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Surface chemical analysis — Secondary ion mass spectrometry — Method for determining yield volume in argon cluster sputter depth profiling of organic materials

Analyse chimique des surfaces — Spectrométrie de masse des ions secondaires — Méthode de détermination du rendement volumique dans le cadre du profilage en profondeur de matériaux organiques par pulvérisation d'argon en grappe

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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.

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Introduction

In many technological and biological samples, it is necessary to understand the distribution of organic chemical species as a function of depth and to combine this with laterally resolved surface chemistry to provide three-dimensional representations. Secondary ion mass spectrometry is a method capable of determining depth distribution combined with lateral information. Argon cluster ion beams can provide sputter depth profiles through most organic materials without causing significant damage and molecular species may be detected and located by mass spectrometry. This approach can provide depth resolutions better than 10 nm and depth profiles which extend over many micrometres in thickness. In order to reconstruct a depth profile or three-dimensional image, it is important to establish the depth scale in the depth profiling experiment. For this purpose, the sputtering yield volume is required.

The sputtering yield volume defines the ratio between the areic dose of sputtering ions and the sputtered depth. Knowledge of the sputtering yield volume enables the depth of features of interest to be determined from the sputtering ion current, the sputtered area and the sputtering time. The sputtering yield volume depends upon the specific experimental conditions such as the sample temperature, the material being sputtered, the cluster source identity, kinetic energy and angle of incidence. However, the prediction of sputtering yield volumes for a particular material is possible using measurements made from the same material under different experimental conditions. Therefore, reliable measurements of sputtering yield volumes are required for accurate measurement of depth, to provide comparability between laboratories and to enable analysts to implement and use sputtering yield volumes reported by others.

This document provides methods to measure sputtering yield volumes of organic test materials using argon cluster ions. The test materials should consist of thin films of known thicknesses between 50 nm and 1 000 nm. The format of the test materials, the measurement of sputtering ion dose, sputtered depth and reporting requirements for sputtering yield volumes are described. Annex A provides informative definitions of sputtered area and sputtering beam width and an example of their measurement. Annex B provides informative examples of typical depth profiles and an example calculation of sputtering yield volume. Annex C provides informative methods to estimate sputtering yield volumes under different sputtering conditions.

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Surface chemical analysis — Secondary ion mass spectrometry — Method for determining yield volume in argon cluster sputter depth profiling of organic materials

1 Scope

This document specifies a method for measuring and reporting argon cluster sputtering yield volumes of a specific organic material. The method requires one or more test samples of the specified material as a thin, uniform film of known thickness between 50 and 1 000 nanometres on a flat substrate which has a different chemical composition to the specified material. This document is applicable to test samples in which the specified material layer has homogeneous composition in depth and is not applicable if the depth distribution of compounds in the specified material is inhomogeneous. This document is applicable to instruments in which the sputtering ion beam irradiates the sample using a raster to ensure a constant ion dose over the analysis area.

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.

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ISO 18115-1:2013, *Surface chemical analysis — Vocabulary — Part 1: General terms and terms used in spectroscopy*

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3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 18115-1:2013 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)

a number of secondary ions used to determine Y

b number of layers of different thicknesses used to determine Y

A area of the raster pattern of the sputtering beam on the sample

d known thickness of material for which the sputtering yield volume is to be measured

D areic dose of ions used for sputtering

δ_A relative standard uncertainty in A

δ_d relative standard uncertainty in d for a specified layer

δ_J relative standard uncertainty in the current during a specified profile

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δ_n relative standard uncertainty in average cluster size during a specified profile

δ_τ relative standard uncertainty in sputtering time in a specified profile

δ_y relative standard uncertainty in the sputtering yield volume for a specified profile

δ_Y relative standard uncertainty in the sputtering yield volume

Δt absolute difference in time between $t(16)$ and $t(84)$

Δt_1 width of the first interface in sputtering time

Δt_2 width of the second interface in sputtering time

e elementary charge, $1{,}602 \times 10^{-19}$ Coulombs

E kinetic energy of primary argon cluster ions used for sputtering in eV

FWHM full width at half maximum

i identifier for the secondary ions monitored in experiments

J_1 current of ions used for sputtering measured before analysis in Amperes

J_2 current of ions used for sputtering measured after analysis in Amperes

J_a current of primary ions used for analysis in Amperes

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k identifier for the different layers of the test material

l side length of the analysed area on the sample

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L minimum side length of sputtering area on the sample

n average number of argon atoms per ion in the primary cluster

n_1 average number of argon atoms per ion in the primary argon cluster measured before analysis

n_2 average number of argon atoms per ion in the primary argon cluster measured after analysis

N number of lines in sputtering beam raster pattern

q number of elementary charges on a primary ion

σ_d standard uncertainty in d for a specified layer

σ_Y standard uncertainty in Y

t_1 sputtering time to reach the first interface of the organic material in seconds

t_2 sputtering time to reach the second interface of the organic material in seconds

$t(x)$ sputtering time for secondary ion intensity to reach x % of that in the pure material in seconds

τ sputtering time through a layer of thickness d in a specified profile in seconds

