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Surface chemical analysis — Secondary-ion mass spectrometry — Determination of relative sensitivity factors from ion-implanted reference materials

Analyse chimique des surfaces — Spectrométrie de masse des ions secondaires — Détermination des facteurs de sensibilité relative à l'aide de matériaux de référence à ions implantés

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

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

This second edition cancels and replaces the first edition (ISO 18114:2003), which has been technically revised.

The main changes compared to the previous edition are as follows:

— Formula (1) has been corrected;

— in Clause 5, Notes 1 and 2 have been modified.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Ion-implanted materials are commonly used in secondary-ion mass spectrometry for the calibration of instruments. This document was prepared to provide a uniform method for determining the relative sensitivity factor of an element in a specified matrix from an ion-implanted reference material, and to show how the concentration of the element in a different sample of the same matrix material can be determined.

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Surface chemical analysis — Secondary-ion mass spectrometry — Determination of relative sensitivity factors from ion-implanted reference materials

1 Scope

This document specifies a method of determining relative sensitivity factors (RSFs) for secondary-ion mass spectrometry (SIMS) from ion-implanted reference materials.

The method is applicable to specimens in which the matrix is of uniform chemical composition, and in which the peak concentration of the implanted species does not exceed one atomic percent.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 (all parts), *Surface chemical analysis — Vocabulary*

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 Symbols and abbreviated terms

$C_i^{A,M}$	atomic concentration of the analyte element A in the matrix M at cycle i of a depth profile, expressed in atoms per unit volume
d	depth over which the depth profile is integrated, expressed in length units
$I_i^{A_j}$	detected count rates of the analyte ion of isotope A_j at measurement cycle i, expressed in counts per second
$I_i^{M_k}$	detected count rates of the reference isotope M_k at measurement cycle i, expressed in counts per second
I_{BG}	mean background count rate of species A_j, expressed in counts per second
N^{A_j}	fractional isotopic abundance of the analyte isotope A_j in the unknown sample
n	number of cycles over which the depth profile is integrated

Φ implanted fluence of isotope A_j, expressed in atoms per unit area

RSF   relative sensitivity factor, expressed in atoms per unit volume

SIMS   secondary-ion mass spectrometry

5 Principle

An isotopic relative sensitivity factor (RSF) for a particular element-matrix combination is derived from a SIMS depth profile of an ion-implanted external standard. This RSF can then be used to quantify the concentration of the same element as a function of depth in a different sample of the same matrix material, using Formula (1):

$$C_i^{A,M} = \frac{\mathrm{RSF}^{A_j, M_k} \times \left(I_i^{A_j} - I_{\mathrm{BG}} \right)}{I_i^{M_k} \times N^{A_j}} \tag{1}$$

This procedure only applies to samples and reference materials in which the detected count rate of the analyte ion is directly proportional to its concentration. Causes of divergence from the proportionality are matrix effect when analyte concentration is too high, or detector saturation when the single ion counter is used as the detector system. In practice, to avoid divergence from the proportionality caused by matrix effect, the upper limit of analyte concentration is generally assumed to be one atomic percent. When the cause of divergence from the proportionality is only the detector saturation and when detected intensity can be corrected according to ISO 20411, this procedure can apply after intensity correction. The measurement conditions used for the analysis of reference materials and unknown samples shall be the same.

NOTE 1   As for $I_i^{M_k}$ in Formula (1), the signal is much higher than the background, so the background is negligible. Formula (1) solves for elemental concentration.

NOTE 2   As far as matrix effect concerned, one atomic percent is acceptable for most case, but some cases it is not. For example, an addition of 1 % potassium to a metal catalyst does change the electronic structure of the entire metal phase such that influences on the chemical reactivity and the RSF can be expected.

6 Apparatus

The procedure described here can be used to determine an RSF from data obtained with any SIMS instrument that is capable of obtaining depth profiles. Follow the instrument manufacturer's instructions or local documented procedures for setting up the instrument to obtain the best quality depth profile data.

7 Ion-implanted reference materials

Ion-implanted reference materials for this procedure shall have peak concentrations below one atomic percent, but at least a factor of 100 greater than the background intensity or detection limit of the analyte element in the SIMS instrument. The depth of the peak in the depth distribution of the analyte shall be at least a factor of two below the depth of the onset of steady-state sputtering conditions as indicated by the stability of a matrix reference signal. Use certified reference materials (CRMs) or secondary materials derived from CRMs, when available. If no CRM is available, use reference materials for which the implanted fluence has been measured by an independent method such as Rutherford backscattering spectrometry or neutron activation analysis, if possible.

8   Procedure

Determine the count rates of the ions of analyte isotope A_j, $I_i^{A_j}$, and reference isotope M_k, $I_i^{M_k}$, at each measurement cycle i of the depth profile of the implanted reference material. Calculate the relative sensitivity factor for isotopic species A_j of element A in matrix M with isotopic reference species M_k from Formula (2):

$$\text{RSF}^{A_j, M_k} = \frac{\Phi \times n}{\sum_{i=1}^{n} \left[\left(I_i^{A_j} - I_{\text{BG}} \right) \middle/ I_i^{M_k} \right] \times d} \tag{2}$$

The depth d is typically calculated by measuring the depth of the sputtered crater in the sample with a calibrated stylus profilometer and assuming that a constant depth is removed over each measurement cycle.

If the variation of the reference species count rate is smaller than a specified tolerance level, it may be regarded as constant and its value I^{M_k} need be measured only once during the depth profile. In this case, Formula (3) is used to calculate the RSF:

$$\text{RSF}^{A_j, M_k} = \frac{\Phi \times n \times I^{M_k}}{\sum_{i=1}^{n} \left(I_i^{A_j} - I_{\text{BG}} \right) \times d} \tag{3}$$

9   Test report

The following information shall be recorded when a relative sensitivity factor is determined by this method:

a)   a reference to this document (i.e. ISO 18114:2021);

b)   the relative sensitivity factor;

c)   the model of SIMS instrument used;

d)   the primary-beam species, incidence angle, and energy;

e)   the secondary-ion species and polarity;

f)   the reference ion isotope and species;

g)   any special spectrometer conditions (e.g. mass resolution, kinetic energy filtering);

h)   any special analytical conditions (e.g. oxygen flooding of surface)

i)   any deviations from the procedure;

j)   any unusual features observed;

k)   the date of the test.

Bibliography

[1] ISO 20411, *Surface chemical analysis — Secondary ion mass spectrometry — Correction method for saturated intensity in single ion counting dynamic secondary ion mass spectrometry*

[2] WILSON R.G., STEVIE F.A., MAGEE C.W. *Secondary Ion Mass Spectrometry — A Practical Handbook for Depth Profiling and Bulk Impurity Analysis*, John Wiley and Sons, New York, 1989, Section 3.1.

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