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

*Analyse chimique des surfaces — Profilage d'épaisseur par  
bombardement — Optimisation à l'aide de systèmes mono- ou  
multicouches comme matériaux de référence*

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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)).

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](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 third edition cancels and replaces the second edition (ISO 14606:2015), which has been technically revised.

The main changes are as follows:

- [Clause 3](#): terms and definitions have been simplified using normative references;
- [6.2](#): descriptions have been added that it is only necessary to sputter off a few certain periods of layers from the topmost surface to obtain average sputtering rate for practical use of a multi-layered system;
- [7.1.3](#), [7.1.4](#) and [7.1.5](#): descriptions have been added to mention the use of the procedures provided by manufacturer of apparatuses for alignment of sputtered area with a smaller analysis area;
- [Table B.1](#) and [Table C.1](#): contents have been updated.

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

Reference materials are useful in optimizing the depth resolution of sputter profiling methods in materials such as silicon wafers, multilayered devices (e.g. AlGaAs double-hetero lasers, high electron mobility transistors) and alloy-galvanized steel for corrosion-resistant car bodies.

The specific applications of this document are as follows.

- a) Single-layered and multilayered systems on a substrate as reference materials are useful for the optimization of depth resolution as a function of instrument settings in Auger electron spectroscopy, X-ray photoelectron spectroscopy and secondary ion mass spectrometry.
- b) These systems are useful for illustrating the effects of the evenness of the sputter crater, the inclination of the crater bottom, the sample drift, the drift of sputter conditions (e.g. ion beam current density) on depth resolution.
- c) These systems are useful for illustrating the effects of sputter-induced surface roughening and sputter-induced atomic mixing on depth resolution.
- d) These systems are useful for the evaluation of instrument performance for instrument suppliers and users.
- e) This document is timely and topical, and can be used for a basis of future development of sputter depth profiling.

A list of ISO Guides related to this document is given in the Bibliography<sup>[1][2][3][4][5]</sup>.

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# Surface chemical analysis — Sputter depth profiling — Optimization using layered systems as reference materials

## 1 Scope

This document gives guidance and requirements on the optimization of sputter-depth profiling parameters using appropriate single-layered and multilayered reference materials, in order to achieve optimum depth resolution as a function of instrument settings in Auger electron spectroscopy, X-ray photoelectron spectroscopy and secondary ion mass spectrometry.

This document is not intended to cover the use of special multilayered systems such as delta doped layers.

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

## 3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 18115-1 and the following apply.

ISO and IEC maintain terminology 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

#### **image depth profile**

three-dimensional representation of the spatial distribution of a particular elemental or molecular species (as indicated by emitted secondary ions or electrons) as a function of depth or material removed by sputtering

### 3.2

#### **plateau region**

region in which the signal remains constant or without significant variation with sputtering time

### 3.3

#### **sputter depth profile**

depth profile obtained when the surface is measured as material is removed by sputtering

## 4 Symbols and abbreviated terms

$\Delta z$	depth resolution
$I$	signal intensity

$\bar{z}$	sputtering rate
AES	Auger electron spectroscopy
SEM	scanning electron microscopy
SIMS	secondary ion mass spectrometry
XPS	X-ray photoelectron spectroscopy

## 5 Setting parameters for sputter depth profiling

### 5.1 General

For the purposes of this document, typical probing and sputtering parameters for sputter depth profiling in AES, XPS and SIMS are given in [Table 1](#) and [Table 2](#). These parameters represent a range which covers many different types of instrumentation. Recommended conditions for a particular instrument may be available from the respective instrument manufacturers and optimized by experimentation on the laboratory instrument using the information included in this document.

