



Designation: F 1528 – 94 (Reapproved 1999)

Standard Test Method for Measuring Boron Contamination in Heavily Doped N-Type Silicon Substrates by Secondary Ion Mass Spectrometry¹

This standard is issued under the fixed designation F 1528; 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 (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of total trace boron contamination in the bulk of single crystal, heavily doped *n*-type 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 by 10^{20} atoms/cm³) for antimony, arsenic or phosphorus. This test method is especially applicable for silicon where boron, the *p*-type dopant, is an unintentional contaminant at trace levels (<5 by 10^{14} atoms/cm³).

1.3 This test method can be used for silicon in which the boron contamination is greater than two times the SIMS detection limits that is approximately between 5 by 10^{12} atoms/cm³ and 5 by 10^{13} atoms/cm³ depending upon the instrumentation type described herein.

1.4 In principle, different sample surfaces can be used, but the precision estimate was taken from data on polished etched surfaces.

1.5 *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:*

E 122 Practice for Choice of Sample Size to Estimate a Measure of Quality of a Lot or Process²

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 Secondary ion mass spectrometry (SIMS) is utilized to determine the bulk contamination of boron in single crystal, heavily doped *n*-type silicon substrates. Specimens of single crystal silicon (one silicon specimen with very low boron concentration, such as a high resistivity *n*-type float-zone silicon specimen, to be used as an instrumental BLANK; one calibration specimen made of a bulk-doped boron silicon wafer; and the test specimens) are loaded into a sample holder. An oxygen primary ion beam is used to bombard each specimen. The positive secondary ions are mass analyzed. The BLANK silicon sample is sputtered to achieve instrumental background. The samples are then analyzed for boron and silicon in a sequential manner throughout the holder. The ratio of the measured boron and silicon secondary ion intensities (B^+/S^+) is calculated for each specimen. The (B^+/Si^+) ratios of the test specimens are then converted to boron concentrations by linear scaling from the (B^+/Si^+) ratio of the calibration specimen that has a known boron concentration. No crater measurement is required.

5. Significance and Use

5.1 SIMS can measure the boron contamination in heavily-doped *n*-type substrates where this contamination can result in autodoping at the epitaxial silicon-substrate interface.

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

6. Interferences

6.1 Boron adsorbed on the surface can interfere with the boron measurement.

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

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² *Annual Book of ASTM Standards*, Vol 14.02.



6.2 Boron adsorbed from the SIMS instrument chamber to the surface can interfere with the boron measurement.

6.3 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.

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

6.5 Variability of boron in the calibration standards can limit the measurement precision.

6.6 Bias in the assigned boron concentration of the calibration standard can introduce bias into the SIMS measured boron.

7. Apparatus

7.1 *Magnetic Sector SIMS Instrument*, equipped with an oxygen primary ion source, electron multiplier detector, and Faraday cup detector or daley detector capable of measuring positive secondary ions. The SIMS instrument should be adequately prepared (that is, baked) so as to provide the lowest possible instrumental background.

7.2 *Liquid Nitrogen or Liquid Helium Cooled Cryopanel*, that surrounds the test specimen holder in the analysis chamber if the analysis chamber vacuum is 10^{-8} torr or higher. For instruments with vacuum less than 10^{-8} torr the cooling is not required.

7.3 *Test Specimen Holder*—A test specimen holder that keeps the specimen(s) analysis surface planar and perpendicular to the several kV extraction field depending on the instrument type.

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. The surface shall have a polish etch surface or better (that is, chem-mechanically polished).

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

9.3 A bulk boron-doped silicon calibration specimen is required.

9.4 A BLANK silicon specimen with boron concentration below $5 \text{ by } 10^{12} \text{ atoms/cm}^3$ must be present.

10. Calibration

10.1 The calibration standard must be present with bulk boron concentration between 1 and $10 \text{ by } 10^{14} \text{ atoms/cm}^3$ as determined by some other measurement that is agreed upon by the parties.

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

10.3 Each BLANK specimen must be the same size and have the same polished surface as the test specimen.

11. Procedure

11.1 Specimen Loading:

11.1.1 Load the specimens into the SIMS sample holder checking to see that the specimens are flat against the backs of the windows and cover the windows as much as is possible. A specimen load includes: one BLANK silicon specimen, one calibration specimen, and the test specimens.

11.2 Instrument Tuning:

11.2.1 Turn on the instrument in accordance with the manufacturer's instructions.

11.2.2 Fill the liquid nitrogen or helium cold trap if cooling equipment is used as stated in 7.2.

11.2.3 Analytical Conditions:

11.2.3.1 Use a focused oxygen primary-ion current and adjust the contrast diaphragm and field aperture to maximize the $^{30}\text{Si}^+$ ion count rate. The rate must be greater than $1 \text{ by } 10^8$ counts/s as measured on the Faraday cup or daley detector, and with no raster.

11.2.3.2 Typical first raster test conditions are several hundred microns by several hundred microns depending upon the beam radius, (typical condition is $250 \text{ by } 250\text{-}\mu\text{m}$ raster) to remove surface boron in the native oxide. A second raster test condition is reduced several times from the first raster condition. A typical second raster condition is $50 \text{ by } 50 \mu\text{m}$ for the actual measurement. Integrations are 1 s.

11.3 Analysis of Specimen:

11.3.1 Position the specimen holder such that the sputtered crater in the specimen will form near the center of the window.

11.3.2 Center the primary ion beam and begin a SIMS profile. Sputter the sample using a first raster condition for 50 to 100 cycles of the magnet until the boron intensity is stable. This step is to remove any residual surface contamination found typically in the native oxide of all wafers. Decrease the rastered area to a second raster condition and continue sputtering until the boron signal is stable.

11.3.3 At the end of the profile measure and record the $^{11}\text{B}^+$ count rate on the electron multiplier detector and the $^{30}\text{Si}^+$ matrix intensity on the Faraday cup detector or daley detector, averaged over the last 15 cycles.

11.3.4 Repeat the above steps for all the specimens in the holder until all the specimens have been analyzed.

11.3.5 Calculate the ratio ($^{11}\text{B}^+/\text{Si}^+$) of boron count rate to silicon intensity using the recorded secondary ion intensities at the end of each profile.

11.3.6 If the measured ratio ($^{11}\text{B}^+/\text{Si}^+$) for the BLANK specimen exceeds 20 to 50 % times the ratio ($^{11}\text{B}^+/\text{Si}^+$) of the other specimens, abort the analysis and find the cause of the high instrumental background.

