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

Analyse chimique des surfaces — Spectrométrie de masse des ions secondaires — Dosage du bore dans le silicium par profilage d'épaisseur

Reference number
ISO 17560:2002(E)

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Contents

Page

Annex

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 17560 was prepared by Technical Committee ISO/TC 201, *Surface chemical analysis*, Subcommittee SC 6, *Secondary ion mass spectrometry*.

Annex A of this International Standard is for information only.

Introduction

This International Standard was prepared for the quantitative depth profiling of boron in silicon by secondary-ion mass spectrometry (SIMS).

For quantitative depth profiling, calibration is necessary both for the concentration and the depth scales of the profile measured. A procedure for the determination of boron in silicon has been established as an International Standard, ISO 14237. Thus, the calibration of boron atomic concentration is performed by following ISO 14237.

In this International Standard, standard procedures are described for depth profiling of boron in single-crystal, poly-crystal or amorphous silicon using SIMS and for depth scale calibration using stylus profilometry or optical interferometry.

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

1 Scope

This International Standard specifies a secondary-ion mass spectrometric method using magnetic-sector or quadrupole mass spectrometers for depth profiling of boron in silicon, and using stylus profilometry or optical interferometry for depth scale calibration. This method is applicable to single-crystal, poly-crystal or amorphous-silicon specimens with boron atomic concentrations between 1×10^{16} atoms/cm^3 and 1×10^{20} atoms/cm^3, and to crater depths of 50 nm or deeper.

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 14237:2000, *Surface chemical analysis — Secondary-ion mass spectrometry — Determination of boron atomic concentration in silicon using uniformly doped materials*

3 Symbols and abbreviated terms

C_i	total boron atomic concentration in measurement cycle i, expressed in atoms per cubic centimetre (atoms/cm^3)
C_i^{10}	atomic concentration of the boron isotope with mass number 10 in measurement cycle i, expressed in atoms per cubic centimetre (atoms/cm^3)
C_i^{11}	atomic concentration of the boron isotope with mass number 11 in measurement cycle i, expressed in atoms per cubic centimetre (atoms/cm^3)
d_i	depth measured in measurement cycle i, expressed in micrometres (µm) or nanometres (nm)
d_t	crater depth, expressed in micrometres (µm) or nanometres (nm)
I_i^{10}	ion intensity of the boron isotope with mass number 10 in measurement cycle i
I_i^{11}	ion intensity of the boron isotope with mass number 11 in measurement cycle i
I_i^{Si}	ion intensity of silicon matrix in measurement cycle i
J_i^{10}	boron to silicon ion intensity ratio for the boron isotope with mass number 10 in measurement cycle i
J_i^{11}	boron to silicon ion intensity ratio for the boron isotope with mass number 11 in measurement cycle i
J_{BG}^{10}	mean background boron to silicon ion intensity ratio for the boron isotope with mass number 10 in measurement cycle i

J_{BG}^{11}	mean background boron to silicon ion intensity ratio for the boron isotope with mass number 11 in measurement cycle i
N	total number of measurement cycles
T	total measurement time, expressed in seconds (s)
t_i^{B}	starting time of boron-ion acquisition in measurement cycle i, expressed in seconds (s)
Δt^{B}	duration of boron-ion acquisition in each measurement cycle, expressed in seconds (s)
δ	mass discrimination correction factor
λ	wavelength of the light for optical interferometry, expressed in micrometres (µm) or nanometres (nm)
$\mathrm{RSF}^{\mathrm{work}}$	working relative-sensitivity factor
SIMS	secondary-ion mass spectrometry

4 Principle

An oxygen-ion beam or caesium-ion beam is scanned over the specimen surface and the emitted secondary ions of boron and silicon from a gated region within the area scanned by the ion beam are detected and mass-analysed. The intensities of these mass-analysed signals are monitored as a function of sputtering time. The depth of the crater formed by the ion beam is measured by stylus profilometry or optical interferometry for depth scale calibration.

NOTE Optical interferometry is generally applicable to crater depths in the range from 0,5 µm to 5 µm.

5 Reference materials

5.1 Reference materials for determination of relative-sensitivity factors

Follow clause 4 of ISO 14237:2000.