**Table 1 — Typical probing parameters for sputter depth profiling**

	AES	XPS	SIMS
Probing species	Electrons	Photons: Mg K $\alpha$ , Al K $\alpha$	Primary ions: Cs <sup>+</sup> , O <sup>-</sup> , O <sub>2</sub> <sup>+</sup> , Ga <sup>+</sup>
Energy of probing species	1 keV to 25 keV	1,253 keV, 1,486 keV	0,1 keV to 25 keV
Current or power	1 nA to 10 <sup>3</sup> nA (Faraday cup)	1 W to 10 <sup>4</sup> W (Source power)	1 nA to 10 <sup>4</sup> nA (Faraday cup)
Angle of incidence	0° ≤ $\theta$ < 90°	0° ≤ $\theta$ < 90°	0° ≤ $\theta$ < 90°
Analysed species	Auger electrons in eV (kinetic energy)	Photoelectrons in eV (kinetic or binding energy)	Secondary ions in AMU (mass or mass/charge)
Energy range of emission species	0 keV to 3 keV	0 keV to 1,5 keV	0 keV to 0,125 keV
Angle of emission	0° ≤ $\theta$ < 90°	0° ≤ $\theta$ < 90°	0° ≤ $\theta$ < 90°
Analysis area	10 <sup>-8</sup> mm <sup>2</sup> to 10 <sup>-2</sup> mm <sup>2</sup>	10 <sup>-4</sup> mm <sup>2</sup> to 10 mm <sup>2</sup>	10 <sup>-6</sup> mm <sup>2</sup> to 10 <sup>-2</sup> mm <sup>2</sup>

**Table 2 — Typical sputtering parameters for sputter depth profiling**

	Typical operating parameters	Remarks
Ion species	Ar <sup>+</sup> , Kr <sup>+</sup> , Xe <sup>+</sup> , O <sup>-</sup> , O <sub>2</sub> <sup>+</sup> , Ga <sup>+</sup> , Cs <sup>+</sup>	Inert or reactive gas ions or metal ions
Ion energy	0,1 keV to 25 keV	
Ion beam current	1 nA to 10 <sup>4</sup> nA	Faraday cup
Angle of incidence	0° ≤ $\theta$ < 90°	
Sputtered area	10 <sup>-2</sup> mm <sup>2</sup> to 10 <sup>2</sup> mm <sup>2</sup>	Raster scan of focused ion beam

NOTE The ion gun parameters and vacuum conditions can also affect the depth resolution. For example, the gas pressure in the ion source can change during the course of the analysis.

## 5.2 Auger electron spectroscopy

Important parameters for a depth profile measurement of a single layered or an A/B/A/B/... multilayered system by AES with ion sputtering are the following<sup>[6]</sup>.

- a) Probing parameters (important for analysis): Electron energy, electron beam current, angle of incidence, analysis area (i.e. beam diameter or raster area).
- b) Sputtering parameters (important for depth resolution): Ion species, ion energy, ion beam current<sup>[7]</sup>, angle of incidence, sputtered or raster area. Sample stage is in a stationary or rotational mode.
- c) Measurement parameters:
  - 1) Kinetic energies of Auger electrons from both overlayer and substrate elements, or from elements A and B (important for both analysis and depth resolution).
  - 2) Direct mode,  $N(E)$  or  $EN(E)$ , or differential mode,  $dN(E)/dE$  or  $dEN(E)/dE$  (important for analysis).

NOTE 1  $N(E)$ ,  $EN(E)$ ,  $dN(E)/dE$  and  $dEN(E)/dE$  refer to different kinds of Auger spectra where the Auger electron intensity,  $N$ , is plotted as a function of the electron kinetic energy,  $E$ . In  $N(E)$  spectra, signal intensities are measured as the heights of the Auger peaks above background. In  $dN(E)/dE$  spectra, signal intensities are measured as the peak-to-peak heights of the Auger signals or the differential spectra of  $N(E)$ . With certain types of analyser (e.g. the cylindrical mirror analyser), Auger electron intensities are presented in  $EN(E)$  and  $dEN(E)/dE$  formats, in which the spectrum approximates  $E$  times  $N(E)$ .

- 3) With ion sputtering, data may be collected in either an alternating mode or continuous mode. If the continuous mode is used, it is preferable to ensure that the ion-induced Auger electron signals are negligible.

NOTE 2 The problem of ion-induced Auger electrons seems only significant for Auger electron peaks below 100 eV<sup>[8][9]</sup>.

