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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

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](http://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](http://www.iso.org/patents)).

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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](http://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](http://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
$\Delta(d_A^U)$	uncertainty of the thickness of the layer A
$\sigma(\bar{t}_A^U)$	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><sup>[1]</sup>.

NOTE Sample rotation during ion sputtering is shown to reduce surface roughness development especially of polycrystalline films<sup>[2]</sup> 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<sup>[3],[4]</sup>.

**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<sub>2</sub> on Si and Ta<sub>2</sub>O<sub>5</sub> 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<sup>[5]</sup> and ISO 18117<sup>[6]</sup>.

## 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 \(1\)](#):

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

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.

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

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

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;

$t_B^R$  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.

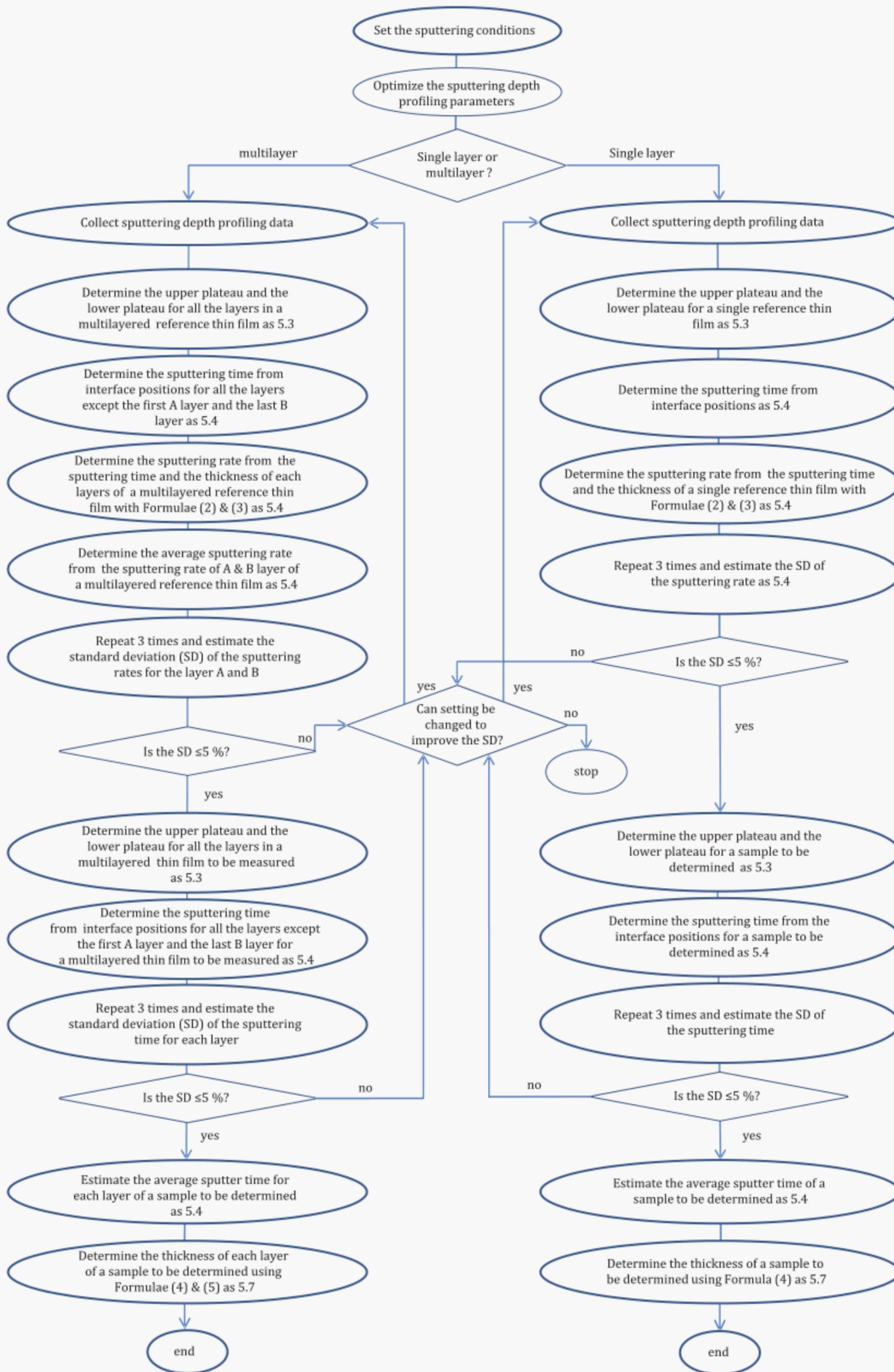


Figure 1 — Determination of sputtering rate

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.

**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 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,  $d_A^U$ , and that of a B layer,  $d_B^U$ , in a multi-layered thin film to be measured can be determined by multiplying the average sputtering time of the layer A,  $\bar{t}_A^U$ , and that of the layer B,  $\bar{t}_B^U$ , in a multi-layered thin film to be measured from three consecutive sputter depth profiles and the average sputtering rates of layers of A,  $\bar{z}_A$ , and B,  $\bar{z}_B$ , determined from the reference multi-layered thin films as in 5.4, respectively, using [Formula \(4\)](#) and [Formula \(5\)](#). The sputtering time of layer A,  $t_A^U$ , 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,  $t_B^U$ , 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.

$$d_A^U = \bar{t}_A^U \times \bar{z}_A \quad (4)$$

$$d_B^U = \bar{t}_B^U \times \bar{z}_B \quad (5)$$

where

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

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

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

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

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

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

[Formula \(4\)](#) and [Formula \(5\)](#) 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 thickness of single-layer thin films to be measured is calibrated with [Formula \(4\)](#).

**5.8** The uncertainty,  $\Delta(d_A^U)$ , of the thicknesses of the layer A,  $d_A^U$ , is estimated from the standard deviations,  $\sigma(\bar{t}_A^U)$ , of average sputter time of the layer A from three consecutive sputter profiles of the thin film to be measured and the standard deviation,  $\sigma(\bar{z}_A)$ , from [5.5](#) with [Formula \(6\)](#).

$$\left(\Delta(d_A^U)/d_A^U\right)^2 = \left(\sigma(\bar{t}_A^U)/\bar{t}_A^U\right)^2 + \left(\sigma(\bar{z}_A)/\bar{z}_A\right)^2 \quad (6)$$

where

$\Delta(d_A^U)$  is the uncertainty of the thickness of the layer A;

$\sigma(\bar{t}_A^U)$  is the standard deviation of  $\bar{t}_A^U$ ;

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

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

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

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

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](#).

