INTERNATIONAL STANDARD

ISO 17109

Second edition
2022-03

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

Reference number
ISO 17109:2022(E)

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

Page

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

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.

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.

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.

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

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_A^R thickness of layer A of a single- or multi-layer reference thin film

d_B^R thickness of layer B of a multi-layer reference thin film

t_A^R sputtering time of layer A of a single- or multi-layer reference thin film

t_B^R sputtering time of layer B of a multi-layer reference thin film

$\bar{z}_A$ average sputtering rate of layer A

$\bar{z}_B$ average sputtering rate of layer B

d_A^U thickness of layer A in a single- or multi-layered thin film to be measured

d_B^U thickness of layer B in a multi-layered thin film to be measured

$\bar{t}_A^U$ average sputtering time from three consecutive sputter depth profiles of the layer A in a multi-layered thin film to be measured

$\bar{t}_B^U$ average sputtering time from three consecutive sputter depth profiles of the layer B in a multi-layered thin film to be measured

t_A^U sputtering time of layer A in a multi-layered thin film to be measured

t_B^U sputtering time of layer B in a multi-layered thin film to be measured

$\Delta\left(d_A^U\right)$ uncertainty of the thickness of the layer A

$\sigma\left(\bar{t}_A^U\right)$ standard deviation of $\bar{t}_A^U$

$\sigma(\bar{z}_A)$ standard deviation of $\bar{z}_A$

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.

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 using relevant references[3],[4].

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 SiO_2 on Si and Ta_2O_5 on Ta which are stable and remain clean or can easily be cleaned are recommended. Guidelines on how to clean thin film surface are available from ISO 18116[5] and ISO 18117[6].

5 Determination of sputtering rate

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.

NOTE 1 A typical measurement procedure and result of depth profiling measurement with AES, XPS, and SIMS using multi-layered thin films are illustrated in Annex A.

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 or of lower concentration and the upper plateau level for the layer where it is present with higher concentration present. The determination of the interface position by this procedure is applied to this document until the development of an ISO Standard for interface position. The average intensity in the upper plateau region is the plateau intensity (I_U). 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_L). 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 a)];

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

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

e) add the minimum intensity to the average calculated in d) to arrive at I_L.

The 50 % signal intensity shall be calculated using Formula (1):

$$I_{50} = (I_U - I_L)/2 \tag{1}$$

where

I_{50} is the 50 % signal intensity of sputter depth profile, in per cent;

I_U is the average intensity in the upper plateau region of the depth profile;

I_L is the average intensity in the lower plateau region of the depth profile.

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 document shall not be used for sputter rate determination.

NOTE 1 The 50 % of the plateau level is mentioned in ISO/TR 15969[7].

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, using Formulae (2) and (3). The unit of sputtering rate is nm/s.

$$z_A = \left(\frac{d_A^R}{t_A^R}\right) \tag{2}$$

$$z_B = \left(\frac{d_B^R}{t_B^R}\right) \tag{3}$$

where

z_A is the sputtering rate of layer A;

z_B is the sputtering rate of layer B;

d_A^R is the thickness of layer A of a single- or multi-layer reference thin film;

d_B^R is the thickness of layer B of a single- or multi-layer reference thin film;

t_A^R is the sputtering time of layer A of a single- or multi-layer reference thin film;

t_A^R is the sputtering time of layer B of a single- or multi-layer reference thin film.

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