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Surface chemical analysis — Secondary-ion mass spectrometry — Determination of boron atomic concentration in silicon using uniformly doped materials

Analyse chimique des surfaces — Spectrométrie de masse des ions secondaires — Dosage des atomes de bore dans le silicium à l'aide de matériaux dopés uniformément

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Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 14237 was prepared by Technical Committee ISO/TC 201, *Surface chemical analysis*, Subcommittee SC 6, *Secondary ion mass spectrometry*.

This second edition cancels and replaces the first edition (ISO 14237:2000), which has been technically revised. The revision includes, in particular, the replacement of old Annex D concerning procedures for the depth profiling of NIST standard reference material SRM 2137 by references to ISO 17560 and ISO 18114.

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Introduction

This International Standard was prepared for the determination by secondary ion mass spectrometry (SIMS) of boron atomic concentrations in uniformly doped silicon wafers.

SIMS needs reference materials to perform quantitative analyses. Certified reference materials are only available for limited matrix-impurity combinations, and they are costly. SIMS inevitably consumes these reference materials at every measurement. Thus, secondary reference materials which can be prepared by each laboratory and calibrated using a certified reference material are useful for daily analyses.

In this International Standard, a standard procedure is described for boron quantitative analysis in single-crystalline silicon using secondary reference materials calibrated by a certified reference material implanted with boron.

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Surface chemical analysis — Secondary-ion mass spectrometry — Determination of boron atomic concentration in silicon using uniformly doped materials

1 Scope

This International Standard specifies a secondary-ion mass spectrometric method for the determination of boron atomic concentration in single-crystalline silicon using uniformly doped materials calibrated by a certified reference material implanted with boron. This method is applicable to uniformly doped boron in the concentration range from 1×10^{16} atoms/cm^3 to 1×10^{20} atoms/cm^3.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 17560, *Surface chemical analysis — Secondary-ion mass spectrometry — Method for depth profiling of boron in silicon*

ISO 18114, *Surface chemical analysis — Secondary-ion mass spectrometry — Determination of relative sensitivity factors from ion-implanted reference materials*

3 Principle

An oxygen-ion beam or a caesium-ion beam is impinged onto the sample surface and the emitted secondary ions of boron and silicon are mass-analysed and detected.

Uniformly doped secondary reference materials are calibrated by using an ion-implanted primary reference material and are used as working reference materials.

4 Reference materials

4.1 Primary reference material

A primary reference material is used for the determination of the boron atomic concentration of the secondary reference materials. The primary reference material shall be a certified reference material (CRM) of silicon implanted with boron.

NOTE At the time of publication of this International Standard, NIST standard reference material SRM 2137 (referred to hereinafter as NIST SRM) was the only ion-implanted CRM of boron in silicon.

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4.2 Secondary reference materials

4.2.1 Secondary reference materials are used for the determination of boron atomic concentrations in test specimens. At least one boron-doped reference material together with one non-boron-doped reference material shall be used for daily analysis. Two other different boron-doping levels are recommended to be used to confirm the performance of the apparatus (see Annex C).

4.2.2 The secondary reference materials (referred to hereinafter as bulk RMs) shall be single-crystal silicon wafers or epitaxial silicon wafers with a ca. 100 μm thick epitaxial layer, and shall be uniformly doped with natural-isotopic boron.

4.2.3 Boron-doped wafers with boron atomic concentrations between 1×10^{16} atoms/cm^3 and 1×10^{20} atoms/cm^3 shall be obtained. It is recommended that the three doping levels given in Table 1 are used. When only one level is used, RM-B or RM-C should be chosen. A non-boron-doped wafer shall be obtained for background checking.

Wafers with small boron concentration gradients shall be selected. The boron concentration gradient shall be less than 5 % per cm.

NOTE Approximate boron atomic concentrations can be determined as carrier densities from the resistivity of the wafers. The resistivity measurement procedures and the procedure for converting between resistivity and carrier density are presented in Annex A.

Table 1 — Bulk reference materials

Name	Boron doping level atoms/cm^3	
RM-A	low	1×10^{16} to 1×10^{17}
RM-B	middle	5×10^{17} to 5×10^{18}
RM-C	high	1×10^{19} to 1×10^{20}
RM-BG	none	$< 1 \times 10^{14}$

4.2.4 The isotope ratio of ^{11}B to ^{10}B in the bulk RM chosen in 4.2.3 shall be determined by one of following methods.

a) The isotope ratio shall be evaluated by a magnetic-sector SIMS instrument detecting BSi^- ions. The measurement procedure stipulated in 7.5.2 shall be used for this purpose.

b) The bulk RM shall be assumed to have the accepted nominal natural isotopic composition of 19,9 atomic percent ^{10}B and 80,1 atomic percent ^{11}B, i.e. a ratio of ^{11}B atoms to ^{10}B atoms of 4,025. The boron isotope ratio in a specific material, however, can have ± 5 % deviation from the natural isotope ratio.

