BS ISO 17560:2014

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

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National foreword

This British Standard is the UK implementation of ISO 17560:2014. It supersedes BS ISO 17560:2002 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee CII/60, Surface chemical analysis.

A list of organizations represented on this committee can be obtained on request to its secretary.

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

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 201, *Surface chemical analysis*, Subcommittee SC 6, *Secondary ion mass spectrometry*.

Annex A of this International Standard is for information only.

This second edition cancels and replaces the first edition (ISO 17560:2002), which has been technically revised. The revision also includes editorial correction.

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.

This International Standard describes standard procedures 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.

INTERNATIONAL STANDARD **ISO 17560:2014(E)**

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 documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 14237:2010, *Surface chemical analysis — Secondary-ion mass spectrometry — Determination of boron atomic concentration in silicon using uniformly doped materials*

3 Symbols and abbreviations

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 centimeter (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 centimeter (atoms/cm^3)
d_i	depth measured in measurement cycle *i*, expressed in micrometres (μm) or nanometers (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*, expressed in counts per second (counts/s)
I_i^{11}	ion intensity of the boron isotope with mass number 11 in measurement cycle *i*, expressed in counts per second (counts/s)
I_i^{Si}	ion intensity of silicon matrix in measurement cycle *i*, expressed in counts per second (counts/s)
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_i^{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

The reference materials should be as specified in ISO 14237:2010, Clause 4.

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

The apparatus should be as specified in ISO 14237:2010, Clause 5.

6.2 Stylus profilometer

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

6.3 Optical interferometer

Use an optical interferometer with the 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 Procedure

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 vary 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, the background described in Note 1 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 the 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 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 1 × 10^5 counts/s.

NOTE When an amorphous-silicon specimen is analysed, note 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. ≥ 1 × 10^5 counts/s), care shall be taken to avoid unacceptable nonlinearity in the detector response due to pulse overlap. 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).

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 For the linearity of the counting system with an electron multiplier, see References[2] and.[3]

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 ISO 14237:2010, 7.6.2, the working relative-sensitivity factor RSF^{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.

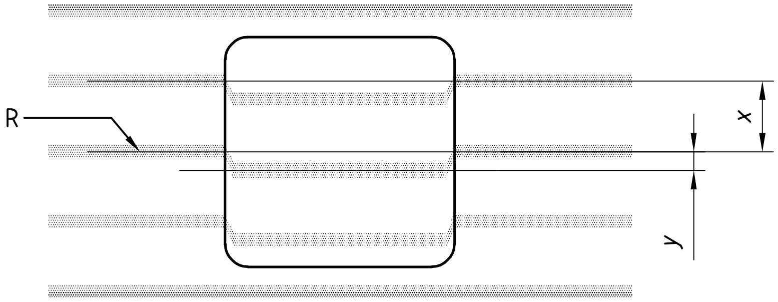

Figure 1 — Schematic drawing of interference fringes at a crater (n = 0)

8.6.3.3 The crater depth shall be obtained using Formula (1):

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

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 Formulae (2) and (3):

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

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

9.2 The boron atomic concentration of the test specimen shall be determined from Formulae (4) through (6) 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 ISO 14237:2010, 7.5.2.6).

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

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

9.4 The depth for measurement cycle shall be determined from Formula (9) 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 Formula (10):

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

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, ${}^{10}C_i$ and/or ${}^{11}C_i$) 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 might 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.[1]

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 grooves 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:

$$s_R^2 = s_r^2 + s_\mathrm{L}^2 \tag{A.1}$$

where

s_r^2 is the repeatability variance;

s_L^2 is the between-laboratory variance;

s_R^2 is the reproducibility variance.

A.6 Results of statistical analysis

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 laboratories	Mean value µm	s_r µm	s_R µm
1	20	2,32	4,98 × 10^{-3}	1,99 × 10^{-2}
2	20	0,27	2,26 × 10^{-3}	3,94 × 10^{-3}
3	14	0,029	7,76 × 10^{-4}	7,98 × 10^{-4}

Bibliography

[1] 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*

[2] SEAH. M. P.: Channel Electron Multipliers — Quantitative Intensity Measurement Efficiency, Gain, Linearity and Bias Effects. *J. Electron Spectrosc. Relat. Phenom.* 1990, **50** pp. 137–157

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

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