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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 — Méthode par 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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Contents

Page

Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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 International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

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

Annexes C and D form a normative part of this International Standard. Annexes A, B and E are for information only.

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 quantitative boron analysis in single-crystalline silicon using secondary reference materials calibrated by a certified reference material implanted with boron.

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 reference

The following normative document contains provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, this publication do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the normative document indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 5725-2:1994, *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.*

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 NIST Standard Reference Material 2137 (referred to hereinafter as NIST SRM) is the only CRM of boron in silicon at this moment.

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 4.2.2 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 type of instrument and 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 C.6 can be replaced by local documented procedures.

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.

NOTE 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.

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.

NOTE 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 annex D. The mean integrated ion intensity ratio of the CRM, A^{imp}, shall be calculated following the procedures stipulated in clause D.7.

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

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

where

$\mathrm{RSF}^{\mathrm{imp}}$ 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.

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:

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

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

where

$I_{i,j}^{11}$ and $I_{i,j}^{\mathrm{Si}}$ 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:

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

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, α_0 = 4,025 (see 4.2.3), 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^{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.

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

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

where

$C_k^{11\text{cal}}$ 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:

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

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.

NOTE 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 the 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:

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

where

RSF^{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:

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

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

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

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. Four 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 E.

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

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.

Annex A
(informative)

Determination of carrier density in silicon wafers

A.1 General

This annex gives information 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 using methods listed in Table A.1. The application of such methods is limited, however, because a Schottky junction is required for them. Also, applicable carrier densities are limited to 4×10^{13} cm^{-3} to 8×10^{16} cm^{-3}.

Table A.1 — ASTM standards for determining carrier density in bulk silicon wafers

F 1392	*Standard Test Method for Determining Net Carrier Density Profiles in Silicon Wafers by Capacitance-Voltage Measurements With a Mercury Probe*
F 1393	*Standard Test Method for Determining Net Carrier Density in Silicon Wafers by Miller Feedback Profiler Measurements With a Mercury Probe*

Consequently, resistivity is generally measured and converted into the carrier density in the whole resistivity range. F 43 and F 84 given in Table A.2 are standard methods for measuring resistivity, while F 723 is a standard practice for conversion between resistivity and carrier density. In general, resistivity is measured by F 84 making reference to F 43 and is converted into carrier density in accordance with F 723.

Table A.2 — ASTM standards for measuring resistivity of bulk silicon wafers and conversion of carrier density

F 43	*Standard Test Methods for Resistivity of Semiconductor Materials*
F 84	*Standard Test Method for Measuring Resistivity of Silicon Wafers With an In-Line Four Point Probe*
F 723	*Standard Practice for Conversion Between Resistivity and Dopant Density for Boron-Doped and Phosphorus-Doped Silicon*

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 standards listed in Table A.3. However, their applicable ranges are limited within the carrier densities ranging from 4×10^{13} cm^{-3} to 8×10^{16} cm^{-3}, due to Schottky junction formation or specimen preparation.

Table A.3 — ASTM standards for determining carrier density in epitaxial silicon layers

F 1392	*Standard Test Method for Determining Net Carrier Density Profiles in Silicon Wafers by Capacitance-Voltage Measurements With a Mercury Probe*
F 1393	*Standard Test Method for Determining Net Carrier Density in Silicon Wafers by Miller Feedback Profiler Measurements With a Mercury Probe*

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 layers as well as their thickness, and multiplying the sheet resistance by the thickness.

A standard test method for the sheet resistance of epitaxial silicon layers is given in Table A.4. F 723 is applied to the conversion of resistivity into carrier density using the epitaxial layer thickness.

Table A.4 — ASTM standard for measuring sheet resistance of epitaxial silicon layers

F 374	*Standard Test Method for Sheet Resistance of Silicon Epitaxial, Diffused, Polysilicon, and Ion-implanted Layers Using an In-Line Four-Point Probe*

A.3.3 Standards for measuring thickness of epitaxial layers

Standard test methods for the thickness of epitaxial layers are listed in Table A.5. F 95 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).