τ' gradient in a graph of τ plotted against d

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W maximum FWHM of sputtering ion beam spot on the sample

y sputtering yield volume determined for a specified ion from the slope of a graph of dose, D, on the x-axis and thickness, d, on the y-axis

Y sputtering yield volume

5 Requirements

5.1 Test samples

The test sample(s) shall consist of one or more layers of organic materials of uniform thicknesses on a flat substrate. The chemical composition of the organic materials and substrate used to construct the test sample(s) shall be known. The thickness of each layer of material for which the sputtering yield volume is to be measured shall be known for each sample. The standard uncertainty in thickness from both the measurement method and variability in thickness shall be known or estimated. If the test sample consists of more than one layer of organic material, the order in which those layers are formed shall be known. The test sample(s) shall be classified according to the sample construction as either:

a) A **surface layer**: if the first interface of the material is the surface of the test sample.

b) A **buried layer**: if the first interface of the material is not at the surface of the test sample.

c) A **series of layers**: if there is more than one layer of the material, with thicknesses spanning at least a factor of 1,5 and having the same first and second interfaces for all layers.

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The first interface is the initial boundary of the material encountered in a sputter depth profile. The second interface is the final boundary of the material encountered in a sputter depth profile. The series of layers is the more accurate method for determination of the sputtering yield volume but requires most effort.

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NOTE 1 Typical methods used to prepare reference samples are vapour deposition, spin casting from appropriate solvents, Langmuir-Blodgett or Langmuir-Schaefer deposition.

NOTE 2 In the case of single layers of organic materials on inorganic substrates, the most appropriate methods to directly measure layer thickness are atomic force microscopy (AFM), ellipsometry and X-ray reflectometry[1]. For multiple layers, these methods typically provide insufficient contrast between different organic materials and other approaches, such as in situ measurements using a quartz crystal microbalance or analysis after each deposition step, are appropriate.

5.2 Sputtering ion source

To ensure that the data required to calculate sputtering yield volumes are useful and that the reporting requirements are met, the following information and conditions are required.

a) The number weighted mean size of the argon clusters, n_1, used for sputtering shall be measured before analysis and the number weighted mean size of the argon clusters, n_2, shall be measured after analysis.

b) The sputtering ion kinetic energy, E, shall be known.

c) The sputtering ion charge, q, shall be known.

d) The incidence angle between the sputtering ion beam and the reference sample surface normal shall be known.

e) The sputtering ion current, J_1, shall be measured using a Faraday cup before analysis of the reference sample and the sputtering ion current, J_2, shall be measured after analysis of the reference sample in the same manner as J_1. See 6.1.

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f) The minimum side length, L, and the area, A, of the raster pattern of the primary ion source on the sample shall be measured.

g) The maximum full width at half maximum (FWHM) of the sputtering ion beam spot on the sample, W, shall be measured.

h) The number of lines in the raster pattern, N, shall be known.

i) The sputtering and analysis cycle shall be set to ensure that raster frames of the sputtering source are complete and without gaps.

j) The ambient sample temperature shall be measured.

The FWHM of the sputtering ion beam spot shall be greater than twice the distance between adjacent lines in the raster pattern, this condition is expressed in Formula (1). If this condition is not met then, either the ion beam shall be defocussed to increase W, or the raster area changed to decrease L until this condition is satisfied.

$$L < 0{,}5NW \tag{1}$$

where

L is the minimum side length of sputtering area on the sample;

N is the number of lines in the sputtering beam raster pattern;

W is the maximum FWHM of the sputtering ion beam spot on the sample.

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The minimum side length of the sputtering source raster, L, shall be larger than the maximum FWHM of the sputtering ion spot, W, by at least a factor of 8. This condition is expressed in Formula (2).

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$$L > 8W \tag{2}$$

If this condition is not met then either the ion beam shall be focussed to decrease W, or the raster size changed to increase L until this condition is satisfied and does not violate the condition described by Formula (1).

NOTE 1 A diagram is provided for information in A.1.1. The area of the raster pattern can be measured by imaging a reference grid positioned at the same height as the test sample. A method is provided in A.1.2 and an example in A.1.3.

NOTE 2 The condition expressed in Formula (1) ensures a constant ion beam dose in the analysed area both on and between raster lines[2].

NOTE 3 The condition expressed in Formula (2) ensures that the central part of the sputtered area on the sample, which has a constant ion beam dose, is large enough to be analysed.

5.3 Analysis conditions

The size and position of the area for analysis shall be within the area irradiated by the sputtering source. The centre of the analysis area shall be aligned with the centre of the sputtered area and have side length, l, meeting the condition expressed in Formula (3). If the sputtering ion beam is used for

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analysis, then the gating conditions to select the signal as a function of raster position shall be selected to meet this criterion.

$$l < L - 6W \tag{3}$$

where l is the side length of the analysed area on sample.

NOTE The condition expressed in Formula (3) is to ensure that the area analysed receives a laterally invariant dose of sputtering ions and that the analysis beam does not sample areas that, due to edge effects, have received a lower dose than the centre. A diagram is provided in A.1.1.