## Annex A (informative)

### Interlaboratory test report

#### A.1 General

This annex gives the interlaboratory test report on the method for sputter rate determination in XPS, AES, and SIMS sputter depth profiling using multi-layer thin films.

#### A.2 Overview

This interlaboratory test was performed to provide data to assist in the development of this document for the calibration of sputter depth by determining ion sputtering rate for depth profiling measurement with AES, XPS, and SIMS using multi-layered thin films.

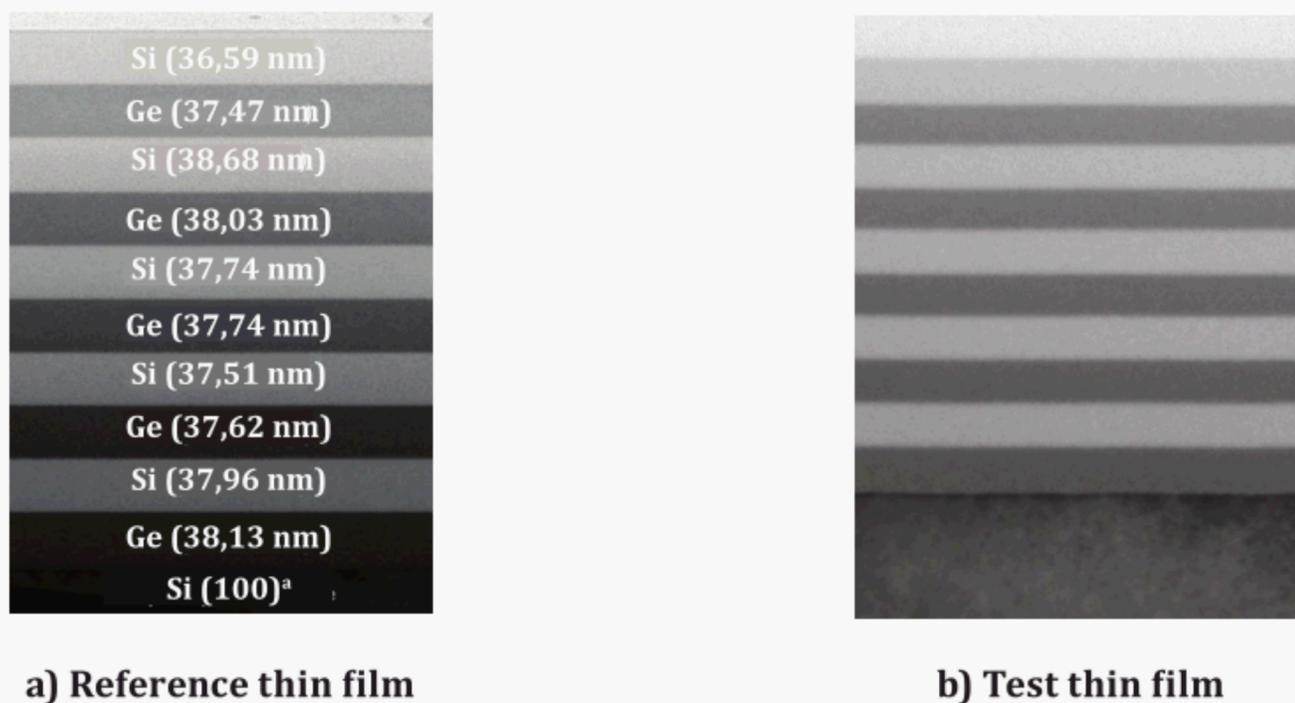
#### A.3 Principle

The sputtering rate of AES, XPS, and SIMS sputter depth profiling is determined using a multi-layer film where the thickness of each layer is determined by transmission electron microscopy (TEM). The interface position is determined by the 50 % drop of the plateau intensity according to ISO/TR 15969.

#### A.4 Sample description

- Reference material (RM): 5 × (Si/Ge)/Si (100) substrate with certified thickness.
- Test sample: 5 × (Si/Ge)/Si (100) substrate with unknown thickness.

The multi-layer films were grown by ion beam sputter deposition. The target materials Si and Ge wafer were sputtered by 1 keV Ar<sup>+</sup> ion beam and deposited on 6" Si (100) wafers at room temperature. The thickness of the samples was certified by TEM based on the lattice constant of the Si (100) substrate as shown in [Figure A.1](#).

**Key**

<sup>a</sup> Substrate.

