# **BSI Standards Publication**

Surface chemical analysis

— Secondary-ion mass
spectrometry — Method for depth profiling of arsenic in silicon



BS ISO 12406:2010 BRITISH STANDARD

#### **National foreword**

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

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# INTERNATIONAL STANDARD

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# 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**

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

## 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.

ISO 12406:2010(E)

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

#### 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  $1 \times 10^{16}$  atoms/cm<sup>3</sup> and  $2.5 \times 10^{21}$  atoms/cm<sup>3</sup>, and to crater depths of 50 nm or deeper.

#### **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

#### Terms and definitions

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

#### Symbols and abbreviated terms

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

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$^{I}$ BG	IIICAII DACKUIUUIIU IUII	HITCHOLD OF THE 6	ai aci ilu-iuli auculca.	expressed in counts/s
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 $J_i$  ion intensity ratio of arsenic to silicon at measurement cycle, i

 $J_{\text{BG}}$  mean background ion intensity ratio of arsenic to silicon

n total number of measurement cycles

RSF<sup>imp</sup> relative-sensitivity factor of implanted arsenic in certified reference material (CRM) (cm<sup>-3</sup>)

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)

 $\Delta t^{As}$  duration of arsenic-ion acquisition in each measurement cycle, expressed in seconds (s)

 $\Phi$  implanted dose of arsenic in CRM, expressed in atoms per square centimetre (atoms/cm<sup>2</sup>)

## 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.

#### 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	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 <sub>2</sub> +
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,  $^{75}$ As $^{-}$  and/or  $^{75}$ As $^{28}$ Si $^{-}$  and/or  $^{75}$ As $^{30}$ Si $^{-}$  shall be detected as secondary-ion species of arsenic. If an oxygen-ion beam is used,  $^{75}$ 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  $5\times 10^{5}$  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  $^{29}\text{Si-ion},\,^{30}\text{Si-ion}$  and their molecular combinations. Therefore, the  $^{28}\text{Si-ion}$  or its molecular ions (e.g.  $^{28}\text{Si}_2^+,\,^{28}\text{Si}_2^-,\,^{28}\text{Si}_3^-)$  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  $5 \times 10^5$  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  $2 \times 10^{20}$  atoms/cm<sup>3</sup> the reference ions  $\mathrm{Si}_2^-$  and  $\mathrm{Si}_3^-$  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.

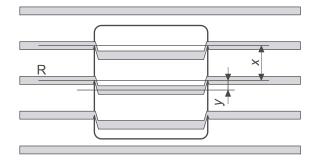


Figure 1 —Schematic drawing of interference fringes at a crater (m = 0)

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

$$d_{t} = \frac{\lambda}{2} \left( m + \frac{y}{x} \right) \tag{1}$$

Use a wavelength value determined by a traceable method.

#### 9.6.3 Calibration of relative-sensitivity factor

- **9.6.3.1** The RSF of implanted arsenic in the CRM, RSF<sup>imp</sup>, shall be calculated following the procedures given in ISO 18114. Calibration and test specimen measurements are preferably done on the same day using the same conditions as the test specimen measurements.
- **9.6.3.2** The measurement shall be continued through the ion-implanted zone until a constant background or the limit of detection is reached.

NOTE When NIST SRM 2134 is used as the CRM, the procedures given in Annex B shall be followed.

**9.6.3.3** The RSF shall be obtained from the following formula, following the procedures in Clause 8 of ISO 18114:2003.

$$RSF^{imp} = \frac{\Phi \times n}{\sum_{i=1}^{n} \left[ \frac{I_i - I_{BG}}{I_i^{Si}} \right] \times d}$$
 (2)

## 10 Expression of results

**10.1** The ion intensity ratios of arsenic to silicon in a test specimen,  $J_i$ , shall be determined for each measurement cycle using the following formula:

$$J_i = \frac{I_i}{I_i^{\text{Si}}} \tag{3}$$

**10.2** The arsenic atomic concentration of test specimens,  $C_i$ , shall be determined from the following formula, using the relative-sensitivity factor, RSF<sup>imp</sup>:

$$C_i = \mathsf{RSF}^{\mathsf{imp}} \times J_i \tag{4}$$

**10.3** If necessary, the background intensity of arsenic shall be subtracted from the intensity ratio. The background intensity ratio,  $J_{BG}$ , can be determined by calculating an average value from Equation (3) for a specimen not doped with arsenic:

$$C_i = \mathsf{RSF}^{\mathsf{imp}} \times (J_i - J_{\mathsf{BG}}) \tag{5}$$

**10.4** The depth for measurement cycle i,  $d_i$ , shall be determined from the following formula using the crater depth,  $d_t$ , measured in 9.6.1 or 9.6.2 and assuming a constant sputtering rate:

$$d_i = \left(t_i^{\text{As}} + \frac{\Delta t^{\text{As}}}{2}\right) \frac{d_{\text{t}}}{T} \tag{6}$$

**10.5** If the total number of measurement cycles, n, is so large that  $\Delta t^{As}/T$  is 0,1 % or less,  $d_i$  may be calculated using the following formula:

$$d_i = (i-1)\frac{d_t}{n} \tag{7}$$

NOTE The sputtering rate can vary in the initial stage of sputtering before the sputtering reaches an equilibrium state.

