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

Analyse chimique des surfaces — Spectrométrie de masse des ions secondaires — Méthode d'estimation des paramètres de résolution en profondeur à l'aide de matériaux de référence multicouches minces

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Foreword

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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.

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ISO 20341 was prepared by Technical Committee ISO/TC 201, *Surface chemical analysis*, Subcommittee SC 6, *Secondary ion mass spectrometry*.

Introduction

Depth resolution is one of the important parameters in SIMS depth profiling. However, sputter depth profiles in SIMS analysis are affected by many factors which may include ion-beam-induced mixing and segregation, charge-driven diffusion, matrix effects, crater shape, surface microtopography, etc. To obtain the best depth resolution, the deterioration of the depth resolution due to these factors should be understood and minimized.

The best depth resolution generally requires special conditions of analysis which may include an ultra-low primary-ion beam energy, glancing incidence angle, specimen rotation, specimen cooling to cryogenic temperature, etc., all of which cannot be easily adopted for daily SIMS analysis. In addition to this, the optimization of the analysis parameters may be quite different for each specimen. Moreover, different aspects of the depth resolution are also affected by instrumental factors such as the crater shape, ion beam homogeneity, removal of the crater edge effect, mass interference, memory effect, residual gas effect, etc.

Therefore, it is not straightforward to estimate the depth resolution under given daily SIMS analysis conditions. In this International Standard, the three essential component parameters of the depth resolution, the leading-edge decay length, the trailing-edge decay length and the Gaussian broadening, are described and procedures are provided for the measurement of each parameter. The depth resolution parameters under daily SIMS analysis conditions can be estimated using multiple delta-layer reference materials.

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

1 Scope

1.1 This International Standard specifies procedures for estimating three depth resolution parameters, viz the leading-edge decay length, the trailing-edge decay length and the Gaussian broadening, in SIMS depth profiling using multiple delta-layer reference materials.

1.2 This International Standard is not applicable to delta-layers where the chemical and physical state of the near-surface region, modified by the incident primary ions, is not in the steady state.

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 18115:2001, *Surface chemical analysis — Vocabulary*

3 Symbols

z depth

z_0 apparent peak depth

λ_L leading-edge decay length

λ_T trailing-edge decay length

σ Gaussian broadening

A_L, A_T scaling factors

B, C scaling coefficients

$I(z)$ intensity of secondary ions as a function of depth

4 Requirements for multiple delta-layer reference materials

4.1 Ideal delta-layers have single atomic layer thickness according to the definition of a delta-layer in ISO 18115. However, it is not always possible to make delta-layers or prove the single atomic layer thickness. If ideal delta-layers are not available, the following criteria are appropriate for non-ideal delta-layers to be used as reference materials.

4.2 The matrix of sputtered surface layers shall not change during SIMS depth profiling so that no significant changes occur in any SIMS matrix effects or in the erosion rate during depth profiling. Constant secondary-ion intensities of the matrix elements through the delta-layers are indicative of a constant matrix.

4.3 The surface and the delta-layers shall be flat and parallel to each other to avoid any distortion of SIMS profiles.

4.4 The thickness of the doped delta-layers shall be sufficiently less than the projected range of the primary ions so that a small variation in the thickness does not affect the profile shape.

4.5 The spacing between adjacent delta-layers shall be large enough so that the secondary-ion intensity at the valley between layers is less than 1 % of the peak intensity.

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

5 Procedures

5.1 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, region analysed, the stability of the primary-ion current, specimen introduction, detected secondary ions, etc., shall be set in accordance with the manufacturer's instructions or a local documented procedure.

In the cases when the SIMS profile shape of each delta-layer in a multiple-delta-doped reference material is not reproducible, the instrument performance shall be checked for items such as the drift of primary-ion current, the scan uniformity, etc.

For good reproducibility of the estimated SIMS depth resolution parameters, SIMS analysis conditions shall be adjusted so that there are more than 10 data points in the top 20 % of the intensity in a SIMS profile, and a SIMS profile shall be recorded down to an intensity below 1 % of the maximum.

5.2 For the use of this International Standard, a SIMS profile of a delta-layer shall be described using an exponential rising edge, a Gaussian-like rounded top and an exponential trailing edge. Assuming the SIMS profile of a delta-layer to be a convolution of the two exponentials $f_{\mathrm{L}}(z)$ for the exponential rising edge as defined in Equation (1) and $f_{\mathrm{T}}(z)$ for the exponential trailing edge as defined in Equation (2), with a Gaussian distribution $g(z)$ as defined in Equation (3), three parameters are required: the leading-edge decay length λ_{L}, the trailing-edge decay length λ_{T}, and the Gaussian broadening σ, usually expressed in nanometres:

$$f_{\mathrm{L}}(z) = A_{\mathrm{L}} \exp\left[\frac{z - z_0}{\lambda_{\mathrm{L}}}\right] \qquad z < z_0 \tag{1}$$

$$f_{\mathrm{T}}(z) = A_{\mathrm{T}} \exp\left[\frac{-(z - z_0)}{\lambda_{\mathrm{T}}}\right] \qquad z > z_0 \tag{2}$$

$$g(z) = \frac{B}{\sqrt{2\pi}\sigma} \exp\left[\frac{-(z - z_0)^2}{2\sigma^2}\right] \tag{3}$$

5.3 Before estimating the SIMS depth resolution parameters, if the background level is higher than 1 % of the peak intensity, any constant background between each peak shall be subtracted before fitting. Using the average value of the sputtering-time intervals between any two delta peaks and the thickness of that interval, convert the sputtering time into the depth in nanometres.

5.4 To estimate the decay lengths and the Gaussian broadening, non-linear curve-fitting software can be used with a user-defined function:

$$I(z) = \frac{C}{\lambda_\mathrm{L} + \lambda_\mathrm{T}} \left\{ (1 + \mathrm{erf}\xi_1) \exp\left[\frac{z - z_0}{\lambda_\mathrm{L}} + 0{,}5 \left(\frac{\sigma}{\lambda_\mathrm{L}} \right)^2 \right] + + (1 + \mathrm{erf}\xi_2) \exp\left[-\frac{z - z_0}{\lambda_\mathrm{T}} + 0{,}5 \left(\frac{\sigma}{\lambda_\mathrm{T}} \right)^2 \right] \right\} \quad (4)$$

where

$$\xi_1 = \frac{1}{\sqrt{2}} \left(-\frac{z - z_0}{\sigma} - \frac{\sigma}{\lambda_\mathrm{L}} \right) \quad (5)$$

$$\xi_2 = \frac{1}{\sqrt{2}} \left(\frac{z - z_0}{\sigma} - \frac{\sigma}{\lambda_\mathrm{T}} \right) \quad (6)$$

and

$$\mathrm{erf}\xi = \frac{2}{\sqrt{\pi}} \int_0^\xi e^{-y^2} \mathrm{d}y \quad (7)$$

If non-linear curve-fitting software is not available, simpler options of estimating one or two SIMS depth resolution parameters shall be used as described in Annex A.

NOTE The derivation of Equation (4) may be found in reference [1] in the Bibliography and example fits of this equation to data both there and in reference [2].

6 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;

c) the conditions of analysis used;

d) the method used to estimate the depth resolution parameters, i.e. Equation (4) or Annex A;

e) the SIMS depth resolution parameters, such as the leading-edge decay length, the trailing-edge decay length and the Gaussian broadening for each delta-layer measured, and the depth of each delta-layer measured;

f) any unusual features noted during the analysis;

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