**Figure A.1 — TEM images of an Si/Ge multi-layer**

## A.5 Measurement procedure

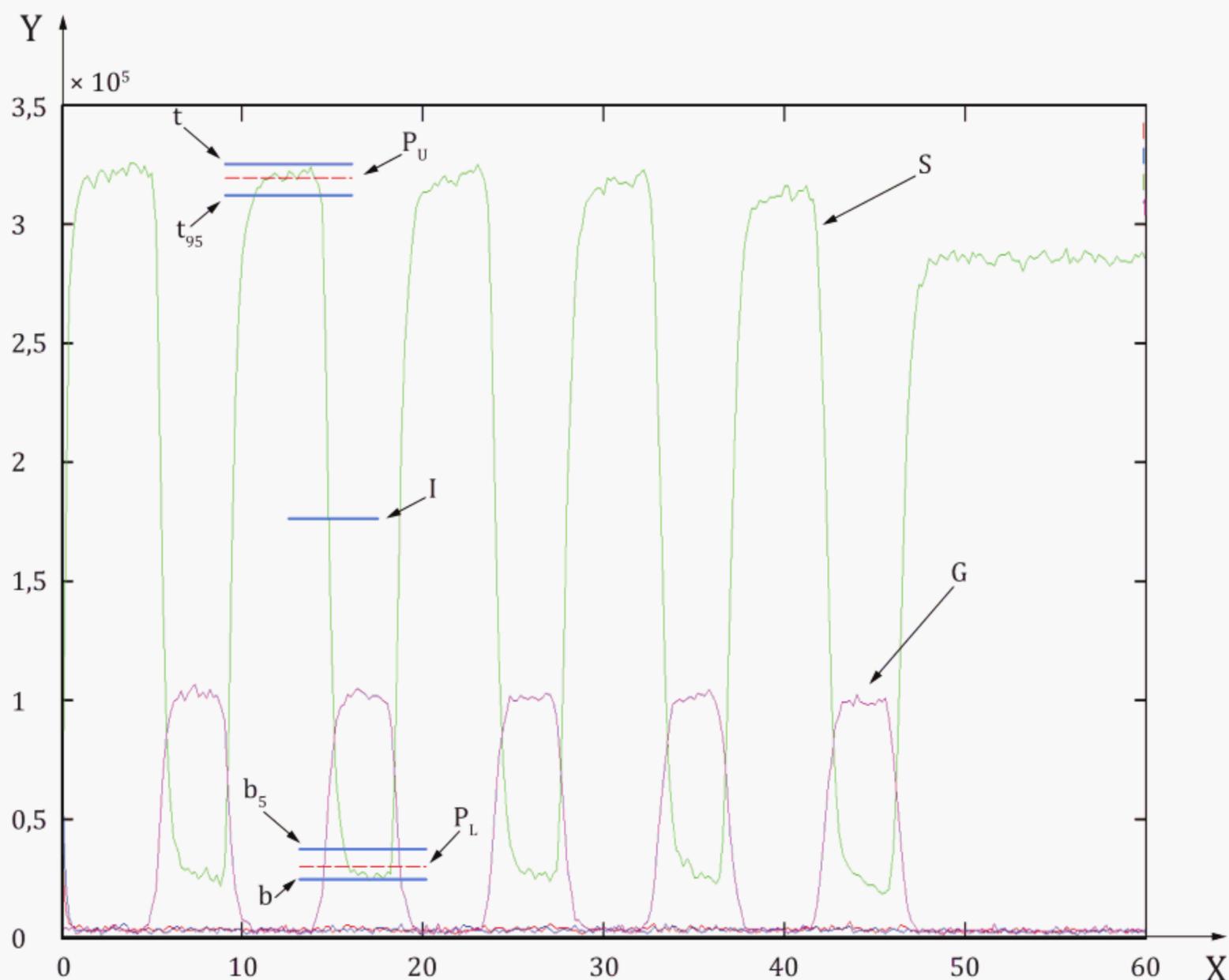
- Optimize the sputter depth profiling parameters according to ISO 14606.
- The reference material and the test sample should be measured in the same day after the instrument is stabilized to minimize uncertainty.
- Measure the sputter depth profile of the Si/Ge multi-layer RM and estimate the sputtering rate of each layer and the mean sputtering rate for Si and Ge, respectively. The interface position is determined by the point where the signal intensity of the element reaches 50 % of its value from the upper plateau level.
- Estimate the depth resolution of each interface according to ISO 14606, and report the data. In ISO 14606, the depth resolution ( $\Delta z$ ) is determined by multiplication of the mean sputtering rate ( $\bar{z}_{av}$ ) by the time interval  $\Delta t$  which is the time period where the signal intensities change from 16 % to 84 % (or 84 % to 16 %) of the intensity corresponding to the 100 % of each of the adjacent layers of a multi-layer system.
- From the mean sputtering rate of Si and Ge, calculate the thickness of each Si and Ge layer in the Si/Ge multi-layer film to be measured.

## A.6 Summary of the interlaboratory test results

- Typical sputter depth profiles.

A typical AES sputter depth profile, XPS sputter depth profile, and SIMS sputter depth profile is given in [Figure A.2](#) to [Figure A.4](#), respectively. For AES and XPS sputter depth profiles, the upper and lower plateau intensities and consequently, the interface position where the signal intensity of the element reaches 50 % of its value from the upper plateau level can be determined. However, for SIMS sputter depth profiling, the interface positions may be significantly affected by a severe matrix effect so that the plateau intensity cannot be clearly determined as shown in [Figure A.4](#). In this case, the average

value intensity of the plateau can be used which can increase the uncertainty of the determined sputter rate. Participants to the interlaboratory test are summarized in [Table A.1](#) with analysis conditions.



