BRITISH STANDARD

BS ISO 23812:2009

Surface chemical analysis — Secondary-ion mass spectrometry — Method for depth calibration for silicon using multiple delta-layer reference materials

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National foreword

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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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Foreword

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ISO 23812 was prepared by Technical Committee ISO/TC 201, *Surface chemical analysis*, Subcommittee SC 6, *Secondary ion mass spectrometry*.

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Introduction

Secondary-ion mass spectrometry (SIMS) is a powerful method for the measurement of depth profiles of dopants in silicon. However, in the near-surface region (< 50 nm), the transient behaviours of the secondary-ion yields and the sputtering rate significantly affect the profile shape [1], thus it is difficult to obtain real profiles. This is caused by the accumulation of implanted primary-ion species, oxygen or caesium, which are essential for enhancing the secondary-ion yields. At the original surface, sputtering of the specimen material occurs with a low concentration of primary-ion species, but, with the progress of sputtering, primary-ion species are incorporated on the surface and sputtered together with the specimen atoms, causing a sputtering-rate change. As a result of the sputtering-rate change in this non-equilibrium zone, a significant profile shift occurs in shallow SIMS depth profiles when a uniform sputtering rate is assumed for depth calibration.

To calibrate the depth scale in such a shallow region, it is essential to evaluate the extent of the above profile shift accurately. In this International Standard, multiple delta-layers are used as a reference material for depth scale calibration in the near-surface region but beyond the non-equilibrium zone, and the procedures for depth scale calibration are described.

This International Standard differs from ISO 20341 in its scope. ISO 20341 specifies procedures for estimating depth resolution parameters in SIMS depth profiling using multiple delta-layer reference materials, whereas this International Standard specifies a procedure for calibrating the depth scale in a shallow region.

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Surface chemical analysis — Secondary-ion mass spectrometry — Method for depth calibration for silicon using multiple delta-layer reference materials

1 Scope

1.1 This International Standard specifies a procedure for calibrating the depth scale in a shallow region, less than 50 nm deep, in SIMS depth profiling of silicon, using multiple delta-layer reference materials.

1.2 This International Standard is not applicable to the surface-transient region where the sputtering rate is not in the steady state.

1.3 This International Standard is applicable to single-crystalline silicon, polycrystalline silicon and amorphous silicon.

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 18115, *Surface chemical analysis — Vocabulary*

ISO 20341, *Surface chemical analysis — Secondary-ion mass spectrometry — Method for estimating depth resolution parameters with multiple delta-layer reference materials*

3 Terms and definitions

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

4 Symbols and abbreviated terms

a intercept of regression line

b slope of regression line

c extra factor of regression line slope

$I(z)$ ion intensity at depth z

k correction factor for the primary-ion current density

l number of delta-layers used for regression analysis

L_s shift distance

$L_s(p)$ shift distance dependent on the definition of delta-layer peak position (see 7.2.1)

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m	maximum delta-layer number used for steady-state sputtering-rate determination
n	minimum delta-layer number where steady-state sputtering rate is reached
r	sputtering rate
r_i	average sputtering rate of ith silicon layer
r_s	steady-state sputtering rate
s_0	standard uncertainty in predicted depth without s_RM and s_c
s_1	standard uncertainty in depth, including s_RM
s_2	standard uncertainty in depth, including s_RM and s_k
s_b	standard uncertainty in b
s_c	standard uncertainty in c
s_k	standard uncertainty in k
s_RM	standard uncertainty in depth of layers in reference material
s_t	standard uncertainty in sputtering time t
$s_{\bar{t}}$	standard uncertainty in $\bar{t}$
$T(m-n-1,\ \alpha)$	critical value obtained from Student's t-distribution table at 100α % for $(m-n-1)$ degrees of freedom
t	sputtering time
$\bar{t}$	mean sputtering time for delta-layers
t_f	sputtering time needed for forming crater
t_i	measured sputtering time to reach the ith delta-layer in the ion-intensity-versus-sputtering-time profile
$\hat{t}_i$	time predicted for ith delta-layer
z	sputtered depth
$\bar{z}$	mean depth of delta-layers
$\hat{z}$	predicted depth
z_{ai}	minimum position in the valley between $(i-1)$th and ith delta profiles
z_{bi}	minimum position in the valley between ith and $(i+1)$th delta profiles
z_c	centroid position
z_d	original delta-layer position
z_f	crater depth
z_i	depth of ith delta-layer
$\langle z_i \rangle$	centroid of the ith delta-layer profile
z_LM	local maximum position

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z_{PC}	peak centre position
Δt_i	sputtering time for ith delta-layer
Δz_i	thickness of ith delta-layer
SIMS	secondary-ion mass spectrometry

5 Requirements on multiple delta-layer reference materials

5.1 The criteria given in 5.2 to 5.8 are appropriate for delta-layers used as the reference material.

5.2 The signal of the matrix containing the delta-layers shall not change during SIMS depth profiling so that no changes occur due to any SIMS matrix effects or due to the sputtering-rate change at delta-layers. Constant secondary-ion intensities of the matrix elements through the delta-layers are indicative of a constant matrix.

5.3 The constancy, across the specimen, of the depths of the delta-layers below the surface shall be comparable to, or better than, the required depth calibration accuracy.

