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Surface chemical analysis — Depth profiling — Method for sputter rate determination in X-ray photoelectron spectroscopy, Auger electron spectroscopy and secondary-ion mass spectrometry sputter depth profiling using single and multi-layer thin films

Analyse chimique des surfaces — Profilage d'épaisseur — Méthode pour la détermination de la vitesse de pulvérisation lors du profilage d'épaisseur par pulvérisation en spectroscopie de photoélectrons par rayons X, spectroscopie d'électrons Auger et spectrométrie de masse des ions secondaires à l'aide de films minces multicouches

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Foreword

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This document was prepared by Technical Committee ISO/TC 201, *Surface chemical analysis*, Subcommittee SC 4, *Depth profiling*.

This second edition cancels and replaces the first edition (ISO 17109:2015), which has been technically revised.

The main changes are as follows:

— in 4.5, reference documents for a cleaning of thin film surface have been added;

— the flowchart in Clause 5 has been revised to improve clarity.

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Introduction

The sputtering rate in surface chemical analysis is generally determined from the quotient of sputtered depth, measured using stylus profilometry, and sputtering time. However, for multi-layered thin films, only the average sputtering rate is determined by this method. Therefore, this method is difficult to apply to multi-layered thin films comprised of materials with different sputtering rates. Sputtering rates are also affected by various experimental parameters so that it is difficult for them to tabulate and to be used for sputter depth calibrations. For higher accuracies, it is important for sputtering rates to be determined under specific experimental conditions for each laboratory for sputter depth calibration. Sputter rates should be determined using single-layers that are much thicker than the projected range of the sputtering ions so that the surface transient effect is negligible or by using multi-layered thin films where the effect of surface transient phenomena can be excluded, and interface transients can be minimized.

This document is developed for the calibration of sputtered depth by determining the ion sputtering rate for depth profiling measurement with Auger electron spectroscopy (AES), X-ray photoelectron spectroscopy (XPS), and secondary ion mass spectrometry (SIMS) using single- and multi-layer thin films. The measured ion sputtering rate can be used for the prediction of ion sputtering rates for a wide range of other materials so that depth scales or sputtering times can be estimated in day-to-day samples through tabulated values of sputtering yields and bulk densities.

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Surface chemical analysis — Depth profiling — Method for sputter rate determination in X-ray photoelectron spectroscopy, Auger electron spectroscopy and secondary-ion mass spectrometry sputter depth profiling using single and multi-layer thin films

1 Scope

This document specifies a method for the calibration of the sputtered depth of a material from a measurement of its sputtering rate under set sputtering conditions using a single- or multi-layer reference sample with layers of the same material as that requiring depth calibration. The method has a typical accuracy in the range of 5 % to 10 % for layers 20 nm to 200 nm thick when sputter depth profiled using AES, XPS and SIMS. The sputtering rate is determined from the layer thickness and the sputtering time between relevant interfaces in the reference sample and this is used with the sputtering time to give the thickness of the sample to be measured. The determined ion sputtering rate can be used for the prediction of ion sputtering rates for a wide range of other materials so that depth scales and sputtering times in those materials can be estimated through tabulated values of sputtering yields and atomic densities.

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 14606, *Surface chemical analysis — Sputter depth profiling — Optimization using layered systems as reference materials*

3 Terms, definitions, symbols and abbreviated terms

3.1 Terms and definitions

For the purposes of this document, the following terms and definitions 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 https://www.electropedia.org/

3.1.1
upper plateau
region exhibiting intensities higher than 95 % of the maximum intensity of the characteristic signal for that layer and covering more than half the thickness of that layer

3.1.2
lower plateau
region exhibiting intensities lower than the minimum intensity plus 5 % of the maximum intensity of the characteristic signal for that layer and covering more than half the thickness of that layer

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3.2 Symbols and abbreviated terms

For the purposes of this document, the following symbols and abbreviated terms apply.

SD	standard deviation
I_{50}	50 % signal intensity of sputter depth profile
I_{U}	average intensity in the upper plateau region of the depth profile
I_{L}	average intensity in the lower plateau region of the depth profile
z_{A}	sputtering rate of layer A
z_{B}	sputtering rate of layer B
$d_{\mathrm{A}}^{\mathrm{R}}$	thickness of layer A of a single- or multi-layer reference thin film
$d_{\mathrm{B}}^{\mathrm{R}}$	thickness of layer B of a multi-layer reference thin film
$t_{\mathrm{A}}^{\mathrm{R}}$	sputtering time of layer A of a single- or multi-layer reference thin film
$t_{\mathrm{B}}^{\mathrm{R}}$	sputtering time of layer B of a multi-layer reference thin film
$\bar{z}_{\mathrm{A}}$	average sputtering rate of layer A
$\bar{z}_{\mathrm{B}}$	average sputtering rate of layer B
$d_{\mathrm{A}}^{\mathrm{U}}$	thickness of layer A in a single- or multi-layered thin film to be measured
$d_{\mathrm{B}}^{\mathrm{U}}$	thickness of layer B in a multi-layered thin film to be measured
$\bar{t}_{\mathrm{A}}^{\mathrm{U}}$	average sputtering time from three consecutive sputter depth profiles of the layer A in a multi-layered thin film to be measured
$\bar{t}_{\mathrm{B}}^{\mathrm{U}}$	average sputtering time from three consecutive sputter depth profiles of the layer B in a multi-layered thin film to be measured
$t_{\mathrm{A}}^{\mathrm{U}}$	sputtering time of layer A in a multi-layered thin film to be measured
$t_{\mathrm{B}}^{\mathrm{U}}$	sputtering time of layer B in a multi-layered thin film to be measured
$\Delta\left(d_{\mathrm{A}}^{\mathrm{U}}\right)$	uncertainty of the thickness of the layer A
$\sigma\left(\bar{t}_{\mathrm{A}}^{\mathrm{U}}\right)$	standard deviation of $\bar{t}_{\mathrm{A}}^{\mathrm{U}}$
$\sigma\left(\bar{z}_{\mathrm{A}}\right)$	standard deviation of $\bar{z}_{\mathrm{A}}$

4 Requirement of single- and multi-layer reference thin films

4.1 The thickness of each layer in multi-layer thin films and the thickness of single-layer thin films shall be sufficiently thicker than the sum of the projected range of the sputtering ions and the information depth of the analytical method, so that an upper plateau and a lower plateau shall be obtained for each layer in sputter depth profiling. The projected range can be simply calculated using SRIM code which is available from http://www.srim.org[1].

NOTE Sample rotation during ion sputtering is shown to reduce surface roughness development especially of polycrystalline films[2] leading to sharper interfaces and a better estimate of sputtering rates.

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