F110 describes a conventional method using angle lapping and stain etching. This method is applicable when there is a 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 which cannot be measured by F 95. It is, however, difficult to apply F 110 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 the epitaxial layer and the 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).

Table A.5 — ASTM standards for measuring thickness of epitaxial silicon layers

F 95	*Standard Test Method for Thickness of Lightly Doped Silicon Epitaxial Layers on Heavily Doped Silicon Substrates Using an Infrared Dispersive Spectrophotometer*
F 110	*Standard Test Method for Thickness of Epitaxial or Diffused Layers in Silicon by the Angle Lapping and Staining Technique*

A.3.4 Alternative method for determining thickness of epitaxial layers

Table A.6 gives a standard test method for measuring resistivity profiles in a silicon wafer using a spreading resistance probe, and a standard practice for preparing the specimen for the measurement. When F 672 is

employed, the depth of the p-n junction or the boundary between layers of different resistivity can be measured, which enables the determination of the thickness of epitaxial layers.

This method 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.

Table A.6 — ASTM standards for measuring spreading resistance of silicon

F 672	*Standard Test Method for Measuring Resistivity Profiles Perpendicular to the Surface of a Silicon Wafer Using a Spreading Resistance Probe*
F 674	*Standard Practice for Preparing Silicon for Spreading Resistance Measurements*

A.4 Summary

A.4.1 Bulk silicon specimens

Resistivity is measured by F 84 making reference to F 43, and the result is converted into carrier density by F 723.

If the carrier density is in the range where a Schottky junction can be formed, F 1392 or F 1393 is applicable.

A.4.2 Epitaxial silicon specimens

If the carrier density is in the range where a Schottky junction can be formed, F 1392 or F 1393 is applicable.

If the carrier density is out of this range, it is preferable to convert from resistivity to carrier density by F 723 after determining the resistivity of the epitaxial layer by measuring the sheet resistance by F 374 and the thickness of the epitaxial layer by F 672.

Annex B
(informative)

Boron isotope ratio measured by SIMS

B.1 General

The boron isotope ratio was evaluated in an 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 were 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 $\times$ 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 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.

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 result of the precise isotopic analysis is as follows:

$$\alpha_0 = 3{,}9221 \pm 0{,}0004\,(2\sigma)$$

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

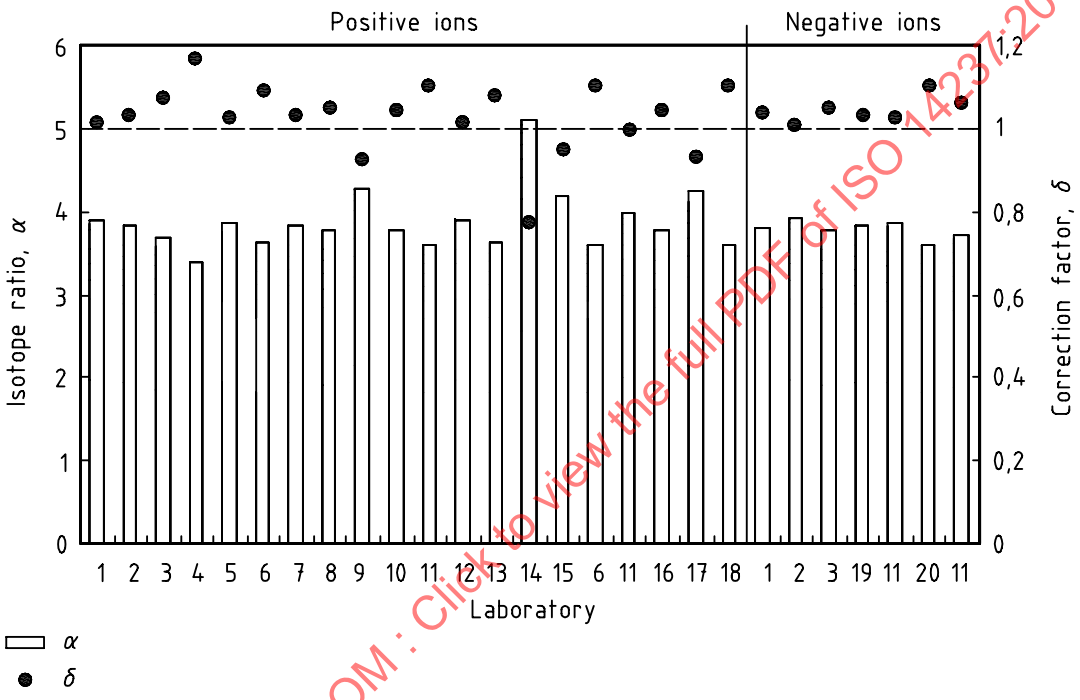

Figure B.1 — Measured isotope ratios and correction factors

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 intensity ratio of boron-ion (or boron cluster-ion) to silicon-ion (or silicon cluster-ion) 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 intensity ratio of boron-ion (or boron cluster-ion) to silicon-ion (or silicon cluster-ion) 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:

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

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:

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

where

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

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

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

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

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.

Annex D
(normative)

Procedures for the depth profiling of NIST SRM 2137

D.1 Adjustment of secondary-ion mass spectrometer

Follow the procedures stipulated in 7.1 except for the primary-ion scan area. The primary-ion scan area shall be 10 times the analysed area or larger.

D.2 Optimizing the secondary-ion mass spectrometer settings

Follow the procedures stipulated in 7.2.

D.3 Data sampling range

The depth profile shall be obtained from the surface to at least 0,4 μm (see D.5).

D.4 Detected ions

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

The ion species of silicon shall be the same as that for the bulk RMs.

D.5 Depth profiling procedure

Since the boron intensity at the peak position is high, care shall be taken so as not to saturate the detector. If the boron intensity at the peak position is higher than 1×10^5 counts/s, the primary-ion intensity shall be reduced. The primary-ion beam current and the beam scan area shall be chosen so that at least 50 measurement cycles are in the 0 μm to 0,4 μm range (sputtering rate is 8 nm/cycle or less).

The secondary-ion intensities for boron and silicon shall be measured alternately, for at least one second for boron per cycle. The measurement shall be continued until a depth of 0,4 μm is reached (determine the approximate sputter depth referring to the profile shown in Figure D.1), or to a measurement cycle 20 cycles or more after the constant background or limit of detection is reached. The measurement shall be repeated three times at different points on the specimen surface.

D.6 Determination of depth scale

In the ion intensity measurement cycle (or time) profile, the midpoint of the width at 0,2 of the maximum of the implanted profile shall be determined. The midpoint shall be 0,167 μm (see Figure D.1). The depth scale shall be calibrated using this midpoint.

NOTE The above procedure describes a tentative method of depth scale calibration. When an International Standard for depth scale calibration in depth profiling is established, this procedure will be revised. An interlaboratory programme of depth

scale measurements showed the difference in depth scales between those determined by this procedure and by crater depth measurement was 2,6 %, which was within experimental error.

D.7 Determination of integrated intensity

Ion intensity ratios of boron to silicon shall be determined for each measurement cycle by cycle at one measurement position, and then an integrated intensity ratio for all the measurement cycles shall be calculated using the following formula. Any intensity anomaly in the surface region caused by native oxide or surface contaminants shall be excluded from integration. The integrated intensity ratio obtained shall be further averaged for the three measurement positions.

$$A^{\mathrm{imp}} = \frac{1}{3}\sum_{j=1}^{3}\left[\sum_{i=1}^{n}\left(\frac{I_{i,j}^{10} - I_{\mathrm{BG}}}{I_{i,j}^{\mathrm{Si}}}\right)\Delta z\right]$$

where

A^{imp} is the mean integrated ion intensity ratio of the NIST SRM;

n is the total number of depth profiling measurement cycles;

$I_{i,j}^{10}$ and $I_{i,j}^{\mathrm{Si}}$ are the boron-ion intensity and the silicon-ion intensity, respectively, in the NIST SRM at measurement cycle i and measurement position j;

I_{BG} is the mean background ion intensity in the NIST SRM;

Δz is the depth profiling depth interval [= (total depth)/n].