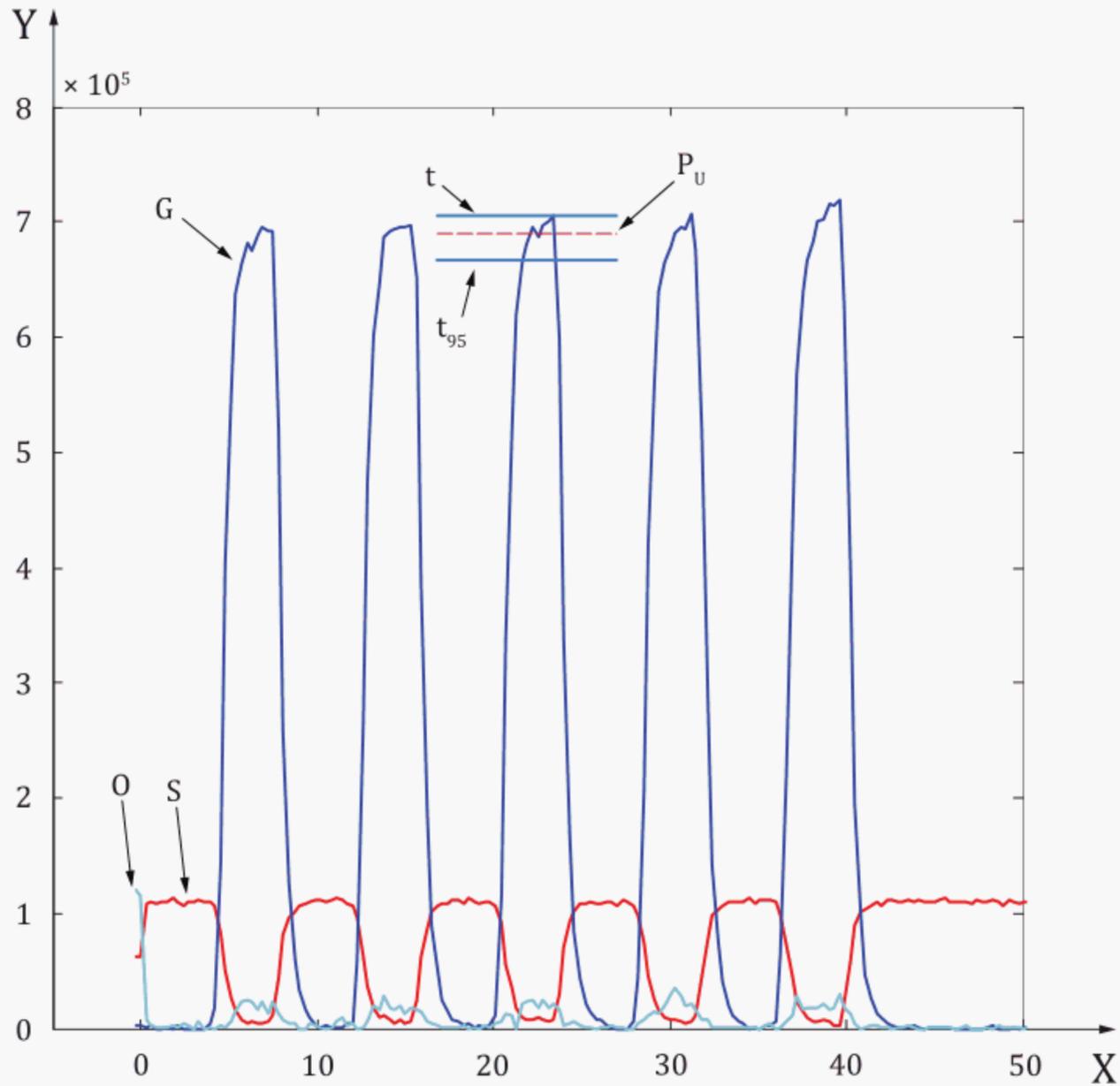
### Key

- X sputter time, expressed in minutes
- Y intensity of auger peak, expressed in counts
- S depth profile of Si LVV
- G depth profile of Ge LVV
- t maximum intensity of Si LVV at second Si layer
- $t_{95}$  95 % of t
- b minimum intensity of Si LVV at second Ge layer
- $b_5$   $b + 5\%$  of t
- $P_U$  upper plateau of Si LVV depth profile
- $P_L$  lower plateau of Si LVV depth profile
- I interface position

**Figure A.2 — Typical AES sputter depth profile of an Si/Ge multi-layer on an Si substrate**

The nominal thickness of Si and Ge layers is 38 nm, respectively. The upper plateau intensity of Si is higher than that of the Si crystalline substrate, probably due to the matrix effect of the amorphous Si/Ge multi-layer grown by ion beam sputter deposition. The peak heights of Si LVV and Ge LVV Auger peaks were used for sputter depth profiling with 1 keV Ar<sup>+</sup> ions with the incidence angle of 42° from the surface normal. To demonstrate an example of how to determine the upper plateau level of Si, indicated

are the maximum intensity and 95 % of the maximum intensity which defines the upper plateau region. The average intensity in the upper plateau region is the upper plateau intensity which can be calculated from intensities of each sputter time intervals or can be determined by visual inspection between the maximum intensity and 95 % of the maximum intensity. The lower plateau intensity can be determined in the similar procedure.

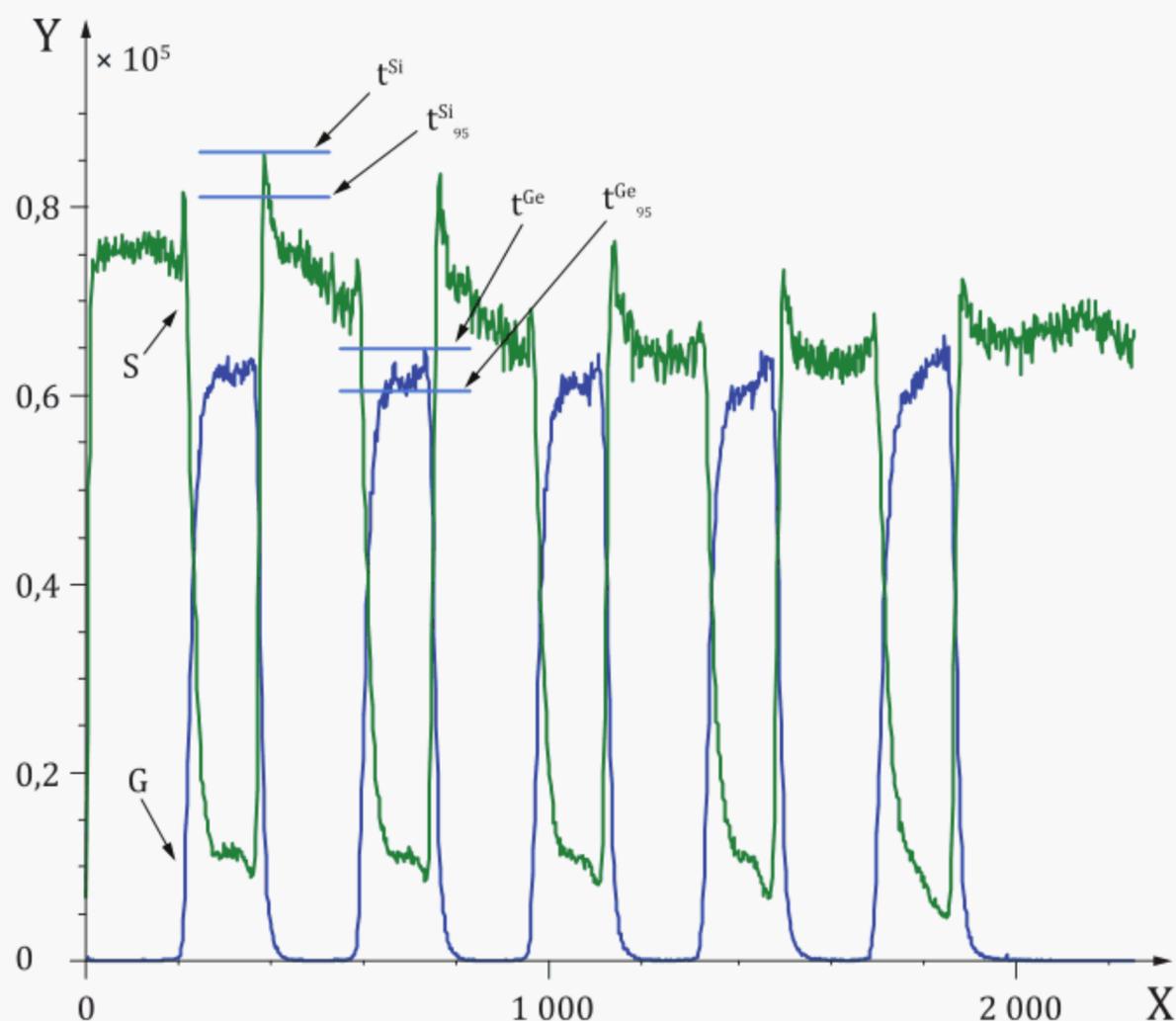


**Key**

- X sputter time, expressed in minutes
- Y XPS peak area intensity, expressed in counts
- G depth profile of Ge 2p<sub>3/2</sub>
- O depth profile of O 1s
- S depth profile of Si 2p
- t maximum intensity of Ge 2p<sub>3/2</sub> at third Ge layer
- t<sub>95</sub> 95 % of t
- P<sub>U</sub> upper plateau of Ge 2p<sub>3/2</sub> depth profile

**Figure A.3 — Typical XPS sputter depth profile of an Si/Ge multi-layer on an Si substrate**

The peak areas of Si 2p (95 eV-115 eV) and Ge 2p<sub>3/2</sub> (1212 eV-1232 eV) peaks were used for sputter depth profiling with 2 keV Ar<sup>+</sup> ions with the incidence angle of 45° from the surface normal.