5.4 The thickness of each doped delta-layer shall be sufficiently less than the projected range of the primary ions (see Annex A for examples for O_2^+ ions) so that a small variation in the thickness does not affect the profile shape.

5.5 The spacing between adjacent delta-layers shall be sufficiently large that the peak-to-valley ratio of the adjacent delta-layer profiles shall be 10 or greater.

5.6 At least three delta-layers shall be contained in the reference material to confirm that a steady-state sputtering rate is reached in the delta-doped region.

5.7 The thickness, the position and the interface roughness of the delta-layers shall be determined by cross-sectional transmission electron microscopy, grazing incidence X-ray reflectivity, medium-energy ion scattering spectrometry or another appropriate method.

5.8 The thickness of the surface oxide layer shall be comparable with that of the native oxide layer on the crystalline-silicon surface. The oxide thickness shall be determined by a valid measurement method.

6 Measurement procedures

6.1 For precise calibration of the depth scale, multiple delta-layer reference materials shall be measured with test specimens using the same energy and incident angle of the primary ions. Once the sputtering rate, the depth correction factor and the shift distance (see 7.2.3) are determined for a specific set of ion-irradiation conditions (for energy, angle of incidence, ion current and scan area), the predetermined values can be used for the determination of the depth scale. It should be noted that the depth scale can have a deviation due to limited reproducibility of the sputtering rate (see 6.5).

6.2 For adjustment and optimization of the secondary-ion mass spectrometer settings, the analysis conditions, such as ion energy, ion species, ion current, secondary-ion polarity, primary-ion scan region, analysed region, stability of the primary-ion current, specimen introduction and detected secondary ions, shall be set in accordance with the manufacturer's instruction or a local documented procedure.

6.3 The SIMS depth profile of multiple delta-layer reference materials shall be measured from the surface to near the interface between the multiple delta-layers and the substrate, or to a depth described in the instructions for the reference materials. However, if a change in secondary-ion intensity occurs due to surface roughness induced by ion bombardment before the above depth is reached, depth profiling shall be terminated before the surface roughening occurs.

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6.4 The measurement conditions, such as the primary-ion intensity and scanning area, shall be determined so that there are more than 20 measurement points for a delta-layer.

6.5 When confirming reproducibility, the SIMS depth profile of a multiple delta-layer reference material shall be measured three times, and the repeatability of the profile shall be checked. The time to each position of the delta-layers shall be reproduced with the accuracy of the analytical requirement. If the SIMS profile shapes of the multiple delta-layer reference material are not reproducible, the instrument performance shall be checked for items such as the drift of primary-ion current and the scan uniformity.

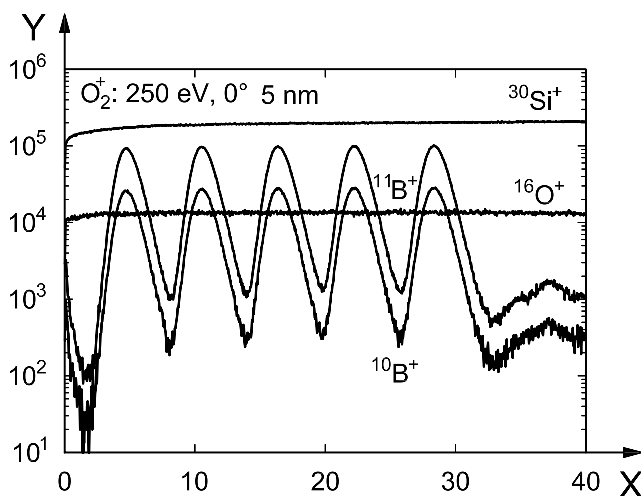
6.6 The SIMS depth profile of test specimens shall be measured using the same measurement conditions as those used for the multiple delta-layer reference materials. If necessary, the primary-ion current density can be changed by changing the primary-ion current and/or the primary-ion scan area.

7 Calibration procedures

7.1 Principle of calibration

7.1.1 This International Standard gives equations for calibrating the depth scale by correcting for the change in sputtering rate in the non-equilibrium zone in the initial stage of sputtering.

7.1.2 Sputter-depth profiling causes the change in shape of the delta-layer as shown in Figure 1, and the shift of the delta-peak position even after the equilibrium of sputtering rate is established. Estimations of the peak shifts due to atomic mixing and peak coalescence are given in Annex B and Annex C, respectively.



Key

X depth, in nm

Y ion intensity, in counts/s

Figure 1 — SIMS depth profile of boron delta-layers in silicon [2]

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7.2 Determination of sputtering rate for reference material

7.2.1 The positions of the delta-layers in the reference material shall be determined in the measured profile for ion intensity versus sputtering time. The position can be determined by one of four methods which generate different shift distances. Select the method that is most consistent with the type of data measured in the working specimen.

a) The position can be determined as the local maximum in a delta-layer profile.

b) The position can be determined as the centre of the full width at half maximum of each delta-layer profile.

c) The position can be determined as the centroid $\langle z_i \rangle$ of the ith delta-layer by using the following equation:

$$\langle z_i \rangle = \frac{\sum_{z_{ai}}^{z_{bi}} z I(z)}{\sum_{z_{ai}}^{z_{bi}} I(z)} \qquad (1)$$

where z_{ai} and z_{bi} are the minima in the valleys between the $(i - 1)$th and ith delta profiles and between the ith and $(i + 1)$th delta profiles, respectively, and $I(z)$ is the ion intensity at z.

d) The position can be determined as z_0 in Equation C.1 (see Annex C) for each delta peak by following the procedures described in ISO 20341.