NOTE SIMS will generally measure a deviated isotope ratio depending on the type of instrument and the detected ions. The deviation is smaller between $^{10}B^{28}Si^-$ and $^{11}B^{28}Si^-$ than between $^{10}B^+$ and $^{11}B^+$ in a magnetic-sector mass spectrometer (see Annex B).

5 Apparatus

Secondary-ion mass spectrometry apparatus equipped with an oxygen-ion source and/or a caesium-ion source shall be used.

Whenever the apparatus performance is required to be confirmed, the procedures stipulated in Annex C shall be carried out. The procedures for linearity of measurement stipulated in Clause C.6 can be replaced by local documented procedures.

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6 Specimen

The analysed specimen shall have a mirror-polished surface. The specimen shall be cut into an appropriate size for analysis and further degreased and washed if necessary.

7 Procedure

7.1 Adjustment of secondary-ion mass spectrometer

For oxygen-ion beam use, see Table 2. For caesium-ion beam use, see Table 3. Other conditions not shown here shall be set in accordance with the manufacturer's instructions or a local documented procedure.

Table 2 — Measurement conditions for oxygen-ion beam

Element	Characteristic
Primary-ion species	O_2^+
Secondary-ion polarity	Positive
Analysed area	$> 100\ \mu m^2$
Primary-ion scan area	4 times the analysed area or larger

Table 3 — Measurement conditions for caesium-ion beam

Element	Characteristic
Primary-ion species	Cs^+
Secondary-ion polarity	Negative
Analysed area	$> 100\ \mu m^2$
Primary-ion scan area	4 times the analysed area or larger

7.2 Optimizing the secondary-ion mass spectrometer settings

7.2.1 Set the required instrument parameters and align the ion optics in accordance with the manufacturer's instructions or a local documented procedure.

7.2.2 Ensure the stability of the primary-ion current and the mass spectrometer in accordance with the manufacturer's instructions or a local documented procedure.

7.3 Specimen introduction

Immediately prior to introducing the specimens into the SIMS apparatus, dust particles shall be removed from the surfaces with a pressurized duster. After introducing the specimens into the analysis chamber, analysis shall not start until the pressure has recovered to the normal value recommended by the manufacturer or a local documented procedure.

Residual gases in the analysis chamber can produce a $^{10}B^{28}Si^{1}H^{-}$ background signal which interferes in $^{11}B^{28}Si^{-}$ detection, so care should be taken to obtain a better vacuum condition when a caesium-ion beam is used.

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7.4 Detected ions

7.4.1 When an oxygen-ion beam is used, both $^{10}B^+$ and $^{11}B^+$ shall be detected as secondary-ion species of boron. When a caesium-ion beam is used, both $^{10}B^{28}Si^-$ and $^{11}B^{28}Si^-$ shall be detected as secondary-ion species of boron.

7.4.2 The ion species of silicon which has an appropriate ion intensity shall be detected, following the manufacturer's instructions or a local documented procedure.

If the instrument has an electrometer detection mode, it is recommended that $^{28}Si^+$ be detected as the reference ion of B^+ using the electrometer. For the pulse-counting mode, the silicon-ion intensity should be less than 1×10^5 counts/s. For BSi^- detection, Si_2^- is preferable as the reference ion.

7.5 Calibration

7.5.1 Measurement procedure for CRM

7.5.1.1 The depth profile of boron (either ^{10}B or ^{11}B) in the CRM shall be measured using the same conditions as those for the bulk RMs on the same day as the bulk RM measurements, following the procedures stipulated in ISO 17560. The mean integrated ion intensity ratio of the CRM, A^{imp}, shall be calculated following the procedures stipulated in ISO 18114.

7.5.1.2 The relative-sensitivity factor of the CRM shall be obtained from the following formula:

$$\text{RSF}^{\text{imp}} = \frac{\Phi}{A^{\text{imp}}}$$

where

RSF^{imp} is the isotopic relative-sensitivity factor obtained from the CRM;

Φ is the implanted boron (either ^{10}B or ^{11}B) dose of the CRM.

7.5.2 Measurement procedure for bulk RMs

7.5.2.1 Measurements shall be made from the central region of the specimen holder window. When the boron-ion intensity of the bulk RM is high, care shall be taken so as not to saturate the detector. If the boron-ion intensity is higher than 1×10^5 counts/s, the primary-ion intensity shall be reduced.

7.5.2.2 Depth profiles of boron and silicon shall be measured for all the bulk RMs. The data sampling described below shall start after any surface contamination is removed and the secondary-ion intensities reach stationary values, but shall be concluded before a change in secondary-ion intensity occurs due to surface roughening induced by ion bombardment.

7.5.2.3 The secondary-ion intensities of boron and silicon shall be measured for at least 10 cycles alternately, for at least 1 s for each boron isotope per cycle, at the same analysis position. This procedure shall be repeated three times at different positions on the same specimen surface. Then another specimen shall be measured.

