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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

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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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Contents

Page

Foreword .... **iv**

Introduction .... **v**

1 Scope **1**

2 Normative references **1**

3 Terms and definitions **1**

4 Requirement of single- and multi-layer reference thin films **1**

5 Determination of sputtering rate **2**

Annex A (informative) **Report of international Round Robin Test** **6**

Annex B (informative) **Prediction of the rates for a wide range of other materials through tabulated values of sputtering yields** **15**

Bibliography **16**

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Foreword

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The committee responsible for this document is ISO/TC 201, *Surface chemical analysis*, Subcommittee SC 4, *Depth profiling*.

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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 International Standard 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 International Standard 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 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.

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2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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 14606, *Surface chemical analysis — Sputter depth profiling — Optimization using layered systems as reference materials*

3 Terms and definitions

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

3.1
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

3.2
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

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.[7]

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NOTE Sample rotation during ion sputtering is shown to reduce surface roughness development especially of polycrystalline films[11] leading to sharper interfaces and a better estimate of sputtering rates.

4.2 The surface and the interfaces shall be flat and parallel to each other to avoid any distortion of sputter depth profiles. The surface roughness is often measured using atomic force microscopy and the thickness variation using transmission electron microscopy. The surface roughness of sample and the thickness variation of each layer shall be smaller than the sum of the projected range of the sputtering ions and the information depth of the analytical method.

4.3 The thickness of each layer in multi-layer thin films and the thickness of single-layer thin films shall be determined by high resolution cross-sectional transmission electron microscopy, grazing incidence X-ray reflectivity, medium energy ion scattering spectroscopy, or other appropriate methods for which an accurate uncertainty of measurement can be evaluated.

4.4 The number of A/B layer pairs in the multi-layered reference thin films shall be greater than two since profiles of the first layer A and the last layer B shall not be used due to the surface and the final interface transient effects.

4.5 For single-layer thin films, to minimize any likely contamination or surface oxidation problems, materials like gold on Si or SiO_2 on Si and Ta_2O_5 on Ta which are stable and remain clean or can easily be cleaned are recommended.

5 Determination of sputtering rate

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5.1 Set the sputtering conditions to be those for which the sputtering rates are required. Changes in the sputtering species, the impact energy, and beam current will change the sputtering rates. The sputter depth profiling parameters are optimized according to ISO 14606.

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NOTE 1 A typical AES, XPS, and SIMS sputter depth profile is given in Figure A.2 to Figure A.4, respetively.

NOTE 2 The ordinate axis units can be intensity, atomic fraction, an intensity ratio, concentration, or whatever is the unit most linearly related to the amount of substance present at each depth.

5.2 The sputter depth profiles shall be measured after the instrument has stabilized to minimize uncertainty due to instrumental fluctuation. Inspect the data, identify, and then ignore, in what follows, any noise spikes.

5.3 Measure the sputter depth profile of a single- or multi-layer reference thin film and determine the interface position by the point where the signal intensity of the element reaches 50 % of its value between the lower plateau where the element is essentially absent and the upper plateau level for the layer where it is present. The determination of the interface position by this procedure is applied to this International Standard until the development of an ISO Standard for interface position. The average intensity in the upper plateau region is the plateau intensity (I_{upper}). This shall be calculated by summing the intensity for each measurement where the intensity is greater than 95 % of the maximum intensity and dividing by the number of measurements used in the summation.

A similar procedure shall be adopted for the determination of the lower plateau for each constituent of the profile (I_{lower}). The average intensity shall be calculated as follows:

a) subtract the minimum intensity value in this part of the profile from all readings;

b) calculate the value of 5 % of the maximum intensity (following the subtraction in one, above);

c) sum all of the intensities which are less than the 5 % value calculated in two, above;

d) divide the sum by the number of readings to get an average;

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e) add the minimum intensity to the average calculated in four to arrive at I_{lower}.

The 50 % signal intensity shall be calculated as follows: $I_{50\,\%} = (I_{upper} - I_{lower})/2$.

Examples of determining the upper plateau level and the lower plateau level are demonstrated in Figure A.2 to Figure A.4 for AES, XPS, and SIMS depth profiling, respectively. For single-layer thin films, the beginning of the sputter time is defined by the time where the intensity for the given element reaches 50 % of the upper plateau level in the similar manner.

For some sputter depth profiling, often by SIMS, the interface positions may be significantly affected by changes in the matrix effect in the interface region. If the upper plateau defined with intensities higher than 95 % of the maximum intensity is less than half of the layer thickness due to large distortions at interfaces, this standard shall not be used for sputter rate determination.

NOTE 1 The 50 % of the plateau level is mentioned in ISO/TR 15969.[2]

NOTE 2 A flow chart is given to guide the sputter rate determination of multi-layered thin films and single-layer thin films as below.