5.2 Reference materials for calibration of depth scale

For stylus profilometry calibration, certified reference materials or reference materials which are traceable to certified reference materials shall be used.

6 Apparatus

6.1 Secondary-ion mass spectrometer

As specified in clause 5 of ISO 14237:2000.

6.2 Stylus profilometer

Use a stylus profilometer with a sensitivity and tip shapes suitable for the crater shapes to be measured.

6.3 Optical interferometer

Use an optical interferometer with a sensitivity and functions suitable for the crater shapes to be measured.

7 Specimen

The specimen shall be cut to an appropriate size for analysis and degreased and washed if necessary.

NOTE The accuracy of crater depth measurement is largely influenced by surface roughness. A mirror-polished wafer is preferable when accurate determination of the depth scale is necessary.

8 Procedures

8.1 Adjustment of secondary-ion mass spectrometer

8.1.1 For oxygen-ion beam use, see Table 1. For caesium-ion beam use, see Table 2. Other conditions not shown here shall be set in accordance with the manufacturer's instructions or a local documented procedure.

Table 1 — Measurement conditions for oxygen-ion beam

Primary-ion species	O_2^+
Secondary-ion polarity	Positive
Primary-ion scan region	$>$ three times the linear dimension of the analysed region in all directions
Analysed region	Centred in the primary-ion scan region

Table 2 — Measurement conditions for caesium-ion beam

Primary-ion species	Cs^+
Secondary-ion polarity	Negative
Primary-ion scan region	$>$ three times the linear dimension of the analysed region in all directions
Analysed region	Centred in the primary-ion scan region

8.1.2 For the primary-ion beam, the beam current and scan region can be varied from specimen to specimen (see 8.5.2). However, when oxygen gas is introduced into the chamber during oxygen-beam irradiation, the oxygen pressure and all the primary-ion beam irradiation conditions shall be the same for the measurements on all specimens.

8.2 Optimizing the secondary-ion mass spectrometer settings

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

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

8.2.3 For a mass spectrometer whose transmission can be varied, use the same transmission for the measurements on both reference materials and test specimens.

8.3 Specimen introduction

Immediately prior to introduction of 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 1 Residual gases in the analysis chamber can produce a $^{10}B^{28}Si^{1}H^{-}$ background signal which interferes with the $^{11}B^{28}Si^{-}$ signal. This background can be reduced by improving the vacuum in the analysis chamber.

NOTE 2 With an amorphous-silicon specimen, it should be noted that the above background will be present because of hydrogen in the specimen.

8.4 Detected ions

8.4.1 When an oxygen-ion beam is used, both $^{10}B^{+}$ and $^{11}B^{+}$ shall be detected. When a caesium-ion beam is used, both $^{10}B^{28}Si^{-}$ and $^{11}B^{28}Si^{-}$ shall be detected.

8.4.2 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 1 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 BSi^{-} detection, Si_2^{-} is preferable as the reference ion. For the pulse-counting mode, the instantaneous silicon-ion count rate should be less than 5×10^5 counts/s.

NOTE 2 When an amorphous-silicon specimen is analysed, it should be noted that Si-H cluster ions can interfere with the ^{29}Si-ion, ^{30}Si-ion and their molecular-ion signals. Therefore, the ^{28}Si-ion or its molecular ions (e.g. $^{28}Si^{+}$, $^{28}Si_2^{+}$) are preferable as the matrix ion.

8.5 Measurement of test specimen

8.5.1 Measurements shall be made in the central region of the specimen holder window.

8.5.2 The primary-ion beam current and the beam scan area shall be chosen so that sufficient data points are obtained to record the details of the profile. It is useful to measure a test profile to determine these conditions. When the boron-ion intensity of the specimen is high (e.g. $\geqslant 1 \times 10^5$ counts/s), care shall be taken so as not to saturate the detector. If the boron-ion intensity is higher than 5×10^5 counts/s in the analysed area (i.e. gated area), the primary-ion current shall be reduced or, alternatively, the transmission of the mass spectrometer shall be reduced (see 8.2.3).