In cases a) and b), it is desirable to use a proper smoothing procedure taking into account statistical fluctuations.

NOTE 1 These positions may shift from the original delta position due to atomic mixing, and the direction of shift depends on the relative knock-on of matrix and analyte atoms. The amount of the shift depends on the primary-ion energy and angle of incidence. To reduce the shift, an appropriate primary-ion energy and angle of incidence have to be selected. Assuming the effect of atomic mixing is the same for matrix and analyte atoms, estimations of peak shift due to atomic mixing can be made as indicated in Annex B. Boron in silicon can be approximated to the simple case.

NOTE 2 Peaks of delta-layers are reported to show a much more pronounced non-linear energy-dependent shift than centroids up to a factor of 2 stronger [3]. When coalescence of adjacent peaks is negligible (see Note 1 to 7.2.3), and the effect of atomic mixing is the same for matrix and analyte atoms, the centroid almost represents the original delta position.

7.2.2 The sputtering time necessary to reach the ith delta-layer from the surface is defined as t_i (see Figure 2). The difference between adjacent positions, Δt_i $(= t_i - t_{i-1})$, denotes the time needed to sputter off the ith silicon layer from the surface, where $t_0 = 0$. The corresponding depth is denoted as z_i and the thickness of the ith silicon layer is Δz_i $(= z_i - z_{i-1})$. The average sputtering rate of the ith layer is given by

$$r_i = \frac{\Delta z_i}{\Delta t_i} \qquad (2)$$

Due to the transient behaviour of ion yield, determination of positions for the first one or two delta-layers may not be possible. In such a case, depth calibration shall be performed using delta-layers whose positions can be determined. For the region shallower than the first detectable delta-layer, the sputtered depth shall be approximated by using the average sputtering rate from the surface to the first detectable delta-layer.

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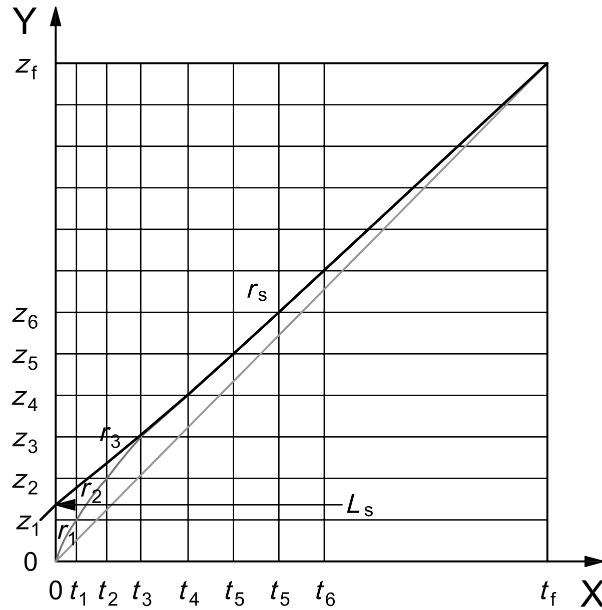

Key

X sputtering time

Y sputtered depth

Figure 2 — Schematic drawing showing the relationship between the sputtered depth and sputtering time in the shallow region

7.2.3 The average sputtering rate for each delta-layer shall be determined. In general, the average sputtering rate is larger at or near the surface, but reaches a steady-state value (the steady-state sputtering rate, r_s) after a few nanometres. When r_i can be regarded as r_s for the nth layer and deeper, the sputtered depth z for the sputtering time t is given by

$$\begin{aligned} z &= \sum_{i=1}^{n-1} r_i \left(t_i - t_{i-1}\right) + r_s \left(t - t_{n-1}\right) \\ &= \sum_{i=1}^{n-1} r_i \left(t_i - t_{i-1}\right) - r_s t_{n-1} + r_s t \qquad (z \geqslant z_n) \\ &= L_s + r_s t \end{aligned} \tag{3}$$

where

$$\begin{aligned} L_s &= \sum_{i=1}^{n-1} r_i \left(t_i - t_{i-1}\right) - r_s t_{n-1} \\ &= \sum_{i=1}^{n-1} \Delta z_i \left(1 - \frac{r_s}{r_i}\right) \end{aligned} \tag{4}$$

L_s is the shift distance and is independent of the primary-ion current density.

NOTE 1 For a short-period multiple delta-layer specimen, measured delta profiles may coalesce with each other so that 5.5 is not fulfilled. When the profile overlapping is significant, the measured delta position is affected by the coalescence. However, the peak shift caused by the coalescence reaches a steady-state value for deeper deltas. Thus, the delta position interval, Δz_i, is usable for depth calibration. When the peak-to-valley ratio of the adjacent delta-layer

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profiles is larger than 2, the third delta or deeper can be used for depth calibration. When the peak-to-valley ratio is 10 or greater, each layer can be regarded as being isolated. In this case, even the first delta can be used if sputtering reaches a steady state before the first layer. Estimations of peak shifts due to delta profile coalescence are presented in Annex C.

NOTE 2 The value of L_s may depend on the choice of procedure a), b), c) or d) in 7.2.1.