#### Key

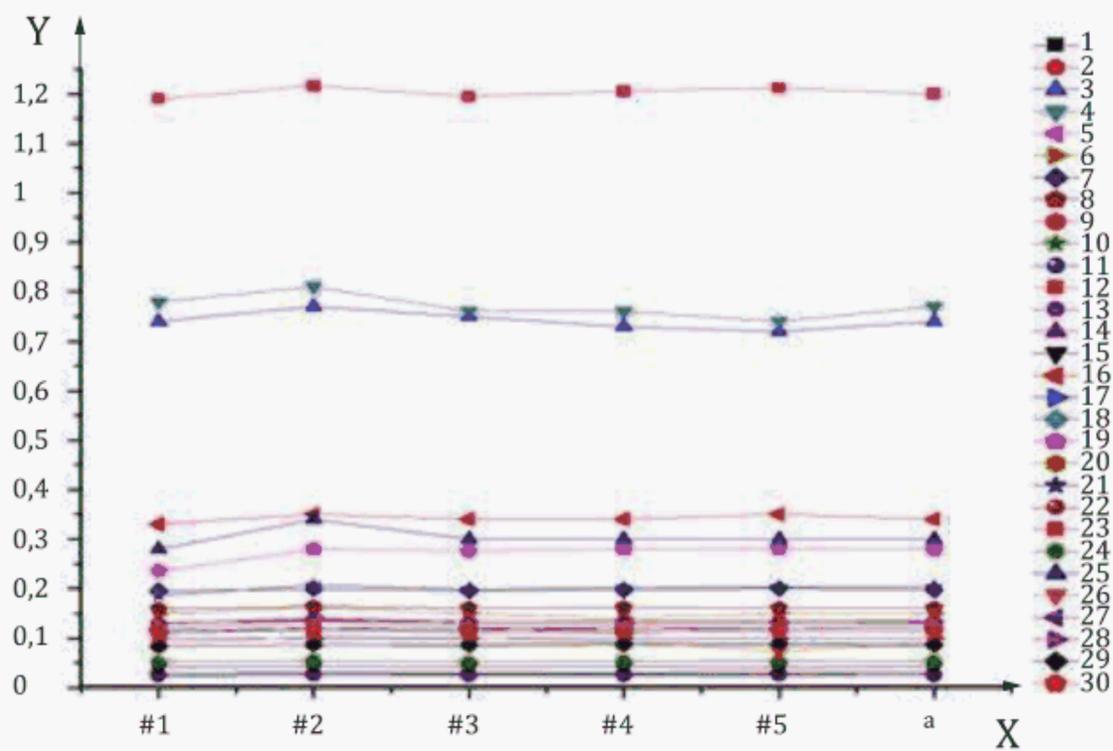
- X sputter time, expressed in seconds
- Y secondary ion intensity, expressed in counts
- S depth profile of  $(^{28}\text{Si}+^{133}\text{Cs})^+$
- G depth profile of  $(^{74}\text{Ge}+^{133}\text{Cs})^+$
- $t^{\text{Si}}$  maximum intensity of  $(^{28}\text{Si}+^{133}\text{Cs})^+$  at second Si layer
- $t^{\text{Si}}_{95}$  95 % of  $t^{\text{Si}}$
- $t^{\text{Ge}}$  maximum intensity of  $(^{74}\text{Ge}+^{133}\text{Cs})^+$  at second Ge layer
- $t^{\text{Ge}}_{95}$  95 % of  $t^{\text{Ge}}$

**Figure A.4 — Typical SIMS sputter depth profile of an Si/Ge multi-layer on an Si substrate**

2,0 keV Cs<sup>+</sup> ions with the incidence angle of 45° from the surface normal were used for primary ions and  $(^{28}\text{Si}+^{133}\text{Cs})^+$  ions and  $(^{74}\text{Ge}+^{133}\text{Cs})^+$  were analysed as secondary ions with a quadrupole mass analyser without normalization. In this SIMS profile, an upper plateau cannot be defined for the second Si peak due to the severe matrix effect in the interface. The thickness of the upper plateau region, as defined in this document, is much smaller than the half of the layer thickness. However, for the second Ge peak, an upper plateau can be defined according to this document.

b) Sputtering rate of Si and Ge layers.

Sputtering rates reported are plotted for Si layers in [Figures A.5](#) and [A.6](#). Sputtering rates range from 1,2 nm/s to 0,02 nm/s.