If the variation of silicon-ion intensity for one measurement point is less than the value guaranteed by the manufacturer or that determined to be acceptable by local documented procedures, it can be regarded as constant. In this case, it is not necessary to measure the silicon-ion intensity cycle by cycle. It can be measured at any one cycle for each analysis position.

7.5.2.4 The detected secondary-ion intensity of boron in RM-BG shall be used as the background level of the analysis.

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7.5.2.5 Ion intensity ratios of boron to silicon for each bulk RM shall be determined for each measurement cycle by cycle at one measurement position, and then a mean value for all the measurement cycles shall be calculated, and the mean value obtained further averaged for three measurement positions, using the following formulae:

$$J_{i,j}^{11} = \frac{I_{i,j}^{11}}{I_{i,j}^{\text{Si}}}$$

$$J^{11} = \frac{1}{3}\sum_{j=1}^{3}\left(\frac{1}{n}\sum_{i=1}^{n} J_{i,j}^{11}\right)$$

where

$I_{i,j}^{11}$ and $I_{i,j}^{\text{Si}}$ are the ^{11}B-ion intensity and the silicon-ion intensity in each RM, respectively, at measurement cycle i and measurement position j;

J^{11} is the mean ion intensity ratio for ^{11}B in each bulk RM;

n is the total number of measurement cycles for each bulk RM.

The same procedure shall be used to determine the mean intensity ratio, J^{10}, for ^{10}B.

7.5.2.6 The experimental boron isotope ratio for the SIMS instrument shall be determined using one of the bulk RMs. Since there is a possible mass-spectral interference between $^{10}B^{+}$ and $^{30}Si^{3+}$ that may be significant for lower boron atomic concentration specimens, it is recommended that a bulk RM be used which has a boron atomic concentration greater than 1×10^{17} atoms/cm^3 with a known isotope ratio. The measured isotope ratio shall be calculated using the following formula:

$$\alpha = \frac{J^{11} - J_{\text{BG}}^{11}}{J^{10} - J_{\text{BG}}^{10}}$$

where

α is the measured isotope ratio of ^{11}B to ^{10}B;

J_{BG}^{11} and J_{BG}^{10} are the mean background ion intensity ratios for ^{11}B and ^{10}B, respectively, derived from RM-BG.

A correction factor for the measured isotope ratio shall be determined using the following formula:

$$\delta = \frac{\alpha_0}{\alpha}$$

where α_0 is the actual isotope ratio in the bulk RM. If α_0 is not known, the natural isotope ratio, $\alpha_0 = 4{,}025$ (see 4.2.4) shall be used.

δ shall be used to correct the experimental mass discrimination between ^{10}B and ^{11}B.

7.5.3 Calibration of bulk RMs

The value of RSF$^{\text{imp}}$ obtained in 7.5.1 shall be used as the calibration relative-sensitivity factor. The ^{11}B atomic concentration in each bulk RM shall be calibrated using the calibration relative-sensitivity factor.

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If the CRM is implanted with ^{10}B, the mass discrimination correction factor obtained in 7.5.2 shall be used:

$$C_k^{11\mathrm{cal}} = \mathrm{RSF}^{\mathrm{imp}} \delta (J_k^{11} - J_{\mathrm{BG}}^{11})$$

where

$C_k^{11\mathrm{cal}}$ is the calibrated ^{11}B atomic concentration in each bulk RM;

J_k^{11} is the mean ion intensity ratio for ^{11}B in each bulk RM.

If the CRM is implanted with ^{11}B, mass discrimination correction is not necessary:

$$C_k^{11\mathrm{cal}} = \mathrm{RSF}^{\mathrm{imp}} (J_k^{11} - J_{\mathrm{BG}}^{11})$$

7.6 Measurement of test specimen

7.6.1 Measurement procedure

Test specimens shall be measured under the same conditions as stipulated in 7.5.2.

Ion intensity ratios of boron to silicon shall be determined for each measurement cycle by cycle at one measurement position, and then a mean value for all the measurement cycles shall be calculated. The mean value obtained shall be further averaged for three measurement positions.

7.6.2 Determination of working relative-sensitivity factor

7.6.2.1 Use one of the previously calibrated bulk RMs to determine the working relative-sensitivity factor and the mass discrimination correction factor for the test specimen measurement. It is recommended that the bulk RM be selected whose boron-ion intensity is as close to those in the test specimens as possible. Use the calibrated boron atomic concentration determined in 7.5.3 as the reference value.

The use of RM-A is not recommended when boron-ion intensities for the sample are lower than 1×10^2 counts/s.

7.6.2.2 The bulk RM chosen and RM-BG shall be measured under the same conditions as the test specimens on the same day, following the procedures stipulated in 7.5.2.

Ion intensity ratios of each boron isotope to silicon shall be determined for each measurement cycle by cycle at one measurement position, and then a mean value for all the measurement cycles shall be calculated. The mean value obtained shall be further averaged for three measurement positions.