7.2.4 When delta-layers from the nth layer to the mth layer are used, L_s and r_s shall be determined by using linear regression of the $(n - m + 1)$ sputtering times referencing the certified depths of the delta-layers for $z \geqslant z_n$.

$$L_\mathrm{s} = \frac{\sum_{i=n}^{m} t_i^2 \sum_{i=n}^{m} z_i - \sum_{i=n}^{m} t_i \sum_{i=n}^{m} t_i z_i}{(m-n+1)\sum_{i=n}^{m} t_i^2 - \left(\sum_{i=n}^{m} t_i\right)^2} \tag{5}$$

$$r_\mathrm{s} = \frac{(m-n+1)\sum_{i=n}^{m} t_i z_i - \sum_{i=n}^{m} t_i \sum_{i=n}^{m} z_i}{(m-n+1)\sum_{i=n}^{m} t_i^2 - \left(\sum_{i=n}^{m} t_i\right)^2} \tag{6}$$

The error in L_s associated with the shift caused by atomic mixing shall be taken into account for depth calibration (see Note 1 to 7.2.1).

7.3 Calibration of the depth scale for test specimens

7.3.1 Calibration under the same sputtering conditions as used for the reference material

When the sputtered depth is calibrated by using the sputtering rate determined from the multiple delta-layer reference material, the following equation shall be used for depth calibration for $z \geqslant z_n$. In this case, the sputtering condition for test specimens shall be the same as those for the reference material.

$$z = L_\mathrm{s} + r_\mathrm{s} t \tag{7}$$

7.3.2 Calibration using a sputtering rate different from that used for the test specimen

When the primary-ion current density differs from that of the reference material by a factor k, the sputtered depth is given by the following equation, provided other sputtering conditions are unchanged:

$$\begin{aligned} z &= \sum_{i=1}^{n-1} k r_i \left(\frac{t_i}{k} - \frac{t_{i-1}}{k} \right) + k r_\mathrm{s} \left(t - \frac{t_{n-1}}{k} \right) \\ &= \sum_{i=1}^{n-1} r_i \left(t_i - t_{i-1} \right) - r_\mathrm{s} t_{n-1} + k r_\mathrm{s} t \qquad (z \geqslant z_n) \\ &= L_\mathrm{s} + k r_\mathrm{s} t \end{aligned} \tag{8}$$

The sputtered depth for $z \geqslant z_n$ shall be determined by Equation (8), using the L_s and r_s values determined from the reference material.

NOTE Equation (8) can be used when the sputtering rate of the test specimen differs from that of the reference material for the same primary-ion current density. This can happen when the density of silicon is different between test specimen and reference material.

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7.4 Uncertainty in calibrated depth

7.4.1 The shift of the delta position depends on the method of peak position determination described in 7.2.1. Thus, L_{s} may vary depending on the method used. Annex B gives an estimation of the variation, but the actual shift also depends on the delta-layer element as well as the ion irradiation condition, and a general equation might not be available. Here, the definition-dependent shift distance is expressed as $L_{\mathrm{s}}(p)$.

7.4.2 When r_{s} is determined using delta-layers from the nth layer to the mth layer, the $100 \times (1 - 2\alpha)$ % confidence interval for the calibrated depth for Equation (7) is given by

$$L_{\mathrm{s}}(p) + r_{\mathrm{s}} t \pm T(m-n-1, \alpha) \times s_1 \tag{9}$$

where $T(m - n - 1, \alpha)$ is the critical value obtained from Student's t-distribution table at 100α % for $(m - n - 1)$ degrees of freedom and s_1 is calculated by using the following equation with the uncertainty for the reference material s_{RM}:

$$s_1 = r_{\mathrm{s}} \times \left[1 + \frac{1}{m-n+1} + \frac{r_{\mathrm{s}}^2 \left(t - \frac{1}{m-n+1} \sum_{i=n}^{m} t_i \right)^2}{\sum_{i=n}^{m} \left(z_i - \frac{1}{m-n+1} \sum_{i=n}^{m} z_i \right)^2} \right]^{\frac{1}{2}} \times \left[\frac{1}{m-n-1} \sum_{i=n}^{m} \left(t_i + \frac{L_{\mathrm{s}}(p)}{r_{\mathrm{s}}} - \frac{1}{r_{\mathrm{s}}} z_i \right)^2 + \frac{s_{\mathrm{RM}}^2}{r_{\mathrm{s}}} \right]^{\frac{1}{2}} \tag{10}$$

For Equation (8), the $100 \times (1 - 2\alpha)$ % confidence interval for the calibrated depth is given by

$$L_{\mathrm{s}}(p) + k r_{\mathrm{s}} t \pm T(m-n-1, \alpha) \times s_2 \tag{11}$$

where

$$s_2 = k r_{\mathrm{s}} \times \left\{ \left[1 + \frac{1}{m-n+1} + \frac{r_{\mathrm{s}}^2 \left(t - \frac{1}{m-n+1} \sum_{i=n}^{m} t_i \right)^2}{\sum_{i=n}^{m} \left(z_i - \frac{1}{m-n+1} \sum_{i=n}^{m} z_i \right)^2} \right] \times \left[\frac{1}{m-n-1} \sum_{i=n}^{m} \left(t_i + \frac{L_{\mathrm{s}}(p)}{r_{\mathrm{s}}} - \frac{1}{r_{\mathrm{s}}} z_i \right)^2 + \frac{s_{\mathrm{RM}}^2}{r_{\mathrm{s}}} \right] + k^2 \left(t - \frac{1}{m-n+1} \sum_{i=n}^{m} t_i \right)^2 s_k^2 \right\}^{\frac{1}{2}} \tag{12}$$

and s_k is the uncertainty in k.