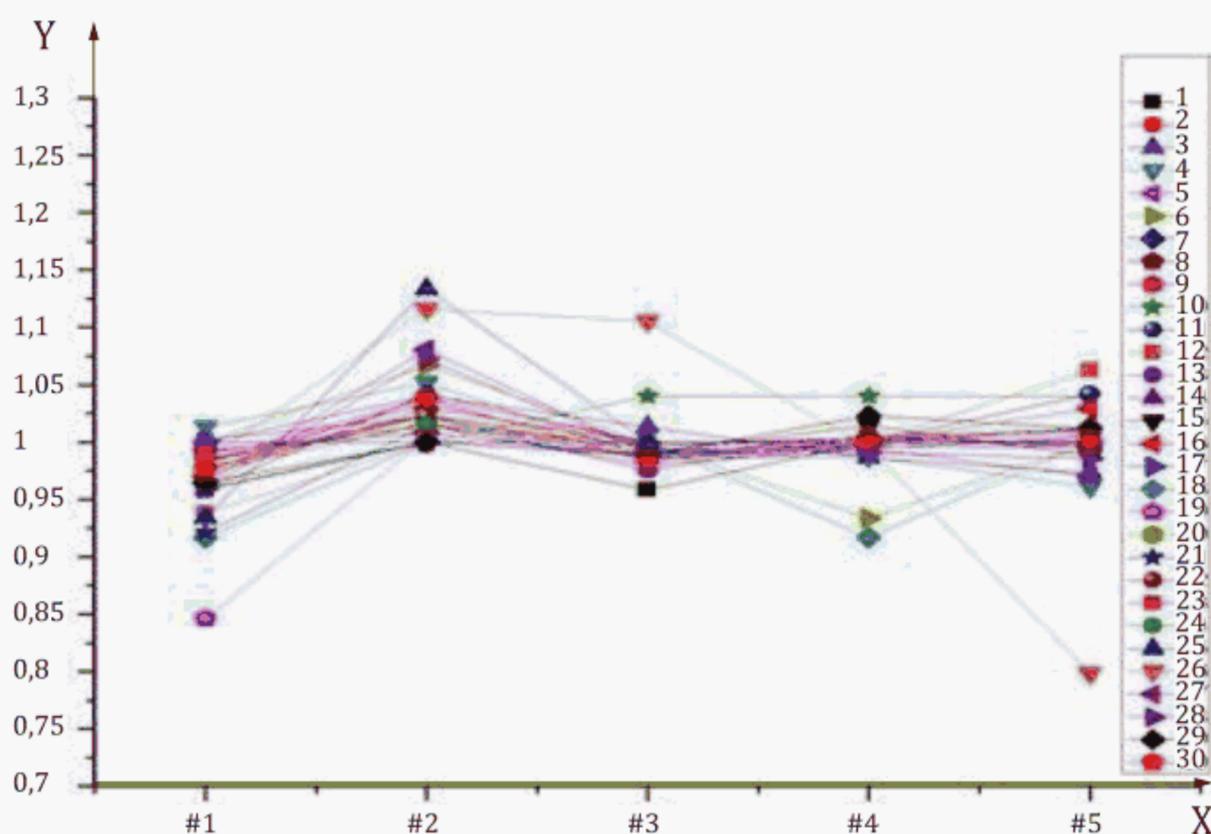


**Key**

- X layer number
- Y Si sputtering rate (nm/s)
- a Mean.

NOTE The numbers refer to the participants in the [Table A.1](#).

**Figure A.5 — Si sputtering rates for each Si layer — Absolute rates**



#### Key

X layer number

Y ratio of the rates to the average

NOTE The numbers refer to the participants in the [Table A.1](#).

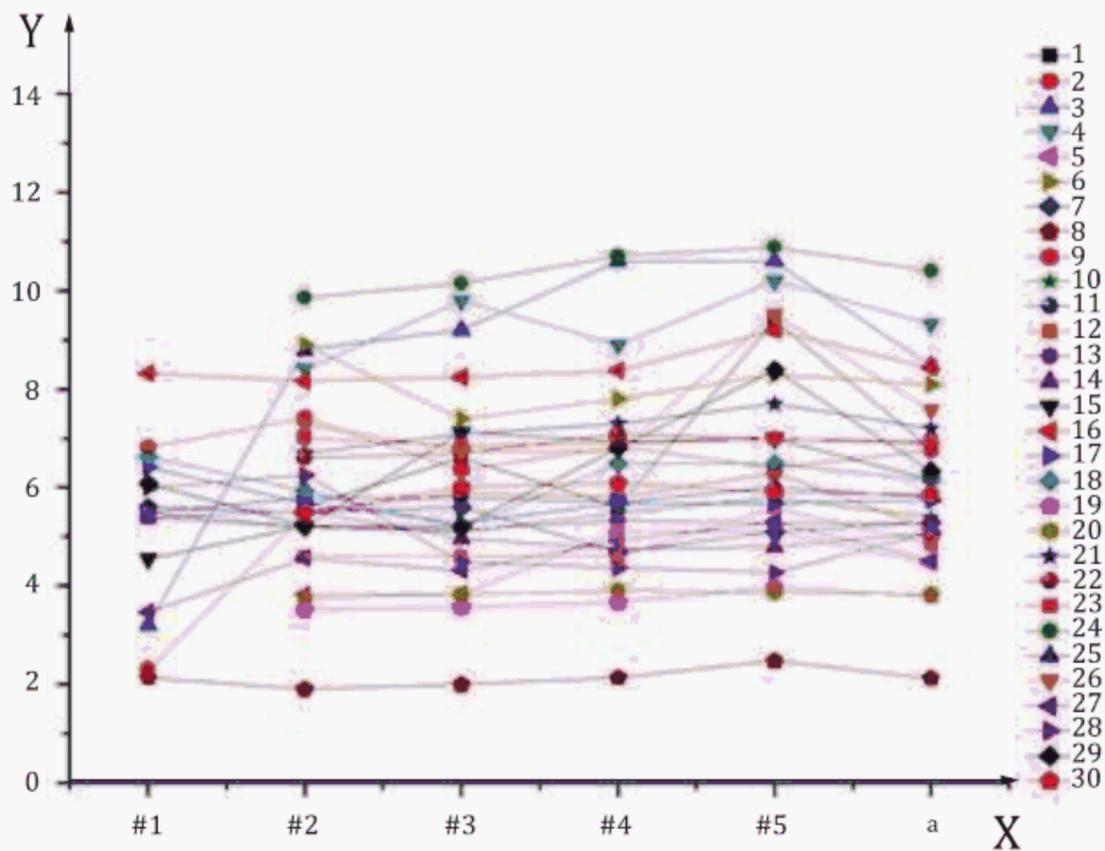
**Figure A.6 — Si sputtering rates for each Si layer — Ratio of the rates to the average rate**

As can be seen in [Figures A.5](#) and [A.6](#), the sputtering rates for five Si layers do not change significantly for most of the sputter depth profiling. If the two results using oxygen primary ions (laboratory 19 and 26) are ignored, they show an average relative standard deviation of 5 %. It shows that the equipment in many laboratories can provide consistent depth profiling rates that make this International Standard worthwhile.

The Ge sputtering rates show a similar consistency to the Si sputtering rates in [Figures A.5](#) and [A.6](#) as 5 %. The Si sputtering rate from the first layer is lower by -4 % due to the surface transient effect and the Ge sputtering rate from the last layer is lower by -6 % due to the interface transient effect between the last Ge layer and the Si substrate for AES sputter depth profiling. The interface between the last Ge layer and the Si substrate can be different from other Ge/Si interfaces because the Si substrate is crystalline and have a native oxide. XPS and SIMS sputter depth profiling show results similar to each other.

c) Depth resolution of Si and Ge profiles.