## 5.3 X-ray photoelectron spectroscopy

Important parameters for a depth profile measurement of a single layered or an A/B/A/B/... multilayered system by XPS with ion sputtering are the following.

- a) Probing parameters (important for analysis): Photon energy (X-ray source), X-ray source power (i.e. voltage and current), angle of incidence, analysis area (i.e. beam diameter or selected area).
- b) Sputtering parameters (important for depth resolution): Ion species, ion energy, ion beam current, angle of incidence, sputtered or raster area. The sample stage can be in a stationary or rotational mode.
- c) Measurement parameters (important for both analysis and depth resolution):
  - 1) kinetic energies of photoelectrons and/or the respective electron binding energies of both overlayer and substrate elements or both elements A and B;
  - 2) area of measurement for selected area XPS;
  - 3) emission angle of electrons.

NOTE Usually, XPS signal intensities are measured as a function of sputtering time in an alternating mode with ion sputtering.

## 5.4 Secondary ion mass spectrometry

Important parameters for a depth profile measurement of a single layered or an A/B/A/B/... multilayered system by SIMS are the following.

- a) Probing and simultaneously sputtering parameters (important for both analysis and depth resolution): Primary ion species, ion impact energy, ion beam current, angle of incidence, analysis area (i.e. gated area), sputtered area. The sample stage can be a stationary or rotational mode.

NOTE 1 In some SIMS systems, the beam energy is given for the source potential with respect to the ground but the sample potential is not at ground. The impact energy takes account of the sample potential.

NOTE 2 Some time-of-flight SIMS instruments use dual beams. In this case, all parameters for both beams are noted.

- b) Measurement parameters (important for both analysis and depth resolution):
- 1) positive or negative secondary ion species (atomic or molecular) of both overlayer and substrate elements or both elements A and B;
  - 2) settings of gates (i.e. electronic, optical, etc.).

NOTE 3 Usually, secondary ion signal intensities are measured as a function of sputtering time in a continuous mode with primary ion sputtering. In some SIMS instruments, an interrupted mode (primary ion gating) is used where different ion beams are used for sputtering and analysis.

## 6 Depth resolution at an ideally sharp interface in sputter depth profiles

### 6.1 Measurement of depth resolution

For the purposes of this document, the measurement of the depth resolution  $\Delta z$  of sputter depth profiles of a single layered or an A/B/A/B/... multilayered system is as follows<sup>[10][11][12]</sup>.

For SIMS, where matrix effects are significantly different between the two layers,  $\Delta z$  may still be used for optimization but may not relate closely to the real depth resolution of the underlying chemical composition.

NOTE The definition of depth resolution  $\Delta z$  in this clause applies only for optimization of setting parameters in depth profiling. The definition and measurement procedures of depth resolution will be described in International Standards to be developed by ISO/TC 201/SC 1 and SC 4, respectively, in the future.

### 6.2 Average sputtering rate $\bar{z}_{av}$

The average sputtering rate  $\bar{z}_{av}$  is given by [Formula \(1\)](#):

$$\bar{z}_{av} = z_{tot} / t_{tot} \quad (1)$$

where

$z_{tot}$  is the total thickness of a single overlayer or multilayered system on a substrate;

$t_{tot}$  is the total sputtering time required to sputter from the topmost surface until the overlayer/substrate interface at which the signal intensity of the element reaches 50 % of its value in the adjacent overlayer on a substrate.

In practical cases using a multilayered system, the total thickness of a few certain periods of layers from the topmost surface and the time required to sputter from the topmost surface until the corresponding interface between the final layer of the certain periods of layers and the layer below them may be used as  $z_{tot}$  and  $t_{tot}$ , respectively, to obtain  $\bar{z}_{av}$ .