5.4 The sputtering rates of layers of A, z_A, and B, z_B, are determined by dividing the thicknesses of layer A, d_A^R, and B, d_B^R, by the sputtering times of layers of A, t_A^R, and B, t_B^R, of a reference A/B/A/B.... multi-layer thin film. The unit of sputtering rate is nm/s.

$$z_A = \left(d_A^R / t_A^R\right) \tag{1}$$

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$$z_B = \left(d_B^R / t_B^R\right) \tag{2}$$

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The sputtering time of layers of A, t_A^R, is determined from the time interval from B/A to A/B interfaces and that of layers of B, t_B^R, is determined from the time period from A/B to B/A interfaces. Measure three sputter depth profiles to determine the uncertainties. If the standard deviation (SD) is inappropriate, the sputter depth profiling parameters shall be optimized according to ISO 14606 to improve the uncertainties.

The average sputtering rates of layers of A, $\bar{z}_A$, and B, $\bar{z}_B$, are determined from the average values of the respective sputtering rates z_A and z_B of all layers except the outmost layer A and the last layer B adjacent to the substrate.

For single-layer thin films, the sputtering rates of layer A, z_A, is determined with Formula (1). The sputtering time of layer A, t_A^R, is determined from the time interval from the surface layer A to the A/substrate interface. The average sputtering rate of layer A, $\bar{z}_A$, is determined from the average value of three consecutive profiling of a single-layer thin film.

NOTE 1 A flow chart is given to guide the sputter rate determination of multi-layered thin films and single-layer thin films.

NOTE 2 SD of better than 5 % has been found useful and practicable.

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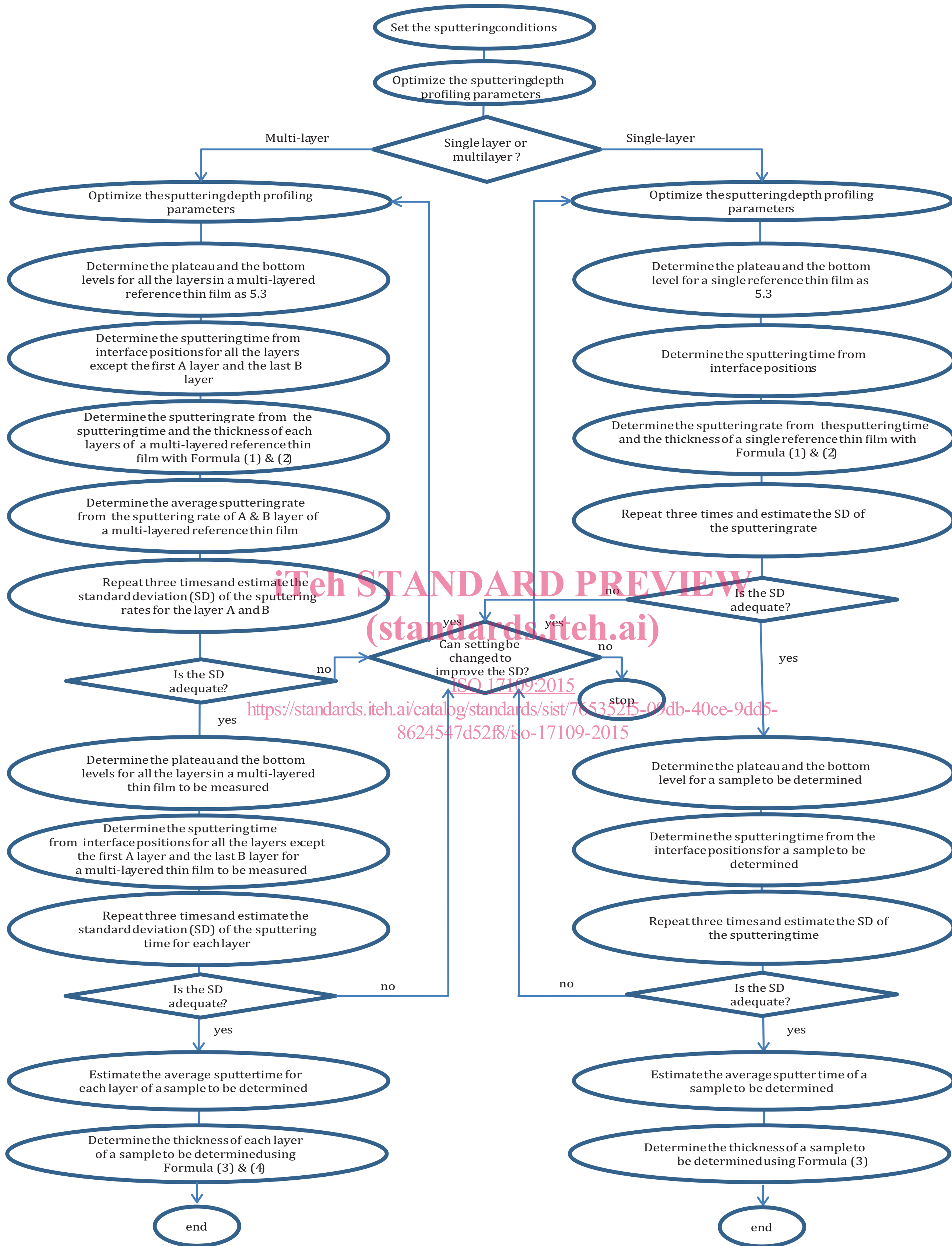

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5.5 For the multi-layered reference thin film, estimate the standard deviation of sputtering rates for layer A and layer B using the three average sputtering rates of layers of A, $\bar{z}_A$, and B, $\bar{z}_B$, from three consecutive sputter depth profiles. For single-layered thin films, the standard deviation of sputtering rate is calculated from three consecutive sputter depth profiles of a single-layered thin film.

