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

*Analyse chimique des surfaces — Spectrométrie de masse des ions
secondaires — Dosage de l'arsenic dans le silicium par profilage
d'épaisseur*

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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 2.

The main task of technical committees is to prepare International Standards. 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 document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 12406 was prepared by Technical Committee ISO/TC 201, *Surface chemical analysis*, Subcommittee SC 6, *Secondary ion mass spectrometry*.

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Introduction

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

For quantitative depth profiling, calibration is necessary both for the concentration and depth scales of the profile measured. A procedure for the determination of boron atomic concentration in silicon has been established as an International Standard, ISO 14237. A procedure for depth profiling of boron in silicon has been established as an International Standard, ISO 17560. A procedure for the determination of relative-sensitivity factors (RSFs) from ion-implanted reference materials has been established as an International Standard, ISO 18114. A vocabulary of general terms and terms used for spectroscopy in surface chemical analysis has been established as an International Standard, ISO 18115-1. The procedure for the quantitative depth profiling of arsenic in silicon makes use of these other International Standards.

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

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Surface chemical analysis — Secondary-ion mass spectrometry — Method for depth profiling of arsenic 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 arsenic in silicon, and using stylus profilometry or optical interferometry for depth calibration. This method is applicable to single-crystal, poly-crystal or amorphous silicon specimens with arsenic atomic concentrations between 1×10^{16} atoms/cm³ and $2,5 \times 10^{21}$ atoms/cm³, and to crater depths of 50 nm or deeper.

2 Normative references

The following referenced documents are indispensable for the application of this document. 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*

ISO 18114:2003, *Surface chemical analysis — Secondary-ion mass spectrometry — Determination of relative sensitivity factors from ion-implanted reference materials*

ISO 18115-1, *Surface chemical analysis — Vocabulary — Part 1: General terms and terms used in spectroscopy*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 18115-1 apply.

4 Symbols and abbreviated terms

C_i	total arsenic atomic concentration at measurement cycle, i , expressed in atoms per cubic centimetre (atoms/cm ³)
d	depth over which depth profile is integrated to determine RSF, expressed in centimetres (cm)
d_i	depth for measurement cycle, i , expressed in centimetres (cm)
d_t	crater depth, expressed in centimetres (cm)
I_i	ion intensity of arsenic species at measurement cycle, i , expressed in counts/s
I_i^{Si}	ion intensity of silicon matrix species at measurement cycle, i , expressed in counts/s

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I_{BG}	mean background ion intensity of the arsenic-ion species, expressed in counts/s
J_i	ion intensity ratio of arsenic to silicon at measurement cycle, i
J_{BG}	mean background ion intensity ratio of arsenic to silicon
n	total number of measurement cycles
RSF^{imp}	relative-sensitivity factor of implanted arsenic in certified reference material (CRM) (cm^{-3})
T	total sputtering time, expressed in seconds (s)
t_i^{As}	starting time of arsenic-ion acquisition at measurement cycle, i , expressed in seconds (s)
Δt^{As}	duration of arsenic-ion acquisition in each measurement cycle, expressed in seconds (s)
Φ	implanted dose of arsenic in CRM, expressed in atoms per square centimetre ($atoms/cm^2$)

5 Principle

A caesium ion beam or oxygen ion beam is scanned over the specimen surface and the emitted secondary ions of arsenic and silicon are mass-analysed and detected from a gated region in the scanned region of the ion beam. 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 0,2 μm to 5 μm .

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6 Reference materials

6.1 Reference materials for calibration of relative-sensitivity factors

A reference material is used for the determination of the arsenic atomic concentration of a test specimen. The reference material shall be a CRM of silicon implanted with arsenic or a secondary reference material of ion-implanted arsenic in silicon, the dose of which is traceable to a CRM.

NOTE At the time of publication of this International Standard, NIST standard reference material 2134 (referred to hereinafter as NIST SRM) and NMIJ CRM 5603-a are the only CRMs of arsenic available.

6.2 Reference materials for calibration of depth scale

For stylus profilometry calibration, reference materials that are traceable to the SI system shall be used.

7 Apparatus

7.1 Secondary-ion mass spectrometer

Follow the specification and procedures in Clause 5 of ISO 14237:2010.

7.2 Stylus profilometer

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

7.3 Optical interferometer

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

8 Specimen

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

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

9 Procedures

9.1 Adjustment of secondary-ion mass spectrometer

9.1.1 For caesium-ion beam use, see Table 1. For oxygen-ion beam use, see Table 2. Other conditions not shown in this clause shall be set according to the manufacturer's instructions or a local documented procedure.

9.1.2 For the primary-ion beam, the beam current and scan region can be varied from specimen to specimen (see 9.5.2). However, when oxygen-gas is introduced in the chamber during oxygen-beam irradiation, the oxygen pressure and all the primary-ion beam irradiation conditions shall be the same for the measurements of all specimens.

Table 1 — Measurement conditions for caesium-ion beam

Primary-ion species	ISO 12406:2010 Cs ⁺
Secondary-ion polarity	Negative
Primary-ion scan region	>3 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 oxygen-ion beam

Primary-ion species	O ₂ ⁺
Secondary-ion polarity	Positive
Primary-ion scan region	>3 times the linear dimension of the analysed region in all directions
Analysed region	Centred in the primary-ion scan region

9.2 Optimizing the secondary-ion mass spectrometer settings

9.2.1 Set the required instrument parameters and align the ion optics according to the manufacturer's instructions or a local documented procedure.

