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## Standard Test Method for Using Computer-Assisted Infrared Spectrophotometry to Measure the Interstitial Oxygen Content of Silicon Slices Polished on Both Sides<sup>1</sup>

This standard is issued under the fixed designation F 1189; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 This test method covers the determination of the interstitial oxygen content of single crystal silicon slices polished on both surfaces, using computer-assisted dispersive infrared (CA-DIR) or Fourier transform infrared (FT-IR) spectrophotometers. It is applicable to silicon slices whose thickness is between 0.3 and 2.5 mm, inclusive.

1.2 This test method can be applied to silicon slices with a resistivity greater than  $0.2 \Omega \cdot \text{cm}$  ( $n$ -type), or greater than  $1.0 \Omega \cdot \text{cm}$  ( $p$ -type).

1.3 The useful range of oxygen concentrations measurable by this test method is  $1 \times 10^{16}$  to  $2 \times 10^{18}$  atoms/cm<sup>3</sup>, depending on the thickness of the specimen. The lower limit is usually attainable for thicker specimens, while the higher limit is attainable for thinner specimens.

1.4 The oxygen concentration obtained using this test method assumes a linear relationship between the interstitial oxygen concentration and the absorption coefficient of the  $1107 \text{ cm}^{-1}$  band associated with interstitial oxygen in silicon.

1.5 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems 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:

- E 1 Specification for ASTM Thermometers<sup>2</sup>
- E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods<sup>3</sup>
- F 120 Practices for Infrared Absorption Analysis of Impurities in Single Crystal Semiconductor Materials<sup>4</sup>
- F 121 Test Method for Interstitial Atomic Oxygen Content of Silicon by Infrared Absorption<sup>4</sup>
- F 612 Practice for Cleaning Surfaces of Polished Silicon Slices<sup>4</sup>

### 3. Summary of Test Method

3.1 An infrared transmittance spectrum of a silicon slice,

which is polished on both sides, is obtained using either a computer-assisted dispersive infrared spectrometer or a Fourier transform infrared spectrometer. This transmittance spectrum is analyzed by a computer, following the algorithms defined in this test method, and the interstitial oxygen content of the silicon slice is determined.

### 4. Significance and Use

4.1 Measurement of the intensity of the  $1107 \text{ cm}^{-1}$  oxygen-in-silicon band with either a CA-DIR or FT-IR spectrophotometer enables the determination of the value of the absorption coefficient and, hence, the content of interstitial oxygen.

4.2 This test method is an automatic, nondestructive procedure for determining the interstitial oxygen content of silicon slices which are polished on both sides. It can be applied on a routine basis to large numbers of silicon wafers. Knowledge of the interstitial oxygen content of silicon wafers is necessary for materials acceptance and control of fabrication processes, as well as for research and development.

4.3 This test method is intended to be used with computer-assisted spectrophotometers; that is, the analysis procedures included in this test method are designed to be embodied in a computer program that can automatically calculate the interstitial oxygen content from the observed spectrum, or from spectra stored in memory. It is intended to be used in laboratories that measure the interstitial oxygen content of large numbers of silicon slices on a routine basis.

### 5. Interferences

5.1 The oxygen absorption band overlaps a silicon lattice band. The absorption due to the lattice band must be subtracted from the oxygen band. Since both the oxygen band and the lattice band can change with the specimen temperature, the temperature inside the spectrometer sample compartment must be maintained at  $27 \pm 5^\circ\text{C}$  during the measurement.

5.2 Free carrier absorption in  $n$ -type silicon with a resistivity below  $0.2 \Omega \cdot \text{cm}$ , or  $p$ -type silicon with a resistivity below  $1.0 \Omega \cdot \text{cm}$ , results in a nonlinear baseline for the infrared spectrum. In this case, the computation techniques used to calculate the net absorption coefficient are no longer applicable.

5.3 The presence of a high concentration of oxide precipitates, that result in absorbance bands at  $1230 \text{ cm}^{-1}$  or  $1073$

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee F-1 on Electronics and is the direct responsibility of Subcommittee F01.04 on Semiconductor Physical Properties.

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<sup>2</sup> Annual Book of ASTM Standards, Vol 14.03.

<sup>3</sup> Annual Book of ASTM Standards, Vol 14.02.

<sup>4</sup> Annual Book of ASTM Standards, Vol 10.05.

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$\text{cm}^{-1}$ , may lead to an error in the interstitial oxygen determination.

## 6. Apparatus

6.1 *Infrared Spectrophotometer*—Operates in the range from 800 to 2200  $\text{cm}^{-1}$ , with a resolution which can be set to 4  $\text{cm}^{-1}$  and 8  $\text{cm}^{-1}$  in the 1000 to 1300  $\text{cm}^{-1}$  range. Either a dispersive or a Fourier transform instrument may be used. Either type must have an associated computer which is used to store and manipulate the spectral data.

6.1.1 *Computer-Assisted Dispersive Infrared (CA-DIR) Spectrophotometer*—Use gratings, or prisms, or both, to obtain data as an amplitude-wavenumber (or wavelength) spectrum.