8 Expression of results

8.1 Method of calculation

8.1.1 The working relative-sensitivity factor shall be obtained from the following formula:

$$\mathrm{RSF}^{\mathrm{work}} = \frac{C_m^{11\mathrm{cal}}}{J_m^{11} - J_{\mathrm{BG}}^{11}}$$

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where

RSF$^{\text{work}}$ is the working relative-sensitivity factor obtained from the bulk RM chosen;

$C_m^{11\text{cal}}$ is the calibrated ^{11}B atomic concentration in the bulk RM chosen;

J_m^{11} is the mean ion intensity ratio for ^{11}B in the bulk RM chosen;

J_{BG}^{11} is the mean background ion intensity ratio for ^{11}B derived from the RM-BG.

8.1.2 The mass discrimination correction factor for the test specimen measurement, δ_m, shall be determined using the following formula:

$$\delta_m = \frac{\alpha_{0m}}{\alpha_m} = \alpha_{0m} \frac{J_m^{10} - J_{\text{BG}}^{10}}{J_m^{11} - J_{\text{BG}}^{11}}$$

where

α_{0m} is the actual isotope ratio in the bulk RM chosen;

J_m^{10} is the mean ion intensity ratio for ^{10}B in the bulk RM chosen;

J_{BG}^{10} is the mean background ion intensity ratio for ^{10}B derived from the RM-BG.

8.1.3 The boron atomic concentration in the test specimens shall be determined from the following formulae, using the working relative-sensitivity factor:

$$C^{11} = \text{RSF}^{\text{work}} (J_t^{11} - J_{\text{BG}}^{11})$$

$$C^{10} = \frac{\text{RSF}^{\text{work}} (J_t^{10} - J_{\text{BG}}^{10})}{\delta_m}$$

$$C = C^{11} + C^{10}$$

where

C^{11} and C^{10} are, respectively, the ^{11}B and ^{10}B atomic concentrations in the test specimens;

C is the total boron atomic concentration in the test specimens;

J_t^{11} and J_t^{10} are, respectively, the mean ion intensity ratios for ^{11}B and ^{10}B in the test specimens.

8.2 Precision

This International Standard was subjected to an interlaboratory test programme involving 12 laboratories in four countries. Three bulk RMs were analysed to cover the scope of the method.

Repeatability and reproducibility were calculated in accordance with the principles of ISO 5725-2.

A statistical report of the interlaboratory test programme is given in Annex D.

It should be noted that the reproducibility data may include errors due to any inhomogeneity of the specimens.

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9 Test report

The test report shall include the following information:

a) all information necessary for the identification of the specimens, the apparatus, the laboratory and the date of analysis;

b) the CRM and the bulk RMs used, as specified in this International Standard;

c) information about isotope ratio correction, as specified in this International Standard;

d) information about apparatus performance and the method of linearity evaluation used, as specified in this International Standard, if apparatus performance was required to be confirmed;

e) the results and the form in which they are expressed;

f) any unusual features noted during the analyses;

g) any operation not specified in this International Standard, as well as any optional operation which may have influenced the results.

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Annex A
(informative)

Determination of carrier density in silicon wafer

A.1 General

This annex gives information on SEMI standards for the determination of boron atomic concentrations in silicon wafers used as the secondary reference materials (see 4.2).

A.2 Determination of carrier density in silicon bulk samples

Carrier densities in bulk silicon crystals can be directly determined by capacitance-voltage measurements with a mercury probe[1]. Application of the method is limited, however, because a Schottky junction is required. Also, applicable carrier densities are limited to 4×10^{13} cm^{-3} to 8×10^{16} cm^{-3}.

Consequently, resistivity is generally measured and converted into the carrier density in the whole resistivity range. SEMI MF43[2] and SEMI MF84[3] are standard methods for measuring resistivity, while SEMI MF723[4] is a standard practice for conversion between resistivity and carrier density. In general, resistivity is measured using SEMI MF84, making reference to SEMI MF43, and is converted into carrier density in accordance with SEMI MF723.

A.3 Determination of carrier density in epitaxial silicon layers

A.3.1 Direct determination of carrier density

Carrier density in epitaxial silicon layers can be directly determined by capacitance-voltage measurements with a mercury probe, as described in SEMI MF1392[1]. However, its applicable range is limited to carrier densities ranging from 4×10^{13} cm^{-3} to 8×10^{16} cm^{-3} due to Schottky junction formation or specimen preparation.

A.3.2 Conversion from resistivity

The resistivity of epitaxial silicon layers can be converted to carrier density. The resistivity of epitaxial layers is generally obtained by growing a desired conductive-type epitaxial layer on a different conductive-type substrate, measuring the sheet resistance of the epitaxial layer as well as its thickness, and multiplying the sheet resistance by the thickness.

SEMI MF374[5] is a standard test method for the sheet resistance of epitaxial silicon layers. SEMI MF723 is applied to the conversion of resistivity into carrier density using the epitaxial-layer thickness.