9.2.2 Ensure the stability of the primary-ion current and the mass spectrometer according to the manufacturer's instructions or a local documented procedure.

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

9.3 Specimen introduction

Immediately prior to introduction of the specimens into the SIMS apparatus, dust particles should be removed from the surfaces with a high purity pressurized gas, such as nitrogen. A commercial pressurized duster can also be used, but it may contaminate the sample surface. 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 $^{29}\text{Si}^{30}\text{Si}^{16}\text{O}$ background signal, which interferes with the $^{75}\text{As}^{\pm}$ signal. This background can be reduced by improving the vacuum in the analysis chamber.

9.4 Detected ions

9.4.1 If a caesium-ion beam is used, $^{75}\text{As}^{-}$ and/or $^{75}\text{As}^{28}\text{Si}^{-}$ and/or $^{75}\text{As}^{30}\text{Si}^{-}$ shall be detected as secondary-ion species of arsenic. If an oxygen-ion beam is used, $^{75}\text{As}^{+}$ shall be detected as secondary-ion species of arsenic.

NOTE When an oxygen-ion beam is used, a $^{29}\text{Si}^{30}\text{Si}^{16}\text{O}$ background signal is produced that interferes with the $^{75}\text{As}^{\pm}$ signal. Specimen offset voltage (detection of secondary-ion with high energy) or high mass resolution operation are effective for decreasing the background signal.

9.4.2 The ion species of silicon which has an appropriate ion intensity shall be detected, following the manufacturer's instructions or a local documented procedure.

If the instrument has an electrometer detection mode, it is recommended to select $^{28}\text{Si}^{\pm}$ as the reference ion for As^{\pm} using the electrometer. If only a pulse-counting detector is available, $^{30}\text{Si}^{\pm}$ should be selected as the reference ion for As^{\pm} . For AsSi^{-} detection, either Si_2^{-} or Si_3^{-} are suitable reference ions, as determined in a round-robin study described in Reference [2] and Annex A. A variety of isotopic species with different count rates can be selected for measurement as reference ions because silicon has three stable isotopes. For pulse counting mode, the isotopic species, the primary-ion beam current and the secondary-ion transmission should be selected so that instantaneous count rates for all measured species are less than 5×10^5 counts/s throughout the depth profile.

NOTE When an amorphous silicon specimen is analysed, it is noted that Si-H cluster ions can interfere with the ^{29}Si -ion, ^{30}Si -ion and their molecular combinations. Therefore, the ^{28}Si -ion or its molecular ions (e.g. $^{28}\text{Si}^{\pm}$, $^{28}\text{Si}_2^{-}$, $^{28}\text{Si}_3^{-}$) are preferable as the matrix ion in this case.

9.5 Measurement of test specimen

9.5.1 Measurements shall be done in the central region of the specimen holder window.

9.5.2 The primary-ion beam current and the beam scan area shall be determined so that the data points necessary to record details of the profile are in the acceptable measurement range. It is useful to measure a test profile to aid in the determination of these conditions. When the arsenic-ion intensity of a specimen is high, care should be taken to avoid significant counting losses caused by pulse pile-up in the detector. If the instantaneous arsenic-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 9.2.3).

In an ion microscope type instrument, average ion intensities may be reduced in several ways. Increasing the raster size or selecting a smaller field aperture would not reduce the instantaneous ion intensity from the analysed region. The signal from the analysed region should be changed, instead, by reducing the primary-ion current or selecting a smaller entrance slit or contrast aperture.

NOTE 1 For additional information on this point, see Reference [3].

NOTE 2 For a discussion of the linearity of the counting system with an electron multiplier, see References [4] and [5].

9.5.3 The secondary-ion intensities of arsenic and silicon shall be measured alternately, and cyclically.

NOTE 1 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 at any one cycle for each analysis position.

NOTE 2 When the arsenic atomic concentration exceeds 2×10^{20} atoms/cm³ the reference ions Si₂⁻ and Si₃⁻ can be reduced in intensity in the region of high arsenic atomic concentration. In this case, it is necessary to measure the reference ion cyclically, and to perform a cycle-by-cycle normalization (see 10.1). For additional information on this point, see References [2] and [6].

9.6 Calibration

9.6.1 Calibration of depth scale by stylus profilometry

9.6.1.1 Calibrate the stylus profilometer for crater depth measurements using the reference materials stipulated in 6.2, in accordance with the manufacturer's instructions or a local documented procedure.

9.6.1.2 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.

9.6.2 Calibration of depth scale by optical interferometry

9.6.2.1 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.

9.6.2.2 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, m , intersected by line R at the crater edge.

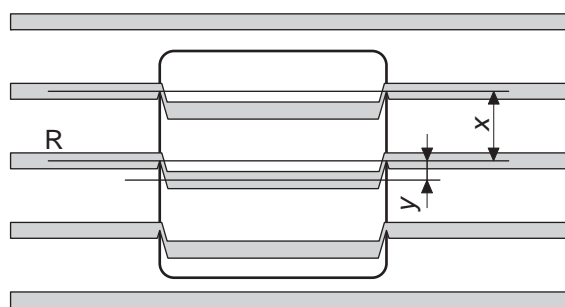


Figure 1 — Schematic drawing of interference fringes at a crater ($m = 0$)

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

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

Use a wavelength value determined by a traceable method.