**10.6** When a graph is used to express measurement results,  $C_i$  shall be plotted as ordinate and  $d_i$  as abscissa. The data range and scaling (linear or logarithmic) shall be selected according to analytical requirements.

### 11 Test report

The test report shall include the following information:

- a) all information necessary for the identification of the specimens, the apparatus, the laboratory and the date of analysis;
- b) the reference materials used, by reference to this International Standard, i.e. ISO 12406:2010 (see Clause 6);
- c) the results and the form in which they are expressed;
- d) any unusual features noted during the analyses;
- e) any operation not specified in this International Standard, or any optional operation which might have influenced the results.

# Annex A

(informative)

# Report of round robin test of depth profiling of arsenic in silicon

#### A.1 General

This International Standard was subjected to an interlaboratory test programme involving 16 laboratories in 7 countries. Three specimens of ion-implanted arsenic in silicon were analysed to cover the scope of the method. Repeatability and reproducibility of the measured arsenic dose were calculated according to the principles of ISO 5725-2.

## A.2 Design of test programme

Each participating laboratory was asked to perform SIMS depth profile measurements of the three test samples and one reference sample and to make between three and five independent measurements on each sample. The determined ion-implanted arsenic dose was requested for each measurement of the test samples.

### A.3 Test samples

Three single crystal silicon wafers that had been ion-implanted with arsenic at an energy of 100 keV and at doses of approximately  $2\times10^{13}$  atoms/cm²,  $8\times10^{14}$  atoms/cm² and  $2\times10^{16}$  atoms/cm² were prepared by cutting them into 1 cm  $\times$  1 cm sections. The peak arsenic concentrations were approximately  $2.5\times10^{18}$  atoms/cm³,  $1\times10^{20}$  atoms/cm³, and  $2.5\times10^{21}$  atoms/cm³, respectively. The implanted doses had previously been measured by neutron activation analysis (NAA). Two pieces of each sample were distributed to each of the participating laboratories. Each laboratory was requested to use NIST SRM 2134 as a reference material for determining the RSF of arsenic to silicon.

## A.4 Procedure for SIMS analysis

Laboratories were requested to determine the ion-implanted doses of arsenic in the test specimens using the procedures described in 9.4 and 9.5 and the calibrations described in 9.6. They were requested to use a Cs<sup>+</sup> ion beam with an impact energy of 3 keV and an angle of incidence of 60° from normal, but if these conditions were not possible they could be modified. They were requested to use AsSi<sup>-</sup> as the analyte ion and to report results with both  $\rm Si_2^-$  and  $\rm Si_3^-$  as the matrix ion. Ion intensity ratios in the test specimen were calculated as cycle-by-cycle ratios to the matrix ion signal as in 10.1, and also as ratios to the average matrix ion signal measured in a region of the profile where the signal is constant.

Laboratories could optionally report additional measurements using an  $O_2^+$  ion beam with  $As^+$  as the analyte ion and  $Si^+$  as the matrix ion.

#### A.5 Statistical procedures

### A.5.1 Scrutiny for consistency and outliers

Cochran's test and Grubbs' test were applied to the data in accordance with ISO 5725-2. Only one outlying measurement was detected and it was excluded from further analysis.

### A.5.2 Computation of repeatability and reproducibility

The results from each laboratory were treated according to 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:  $s_R^2 = s_r^2 + s_L^2$