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5.6 If the measured standard deviation in the sputter rates is greater than 5 %, then the experimental parameters shall be adjusted and the depth profile measurements repeated. The standard deviation of sputter rates from each layer can be used for evaluating the constancy of the sputtering rate as a function of depth or of the time that the gun has been running. Additionally, the change of the sputter rates from each layer excluding the first A layer and the last B layer can be used for evaluation of both the short- and long-term drifts of the ion beam current as a function of the time that the gun has been running.

5.7 The average sputtering rate of layer A from a single-layer or layers A and B from the multi-layer reference thin films determined in 5.4 can be used for the calibration of the sputter depth or the thickness of single-layer thin films to be measured or each layer of multi-layered thin films to be measured. To use the average sputtering rate determined using a multi-layered reference thin film for the calibration of the sputter depth profile of a multi-layered thin film to be measured, the two samples should be sputter depth profiled consecutively after stabilization of the instrument. The thicknesses of layer A, $d_{\mathrm{A}}^{\mathrm{U}}$, and that of a B layer, $d_{\mathrm{B}}^{\mathrm{U}}$, in a multi-layered thin film to be measured can be determined by multiplying the average sputtering time of the layer A, $\bar{t}_{\mathrm{A}}^{\mathrm{U}}$, and that of the layer B, $\bar{t}_{\mathrm{B}}^{\mathrm{U}}$, in a multi-layered thin film to be measured from three consecutive sputter depth profiles and the average sputtering rates of layers of A, $\bar{z}_{\mathrm{A}}$, and B, $\bar{z}_{\mathrm{B}}$, determined from the reference multi-layered thin films as in 5.4, respectively, using Formula (3) and Formula (4). The sputtering time of layer A, $t_{\mathrm{A}}^{\mathrm{U}}$, in a multi-layered thin film to be measured is determined from the time interval from B/A to A/B interfaces and that of a B layer, $t_{\mathrm{B}}^{\mathrm{U}}$, in a multi-layered thin film to be measured is determined from the time period from A/B to B/A interfaces as described for reference thin films in 5.4.

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$$d_{\mathrm{A}}^{\mathrm{U}} = \bar{t}_{\mathrm{A}}^{\mathrm{U}} \times \bar{z}_{\mathrm{A}} \qquad (3)$$

$$d_{\mathrm{B}}^{\mathrm{U}} = \bar{t}_{\mathrm{B}}^{\mathrm{U}} \times \bar{z}_{\mathrm{B}} \qquad (4)$$

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Formula (3) and Formula (4) can be applied for all the layers in multi-layered thin films of unknown thickness except the outmost layer A and the layer B adjacent to the substrate.

For single-layer thin films, the sputter depth or the thickness of single-layer thin films to be measured is calibrated with Formula (3).

5.8 The uncertainty, $\Delta\left(d_{\mathrm{A}}^{\mathrm{U}}\right)$, of the thicknesses of the layer A, $d_{\mathrm{A}}^{\mathrm{U}}$, is estimated from the standard deviations, $\sigma\left(\bar{t}_{\mathrm{A}}^{\mathrm{U}}\right)$, of average sputter time of the layer A from three consecutive sputter profiles of the thin film to be measured and the standard deviation, $\sigma\left(\bar{z}_{\mathrm{A}}\right)$, from 5.5 with Formula (5).

$$\left(\Delta\left(d_{\mathrm{A}}^{\mathrm{U}}\right)/d_{\mathrm{A}}^{\mathrm{U}}\right)^2 = \left(\sigma\left(\bar{t}_{\mathrm{A}}^{\mathrm{U}}\right)/\bar{t}_{\mathrm{A}}^{\mathrm{U}}\right)^2 + \left(\sigma\left(\bar{z}_{\mathrm{A}}\right)/\bar{z}_{\mathrm{A}}\right)^2 \qquad (5)$$

For estimation of the thickness of layer A with the minimal uncertainty, the sputter rate of the layer A should be measured accurately from reference thin films and the sputter time of layer A in a multi-layered thin film to be measured accurately as well.

NOTE The average sputtering rate of layer A from a single-layer sample or layers A and B from a multi-layer sample determined in 5.4 can be used for the prediction of the rates for a wide range of other materials through tabulated values of sputtering yields and bulk densities as described in Annex B.

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