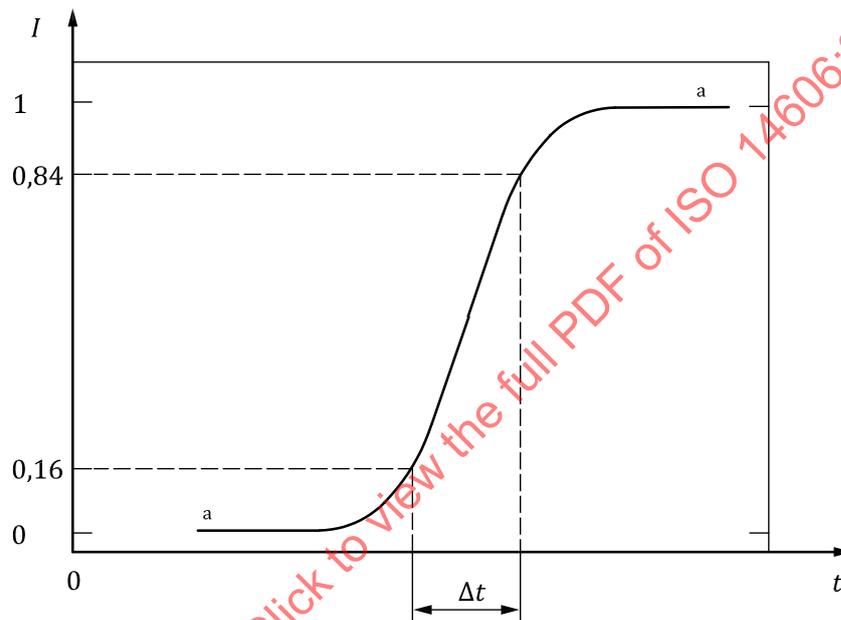
### 6.3 Depth resolution $\Delta z$

Depth resolution,  $\Delta z$ , is given by [Formula \(2\)](#):

$$\Delta z = \bar{z}_{av} \times \Delta t \quad (2)$$

where  $\Delta t$  is the sputtering time interval in which the signal intensities change from 16 % to 84 % (or 84 % to 16 %) of the intensity corresponding to 100 % of each of the overlayer and the substrate of a single-layered system or each of the adjacent layers of a multilayer system.

The measurement of  $\Delta t$  is only applicable where plateau regions have been obtained for both maximum and minimum intensities (see [Figure 1](#)).



#### Key

- $I$  signal intensity
- $t$  sputtering time
- $a$  Plateau region.