A.3.3 Standards for measuring thickness of epitaxial layers

Standard test methods for the thickness of epitaxial layers are described in SEMI MF95[6] and SEMI MF110[7]. SEMI MF95 has good reproducibility, but is not applicable if the dopant density of the epitaxial layer is high (max. $2{,}8 \times 10^{17}$ cm^{-3} for p-type and $7{,}8 \times 10^{16}$ cm^{-3} for n-type). The dopant density of the substrate should also be high (at least $3{,}2 \times 10^{18}$ cm^{-3} for p-type and $1{,}3 \times 10^{18}$ cm^{-3} for n-type).

SEMI MF110 describes a conventional method using angle lapping and stain etching. This method is applicable when there is conductive-type difference or resistivity difference of more than one order of magnitude between the epitaxial layer and the substrate, even for epitaxial layers with a high carrier density

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which cannot be measured by SEMI MF95. It is, however, difficult to apply SEMI MF110 to measurements of epitaxial layers over 25 µm thick since the angle-lapped surface inclines too much towards the optical system of the interference microscope used for such thickness measurements.

In either of these standards, the width of the transition region between an epitaxial layer and a substrate can affect the measured value of the epitaxial-layer thickness. The effect of such a transition region on the thickness determination is larger for thin epitaxial layers (e.g. 5 µm) than for thicker ones (e.g. 100 µm).

A.3.4 Alternative method for measuring thickness of epitaxial layers

SEMI MF672[8] describes a standard test method for measuring resistivity profiles in a silicon wafer using a spreading resistance probe. SEMI MF674[9] gives a standard practice for preparing the specimen for the measurement. When SEMI MF672 is employed, the depth of a p-n junction or the boundary between layers of different resistivity can be measured, which enables the determination of the thickness of epitaxial layers.

SEMI MF672 is applicable to determining the thickness of epitaxial layers regardless of the carrier density of the specimen. Furthermore, the thickness of thick epitaxial layers (e.g. 100 µm) can be determined. Application of this method is preferable when the carrier density is so high that a Schottky junction for CV measurements is hard to form.

A.4 Summary

A.4.1 Bulk silicon specimens

Resistivity is measured by SEMI MF84, making reference to SEMI MF43, and the result is converted into carrier density using SEMI MF723.

If the carrier density is in the range where a Schottky junction can be formed, SEMI MF1392 is applicable.

A.4.2 Epitaxial silicon specimens

If the carrier density is in the range where a Schottky junction can be formed, SEMI MF1392 is applicable.

If the carrier density is outside this range, it is preferable to convert from resistivity to carrier density using SEMI MF723 after determining resistivity of the epitaxial layer by means of measuring the sheet resistance by SEMI MF374 and the thickness of the epitaxial layer by SEMI MF672.

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Annex B
(informative)

Boron isotope ratio measured by SIMS

B.1 General

The boron isotope ratio was evaluated in the interlaboratory test programme. A boron-doped bulk silicon specimen with a boron atomic concentration of ca. 1×10^{19} atoms/cm^3 was analysed by SIMS in 20 laboratories. The results are compared with that of a precise isotopic analysis.

B.2 Test specimen

Two boron-doped bulk silicon wafers were cut from adjacent positions in an ingot doped with ca. 1×10^{19} atoms/cm^3 of boron. One of the wafers was cut into 7 mm × 7 mm pieces and subjected to SIMS analysis in each participating laboratory.

The other wafer was subjected to isotopic analysis using the method presented in Reference [10].

B.3 Procedure of SIMS analysis

SIMS analyses were done following the procedures stipulated in 7.5.2.

B.4 Results of isotope ratio analysis

B.4.1 The results of the isotope ratio analysis for each laboratory are given in Table B.1 and Figure B.1.

B.4.2 The mean value and repeatability (2σ) of the ratio of ^{11}B atoms to ^{10}B atoms are as follows:

$3{,}922\ 1 \pm 0{,}000\ 4\ (2\sigma)$

Since NIST SRM 951 has a relative uncertainty in the boron isotopic ratio of 0,08 % (2σ), the uncertainty of the measurement is determined by the uncertainty inherent in NIST SRM 951. Therefore

$\alpha_0 = 3{,}922\ 1 \pm 0{,}003\ 1\ (2\sigma)$

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B.4.3 The correction factors, δ, for the measured isotope ratio from each laboratory are also given in Table B.1.

Table B.1 — Measured isotope ratio, α, and correction factor, δ

Laboratory	Detection mode	Instrument	α	δ
1	Positive ions	Magnetic sector	3,878	1,011
2			3,808	1,030
3			3,669	1,069
4			3,367	1,165
5			3,845	1,020
6			3,608	1,087
7			3,808	1,030
8			3,739	1,049
9			4,263	0,920
10			3,762	1,043
11			3,566	1,100
12			3,878	1,011
13			3,615	1,074
14			5,098	0,769
15		Quadrupole	4,155	0,944
6			3,566	1,100
11			3,950	0,993
16			3,762	1,043
17			4,236	0,926
18			3,566	1,100
1	Negative ions	Magnetic sector	3,785	1,036
2			3,902	1,005
3			3,751	1,046
19			3,808	1,030
11			3,831	1,024
20		Quadrupole	3,566	1,100
11			3,695	1,061