If a different primary ion beam is used for analysis the alignment of the two ion beams on the sample shall be within a distance $W/2$. In this case, the primary ion source used for analysis shall have a dose rate much smaller than the sputtering source. The time-averaged current of the analytical primary ion beam on the sample shall meet the criterion expressed in Formula (4) see also NOTE 1 below.

$$J_a < 0{,}05 J_1 \frac{l^2}{A} \tag{4}$$

where

J_a is the time averaged current of primary ions used for analysis;

J_1 is the current of primary ions used for sputtering measured before analysis;

l is the side length of the analysed area on sample;

A is the area of the raster pattern of the sputtering beam on sample.

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If this condition is not met then J_a or A shall be reduced or J_1 or l increased until the condition is met, whilst also meeting the conditions expressed in Formulae (1), (2) and (3). The ratio of J_a to J_1 can be altered by changing the ratio of sputtering cycles to analysis cycles, or the duty cycle of the analytical primary ion beam in time-of-flight systems.

Charge compensation using an electron flood gun is usually required. If it is necessary to use electrons for charge compensation the electron beam current should be kept as low as possible to avoid unintended damage to the sample.

It is usually necessary to use an area of the test sample to optimise the analysis conditions. If this is the case, the position of the area used for optimisation shall be selected to be as far as possible from areas that will be used for the determination of sputtering yield volume.

If it is suspected that the primary ions used for analysis have a significantly higher sputtering yield volume than that of the sputtering ions then the factor 0,05 in Formula (4) shall be replaced by the product of 0,05 and the ratio of the expected sputtering yield volume of the sputtering ion to that of the analysis ion.

NOTE 1 The condition expressed in Formula (4) is derived from reference [3] where the sputtering and analysis ions have similar sputtering yield volumes in the test material.

6 Data Acquisition

6.1 Analysis

The analysis position on the sample shall be chosen to maintain a suitable distance from the edge of the sample and other areas where non-homogeneous thickness or surface damage is suspected. If an electron flood source is used, the separation of areas used for instrument set up and analysis shall be as large as possible to avoid areas previously irradiated by the electron flood gun. The sputtering ion current J_1 shall be recorded using a Faraday cup prior to the start of each profile. Then the depth profile

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is performed at the selected analysis position, see 6.2. Finally, the sputtering ion current J_2 shall be recorded using a Faraday cup immediately after the profile is complete. If the ratio of J_1 and J_2 is greater than 1,1 or less than 0,9 then the data from the depth profile shall not be used to calculate a sputtering yield volume.

NOTE 1 The effect of electron-induced surface damage on sputtering yields is described in reference [4].

6.2 Minimum data requirements

The intensities of secondary ions which display visibly and identifiably greater intensity for the material of interest in the sample, compared to the other materials, shall be measured as a function of sputtering time. More than one secondary ion from the material for which the sputtering yield volume is to be determined shall have their intensities measured. This condition applies even in the case of a surface or buried layer, where only one secondary ion is employed for the measurement of sputtering yield volume, because selected secondary ions may be rejected in the data quality check in 6.3.

By preference, one of the secondary ions unique to the material for which the sputtering yield volume is to be determined should be atomic or diatomic.

NOTE 1 Atomic or diatomic secondary ions are likely to exhibit smaller matrix effects than molecular secondary ions as demonstrated in reference [5].

6.3 Data quality

After the depth profiles are complete, the intensities of the selected secondary ions shall be plotted as a function of sputtering time and inspected visually. The secondary ion intensities shall have plateaus of relatively constant (±10 %) intensity before and after each interface for a longer sputtering time than the transition in intensity at the interfaces.

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If there is an increase in secondary ion intensity at the interface above the plateau value for the pure material and only one layer is being used to determine sputter yield volume, then the secondary ion shall not be used to determine the sputtering yield volume: a different secondary ion shall be chosen.

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NOTE 1 An increase in secondary ion intensity above the plateau for interfaces between two organic materials indicates that significant matrix effects are occurring[6], whereas for a second interface between an organic material and an inorganic material such effects are usually caused by an enhancement in sputtering yield close to the interface. For secondary ions which are not specific to the material, interfacial contaminants can also produce such features.

NOTE 2 If there is a decline in secondary ion intensity in the plateau region of all profiles and the absolute intensities in the repeated profiles are similar then this strongly indicates that the sputtering yield volume is not constant[7]. If there is a decline in secondary ion intensity in the plateau region of all profiles and a decline in absolute intensities in subsequent experiments, then this indicates that there is a decline in the primary ion current used for analysis.

7 Calculation of sputtering yield volume

7.1 Introduction

In the case that only one thickness of material (b = 1) has been analysed, the procedure to locate interfaces in sputtering time is to measure the sputtering time, through the layer of interest, τ and calculate the sputtering yield volume, Y, as described in 7.2. In this case only one secondary ion is required because the uncertainty in sputtering time is dominated by matrix effects. Without additional knowledge, the uncertainty is not reduced by including more than one secondary ion in the analysis.

In the case that more than one thickness of material (b > 1) has been analysed, the procedure to locate interfaces in sputtering time, measure sputtering time, τ, and calculate sputtering yield volume, Y, is described in 7.3.

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