The derivation of these equations is presented in Annex D.

8 Expression of results

8.1 Calibration under the same sputtering conditions as used for the reference material

The shift distance, L_{s}, and steady-state sputtering rate, r_{s}, shall be determined by using linear regression of the measured delta sputtering times referencing the certified depths of the delta-layers, then the sputtering time shall be converted to the sputtered depth using Equation (7).

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8.2 Calibration using a sputtering rate different from that of the test specimen

The shift distance, L_s, and steady-state sputtering rate, r_s, shall be determined by using linear regression of the measured delta sputtering times referencing the certified depths of the delta-layers, then the sputtering time shall be converted to the sputtered depth using Equation (8).

8.3 Calibration with respect to concentration

If necessary, the ion intensity shall be converted to atomic concentration in accordance with other International Standards or other local documented procedures.

9 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 multiple delta-layer reference material used, as specified in this International Standard;

c) the analysis conditions used;

d) any unusual features noted during the analysis;

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

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Annex A
(informative)

Projected range of oxygen-ion in silicon

This annex shows the range of primary ions, O_2^+, in silicon, calculated using SRIM (see http://www.srim.org/).

The effect of native oxide was not included in the calculation.

Table A.1 — Projected range of O_2^+ in silicon

Primary-ion energy eV	**Projected range** nm	
	0° incidence	45° incidence
250	1,3	1,1
500	2,0	1,7
1 000	3,0	2,4
2 000	4,7	3,7

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Annex B
(informative)

Estimations of peak shifts due to atomic mixing

B.1 General

This annex describes the peak shift of delta-layer profiles due to atomic mixing in order to give criteria for ion-sputtering conditions of shallow depth profiling by SIMS. The centroid, local maximum and centre of the full width at half maximum of each delta-layer were derived from measured depth profiles of boron in silicon and compared with the original delta-layer position estimated from a theoretical model. It should be noted that the theoretical model assumes the same atomic mixing of matrix and analyte atoms. Boron in silicon can be treated by the simple model, but measured peak positions can behave differently when the atomic number of the analyte is very different from that of the matrix or when radiation-induced segregation occurs.

B.2 Procedure

SIMS depth profiles of BN multiple delta-layers were analysed based on an atomic mixing, surface roughness, information depth (MRI) model [4]. In this analysis, only two parameters, atomic mixing and surface roughness, were used for fitting the experimental data because the information depth can be regarded to be zero in SIMS [5]. The MRI model can extract the original delta position from measured depth profiles, provided that no segregation of delta-layer material occurs. The centroid, the local maximum and the centre of the full width at half maximum (peak centre) were derived from each delta-layer profile. When mixing is a homogeneous process, which is the assumption of the MRI model, the centroid coincides with the original delta position. So the amount of peak position shift was determined as the difference from the centroid position. The specimen used was one with well-separated BN delta-layers [2]. The Si spacer layer thickness was 18,5 nm. In the calculation, the same parameters were applied to the delta-layer and matrix atoms.

B.3 Results

The position of the centroid, z_c, relative to the original delta-layer position, z_d, is shown in Figure B.1 for various ion energies and incident angles of primary O_2^+ beam. z_c can be regarded as almost unchanged except for the 3 keV data. The positions of the local maximum, z_{LM}, and peak centre, z_{PC}, shift towards the surface significantly compared to the centroid. The shifts of these positions relative to the centroid, $z_{LM} - z_c$ and $z_{PC} - z_c$, are shown in Figures B.2 and B.3, respectively. The amount of the shift depends primarily on the ion energy and is larger for the local maximum than for the peak centre. The shift value of the local maximum is about twice that of the peak centre. For ion energies of 1 keV or less, the shift of the local maximum is less than 1 nm.

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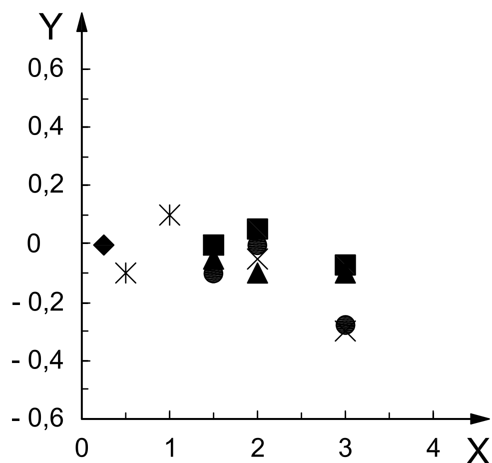