NOTE 1 In an ion microscope type instrument, ion intensities could be reduced by using a small field-limiting aperture. However, this would not reduce the instantaneous count rate from the analysed region. The overall transmission should be changed instead of using a small field aperture.

NOTE 2 For the linearity of the counting system with an electron multiplier, see bibliographic references [1] and [2].

8.5.3 The secondary-ion intensities of boron and silicon shall be measured alternately and cyclically.

NOTE If the variation of silicon-ion intensity during one profile measurement is less than the value determined to be acceptable by the manufacturer's instructions or a local documented procedure, 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 for any one cycle for each analysis position.

8.6 Calibration

8.6.1 Determination of relative-sensitivity factor

Following the procedure in 7.6.2 of ISO 14237:2000, the working relative-sensitivity factor $\mathrm{RSF}^{\mathrm{work}}$ and the mass discrimination correction factor δ shall be obtained using the same measurement conditions as for the test specimen measurements. Calibration and test specimen measurements are preferably done on the same day.

8.6.2 Calibration of depth scale by stylus profilometry

8.6.2.1 Calibrate the stylus profilometer for crater depth measurements using the reference materials stipulated in 5.2, in accordance with the manufacturer's instructions or a local documented procedure.

NOTE Precision of stylus profilometry measurements was evaluated in an interlaboratory test programme. A statistical report of the interlaboratory tests is given in annex A.

8.6.2.2 Measure the crater depth d_t using the calibrated stylus profilometer, in accordance with the manufacturer's instructions or a local documented procedure. The stylus shall be scanned over the crater so that it starts from a negligibly sputtered region, passes through the central region of the crater, and stops at the other end in a negligibly sputtered region.

8.6.3 Calibration of depth scale by optical interferometry

8.6.3.1 Measure the crater depth d_t using the calibrated optical interferometer, in accordance with the manufacturer's instructions or a local documented procedure. The interference fringes used to make the measurement shall pass through the centre and start and finish in negligibly sputtered regions.

8.6.3.2 Detailed procedures for measurement of interference fringes shall be in accordance with the manufacturer's instructions or a local documented procedure. Hereafter, general procedures are given. Draw two lines through the centre of two adjacent fringes (see Figure 1). One of the lines shall cross the centre of the crater (referred to as line R). Measure the distance x (in arbitrary units) between the two lines. Then measure the shift y (in the same units) of the fringe centre at the crater bottom relative to line R. Count the number of fringes n intersected by line R at the crater edge.

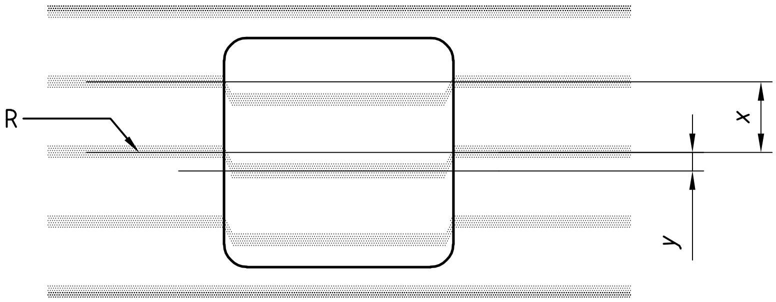

Figure 1 — Schematic drawing of interference fringes at a crater ($n = 0$)

8.6.3.3 The crater depth shall be obtained using the following formula:

$$d_t = \frac{\lambda}{2}\left(n + \frac{y}{x}\right) \tag{1}$$

Use a wavelength value determined by a traceable method.

9 Expression of results

9.1 Ion intensity ratios of boron to silicon shall be determined for each measurement cycle using the following formulae:

$$J_i^{11} = \left(\frac{I_i^{11}}{I_i^{\mathrm{Si}}} \right) \tag{2}$$

$$J_i^{10} = \left(\frac{I_i^{10}}{I_i^{\mathrm{Si}}} \right) \tag{3}$$

9.2 The boron atomic concentration of the test specimen shall be determined from the following formulae using the working relative-sensitivity factor $\mathrm{RSF}^{\mathrm{work}}$:

$$C_i^{11} = \mathrm{RSF}^{\mathrm{work}} \times J_i^{11} \tag{4}$$

$$C_i^{10} = \frac{\mathrm{RSF}^{\mathrm{work}} \times J_i^{10}}{\delta} \tag{5}$$

$$C_i = C_i^{11} + C_i^{10} \tag{6}$$

9.3 When necessary, the background intensity of boron shall be subtracted from the intensity ratio. The background intensity can be determined from a specimen not doped with boron (see 7.5.2.6 of ISO 14237:2000):

$$C_i^{11} = \mathrm{RSF}^{\mathrm{work}} \times \left(J_i^{11} - J_{\mathrm{BG}}^{11} \right) \tag{7}$$

$$C_i^{10} = \frac{\mathrm{RSF}^{\mathrm{work}} \times \left(J_i^{10} - J_{\mathrm{BG}}^{10} \right)}{\delta} \tag{8}$$

9.4 The depth for measurement cycle i shall be determined from the following formula using the crater depth d_t measured in 8.6 and assuming a constant sputtering rate:

$$d_i = \left(t_i^{\mathrm{B}} + \frac{\Delta t^{\mathrm{B}}}{2} \right) \left(\frac{d_t}{T} \right) \tag{9}$$

9.5 When the total number of measurement cycles N is so large that $\Delta t^{\mathrm{B}}/T$ is 0,1 % or less, d_i can be calculated using the following formula:

$$d_i = (i - 1) \frac{d_t}{N} \tag{10}$$

NOTE The sputtering rate can vary in the initial stages of sputtering before the sputtering reaches a steady state.

9.6 When graphical expression of the results is necessary, C_i (if necessary, C_i^{10} and/or C_i^{11}) shall be plotted as ordinate and d_i as abscissa for the necessary measurement cycles.

10 Test report

The test report shall include the following information:

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

b) the reference materials used, as specified in this International Standard (see clause 5);

c) information about isotope ratio correction, as specified in this International Standard (see 8.6.1);

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

e) any unusual features noted during the analysis;

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

Annex A
(informative)

Statistical report of stylus profilometry measurements

A.1 Introduction

The stylus profilometry technique used was subjected to an interlaboratory test programme involving 20 laboratories. A reference material having three types of groove for depth scale calibration was measured. Repeatability and reproducibility were calculated in accordance with the principles of ISO 5725-2[3].

A.2 Design of test programme

Each participating laboratory was asked to measure the depths of three grooves and report three independent results.

A.3 Test specimen

The test specimen used was a commercially available depth scale standard. The certified values of groove depth on the specimen were 2,33 µm (level 1), 0,27 µm (level 2) and 0,029 µm (level 3). The specimen was passed from one laboratory to the next.

A.4 Procedure of stylus measurement

The depths of grooves were measured using stylus profilometry by each participant, in accordance with the manufacturer's instructions or a local documented procedure. Each groove was measured three times.

A.5 Statistical procedures

A.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. An outlier indicated by all the tests was excluded from the analysis of the level 3 specimen.

A.5.2 Computation of repeatability and reproducibility

The number of laboratories completing the test programme was 20 for the 2,33 µm and 0,27 µm grooves and 15 for the 0,029 µm groove (an outlier was excluded). The results from each laboratory were processed 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.

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:

$$s_R^2 = s_r^2 + s_{\mathsf{L}}^2$$

A.6 Results of statistical analysis

A.6.1 The results of the statistical analysis are given in Table A.1.

Table A.1 — Repeatability and reproducibility of groove depth measurement

Level	**Number of labs**	**Mean value** (µm)	s_r (µm)	s_R (µm)
1	20	2,32	$4{,}98 \times 10^{-3}$	$1{,}99 \times 10^{-2}$
2	20	0,27	$2{,}26 \times 10^{-3}$	$3{,}94 \times 10^{-3}$
3	14	0,029	$7{,}76 \times 10^{-4}$	$7{,}98 \times 10^{-4}$

Bibliography

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[2] SEAH, M. P. and TOSA, M.: Linearity in Electron Counting and Detection Systems, *Surf. Int. Anal.*, **18**, pp. 240-246 (1992)

[3] 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*

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