



# Standard Test Method for Measuring Oxygen Concentration in Heavily Doped Silicon Substrates by Secondary Ion Mass Spectrometry<sup>1</sup>

This standard is issued under the fixed designation F 1366; 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.

<sup>ε1</sup> NOTE—Keywords were added editorially in April 1998.

## 1. Scope

1.1 This test method covers the determination of total oxygen concentration in the bulk of single crystal silicon substrates using secondary ion mass spectrometry (SIMS).

1.2 This test method can be used for silicon in which the dopant concentrations are less than 0.2 % ( $1 \times 10^{20}$  atoms/cm<sup>3</sup>) for boron, antimony, arsenic, and phosphorus (see Test Method F 723). This test method is especially applicable for silicon that has resistivity between 0.0012 and 1.0  $\Omega$ -cm for *p*-type silicon and between 0.008 and 0.2  $\Omega$ -cm for *n*-type silicon (see Test Methods F 43).

1.3 This test method can be used for silicon in which the oxygen content is greater than the SIMS instrumental oxygen background as measured in a float zone silicon sample, but the test method has a useful precision especially when the oxygen content is much greater (approximately 10 $\times$  to 20 $\times$ ) than the measured oxygen background in the float zone silicon.

1.4 This test method is complementary to infrared absorption spectroscopy that can be used for the measurement of interstitial oxygen in silicon that has resistivity greater than 1.0  $\Omega$ -cm for *p*-type silicon and greater than 0.1  $\Omega$ -cm for *n*-type silicon (see Test Method F 1188). The infrared absorption measurement can be extended to between 0.02 and 0.1  $\Omega$ -cm for *n*-type silicon with minor changes in the measurement procedure.<sup>2</sup>

1.5 In principle, different sample surfaces can be used, but the precision estimate was taken from data on chemical-mechanical polished surfaces.

1.6 *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:

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee F-01 on Electronics and is the direct responsibility of Subcommittee F01.06 on Silicon Materials and Process Control.

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<sup>2</sup> Hill, D. E., "Determination of Interstitial Oxygen Concentration in Low-Resistivity *n*-type Silicon Wafers by Infrared Absorption Measurements," *Journal of the Electrochemical Society*, Vol 137, 1990, p. 3926.

E 122 Practice for Choice of Sample Size to Estimate a Measure of Quality for a Lot or Process<sup>3</sup>

F 43 Test Methods for Resistivity of Semiconductor Materials<sup>4</sup>

F 723 Practice for Conversion Between Resistivity and Dopant Density for Arsenic-Doped, Boron-Doped, and Phosphorus-Doped Silicon<sup>4</sup>

F 1188 Test Method for Interstitial Atomic Oxygen Content of Silicon by Infrared Absorption<sup>4</sup>

## 3. Terminology

### 3.1 Definitions of Terms Specific to This Standard:

3.1.1 *ion mass spectrometry*—the separation and counting of ions by their mass-to-charge ratio.

3.1.2 *primary ions*—ions created and focussed by an ion gun onto the specimen surface to sputter ionize surface atoms.

3.1.3 *secondary ions*—ions that leave the specimen surface as a result of the primary ion beam sputter ionizing the specimen surface atoms.

3.1.4 *secondary ion mass spectrometry*—mass spectrometry performed upon secondary ions from the specimen surface.

## 4. Summary of Test Method

4.1 SIMS is utilized to determine the bulk concentration of oxygen in single crystal silicon substrate. Specimens of single crystal silicon (one float-zone silicon specimen, two calibration specimens, and the test specimen) are loaded into a sample holder. The holder with the specimens is baked at 100°C in air for 1 h and then transferred into the analysis chamber of the SIMS instrument. A cesium primary ion beam is used to bombard each specimen. The negative secondary ions are mass analyzed. The specimens are presputtered sequentially to reduce the instrumental oxygen background. The specimens are then analyzed, in locations different from the presputtering locations, for oxygen and silicon in a sequential manner throughout the holder. Three measurement passes are made through the holder. The ratio of the measured oxygen and silicon secondary ion intensities ( $O^-/Si^-$ ) is calculated for each specimen. The relative standard deviation (RSD) of the ratio is

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

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

then calculated for each specimen. If any specimen other than the float zone specimen has a RSD of the ratio greater than 3 %, more analyses are performed. The SIMS average O/Si ratios are then converted to infrared-equivalent concentrations by utilizing either the load-line calibration method<sup>5,6</sup> or the load factor calibration method<sup>6</sup> with the calibration specimens in the load.

## 5. Significance and Use

5.1 SIMS can measure the oxygen concentration in heavily-doped silicon substrates used for epitaxial silicon where the free carrier concentration obscures the infrared absorption and prevents the normal use of the infrared measurement as a characterization technique for the commercial production of silicon.