6.1.2 *Fourier Transform Infrared (FT-IR) Spectrophotometers*—Obtain data as an interferogram which 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 transform to obtain an amplitude-wavenumber (or wavelength) spectrum. All FT-IRs are computer equipped.

6.1.2.1 *Resolution*—Most FT-IR instruments will produce a spectrum with a 4  $\text{cm}^{-1}$  resolution by using an optical retardation in the interferometer of approximately 0.26 cm. For a typical FT-IR system, if the data points are collected at every other zero-crossing of the 633 nm He-Ne laser line, this optical retardation is achieved by collecting 4096 data points after the point of zero retardation. At 8  $\text{cm}^{-1}$  resolution, an optical retardation of 0.13 cm and 2048 data points are collected. When the FT-IR system collects a non-symmetrical interferogram, the number of data points collected before the point of zero retardation should be at least 260 but not greater than 500.

6.1.2.2 *Undersampling*—The data collection method should produce interferograms which, when zero-filled and Fourier transformed, will produce a spectrum containing data points approximately 1.9  $\text{cm}^{-1}$  apart in wavenumber at 4  $\text{cm}^{-1}$  resolution, or 3.8  $\text{cm}^{-1}$  apart at 8  $\text{cm}^{-1}$  resolution.

6.1.2.3 *Phase Correction*—The phase correction routine used during Fourier transformation should use at least 256 points on either side of the point of zero retardation in order to produce a phase array which can be used to eliminate phase errors.

6.2 *Equipment and Materials for Slicing and Polishing Crystals to a Thickness Uniformity of 1.0 % or Less and a Surface Flatness Equal to 5  $\mu\text{m}$ .*

6.3 *Thickness Gauge*—or other instrument suitable for measurement of the thickness of a silicon slice to a tolerance of  $\pm 2.5 \mu\text{m}$ .

6.4 *Thermometer*—or other device capable of measuring temperatures in the range from 10 to 50°C to within 1°C, such as 91C, 49C or 98C or equivalent, as described in Specification E 1.

## 7. Test Specimens

7.1 The silicon slices must be between 0.3 and 2.5 mm thick.

7.2 Both surfaces of the silicon slice must be polished in accordance with the Apparatus Section of Practice F 120. The surface must be flat to within 5  $\mu\text{m}$  over the spectrophotometer beam diameter.

7.3 Silicon slices that have undergone high temperature heat treatments may have an oxide film on their surfaces. This film must be completely removed prior to the IR measurement.

7.4 The surfaces of the silicon specimen should be visibly free of contamination or surface oxide films and cleaned within 2 weeks prior to the test (using, for example, the procedures described in Practice F 612).

## 8. Calibration and Standardization

8.1 Determine the short term stability of the spectrophotometer, over a time interval,  $T$ , by the following procedure:

8.1.1 Collect a spectrum without and with a silicon sample in place.

8.1.2 Wait for the time interval,  $T$ , representative of the time required to measure the oxygen content of a set of samples.

8.1.3 Collect another spectrum without and with a silicon sample in place.

8.1.4 Subtract, in units of absorbance (that is, the negative logarithm of the transmittance), the intensities of the two spectra taken without the sample in place, for the wavenumber range from 800 to 1500  $\text{cm}^{-1}$ . Repeat this procedure for the two spectra taken with the silicon sample in place.

8.1.5 Neither of the above difference spectra should deviate from 0 by more than  $\pm 0.01$  absorbance units at any wavenumber within the range from 800 to 1500  $\text{cm}^{-1}$ .

8.2 Verify a proper purge condition for the specimen chamber by monitoring water vapor bands. The water vapor can be monitored at 1521  $\text{cm}^{-1}$ . The instrument should be sufficiently well purged or evacuated so that the absorbance at this location is less than 0.2 absorbance units.

8.3 Under certain conditions, the spectrophotometer may have a non-linear response. Place a flat, double-side polished and high resistivity (greater than 5  $\Omega \cdot \text{cm}$ ) silicon slice in the instrument. In some instruments, this silicon slice may have to be placed so that its normal is at a small angle (not to exceed 10°) to the axis of the incoming beam, in order to minimize undesirable reflections between the silicon surfaces, and the spectrometer components. Initially determine this angle by placing the silicon slice normal to the axis of the IR beam, and then gradually tilting the sample while repeatedly obtaining the transmittance spectrum of the slice above 1600  $\text{cm}^{-1}$ . The optimum angle is reached when a flat baseline, as close as possible to 53.8 % is obtained. The baseline transmittance at 2000  $\text{cm}^{-1}$  must be  $53.8 \pm 2 \%$ .

NOTE 1—Recent experimental evidence, using equivalent samples of different thicknesses, suggests that under some circumstances, even when the  $\pm 2 \%$  criterion is met, system nonlinearities may still affect the results obtained.

## 9. Procedure

9.1 Position the silicon slice in the sample beam of the spectrometer at the optimum angle determined in 8.3. Note that this angle must not exceed 10°.

9.2 *Resolution*—Set the resolution of the instrument to 4  $\text{cm}^{-1}$  for silicon slices with thicknesses between 0.4 and 2.5 mm, inclusive. Set the resolution of the instrument to 8  $\text{cm}^{-1}$  for silicon slices between 0.3 and 0.4 mm.