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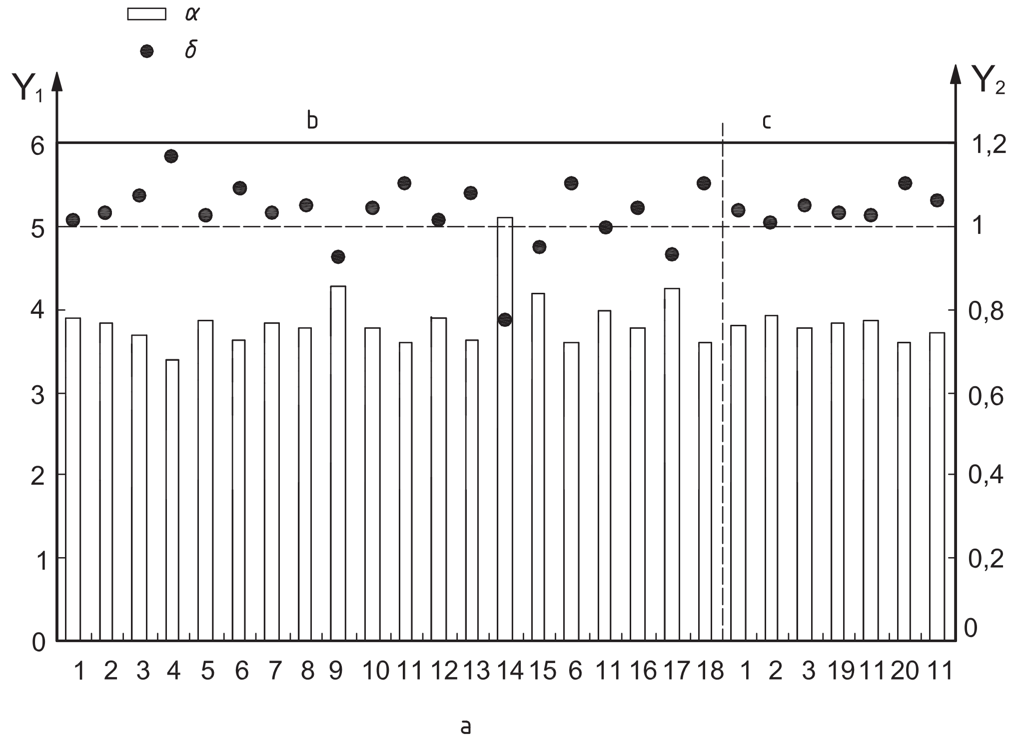

Key

Y_1 isotope ratio, α

Y_2 correction factor, δ

[a] Laboratory.

[b] Positive ions.

[c] Negative ions.

Figure B.1 — Measured isotope ratio and correction factor

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Annex C
(normative)

Procedures for evaluation of apparatus performance

C.1 General

The apparatus performance shall be evaluated using the three levels of boron-doped bulk RMs (RM-A, RM-B and RM-C) and the non-boron-doped RM (RM-BG) given in Table 1. The boron atomic concentrations in the RMs shall be determined using the procedures stipulated in Annex A by an expert capable of implementing the procedures in Annex A.

C.2 Measurement procedure for bulk RMs

Follow the procedures stipulated in 7.5.2.

C.3 Mass resolution

C.3.1 The minimum ion intensity of the valley between the peaks for ^{10}B and ^{11}B in the RM-C shall be less than 1 % of the maximum ion count for the ^{11}B peak.

C.3.2 The minimum ion intensity of the valley between the peaks for $^{29}Si^{30}Si$ and $^{30}Si^{30}Si$ shall be less than 1 % of the maximum ion count for the $^{29}Si^{30}Si$ peak.

C.4 Minimum ion intensity

The secondary boron-ion (or boron cluster-ion) intensity in the RM-A shall be at least three times higher than the background intensity evaluated by using the RM-BG.

C.5 Minimum precision

C.5.1 The ratio of the boron-ion (or boron cluster-ion) intensity to the silicon-ion (or silicon cluster-ion) intensity in the RM-C shall be measured three times at different points on the specimen surface in order to obtain the mean intensity ratio and the standard deviation.

The standard deviation shall not exceed 10 % of the mean intensity ratio.

C.5.2 The ratio of the boron-ion (or boron cluster-ion) intensity to the silicon-ion (or silicon cluster-ion) intensity in the RM-A shall be measured three times at different points on the specimen surface in order to obtain the mean intensity ratio and the standard deviation.

The standard deviation shall not exceed 20 % of the mean intensity ratio.

NOTE An intralaboratory standard deviation is defined by the following formula:

$$\sigma = \sqrt{\frac{1}{n-1}\sum_{i=1}^{n}(X_i - X_{\text{AV}})^2}$$

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where

σ is the standard deviation of X;

n is the total number of measurements of X;

X_{AV} is the mean value of X.

