etched. The back-surface roughness shall be such that:

8.1.1.1 The rms roughness shall be less than 0.9 µm,

8.1.1.2 The transmittance through the wafer at 1107 cm^{-1} shall equal or exceed 25%, or

8.1.1.3 The difference between the absorption coefficient at 1200 cm^{-1} and the absorption coefficient at 950 cm^{-1} shall be positive but less than 5 cm^{-1}.

8.1.2 Wafers shall have thickness in the range specified in SEMI M1 (between 500 µm and 795 µm for wafers with diameter from 100 mm to 300 mm). Measure and record as d_{CZ} the center-point thickness of each test specimen to the nearest µm.

8.1.3 The resistivity of either *n*- or *p*-type test specimens shall be greater than 5 Ω·cm.

8.2 *Oxygen-Free Reference Specimen*

8.2.1 A double-side polished, float-zoned silicon wafer with maximum oxygen content of 1 atoms/cm^3 × 10^{16} atoms/cm^3 or, equivalently, 0.2 parts per million atomic (ppma) and resistivity greater than 5 Ω·cm.

8.2.2 Measure and record as d_{FZ} the thickness to the nearest µm; the thickness of the reference specimen shall be within ±20% of that of the test specimen.

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8.3 *Thin Double-Side Polished, Float-Zoned Wafer* — Approximately 400 µm thick, for use in determining the optimum angle of incidence.

8.4 *Sapphire Wafer* — Greater than or equal to 400 µm-thick, polished on one or both sides.

9 Procedure

9.1 *Stability of FT-IR Spectrometer*

9.1.1 Turn on the spectrometer and allow it to operate long enough to stabilize.

9.1.2 Set the resolution of the spectrometer to 4 cm^{-1}.

9.1.3 Use a minimum of 64 scans for each spectrum collection.

9.1.4 *100% Line Check*

9.1.4.1 Collect a background spectrum $I_{01}(v)$ with the sample beam empty over the wave number range from 900 cm^{-1} to 1300 cm^{-1}.

9.1.4.2 Wait a time interval, *t* minutes, long enough to make the desired measurements on the test and reference specimens, and then again collect a background spectrum $I_{02}(v)$ with the sample beam empty over the wave number range from 900 cm^{-1} to 1300 cm^{-1}. The time interval *t* shall be at least 60 minutes.

9.1.4.3 Determine the ratio $I_{01}(v)/I_{02}(v)$ over the wave number range from 900 cm^{-1} to 1300 cm^{-1}.

9.1.4.4 If the ratio $I_{01}(v)/I_{02}(v) = 1.000 \pm 0.005$ (100% ± 0.5%) over the entire wave number range, the instrument is acceptable for use in any measuring sequence that requires a total elapsed time ≤*t* minutes.

9.1.4.5 If the ratio $I_{01}(v)/I_{02}(v)$ falls outside the range 1.000 ± 0.005 in any part of the wave number range 900 cm^{-1} to 1300 cm^{-1}, reduce the time interval, *t*, and repeat ¶¶ 9.1.4.1 through 9.1.4.4 until the ratio $I_{01}(v)/I_{02}(v) = 1.000 \pm 0.005$ over the entire wave number range.

9.1.4.6 Ensure that any sequence of measurements made using a single background spectrum is completed within the time interval *t* minutes.

9.1.5 *0% Line Check*

9.1.5.1 Collect a background spectrum $I_0(v)$ with the sample beam empty over the wave number range from 900 cm^{-1} to 1300 cm^{-1}.

9.1.5.2 Then collect a spectrum $I_s(v)$ with the sapphire wafer (refer to ¶ 8.1.3) in the sample beam over the wave number range from 900 cm^{-1} to 1300 cm^{-1}.

9.1.5.3 Determine the ratio $I_0(v)/I_s(v)$ over the wave number range from 900 cm^{-1} to 1300 cm^{-1}.

9.1.5.4 If the ratio $I_0(v)/I_s(v) \leq 0.001$ (0.1%) over the entire wave number range, the instrument is acceptable for use.

9.1.5.5 If the ratio $I_0(v)/I_s(v) > 0.001$ (0.1%) over any part of the wave number range, adjust the instrument in accordance with the manufacturer's instructions and repeat the entire procedure beginning with ¶ 9.1.

9.2 *Angle of Incidence*

9.2.1 Use one of the following two methods to determine the best angle of incidence of the *p*-polarized infrared beam.

9.2.2 *Fringe Minimum (FM) Method*

9.2.2.1 Set the resolution of the spectrometer to 1 cm^{-1}.

9.2.2.2 Adjust the angle of the specimen holder so that the angle of incidence is somewhat larger than the Brewster angle, and collect a spectrum $I_{FZ}(v)$ with the thin double-side polished, float-zoned wafer (refer to ¶ 8.3) in the sample beam. Observe the magnitude of the interference fringes in the spectrum.

NOTE 2: If desired, the spectrum $I_{FZ}(v)$ can be ratioed with a background spectrum $I_0(v)$ collected with the sample beam empty.

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