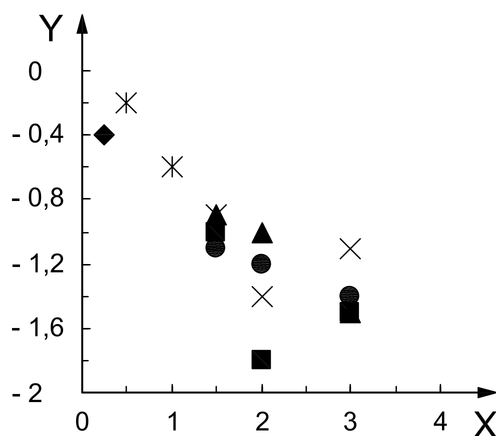
Incident angle	
◆	0°
■	9°
▲	27°
×	45°
✳	45° (O_2 leak)
●	63°

Key

X primary-ion energy, in keV

Y $z_c - z_d$, in nm

Figure B.1 — Shift of centroid positions relative to the original delta-layer position deduced using the MRI model with the same parameters for delta-layer and matrix atoms



Incident angle	
◆	0°
■	9°
▲	27°
×	45°
✳	45° (O_2 leak)
●	63°

Key

X primary-ion energy, in keV

Y $z_{LM} - z_c$, in nm

Figure B.2 — Shift of local maximum position relative to centroid of delta-layers

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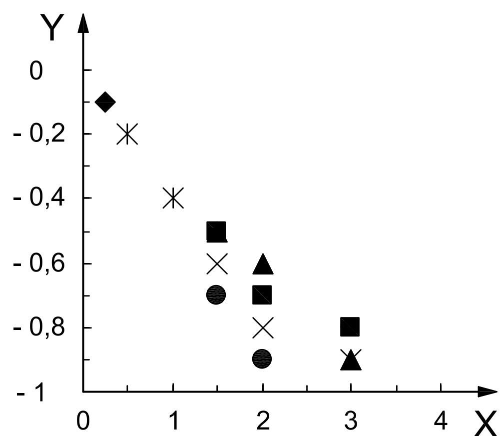

Incident angle	
◆	0°
■	9°
▲	27°
×	45°
✳	45° (O_2 leak)
●	63°

Key

X primary-ion energy, in keV

Y $z_{PC} - z_c$, in nm

Figure B.3 — Shift of peak centre relative to centroid of delta-layers

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Annex C
(informative)

Estimations of peak shift due to peak coalescence

C.1 General

This annex describes the apparent peak shift of delta-layer profiles due to peak coalescence in order to give criteria for multiple delta-layers as the reference material for shallow depth profiling by SIMS. The shifts of the centroid, the local maximum and the centre of the full width at half maximum of delta-layers in steady-state depth profiling were estimated from simulated profiles of short-period multiple delta-layers.

C.2 Procedure

SIMS depth profiles of multiple delta-layers were numerically synthesized using a model profile function of a delta-layer [6]:

$$I(z) = \frac{1}{\lambda_L + \lambda_T}\left\{(1+\operatorname{erf}\xi_1)\exp\left[\frac{z-z_0}{\lambda_L} + 0{,}5\left(\frac{\sigma}{\lambda_L}\right)^2\right] + (1+\operatorname{erf}\xi_2)\exp\left[-\frac{z-z_0}{\lambda_T} + 0{,}5\left(\frac{\sigma}{\lambda_T}\right)^2\right]\right\} \quad \text{(C.1)}$$

where

$$\xi_1 = \frac{1}{\sqrt{2}}\left(-\frac{z-z_0}{\sigma} - \frac{\sigma}{\lambda_L}\right) \quad \text{(C.2)}$$

$$\xi_2 = \frac{1}{\sqrt{2}}\left(\frac{z-z_0}{\sigma} - \frac{\sigma}{\lambda_T}\right) \quad \text{(C.3)}$$

z_0 is the apparent peak depth;

λ_L is the leading-edge decay length;

λ_T is the trailing-edge decay length;

σ is the Gaussian broadening parameter (see ISO 20341).

When the spacing between adjacent delta-layers is reduced, delta peaks begin to coalesce with each other. As a measure of this coalescence for a particular peak, the peak-to-valley ratio of the coalesced peak was used. The apparent positions of the centroid, the local maximum and the centre of the full width at half maximum (peak centre) were derived in the steady state of coalescence, i.e. rejecting the first two or three delta peaks in the pre-steady state of coalescence. The values of the parameters used for the simulation were $\lambda_L = 0{,}1$ nm, $\lambda_T = 0{,}7$ nm and $\sigma = 0{,}4$ nm, and $\lambda_L = 0{,}3$ nm, $\lambda_T = 2$ nm and $\sigma = 0{,}5$ nm. These values correspond to two extremes of O_2^+ beam energy, 250 eV and 2 keV, respectively, which were obtained by the fitting of measured BN multiple delta-layers in Si. The delta-layer spacing was set at 2 nm, 3 nm, 4 nm, 5 nm, and 6 nm in the former case and 3,5 nm, 5 nm, 7 nm, 11 nm and 14 nm in the latter case.