C.6 Linearity of measurement

C.6.1 Determination of relative-sensitivity factors

The relative-sensitivity factor for each boron atomic concentration shall be obtained from the following formula:

$$\mathrm{RSF}_k^{\mathrm{bulk}} = \frac{\alpha_{0_k}}{1+\alpha_{0_k}} \times \frac{C_k^{\mathrm{bulk}}}{J_k^{11} - J_{\mathrm{BG}}^{11}}$$

where

the suffix k denotes RM-A, B and C;

α_{0_k} is the actual isotope ratio in each bulk RM;

$\mathrm{RSF}_k^{\mathrm{bulk}}$ is the relative-sensitivity factor for each bulk RM;

C_k^{bulk} is the boron atomic concentration in each bulk RM determined by resistivity;

J_k^{11} is the mean ion intensity ratio for ^{11}B in each bulk RM;

J_{BG}^{11} is the mean background ion intensity ratio for ^{11}B derived from RM-BG.

If α_{0_k} is not known, the natural isotope ratio ($\alpha_{0_k} = 4,025$) shall be used, giving

$$\mathrm{RSF}_k^{\mathrm{bulk}} = \frac{0,801\, C_k^{\mathrm{bulk}}}{J_k^{11} - J_{\mathrm{BG}}^{11}}$$

C.6.2 Evaluation of linearity

The mean value and the standard deviation of the relative-sensitivity factors for three bulk RMs shall be calculated. The standard deviation shall not exceed 20 % of the mean relative-sensitivity factor. If the value is greater than 20 %, the measurement conditions shall be changed so as to reduce the deviation.

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Annex D
(informative)

Statistical report on interlaboratory test programme

D.1 General

This International Standard was subjected to an interlaboratory test programme involving 12 laboratories in four countries. Four bulk specimens were analysed to cover the scope of the method. Repeatability and reproducibility were calculated in accordance with the principles of ISO 5725-2[11].

D.2 Design of test programme

Each participating laboratory was asked to analyse four specimens and report three independent results.

D.3 Test samples

Three epitaxial silicon wafers doped with ca. 1×10^{16} atoms/cm^3, 8×10^{17} atoms/cm^3 and 5×10^{18} atoms/cm^3 of boron, respectively, and a bulk silicon wafer doped with ca. 1×10^{19} atoms/cm^3 of boron were prepared. An n-type silicon wafer was also prepared for background checking. SIMS specimens 7 mm × 7 mm in size were cut from the central region of these wafers and distributed to participating laboratories. NIST SRM 2137 was used as the primary reference material.

D.4 Procedure of SIMS analysis

Boron atomic concentrations in the four boron-doped specimens were determined following the procedures stipulated in 7.5.

The boron isotope ratio was corrected using the value shown in Annex B (α_0 = 3,922 1) for all the specimens.

D.5 Statistical procedures

D.5.1 Scrutiny for consistency and outliers

Cochran's test, Grubb's test and the graphical consistency technique were applied independently to the data in accordance with ISO 5725-2. If all three tests indicated that a result was an outlier, it was excluded from the analysis.

D.5.2 Computation of repeatability and reproducibility

The numbers of laboratories completing the test programme were 12 for the positive-ion detection mode and 6 for the negative-ion detection mode. The results from each laboratory were treated in accordance with ISO 5725-2 to give the mean value, within-laboratory variance and between-laboratory variance. The corresponding repeatability and reproducibility were calculated.

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The following information was thus obtained:

s_r^2 the repeatability variance;

s_L^2 the between-laboratory variance;

s_R^2 the reproducibility variance, given by

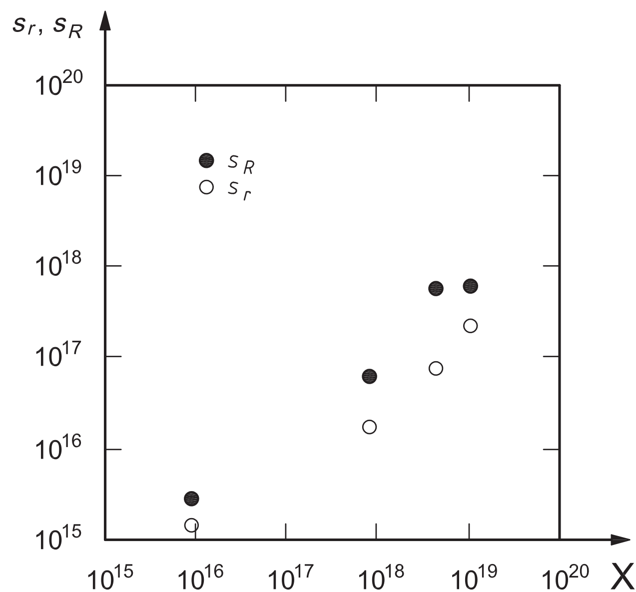
$$s_R^2 = s_r^2 + s_L^2$$

D.6 Results of statistical analysis

D.6.1 The results of the statistical analysis for the positive-ion detection mode are given in Table D.1. A graphical presentation of these data is given in Figure D.1. One laboratory was excluded from each of levels 1, 2 and 4. Two laboratories were excluded from level 3.