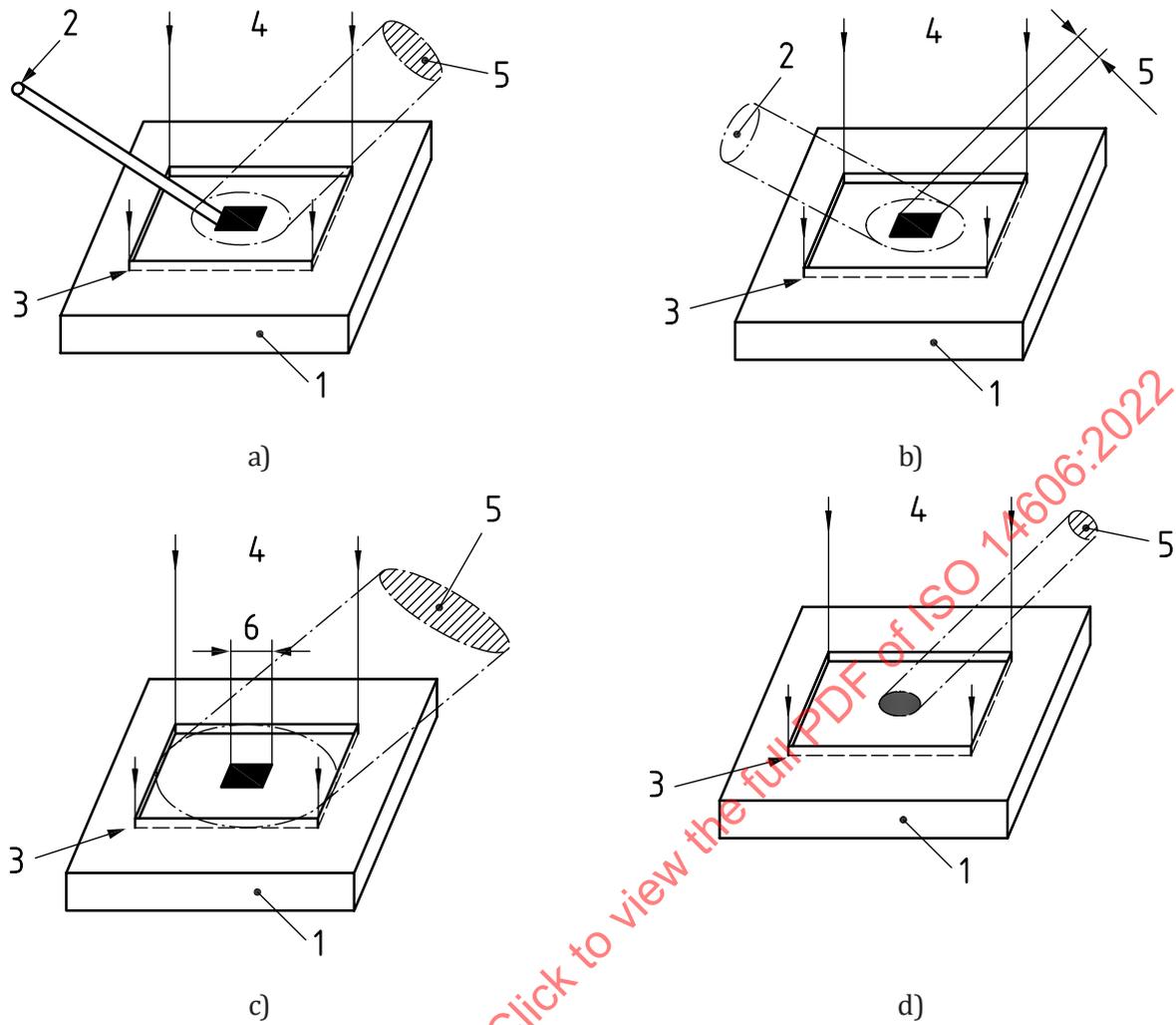
**Figure 1 — Diagram of the measurement of  $\Delta t$  at an ideally sharp interface in a sputter depth profile**

## 7 Procedures for optimization of parameter settings

### 7.1 Alignment of sputtered area with a smaller analysis area

#### 7.1.1 General

The centre of a sputtered area shall be aligned with a smaller analysis area using an appropriate method. The ratio of the analysis area to the sputtered area should be chosen to be as small as possible so as to reduce effects resulting in the depth resolution due to the proximity to the crater edge<sup>[15]</sup>. A number of different situations exist, as shown in [Figure 2](#).



**Key**

- 1 sample
- 2 probe
- 3 sputtered area
- 4 direction of ions
- 5 spectrometer analysis area
- 6 electronic gate

NOTE In some cases, a larger area at the sample surface is used in alignment. For each example, the smaller area is given as a black shaded area in this figure and by “X” in Table 3 whereas the larger area is given by “Y” in Table 3.

**Figure 2 — Methods for aligning the sputtered area with a smaller analysis area**

**Table 3 — Description of sputtering alignment methods**

Figure	Smaller area X	Larger area Y	Example
2 a)	Focused probe beam	Spectrometer analysis area	AES, or XPS with focused monochromator, or time-of-flight SIMS
2 b)	Spectrometer analysis area	Broad probe beam	XPS without monochromator

Table 3 (continued)

Figure	Smaller area X	Larger area Y	Example
2 c)	Electronic gate	Spectrometer analysis area	Dynamic SIMS with electronic gate
2 d)	Optical aperture	Not appropriate	Dynamic SIMS with optical aperture

### 7.1.2 AES

**7.1.2.1** The centre of the sputtered area shall be aligned with the analysis area as defined by the focused electron beam, which may be rastered as shown in [Figure 2 a\)](#). Such correct alignment shall be accomplished according to the procedures provided by respective manufacturer of apparatuses.