Depth resolutions estimated for Si leading edges are given in [Figure A.7](#). It shows a wide range of depth resolution from 2 nm to 11 nm. The distribution of depth resolutions estimated from AES, XPS, and SIMS sputter depth profiling in this interlaboratory test does not show any clear difference.



**Key**

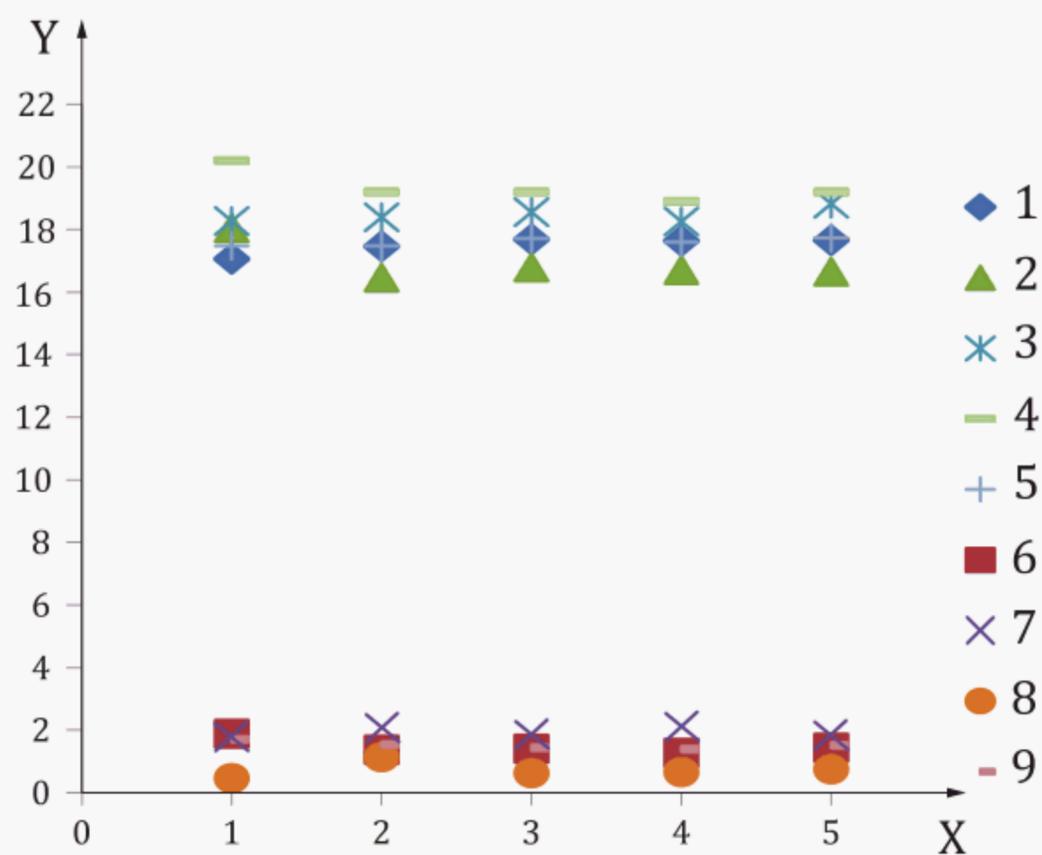
- X layer number
- Y Si depth resolution at leading edge(nm)
- a Mean.

**Figure A.7 — Si depth resolution at leading edge for each layer**

The Si depth resolution at trailing edge for each layer shows a similar distribution. The Ge depth resolution at leading edge and that at trailing edge do not show any significant difference from the Si depth resolution.

d) Si and Ge thickness determined for test thin films.

Si and Ge thickness determined for test thin films are plotted in [Figures A.8](#) and [A.9](#). The standard deviation of Si and Ge thickness determined is 7 % to 8 %. Considering that the stability of Si and Ge sputtering rate for five layers is much better than 7 % to 8 % as shown in [Figures A.5](#) and [A.6](#), the standard deviation of Si and Ge thickness values reported by interlaboratory test participants is quite high. As shown in [Figures A.8](#) and [A.9](#), the standard deviation of thickness values from AES sputter depth profiling seems to be approximately two times higher than that from XPS sputter depth profiling in this interlaboratory test which is not clearly understood at present. The poor standard deviation of thickness from SIMS sputter depth profiling can be explained by the SIMS matrix effect in the interface which makes it difficult to define the plateau intensity and the interface position.