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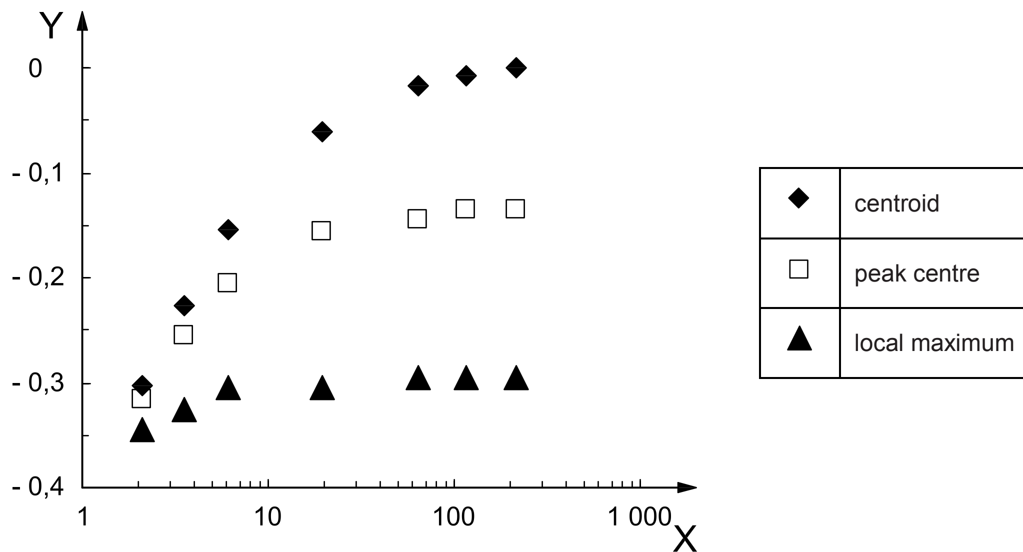
C.3 Results

To illustrate the apparent peak shift depending on the degree of coalescence, the apparent positions of the centroid, the local maximum and the peak centre are plotted against the peak-to-valley ratio in Figures C.1 and C.2 for O_2^+ beam energies of 250 eV and 2 keV, respectively. These shifts are referenced to the centroid positions of the uncoalesced delta-layers. The centroid position is the most sensitive to coalescence, particularly at low peak-to-valley ratios, while the local maximum position is almost unaffected by coalescence. The peak centre position shows an intermediate behaviour. However, even for the centroid, the amount of shift is small when the peak-to-valley ratio is 10 or greater. Under such conditions, the shift is 0,1 nm or less for 250 eV, and less than 0,4 nm for 2 keV. Therefore, the effect of coalescence can be neglected for peak-to-valley ratios of 10 or greater.



Key

X peak-to-valley ratio

Y apparent shift, in nm

Figure C.1 — Apparent shift of peak positions due to coalescence relative to the centroid position of isolated delta-layers for 250 eV O_2^+ (λ_L = 0,1 nm, λ_T = 0,7 nm and σ = 0,4 nm)

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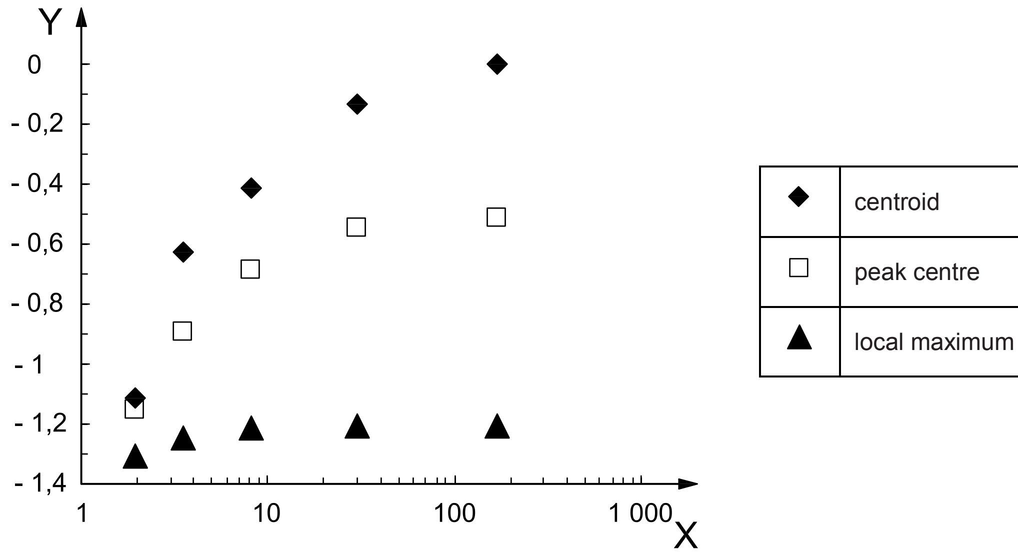

Key

X peak-to-valley ratio

Y apparent shift, in nm

Figure C.2 — Apparent shift of peak positions due to coalescence relative to the centroid position of isolated delta-layers for 2 keV O_2^+ (λ_L = 0,3 nm, λ_T = 2 nm and σ = 0,5 nm)

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Annex D
(informative)

Derivation of uncertainty

When l (= $m - n + 1$) delta-layers from the nth layer to the mth layer of the reference material are used, the linear regression line for the sputtering time, t, and sputtered depth, z, is expressed as

$$t = a + bz = \left(\overline{t} - b\overline{z}\right) + bz = \overline{t} + b\left(z - \overline{z}\right) \tag{D.1}$$

