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

Analyse chimique des surfaces — Spectrométrie de masse des ions secondaires — Méthode pour l'étalonnage de la profondeur pour le silicium à l'aide de matériaux de référence à couches delta multiples

Reference number
ISO 23812:2009(E)

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

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.

INTERNATIONAL STANDARD **ISO 23812:2009(E)**

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
$L_\mathrm{s}(p)$	shift distance dependent on the definition of delta-layer peak position (see 7.2.1)

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

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.

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.

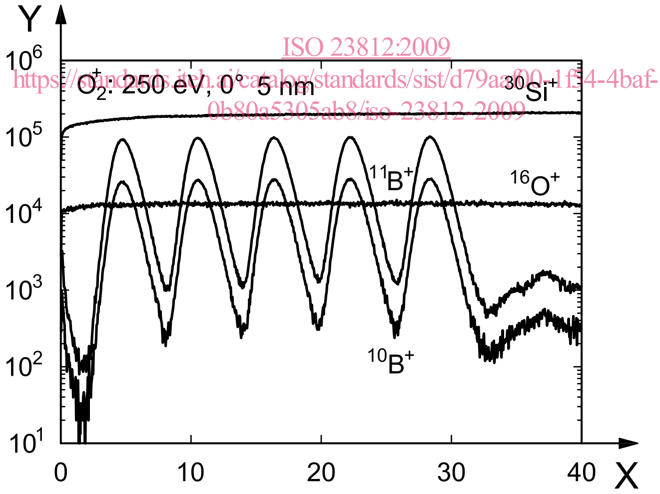
6.6 The SIMS depth profile of test specimens shall be measured using the same measurement conditions as those used for the multiple delta-layer reference materials. If necessary, the primary-ion current density can be changed by changing the primary-ion current and/or the primary-ion scan area.

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]

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:

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

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

$$r_i = \frac{\Delta z_i}{\Delta t_i} \tag{2}$$

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.