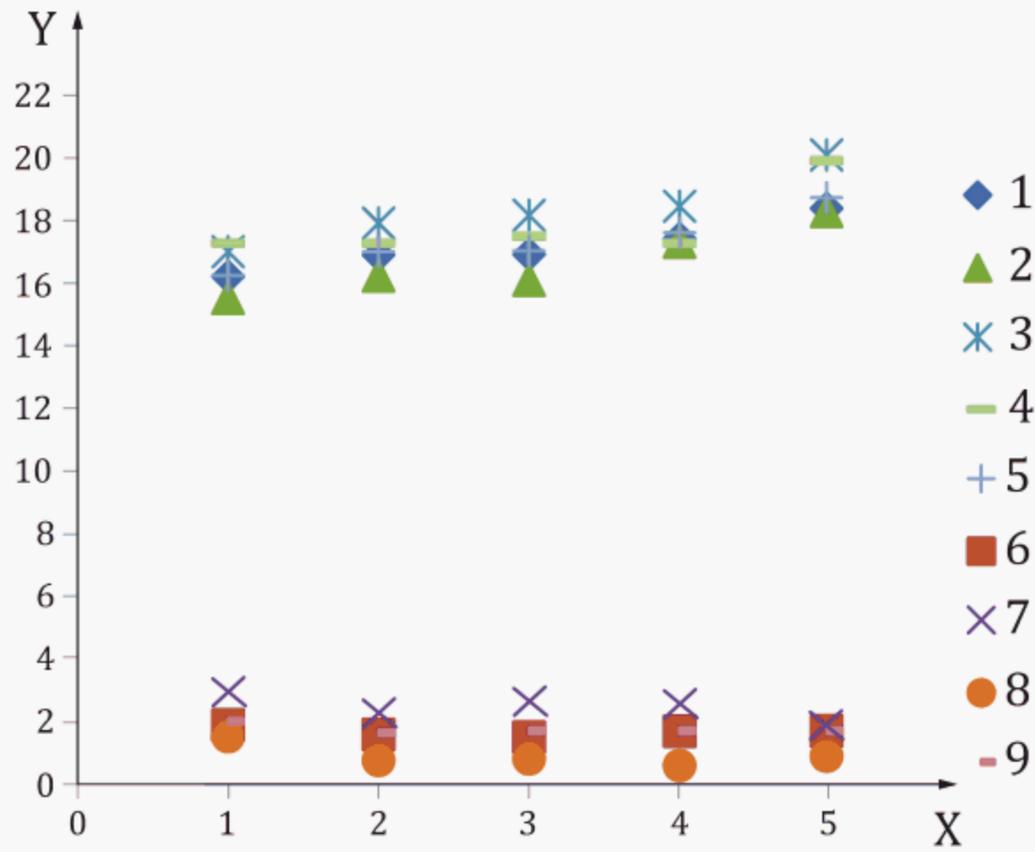


**Key**

- X layer number
- Y Si thickness average and SD (nm)
- 1 AES ave
- 2 SIMS ave
- 3 XPS ave
- 4 total ave
- 5 TEMt
- 6 AES SD
- 7 SIMS SD
- 8 XPS SD
- 9 total SD

NOTE Ave and SD are abbreviations of average and standard deviation, respectively.

**Figure A.8 — Thickness determined for each layers in the test thin films — Statistics of Si**



**Key**  
 X layer number  
 Y Ge average thickness SD (nm)  
 1 AES ave  
 2 SIMS ave  
 3 XPS ave  
 4 total ave  
 5 TEM  
 6 AES SD  
 7 SIMS SD  
 8 XPS SD  
 9 total SD

NOTE Ave and SD are abbreviations of average and standard deviation, respectively.

**Figure A.9 — Thickness determined for each layers in the test thin films — Statistics of Ge**

**A.7 Conclusions**

To develop a standard document for the calibration of sputter depth by determining ion sputtering rate for depth profiling measurement with AES, XPS, and SIMS using multi-layered thin films, an interlaboratory test was performed. The standard deviation of the thickness values in the interlaboratory test with the evaluation of the test sample was 7 % to 8 % which is higher than 5 % for the Si and Ge sputtering rates of five layers. The standard deviation of the thickness values from AES sputter depth profiling seems to be higher than that from XPS sputter depth profiling in this interlaboratory test. The poor standard deviation of thickness from SIMS sputter depth profiling can be from the SIMS matrix effect in the interface.

Table A.1 — Participants to the interlaboratory test with analysis conditions

No.	Technique	Primary ion and energy		Angle of incidence from the surface normal	Sputter area (mm <sup>2</sup> )
1	AES	Ar <sup>+</sup>	1 keV	42°	0,64
2	AES	Ar <sup>+</sup>	1 keV	42°	0,64
3	AES	Ar <sup>+</sup>	3 keV	42°	0,64
4	AES	Ar <sup>+</sup>	3 keV	42°	0,64
5	SIMS	Ar <sup>+</sup>	1 keV	45°	0,09
6	SIMS	Ar <sup>+</sup>	3 keV	45°	0,09
7	AES	Ar <sup>+</sup>	2 keV	47°	2,25
8	SIMS	Cs <sup>+</sup>	2 keV	60°	0,32
9	XPS	Ar <sup>+</sup>	2 keV	45°	4,00
10	AES	Ar <sup>+</sup>	1 keV	55°	X:60, Y:70 (arb. Units)
11	AES	Ar <sup>+</sup>	1 keV	85°	X:60, Y:70 (arb. Units)
12	AES	Ar <sup>+</sup>	1 keV	55°	0,16
13	AES	Ar <sup>+</sup>	1 keV	45°	4,00
14	AES	Ar <sup>+</sup>	1 keV	45°	4,00
15	XPS	Ar <sup>+</sup>	1 keV	45°	4,00
16	AES	Ar <sup>+</sup>	3 keV	47°	4,00
17	XPS	Ar <sup>+</sup>	2 keV	45°	4,00
18	XPS	Ar <sup>+</sup>	2 keV	45°	4,00
19	SIMS	O <sub>2</sub> <sup>+</sup>	3 keV	?	0,04
20	XPS	Ar <sup>+</sup>	0,5 keV	58°	8,00
21	AES	Ar <sup>+</sup>	3 keV	60°	4,00
22	AES	Ar <sup>+</sup>	3 keV	60°	4,00
23	AES	Ar <sup>+</sup>	2 keV	56,5°	3,60
24	XPS	Ar <sup>+</sup>	2 keV	45°	3,00
25	AES	Ar <sup>+</sup>	3 keV	50°	1,00
26	SIMS	O <sub>2</sub> <sup>+</sup>	3 keV	40,8°	0,062 5
27	AES	Ar <sup>+</sup>	0,5 keV	45°	0,25
28	AES	Ar <sup>+</sup>	0,5 keV	45°	0,25
29	AES	Ar <sup>+</sup>	1 keV	66,5°	1,00
30	AES	Ar <sup>+</sup>	3 keV	9°	16,00

## Annex B (informative)

### Prediction of the rates for a wide range of other materials through tabulated values of sputtering yields

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. The sputtering rate of layer C,  $z_C$ , can be approximately predicted from the ratio of tabulated values of sputtering yields and bulk densities as in [Formula \(B.1\)](#).  $Y_C$  and  $Y_A^R$  are the tabulated values of sputtering yields of C and A elements, respectively.  $N_C$  and  $N_A^R$  are the atomic densities in atoms/m<sup>3</sup> of C and A elements.

$$z_C = \bar{z}_A \left( Y_C / Y_A^R \right) \times \left( N_A^R / N_C \right) \quad (\text{B.1})$$

Some sputtering yields are tabulated in References [\[2\]](#), [\[8\]](#) to [\[11\]](#) and [\[12\]](#) to [\[15\]](#). The predicted sputtering rates are not considered to have an accuracy better than probably 10 % but can be used as a guide to setting up the sputtering conditions in day-to-day sputter depth profiling for a wide range of other materials or to give approximate thicknesses. Therefore, when reporting the predicted sputtering rates, the final uncertainty of the predicted sputtering rates should be clearly stated with detailed descriptions of prediction procedures, the source of the tabulated values of sputtering yields and bulk densities, and the prediction results. The composition of the measured thin films should be similar to that of the tabulated data since significant errors may arise if elemental data are applied to compound specimens in [Formula \(B.1\)](#).

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