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Standard Guide for Determining SIMS Relative Sensitivity Factors from Ion Implanted External Standards[1]

This standard is issued under the fixed designation E 1505; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 The purpose of this guide is to provide the secondary ion mass spectrometry (SIMS) analyst with two procedures for determining relative sensitivity factors (RSFs) from ion implanted external standards. This guide may be used for obtaining the RSFs of trace elements (<1 atomic %) in homogeneous (chemically and structurally) specimens. This guide is useful for all SIMS instruments.

1.2 This guide does not describe procedures for obtaining RSFs for major elements (>1 atomic %). In addition, this guide does not describe procedures for obtaining RSFs from implants in heterogeneous (either laterally or in-depth) specimens.

1.3 The values stated in SI units are to be regarded as the standard.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

E 673 Terminology Relating to Surface Analysis[2]

3. Terminology

3.1 *Definitions*—See Terminology E 673 for definitions of terms used in SIMS.

4. Summary of Practice

4.1 This guide will allow calculation of the RSFs of trace elements from plots of SIMS secondary ion intensity (counts/s) versus time (s) that are acquired during the sputtering of ion implanted external standards. Briefly, these plots are obtained in the following manner: an ion beam of a particular ion species, ion energy, and angle of incidence is used to bombard an ion implanted external standard. The beam is rastered or defocussed in order to attempt to produce uniform current density in the analyzed area, which is defined by means of mechanical or electronic gating. The intensities of the secondary ions associated with the implanted element of interest and a reference element (typically, a major element in the specimen matrix, which is distributed homogeneously in the specimen at a known concentration) are monitored with respect to time during the ion sputtering.

4.2 An RSF for a given analyte ion, *A*, and a given reference ion, *R*, is equal to the ratio of their respective useful ion yields, $\tau_A \cdot \tau_R^{-1}$, where τ equals the number of ions detected divided by the number of corresponding atoms sputtered **(1-3)**.[3] An RSF is determined from the secondary ion intensity versus time data obtained from implanted standards using one of two arithmetic models described in the procedure (Section 7) of this guide. A measure of final crater depth is required for RSF determination. This measurement may be performed by another analytical technique (see Section 7).

5. Significance and Use

5.1 The quantification of trace element compositions in homogeneous matrices from first principles requires (*1*) knowledge of the factors influencing ion and sputtering yields and (*2*) understanding of how instrumental parameters influence these yields **(1-3)**. This information is difficult to obtain. Therefore, SIMS operators commonly use external standards to determine RSFs. These RSFs are then used to quantify the composition of trace elements in the specimen of interest through the application of the following equation to each data point of the depth profile of interest **(1-3)**.

$$C_A = I_A \cdot C_R \cdot (I_R \cdot \mathrm{RSF} \cdot N)^{-1} \tag{1}$$

where:

C_A and C_R = concentrations (atoms-cm^{-3}) of the analyte and reference elements, respectively;

I_a and I_R = intensities (counts/s) obtained from the analyte and reference ions, respectively; and

N = natural abundance (expressed as a fraction) of the analyte isotope being examined.

5.2 The most common method of creating external standards is to use an ion accelerator to homogeneously implant a known dose of ions of a particular elemental isotope into a specimen matrix that matches the specimen of interest **(4)**. The implanted ion depth distribution is near-Gaussian (see Fig. 1)

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[1] This guide is under the jurisdiction of ASTM Committee E42 on Surface Analysis and is the direct responsibility of Subcommittee E42.06 on SIMS.

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[2] *Annual Book of ASTM Standards*, Vol 03.06.

[3] The boldface numbers in parentheses refer to the list of references at the end of this guide.

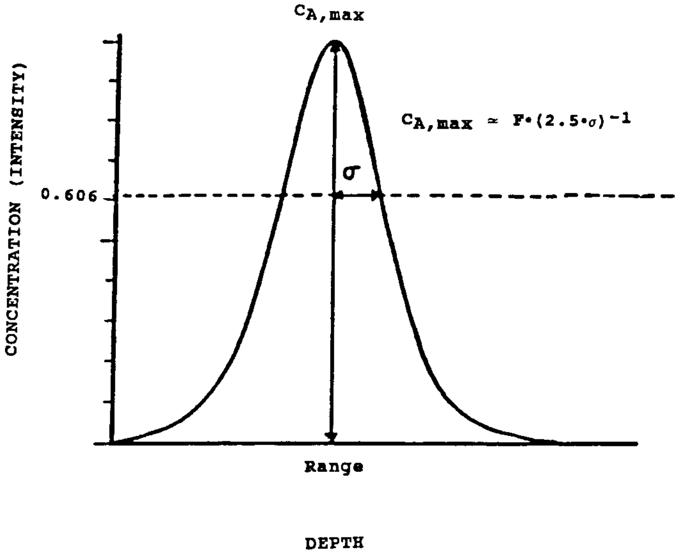

FIG. 1 Parameter for Implant Quantification

and is therefore distinguished readily from background signal intensities. Elemental quantification performed using RSFs obtained from implant standards is generally accurate to ± 15 % relative standard deviation **(4-6)**.