**7.1.2.2** The correct alignment may be checked by a method provided by the instrument manufacturer. The correct alignment may also be checked by either post-profile crater observation or by measurement to ensure that the sputtered area lies centred over the analysis area [see [Figure 2 a\)](#)]<sup>[13]</sup><sup>[14]</sup>.

**7.1.2.3** If an instrument is available with SEM imaging, post-profile crater observation may be performed using a monitor display<sup>[13]</sup>.

NOTE If necessary, alignment and post-profile crater observation is repeated to minimize the depth resolution  $\Delta z$ .

### 7.1.3 XPS with a small probe (e.g. monochromator)

**7.1.3.1** The centre of the sputtered area shall be aligned with the analysis area as defined by the focused X-ray beam [(see [Figure 2 a\)](#)]. Such correct alignment shall be accomplished according to a procedure provided by the respective manufacturer of the apparatus.

**7.1.3.2** The correct alignment may be checked by a method provided by the instrument manufacturer.

### 7.1.4 XPS with a large area source (e.g. without monochromator)

**7.1.4.1** The centre of the sputtered area shall be aligned with the analysis area as defined by the electron spectrometer [see [Figure 2 b\)](#)]. Such the correct alignment shall be realized according to a procedure provided by respective manufacturer of apparatuses.

**7.1.4.2** The correct alignment may be checked by a method provided by the instrument manufacturer.

### 7.1.5 SIMS

**7.1.5.1** If an electronic gate is used in dynamic SIMS, the centre of the sputtered area shall be aligned with the analysis area as defined by the electronic gate [(see [Figure 2 c\)](#)]<sup>[15]</sup>.

**7.1.5.2** If an ion optical aperture is used in dynamic SIMS, the centre of the sputtered area shall be aligned with the analysis area as defined by the ion optical aperture [see [Figure 2 d\)](#)], and this is carried out in a test area as near as possible to the analysis area. For some instruments with an optical aperture and a scanning ion image display, alignment may be carried out during the profile.

**7.1.5.3** If different ion beams are used for sputtering and analysis in a time-of-flight SIMS instrument, the centre of the sputtered area shall be aligned with the analysis area as defined by the focused ion beam [see [Figure 2 a\)](#)].

7.1.5.4 If the post-profile realignment of each column of pixels is used to provide an image depth profile alignment shall not be necessary.

## 7.2 Optimization of parameter settings

7.2.1 If a sample consisting of a single-layered or multilayered structure on a substrate is to be measured, then a similar reference material system should be used for optimization. Some single-layered and multilayered reference materials are listed in [Annex B](#) and [Annex C](#).

7.2.2 See [Table 1](#) and [Table 2](#) for probing and sputtering parameters which shall be optimized in accordance with [7.2.3](#) to [7.2.5](#).

7.2.3 Perform the measurements of sputter depth profiles on a single-layered or multilayered system using the appropriate parameter settings, in particular, those important for depth resolution (see [Table 1](#) and [Table 2](#)).

7.2.4 Determine the depth resolution  $\Delta z$  from the data sets of depth profiles using [Formulae \(1\)](#) and [\(2\)](#) (see [Clause 6](#)).

7.2.5 Choose the parameter settings so as to obtain the optimum depth resolution  $\Delta z$ .

NOTE 1 Useful information for a number of important parameters is given in [Annex A](#).

NOTE 2 Useful information obtained using a multilayered system is listed in [Annex D](#).

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## Annex A (informative)

### Factors influencing the depth resolution

#### A.1 General

Usually, probing and sputtering parameters are taken into account in order to optimize parameter settings for enhancing the depth resolution  $\Delta z$ <sup>[11][16][17][18][19][20][21][22]</sup>.

#### A.2 Sputtering parameters

##### A.2.1 Ion species

The ion species should be considered in order to reduce the sputter-induced roughness, or cone formation, or extent of atomic mixing<sup>[23][24]</sup>. Typical ion species used for sputtering are inert gas ions (e.g. Ar<sup>+</sup>, Kr<sup>+</sup>, Xe<sup>+</sup>), reactive gas ions (e.g. O<sup>-</sup>, O<sub>2</sub><sup>+</sup>) or metal ions (e.g. Ga<sup>+</sup>, Cs<sup>+</sup>).

