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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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Tel. + 41 22 749 01 11  
Fax + 41 22 734 10 79  
E-mail [copyright@iso.ch](mailto:copyright@iso.ch)  
Web [www.iso.ch](http://www.iso.ch)

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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 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.

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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 quantitative boron 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  $1 \times 10^{16}$  atoms/cm<sup>3</sup> to  $1 \times 10^{20}$  atoms/cm<sup>3</sup>.

## 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.

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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  $1 \times 10^{16}$  atoms/cm<sup>3</sup> and  $1 \times 10^{20}$  atoms/cm<sup>3</sup> 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 <sup>3</sup>
RM-A	low $1 \times 10^{16}$ to $1 \times 10^{17}$
RM-B	middle $5 \times 10^{17}$ to $5 \times 10^{18}$
RM-C	high $1 \times 10^{19}$ to $1 \times 10^{20}$
RM-BG	none $< 1 \times 10^{14}$

**4.2.4** The isotope ratio of <sup>11</sup>B to <sup>10</sup>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<sup>-</sup> 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 <sup>10</sup>B and 80,1 atomic percent <sup>11</sup>B, i.e. a ratio of <sup>11</sup>B atoms to <sup>10</sup>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 <sup>10</sup>B<sup>28</sup>Si<sup>-</sup> and <sup>11</sup>B<sup>28</sup>Si<sup>-</sup> than between <sup>10</sup>B<sup>+</sup> and <sup>11</sup>B<sup>+</sup> 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

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**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  $^{10}\text{B}^{28}\text{Si}^1\text{H}^-$  background signal which interferes in  $^{11}\text{B}^{28}\text{Si}^-$  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  $^{10}\text{B}^+$  and  $^{11}\text{B}^+$  shall be detected as secondary-ion species of boron. When a caesium-ion beam is used, both  $^{10}\text{B}^{28}\text{Si}^-$  and  $^{11}\text{B}^{28}\text{Si}^-$  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  $^{28}\text{Si}^+$  be detected as the reference ion of  $\text{B}^+$  using the electrometer. For the pulse-counting mode, the silicon-ion intensity should be less than  $1 \times 10^5$  counts/s. For  $\text{BSi}^-$  detection,  $\text{Si}_2^-$  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  $^{10}\text{B}$  or  $^{11}\text{B}$ ) 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,  $A^{\text{imp}}$ , shall be calculated following the procedures stipulated in clause D.7.

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

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

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where

$\text{RSF}^{\text{imp}}$  is the isotopic relative sensitivity factor obtained from the CRM;

$\Phi$  is the implanted boron (either  $^{10}\text{B}$  or  $^{11}\text{B}$ ) 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  $1 \times 10^5$  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:

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

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

where

$I_{i,j}^{11}$  and  $I_{i,j}^{\text{Si}}$  are the  $^{11}\text{B}$ -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  $^{11}\text{B}$  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  $^{10}\text{B}$ .

**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  $^{10}\text{B}^+$  and  $^{30}\text{Si}^{3+}$  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  $1 \times 10^{17}$  atoms/cm<sup>3</sup> 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

$\alpha$  is the measured isotope ratio of  $^{11}\text{B}$  to  $^{10}\text{B}$ ;

$J_{\text{BG}}^{11}$  and  $J_{\text{BG}}^{10}$  are the mean background ion intensity ratios for  $^{11}\text{B}$  and  $^{10}\text{B}$ , 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  $\alpha_0$  is the actual isotope ratio in the bulk RM.

If  $\alpha_0$  is not known, the natural isotope ratio,  $\alpha_0 = 4,025$  (see 4.2.3), shall be used.

$\delta$  shall be used to correct the experimental mass discrimination between  $^{10}\text{B}$  and  $^{11}\text{B}$ .

### 7.5.3 Calibration of bulk RMs

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

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

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

where

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

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

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

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

## 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

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**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  $1 \times 10^2$  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

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