# A.6 Results of statistical analysis

- **A.6.1** The results of the statistical analysis for  $O_2^+$  ion bombardment and As<sup>+</sup> as the analyte ion are given in Table A.1. The table combines results from one quadrupole SIMS instrument and two magnetic sector SIMS instruments from three different laboratories. One of the laboratories analysed the samples with and without oxygen gas flooding of the sample surface. Another laboratory analysed the samples with and without kinetic energy filtering of the secondary ions (see Note in 9.4.1). All analyses are combined in Table A.1. Only one measurement was made of the low-dose sample, so no statistical analysis could be made in that case.
- **A.6.2** The results of the statistical analysis for Cs<sup>+</sup> ion bombardment and AsSi<sup>-</sup> as the analyte ion are given in Table A.2. The table combines results from eight quadrupole SIMS instruments and eight magnetic sector SIMS instruments. Several of the magnetic sector instruments used bombardment energies of ca. 15 keV but the data from these instruments were not distinguishable from the rest, so all of the data were analysed together.
- **A.6.3** A bias value is given in Tables A.1 and A.2 as a measure of the disagreement between the mean SIMS dose value for a given sample and measurement condition and the value obtained by neutron activation analysis that has an estimated relative uncertainty of 1 %. In all cases, the reproducibility standard deviation is larger than the bias except for the high-dose sample measured with Cs<sup>+</sup> ion bombardment when a constant average matrix signal was used to form the ion intensity ratios. For this reason, calculation of ion intensity ratios from cycle-by-cycle determinations of the matrix ion intensities, as indicated in 10.1, is recommended.

Table A.1 —Repeatability and reproducibility of arsenic dose measurements in mode of  $\rm O_2^+$  bombardment and  $\rm As^+$  detection with two types of normalization

Units of dose are atoms/cm<sup>2</sup>

		Dose	level	
	Me	dium	Hi	igh
Component		Norm	. type	
Component	Constant	Cycle-by-cycle	Constant	Cycle-by-cycle
		No. con	ditions	
	5	5	5	5
NAA value	$9,07 \times 10^{14}$	9,07 × 10 <sup>14</sup>	$1,71 \times 10^{16}$	$1,71 \times 10^{16}$
Mean value	$8,86 \times 10^{14}$	8,84 × 10 <sup>14</sup>	$1,67 \times 10^{16}$	$1,67 \times 10^{16}$
% bias	-2,29 %	-2,52 %	-2,58 %	-2,53 %
$S_r$	2,21 × 10 <sup>13</sup>	2,33 × 10 <sup>13</sup>	5,20 × 10 <sup>14</sup>	5,04 × 10 <sup>14</sup>
s <sub>r</sub> (%)	2,50 %	2,64 %	3,12 %	3,02 %
$s_L$	$4,22 \times 10^{13}$	$3,47 \times 10^{13}$	1,27 × 10 <sup>15</sup>	1,00 × 10 <sup>15</sup>
s <sub>L</sub> (%)	4,76 %	3,93 %	7,60 %	6,01 %
$s_R$	4,76 × 10 <sup>13</sup>	4,19 × 10 <sup>13</sup>	1,37 × 10 <sup>15</sup>	1,12 × 10 <sup>15</sup>
s <sub>R</sub> (%)	5,37 %	4,73 %	8,22 %	6,73 %

Units of dose are atoms/cm<sup>2</sup>

Table A.2 —Repeatability and reproducibility of arsenic dose measurements in mode of Cs⁺ bombardment and AsSi⁻ detection with two types of normalization and two types of matrix species