6. Apparatus

6.1 The procedures described here can be used to determine an RSF from data obtained with virtually any SIMS instrument.

6.2 The procedures described in this guide may be used to obtain RSFs from most implant standards in which the near-Gaussian implant distribution (see Fig. 1) is observed clearly beneath any surface artifacts and above the background intensities observed for the analyte ion. The peak concentration of the implanted ion must be below 1 atomic% in order to avoid matrix effects **(3)**. In order to avoid errors associated with insufficient signal intensity, the intensity at the peak of the implant should be at least a factor of 100 greater than the background intensity. Useful ion fluences vary between 10^{13} and 10^{16} atoms-cm^{-2}. Useful ion energies generally vary between 30 and 400 keV **(4)**.

7. Procedure

7.1 One procedure for determining RSFs from implant standards assumes that the implant distribution is actually Gaussian. Most implant distributions deviate from a Gaussian shape, however, because they are skewed or exhibit channelling artifacts. Therefore, this procedure will result in an approximate RSF. As shown in Fig. 1, the maximum concentration of the implanted analyte atom, A ($C_{A,\max}$; atoms-cm^{-3}), can then be determined using the following relationship **(4)**:

$$C_{A,\max} = F \cdot (2.5 \cdot \sigma)^{-1} \tag{2}$$

where:

F = implant fluence (atoms-cm^{-2}), which is determined during implantation; and

σ = measured standard deviation of the implant distribution (cm), which equals half the peak width at 0.606 of the maximum intensity.

Once $C_{A,\max}$ has been measured, τ_A can be determined using the following relationship:

$$\tau_A = I_{A,\max} \cdot (C_{A,\max} \cdot A_o \cdot z)^{-1} \tag{3}$$

where:

$I_{A,\max}$ = intensity at the peak,

A_o = area (cm^2) analyzed, and

z = sputtering rate (cm/s).

Similarly, τ_R can be determined using the following relationship:

$$\tau_R = I_{R,\text{avg}} \cdot (C_R \cdot A_o \cdot z)^{-1} \tag{4}$$

where:

$I_{R,\text{avg}}$ = average intensity obtained from the reference ion.

From these two expressions for the useful ion yields, the following expression for determining RSFs is obtained:

$$\text{RSF} = \tau_A \cdot \tau_R^{-1} = I_{A,\max} \cdot C_R \cdot (I_{R,\text{avg}} \cdot C_{A,\max})^{-1} \tag{5}$$

With the exception of $C_{A,\max}$, all of these parameters are either known or can be determined directly from the depth profile data. Determination of $C_{A,\max}$ requires a knowledge of F, which is readily available from the implantation parameters, and σ. The value of σ equals half the peak width (in seconds of sputtering) at 0.606 of the maximum intensity, divided by the total sputtering time (s) required for the depth profile, and then multiplied by the final depth (cm) of the sputtered crater. The depth of the sputtered crater is commonly measured using either profilometry or interferometry.

7.2 A second procedure for determining RSFs from implant standards involves integration of the implant signal **(4,6)**. This procedure makes no assumption concerning the shape of the implant profile, and it is therefore more accurate than the procedure described in 7.1. With this procedure, the intensities obtained at each data point (except for those distorted by surface artifacts) of an implant depth profile are added together. This integrated intensity ($I_{A,\text{integ}}$; counts/s) is then used in the following relationship to determine the useful ion yield of the analyte:

$$\tau_A = (I_{A,\text{integ}} - I_{A,\text{bkg}} \cdot n) \cdot T \cdot (F \cdot A_o)^{-1} \tag{6}$$

where:

$I_{A,\text{bkg}}$ = average background intensity (count/s) determined at a depth well below the observed implant distribution;

T = time (s) required to cycle through all of the masses being examined; and

n = number of data points that were integrated to obtain $I_{A,\text{integ}}$.

Using this expression for τ_A and the expression for τ_R described above, the following expression for determining RSFs is obtained:

$$\text{RSF} = \tau_A \cdot \tau_R^{-1} = (I_{A,\text{integ}} - I_{A,\text{bkg}} \cdot n) \cdot T \cdot C_R \cdot z \cdot (F \cdot I_{R,\text{avg}})^{-1} \tag{7}$$

The sputtering rate (z) is the only parameter that cannot be obtained directly from either the depth profile data, depth profile setup parameters, or implantation parameters. The sputtering rate is usually obtained by measuring the final crater depth and dividing it by the total sputtering time used to perform the depth profile. The depth of the sputtered crater is commonly measured using either profilometry or interferometry.

8. Keywords

8.1 SIMS

REFERENCES

(**1**) Benninghoven, A., Rudenauer, F. G., and Werner, H. W., *Secondary Ion Mass Spectrometry*, John Wiley & Sons, Inc., New York, NY, 1987.

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(**4**) Leta, D. P., and Morrison, G. H., "Ion Implanted Standards for Secondary Ion Mass Spectrometric Determination of the 1a–7a Group Elements in Semiconducting Matrices", *Analytical Chemistry*, Vol 52, 1980, p. 514.

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