##### A.2.2 Ion energy

In general, a lower ion energy is preferable in order to reduce the atomic mixing effect and/or the sputter-induced roughness<sup>[25][26][27]</sup>. But this should be balanced against the reduced sputtering yield and lower sputtering rates where contamination from adsorption of residual gas species may be a problem.

##### A.2.3 Angle of incidence

Often, a high angle of incidence (i.e. glancing incidence) may be used to reduce the atomic mixing effect and/or the sputter-induced roughness<sup>[26][28][29]</sup>. In some cases, a higher angle of incidence may lead to increased sputter-induced roughness, for example, when using reactive primary ions and stationary sample stage<sup>[29][30][31][32]</sup>.

##### A.2.4 Sample stage

The rotational mode is generally preferable, in particular, for polycrystalline metallic materials so as to reduce sputter-induced roughness<sup>[28][29][33][34][35][36]</sup>. The speed of rotation is generally chosen to be greater than a critical value so as to ensure an improvement of  $\Delta z$  when compared to stationary conditions<sup>[29]</sup>.

#### A.3 Measurement parameters

##### A.3.1 Kinetic energy ( $E_k$ ) of signal used in AES and XPS

A signal with a lower kinetic energy is preferable in order to reduce the information depth<sup>[37][38]</sup> ( $E_k > 30$  eV).

NOTE “Information depth” is the maximum depth, normal to the surface, from which useful information is obtained. The information depth can be identified with the sample thickness from which a specified percentage (e.g. 95 % or 99 %) of the detected signal originates. In addition, the information depth can be determined from a measured, calculated or estimated emission-depth distribution function for the signal of interest.

### A.3.2 Angle of emission

More grazing emission reduces the sampling depth in AES and XPS as  $\lambda \cos\theta$  and so improves the measured depth resolution, where  $\lambda$  is the attenuation length,  $\theta$  is the angle between surface normal and optical axis of the analyser.

NOTE 1 The depth resolution  $\Delta z$  can be different for the same element if different peaks with different attenuation lengths (ALs) are used. The depth resolution clearly depends on the AL, or more generally speaking on the information depth (AL in AES and XPS, secondary ion emission depth in SIMS).

NOTE 2 Usually molecular ions such as dimers (i.e.  $A_2^+$ ) or trimers (i.e.  $A_3^+$ ) used in SIMS secondary ion signals have nonlinear responses of intensity with concentration and so give artificially low values of depth resolution.

## A.4 Experimental considerations

A.4.1 The sputtering rate of some polycrystalline films has been shown to be nonlinear due to topography development when sputtering films are not rotated<sup>[39]</sup>.

A.4.2 The sputtering rate can be affected in AES by electron irradiation during the sputtering process. The electron beam may be unfocused to avoid this effect which can double the sputtering rate (e.g. on  $SiO_2$  thin films)<sup>[14]</sup>. Similar effects have been reported in XPS where focused X-ray sources can affect sputtering yields.

A.4.3 Ion irradiation produces Auger electrons at energies up to 100 eV. If the electron spectrum is acquired with the ion beam turned on, care should be taken to minimize the presence on the resulting spectrum<sup>[8][9]</sup>. Modulation techniques, such as beam brightness modulation, can minimize this effect by excluding Auger electrons present in the ion beam (giving a DC signal) from the phase-modulated Auger electrons present in the electron-beam blanking.

A.4.4 The roughness of the surface being sputtered affects the depth resolution. For polycrystalline samples the surface roughness increases during ion sputtering. This can be minimized or eliminated by using multiple ion sources on the same instrument<sup>[40]</sup> and by rotating the sample under ion beam irradiation<sup>[28][33][35][39][40]</sup>.

