

Australian Standard[®]

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



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

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PREFACE

This Standard was prepared by the Standards Australia Committee CH-016, Spectroscopy. This Standard is identical with, and has been reproduced from ISO 14237:2000, *Surface chemical analysis—Secondary-ion mass spectrometry—Determination of boron atomic concentration in silicon using uniformly doped materials*.

The objective of this Standard is to ensure that the procedure for the quantitative determination of boron analysis in single-crystalline silicon using secondary reference materials, calibrated by a certified reference material implanted with boron, is achieved.

As this Standard is reproduced from an international standard, the following applies:

- (a) Its number appears on the cover and title page while the international standard number appears only on the cover.
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The terms 'normative' and 'informative' have been used in this Standard to define the application of the annex to which they apply. A 'normative' annex is an integral part of a Standard, whereas an 'informative' annex is only for information and guidance.

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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.

AUSTRALIAN STANDARD

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/cm³ to 1×10^{20} atoms/cm³.

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/cm³ and 1×10^{20} atoms/cm³ 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 ¹¹B to ¹⁰B in the bulk RM chosen in 4.2.2 shall be determined by one of following methods.

- 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.
- The bulk RM shall be assumed to have the accepted nominal natural isotopic composition of 19,9 atomic percent ¹⁰B and 80,1 atomic percent ¹¹B, i.e. a ratio of ¹¹B atoms to ¹⁰B 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 ¹⁰B²⁸Si⁻ and ¹¹B²⁸Si⁻ than between ¹⁰B⁺ and ¹¹B⁺ 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 $^{10}\text{B}^{28}\text{Si}^1\text{H}^-$ 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 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:

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

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.