Then the predicted sputtered depth of the test specimen at time t is given by

$$\hat{z} = \overline{z} + \frac{t - \overline{t}}{b} \tag{D.2}$$

The variance of the predicted depth is calculated as follows, using the standard uncertainties, s, for each variable:

$$s_0^2 \cong \left(\frac{\partial \hat{z}}{\partial t}\right)^2 s_t^2 + \left(\frac{\partial \hat{z}}{\partial \overline{t}}\right)^2 s_{\overline{t}}^2 + \left(\frac{\partial \hat{z}}{\partial b}\right)^2 s_b^2$$

$$= \left(\frac{1}{b}\right)^2 s_t^2 + \left(-\frac{1}{b}\right)^2 s_{\overline{t}}^2 + \left(-\frac{t - \overline{t}}{b^2}\right)^2 s_b^2$$

$$= \frac{s_t^2}{b^2} + \frac{1}{b^2} \times \frac{s_t^2}{l} + \left[\frac{\left(t - \overline{t}\right)^2}{b^4}\right] \times \left[\frac{s_t^2}{\sum_i \left(z_i - \overline{z}\right)^2}\right]$$

$$= \left[1 + \frac{1}{l} + \frac{\left(t - \overline{t}\right)^2}{b^2 \sum_i \left(z_i - \overline{z}\right)^2}\right] \times \frac{s_t^2}{b^2} \tag{D.3}$$

Here, the standard uncertainty in the sputtering time is given by

$$s_t = \left\{\frac{\sum_i \left[t_i - \left(a + bz_i\right)^2\right]}{l - 2}\right\}^{\frac{1}{2}} = \left[\frac{\sum_i \left(t_i - \hat{t}_i\right)^2}{l - 2}\right]^{\frac{1}{2}} \tag{D.4}$$

Thus, the standard uncertainty in the depth is given by

$$s_0 = \frac{1}{b} \times \left[1 + \frac{1}{l} + \frac{\left(t - \overline{t}\right)^2}{b^2 \sum_i \left(z_i - \overline{z}\right)^2}\right]^{\frac{1}{2}} \times \left[\frac{\sum_i \left(t_i - \hat{t}_i\right)^2}{l - 2}\right]^{\frac{1}{2}} \tag{D.5}$$

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The 100 × (1 – 2α) % confidence interval is given by

$$\hat{z} \pm T(l-2, \alpha) \times s_0 \quad \text{(D.6)}$$

where $T(l-2, \alpha)$ is the critical value from Student's t-distribution at 100α % for (l – 2) degrees of freedom.

When the reference material has an uncertainty s_{RM}, the quadratic mean of the uncertainties s_t and s_{RM}, $\left(s_t^2 + bs_{RM}^2\right)^{1/2}$, is used to substitute for s_t in Equation (D.3). Thus, the uncertainty in the predicted depth is given by

$$s_1 = \frac{1}{b} \times \left[1 + \frac{1}{l} + \frac{\left(t - \bar{t}\right)^2}{b^2 \sum_i \left(z_i - \bar{z}\right)^2}\right]^{\frac{1}{2}} \times \left[\frac{\sum_i \left(t_i - \hat{t}_i\right)^2}{\left(l-2\right)} + bs_{RM}^2\right]^{\frac{1}{2}} \quad \text{(D.7)}$$

In this International Standard, $z = L_s + r_s t$ and $t = \frac{-L_s}{r_s} + \frac{1}{r_s} z$.

By substituting $a + bz_i$ for $\hat{t}_i$, $\frac{-L_s}{r_s}$ for a, $\frac{1}{r_s}$ for b and $m - n + 1$ for l in Equation (D.7), we obtain Equation (10).

For calibration using a different sputtering rate, the following equations are used:

$$t = a + bcz = \left(\bar{t} - b\bar{z}\right) + bcz \quad \text{(D.8)}$$

$$\hat{z} = \frac{t - \bar{t} + b\bar{z}}{bc} \quad \text{(D.9)}$$

$$s_2^2 = \left(\frac{1}{bc}\right)^2 s_t^2 + \left(-\frac{1}{bc}\right)^2 s_{\bar{t}}^2 + \left(-\frac{t - \bar{t}}{b^2 c}\right)^2 s_b^2 + \left(-\frac{t - \bar{t}}{bc^2}\right)^2 s_c^2$$

$$= \left(\frac{1}{b^2 c^2}\right) s_t^2 + \left(\frac{1}{b^2 c^2}\right) \times \left(\frac{s_t}{l}\right)^2 + \left[\frac{\left(t - \bar{t}\right)^2}{b^4 c^2}\right] \times \left[\frac{s_t^2}{\sum_i \left(z_i - \bar{z}\right)^2}\right] + \frac{\left(t - \bar{t}\right)^2}{b^2 c^4} s_c^2 \quad \text{(D.10)}$$

$$s_2 = \left(\frac{1}{bc}\right) \left\{\left[1 + \frac{1}{l} + \frac{\left(t - \bar{t}\right)^2}{b^2 \sum_i \left(z_i - \bar{z}\right)^2}\right] \times \left[\frac{\sum_i \left(t_i - \hat{t}_i\right)^2}{l-2} + bs_{RM}^2\right] + \frac{\left(t - \bar{t}\right)^2}{c^2} s_c^2\right\}^{\frac{1}{2}} \quad \text{(D.11)}$$

By substituting $a + bz_i$ for $\hat{t}_i$, $\frac{-L_s}{r_s}$ for a, $\frac{1}{r_s}$ for b, $m - n + 1$ for l and $\frac{1}{k}$ for c in Equation (D.11), we obtain Equation (12).

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