A.4.5 The use of oxygen flooding of the sample surface is sometimes used in SIMS analysis (with an oxygen primary ion beam) to reduce transient ion-yield effects on interfaces. The oxygen-flooding technique may also be used to reduce sputter-induced topography.

A.4.6 Sample contamination should be a consideration when depth profiling because the sample vacuum environment may have an influence on the depth resolution. Accidental contamination of the sample surface can be checked by measuring the residual gases in the vacuum chamber using a residual gas analyser.

A.4.7 Another kind of check can be made for sample contamination with respect to time for AES and XPS techniques. It involves measuring sample contamination on a reactive material surface, such as that from pure titanium. A contamination appearance profile (CAP) measurement is made whereby the sample is first ion-sputter-cleaned until there is no detectable carbon, oxygen, or other surface contaminants. Next, a CAP measurement is made for the time it takes to detect a significant peak of oxygen, carbon or another element of interest. This procedure can be automated using a computer-controlled system whereby a depth profile is collected with the ion beam turned off or by either turning off the ion gun high voltage or by closing the sputtering gas supply. The resulting profile shows the contamination signal versus time relationship. It is noted that this technique is not applicable for SIMS.

A.4.8 If the analysis area is too small, the measured resolution can be decreased. For example, in AES, a small analysis area gives rise to sputter enhancement on the area irradiated by the electron beam and leads to poor depth resolution. This effect is best known to occur with  $SiO_2$  but also occurs with many

compounds. In SIMS, as the measured signal intensity depends on the analysis area, sputtering rate and integration time, the experimental conditions need to be chosen with care to ensure that the signal is sufficiently high so as to obtain a good signal-to-noise ratio but at the same time having sufficient data points obtained for the interface regions so as to allow the depth resolution to be measured.

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## Annex B (informative)

### Typical single-layered systems as reference materials

Certified reference materials from national laboratories and those traceable to them are useful in optimizing the depth resolution and calibrating the sputtering rate for sputter depth profiling in surface-dependent materials.

Typical single-layered systems used as reference materials for sputter depth profiling are given in [Table B.1](#).

NOTE When using the reference sample of Ta<sub>2</sub>O<sub>5</sub>/Ta to measure depth profile by microscopic area AES, they can be affected by irregularity due to grain boundaries that exist in the polycrystalline Ta substrate.

**Table B.1 — Typical single-layered systems as reference materials**

Reference material	Thickness nm	Fabrication method	Crystalline state	Reference
Ta <sub>2</sub> O <sub>5</sub> /Ta	30, 100	Anodic oxidation	Amorphous	BCR-261T <sup>a</sup>

<sup>a</sup> Ta<sub>2</sub>O<sub>5</sub> films on tantalum at thicknesses of 30 nm and 100 nm<sup>[17][18]</sup> are available as BCR-261T from the EC Institute for Reference Materials and Measurements. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

## Annex C (informative)

### Typical multilayered systems used as reference materials

Typical multilayered systems used as reference materials for sputter depth profiling are given in [Table C.1](#).

**Table C.1 — Typical multilayered systems used as reference materials**

Reference material	Thickness nm	Total number of layers	Fabrication method	Crystalline state	Reference
Ni/Cr multilayered	63/53	9	Sputter deposition	Polycrystalline	NIST SRM 2135c, d, e <sup>a</sup>
SiO <sub>2</sub> /Si multilayered	20/20	5	Sputter deposition	Amorphous SiO <sub>2</sub> /polycrystalline Si	NMIJ CRM 5202-a <sup>b</sup>
AlAs/GaAs superlattice	10/10	6	MBE	Single crystal	NMIJ CRM 5203-a <sup>b</sup>

**Key**  
 MBE molecular beam epitaxy

<sup>a</sup> Standard Reference Material (SRM) 2135c, d, e<sup>[19][35][41]</sup> is available from the National Institute of Standards and Technology (NIST). This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

<sup>b</sup> SiO<sub>2</sub>/Si multilayered and AlAs/GaAs superlattices<sup>[22][24][25][26]</sup> are available from the National Metrology Institute of Japan (NMIJ). This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.