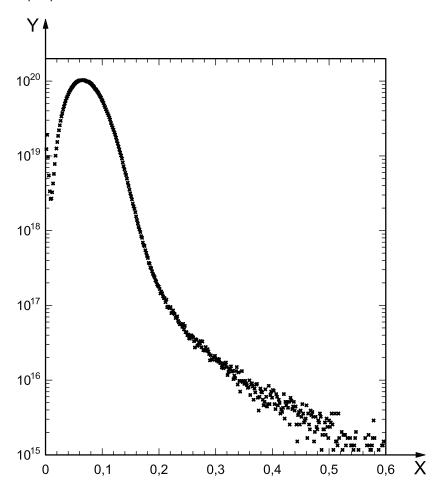
						Dose level	level					
		Pc	Low			Medium	ium			High	h	
						Norm. type	type					
tuonon tu	Con	Constant	Cycle-b	Cycle-by-cycle	Constant	stant	Cycle-by-cycle	y-cycle	Constant	tant	Cycle-by-cycle	y-cycle
						Matrix species	pecies					
	Si <sub>2</sub>	Si <sub>3</sub>	Si <sub>2</sub>	Si <sub>3</sub>	Si <sub>2</sub>	Si <sub>3</sub>	Si <sub>2</sub>	Si <sub>3</sub>	Si <sub>2</sub>	Si <sub>3</sub>	Si <sub>2</sub>	Si <sub>3</sub>
						No. labs	abs					
	14	15	14	15	14	15	14	15	14	14	14	15
NAA value	$2,09\times10^{13}$	$2,09\times10^{13}$	$2,09\times 10^{13} \ \ 2,09\times 10^{13} \ \ 2,09\times 10^{13}$	$2,09\times10^{13}$	$9,07 \times 10^{14}$	$9,07 \times 10^{14}$ $9,07 \times 10^{14}$	$9,07 \times 10^{14}$	$9,07\times 10^{14} \ \ 9,07\times 10^{14} \ \ 1,71\times 10^{16} \ \ 1,71\times 10^{16}$	$1,71 \times 10^{16}$		$1,71\times 10^{16}  1,71\times 10^{16}$	$1,71 \times 10^{16}$
Mean value	$2,09\times10^{13}$	$2,09 \times 10^{13}$ $2,10 \times 10^{13}$ $2,06 \times 10^{13}$	$2,06 \times 10^{13}$	$2,07\times0^{13}$	$8,82\times10^{14}$	$8,81\times10^{14}$	$8,84 \times 10^{14}$	$8,76\times10^{14}$	$1,51\times 10^{16}  1,51\times 10^{16}$	$1,51 \times 10^{16}$	$1,63\times10^{16}$	$1,71\times10^{16}$
% bias	0,16 %	0,32 %	-1,45 %	-0,92 %	-2,72 %	-2,85 %	-2,55 %	-3,39 %	-11,78 %	-11,62 %	-4,50 %	-0,34 %
$S_F$	$5,69 \times 10^{11}$		$5.01\times 10^{11}  5.30\times 10^{11}$	$5,63\times10^{11}$	$2,23\times10^{13}$	$2,12\times10^{13}$	$2,38\times10^{13}$	$2,72\times10^{13}$	$4,82\times10^{14}$	$2,65\times10^{14}$	$6,26 \times 10^{14}$	$4,25\times10^{14}$
$S_{r}$ (%)	2,72 %	2,39 %	2,58 %	2,72 %	2,53 %	2,41 %	% 69'Z	3,10 %	3,19 %	1,75 %	3,83 %	2,49 %
$T_S$	$1,53 \times 10^{12}$	$1,53 \times 10^{12}$ $1,29 \times 10^{12}$ $1,39 \times 10^{12}$	$1,39 \times 10^{12}$	$1,04 \times 10^{12}$	$2,62\times10^{13}$	$4,01 \times 10^{13}$	$3,29\times10^{13}$	$3,18\times10^{13}$	$7,22\times 10^{14}  1,13\times 10^{15}$	$1,13\times10^{15}$	$8,16\times10^{14}$	$6,69 \times 10^{14}$
$S_L$ (%)	7,30 %	6,17 %	6,77 %	5,04 %	2,97 %	4,55 %	3,72 %	3,63 %	4,78 %	7,44 %	4,99 %	3,92 %
$S_R$	$1,63 \times 10^{12}$	$1,39 \times 10^{12}$	$1,49 \times 10^{12}$	$1,49 \times 10^{12}$ $1,19 \times 10^{12}$	$3,44 \times 10^{13}$	$4,53\times10^{13}$	$4,06 \times 10^{13}$	$4,19\times10^{13}$	$8,69 \times 10^{14}$	$1,16 \times 10^{15}$	$1,03\times10^{15}$	$7,92\times10^{14}$
$s_R$ (%)	7,79 %	6,61 %	7,24 %	5,72 %	3,90 %	5,15 %	4,59 %	4,78 %	2,75 %	7,64 %	6,29 %	4,64 %

# Annex B (normative)

# Procedures for the depth profiling of NIST SRM 2134

# **B.1 Data sampling range**

The depth profile shall be obtained from the surface to at least 0,5  $\mu$ m (see B.2). Figure B.1 shows an example of a SIMS depth profile of NIST SRM 2134.



#### Key

- X depth ( $\mu m$ )
- Y <sup>75</sup>As concentration (atoms/cm<sup>3</sup>)

Figure B.1 —SIMS depth profile of arsenic in NIST SRM 2134

## **B.2 Procedures for depth profiling**

Since the arsenic concentration at the peak position is approximately  $1\times 10^{20}$  atoms/cm³, care should be taken so as not to saturate the detector. If the instantaneous arsenic intensity at the peak position is higher than  $5\times 10^5$  counts/s in the gated area, the primary-ion intensity shall be reduced. The primary-ion beam current and the beam scan area shall be determined so that at least 50 measurement cycles are in the 0  $\mu$ m to 0,5  $\mu$ m range (sputtering rate is 10 nm/cycle or less).

The secondary-ion intensities of arsenic and silicon shall be measured alternately, for at least one second for arsenic per cycle. The measurement shall be continued until a depth of  $0.5 \, \mu m$  is reached or to a measurement cycle 20 cycles or more after the constant background or limit of detection is reached.

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