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

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This document comprises a front cover, an inside front cover, the ISO title page, pages ii to v, a blank page, pages 1 to 4, an inside back cover and a back cover.

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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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ISO 18114 was prepared by Technical Committee ISO/TC 201, *Surface chemical analysis*, Subcommittee SC 6, *Secondary ion mass spectrometry*.

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Introduction

Ion-implanted materials are commonly used in secondary-ion mass spectrometry for the calibration of instruments. This international Standard 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 International Standard 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 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*

3 Terms and definitions

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

4 Symbols and abbreviated terms

$C_i^{A,M}$ the 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 the depth over which the depth profile is integrated, expressed in length units

$I_i^{A_j}$ the detected count rates of the analyte ion of isotope A_j at measurement cycle i, expressed in counts/s

$I_i^{M_k}$ the detected count rates of the reference isotope M_k at measurement cycle i, expressed in counts/s

I_{BG} the mean background count rate of species A_j, expressed in counts/s

N^{A_j} the fractional isotopic abundance of the analyte isotope A_j in the unknown sample

n the number of cycles over which the depth profile is integrated

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

RSF the relative sensitivity factor, expressed in atoms per unit volume

SIMS secondary-ion mass spectrometry

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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 the equation:

$$C_i^{A,M} = \frac{\text{RSF}^{A_j,M_k} \times I_i^{A_j}}{I_i^{M_k} \times N^{A_j}} \tag{1}$$

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. In practice, the upper limit of analyte concentration for which this proportionality holds is generally assumed to be one atomic percent. The measurement conditions used for the analysis of reference materials and unknown samples shall be the same.

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 the equation:

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

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.

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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, the equation used to calculate the RSF becomes:

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

9 Test report

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

a) the relative sensitivity factor;

b) the model of SIMS instrument used;

c) the primary-beam species and energy;

d) the secondary-ion species and polarity;

e) the reference ion isotope and species;

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

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

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Bibliography

[1] WILSON, R.G., STEVIE, F.A., and 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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