5.2 The SIMS measurement allows for the production of controlled oxygen content in heavily-doped silicon crystals.

5.3 This test method can be used for process control, research and development, and materials acceptance purposes.

## 6. Interferences

6.1 Oxygen from silicon oxide, carbon oxide, and water on the surface can interfere with the oxygen measurement.

6.2 Oxygen adsorbed from the SIMS instrument chamber to the surface can interfere with the oxygen measurement.

6.3 There are no effects upon the oxygen ion yield from the dopants for dopant densities less than  $1 \times 10^{20}$  atoms/cm<sup>3</sup>.<sup>7</sup>

6.4 The SIMS oxygen instrumental background as measured on the float zone silicon specimen should be as low as possible and stable before the analyses are begun.

6.5 The specimen surface must be flat in the specimen holder windows so that the inclination of the specimen surface with respect to the ion collection optics is constant from specimen to specimen. Otherwise, the accuracy and precision can be degraded.

6.6 The accuracy and precision of the measurement significantly degrade as the roughness of the specimen surface increases. This degradation can be avoided by using chemical-mechanical polished surfaces.

6.7 Variability of oxygen in the calibration standards can limit the measurement precision.

6.8 Bias in the assigned oxygen of the calibration standards can introduce bias into the SIMS measured oxygen.

## 7. Apparatus

7.1 *SIMS Instrument*, equipped with a cesium primary ion source, electron multiplier detector and Faraday cup detector, and capable of measuring negative secondary ions.

<sup>5</sup> Goldstein, M., and Makovsky, J., "The Calibration and Reproducibility of Oxygen Concentration in Silicon Measurements Using SIMS Characterization Techniques," *Semiconductor Fabrication: Technology and Metrology*, ASTM STP 990, Dinesh C. Gupta, Ed., ASTM, 1988, pp. 350–360.

<sup>6</sup> Makovsky, J., Goldstein, M., and Chu, P., "Progress in the 'Load Line Calibration' Method for Quantitative Determination of [O] in Silicon by SIMS," *Seventh International Meeting on Secondary Ion Mass Spectrometry, SIMS VII*, John Wiley and Sons, 1990, p. 487.

<sup>7</sup> Bleiler, R. J., Chu, P. K., Novak, S. W., and Wilson, R. G., "Study of Possible Matrix Effects in the Quantitative Determination of Oxygen in Heavily Doped Czochralski Silicon Crystals," *Seventh International Meeting on Secondary Ion Mass Spectrometry, SIMS VII*, John Wiley and Sons, 1990, p. 507.

7.1.1 The SIMS instrument should be adequately prepared (that is, baked) so as to provide the lowest possible instrumental background.

7.2 *Cryopanel*, liquid nitrogen- or liquid helium-cooled, which surrounds the test specimen holder in the analysis chamber.

7.3 *Test Specimen Holder*.

7.4 *Oven*, for baking test specimen holder.

## 8. Sampling

8.1 Since this procedure is destructive in nature, a sampling procedure must be used to evaluate the characteristics of a group of silicon wafers. No general sampling procedure is included as part of this test method, because the most suitable sampling plan will vary considerably depending upon individual conditions. For referee purposes, a sampling plan shall be agreed upon before conducting the test. See Practice E 122 for suggested choices of sampling plans.

## 9. Specimen Requirements

9.1 Sample specimens must be flat and smooth on the side used for analysis.

9.2 Sample specimens must be cleaved or diced to fit within the sample specimen holder.

## 10. Calibration

10.1 The two calibration standards in each load must be lightly doped Czochralski silicon<sup>8</sup> in which the oxygen concentration is measured by infrared absorption spectroscopy (see Test Method F 1188), and the measured values of the two standards bracket the expected values for the test specimen (that is, one calibration standard is higher in oxygen and one is lower, compared to the expected value in the test specimen).

10.2 The calibration standards must be measured by infrared absorption to determine the concentration and homogeneity of the oxygen within the standards; each standard is assigned the averaged infrared absorption oxygen value for the substrate.

10.3 Calibration standards that are included in the SIMS analyses must be taken from that portion of the wafer that provided a homogeneous measurement in the Fourier transform infrared spectrophotometer, (FT-IR) analysis; this portion is typically the central portion of the wafer.

10.4 Each calibration standard specimen must be the same size and have the same polished surface as the test specimen.

10.5 The float zone specimens that are included in the SIMS analysis to measure the instrumental oxygen background must be measured by infrared absorption to determine if the oxygen concentration is low enough to measure the instrumental SIMS background. Oxygen concentrations below 0.5 ppma (see Test Method F 1188) in the float zone specimen are normally sufficient.

10.6 Each float zone specimen must be the same size and have the same polished surface as the test specimen.

## 11. Procedure

### 11.1 Specimen Loading:

<sup>8</sup> Czochralski silicon is available from most silicon substrate suppliers.