Table D.1 — Repeatability and reproducibility of boron atomic concentrations in the positive-ion detection mode

Level	Number of labs	Mean value	s_r	s_R
1	11	$9,15 \times 10^{15}$	$1,38 \times 10^{15}$	$2,68 \times 10^{15}$
2	11	$8,05 \times 10^{17}$	$1,73 \times 10^{16}$	$6,05 \times 10^{16}$
3	10	$1,08 \times 10^{19}$	$2,15 \times 10^{17}$	$6,14 \times 10^{17}$
4	11	$4,47 \times 10^{18}$	$7,36 \times 10^{16}$	$5,50 \times 10^{17}$



Key

X mean value

s_r repeatability

s_R reproducibility

Figure D.1 — Plot of s_r and s_R against mean value for data from Table D.1

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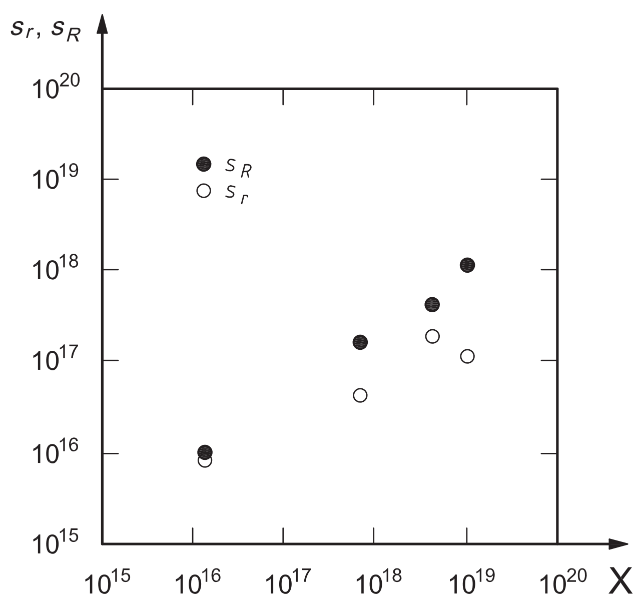
D.6.2 The results of the statistical analysis for the negative-ion detection mode are given in Table D.2. A graphical presentation of these data is given in Figure D.2. One laboratory was excluded from each of levels 1, 3 and 4.

Table D.2 — Repeatability and reproducibility of boron atomic concentrations in the negative-ion detection mode

Level	Number of labs	Mean value	s_r	s_R
1	5	$1,40 \times 10^{16}$	$8,25 \times 10^{15}$	$9,87 \times 10^{15}$
2	6	$7,29 \times 10^{17}$	$4,09 \times 10^{16}$	$1,53 \times 10^{17}$
3	5	$1,06 \times 10^{19}$	$1,12 \times 10^{17}$	$1,11 \times 10^{18}$
4	5	$4,36 \times 10^{18}$	$1,83 \times 10^{17}$	$4,17 \times 10^{17}$



Key

X mean value

s_r repeatability

s_R reproducibility

Figure D.2 — Plot of s_r and s_R against mean value for data from Table D.2

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Bibliography

[1] SEMI MF1392-0307, *Test Method for Determining Net Carrier Density Profiles in Silicon Wafers by Capacitance-Voltage Measurements with a Mercury Probe*

[2] SEMI MF43-0705, *Test Methods for Resistivity of Semiconductor Materials*

[3] SEMI MF84-0307, *Test Method for Measuring Resistivity of Silicon Wafers with an In-Line Four-Point Probe*

[4] SEMI MF723-0307, *Practice for Conversion between Resistivity and Dopant or Carrier Density for Boron-Doped, Phosphorus-Doped, and Arsenic-Doped Silicon*

[5] SEMI MF374-0307, *Test Method for Sheet Resistance of Silicon Epitaxial, Diffused, Polysilicon, and Ion-implanted Layers Using an In-Line Four-Point Probe with the Single-Configuration Procedure*

[6] SEMI MF95-1107, *Test Method for Thickness of Lightly Doped Silicon Epitaxial Layers on Heavily Doped Silicon Substrates Using an Infrared Dispersive Spectrophotometer*

[7] SEMI MF110-1107, *Test Method for Thickness of Epitaxial or Diffused Layers in Silicon by the Angle Lapping and Staining Technique*

[8] SEMI MF672-0307, *Test Method for Measuring Resistivity Profiles Perpendicular to the Surface of a Silicon Wafer Using a Spreading Resistance Probe*

[9] SEMI MF674-0705, *Practice for Preparing Silicon for Spreading Resistance Measurements*

[10] NAKAMURA, E., ISHIKAWA, T., BIRCK, J.-L., and ALLEGRE, C.J., Precise isotopic analysis of natural rock samples using a boron-mannitol complex, *Chemical Geology* (Isotope Geoscience Section), **94** (1992), pp. 193-204

[11] ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

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