
**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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Printed in Switzerland

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 14606 was prepared by Technical Committee ISO/TC 201, *Surface chemical analysis*, Subcommittee SC 4, *Depth profiling*.

Annexes A, B, C and D of this International Standard are for information only.

Introduction

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

The specific applications of this International Standard 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 (for example 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 International Standard 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 International Standard is given in the Bibliography^{[1][2][3][4][5]}.

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

1 Scope

This International Standard gives guidance 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 International Standard is not intended to cover the use of special multilayered systems such as delta doped layers.

2 Terms and definitions

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

NOTE The terms used in this International Standard follow basically ASTM E 673-97^[6]. The definitions of the terms used are to be modified to conform to those being developed by ISO/TC 201/SC 1, *Terminology*.

2.1

analysis area

two-dimensional region of a sample surface measured in the plane of that surface from which the full signal or a specified percentage of that signal is detected

2.2

angle of incidence

angle between the incident beam and the local or average surface normal

2.3

crater edge effect

signals from the crater edge which often originate from depths shallower than the central region of the crater formed in depth profiling

2.4

depth resolution

depth range over which a signal intensity increases or decreases by a specified amount when profiling an ideally sharp interface between two media

NOTE By convention, a measure of the depth resolution is often taken to be the distance over which the signal intensity changes from 16 % to 84 % of the full change between the respective plateau values of the two media^[7].

2.5

gated area

defined area within a larger area from which the signal may be obtained

2.6

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

2.7

plateau region

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

2.8

signal intensity

strength of a signal at the spectrometer output or after some defined data processing

NOTE Examples of signal intensity are the height of the peak above the background or the peak-to-peak heights in AES or the peak areas in XPS.

2.9

sputter depth profile

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

2.10

sputtering rate

quotient of amount of sample material removed as a result of particle bombardment by time

NOTE The rate may be measured as a velocity, a mass per unit area per unit time, or some other measure of quantity per unit time.

3 Symbols and abbreviated terms

Δz 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

4 Setting parameters for sputter depth profiling

4.1 General

For the purposes of this International Standard, 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 International Standard.

Table 1 — Typical probing parameters for sputter depth profiling

	AES	XPS	SIMS
Probing species	Electrons	Photons: Mg K α , Al K α	Primary ions: Cs ⁺ , O ⁻ , O ₂ ⁺ , Ga ⁺
Energy	1 keV to 25 keV	1,253 keV, 1,486 keV	0,1 keV to 25 keV
Current or power	1 nA to 10 ³ nA (Faraday cup)	1 W to 10 ⁴ W (Source power)	1 nA to 10 ⁴ nA (Faraday cup)
Angle of incidence	0° ≤ θ < 90°	0° ≤ θ < 90°	0° ≤ θ < 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	0 keV to 3 keV	0 keV to 1,5 keV	0 keV to 0,125 keV
Angle of emission	0° ≤ θ ≤ 90°	0° ≤ θ ≤ 90°	0° ≤ θ ≤ 90°
Analysis area	10 ⁻⁸ mm ² to 10 ⁻² mm ²	10 ⁻⁴ mm ² to 10 mm ²	10 ⁻⁶ mm ² to 10 ⁻² mm ²

Table 2 — Typical sputtering parameters for sputter depth profiling

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

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

4.2 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^[8].

- Probing parameters** (important for analysis): Electron energy, electron beam current, angle of incidence, analysis area (i.e. beam diameter or raster area).
- Sputtering parameters** (important for depth resolution): Ion species, ion energy, ion beam current^[9], angle of incidence, sputtered or raster area. Sample stage is in a stationary or rotational mode.
- Measurement parameters:**
 - Kinetic energies of Auger electrons from both overlayer and substrate elements, or from elements A and B (important for both analysis and depth resolution).
 - Direct mode, $N(E)$ or $EN(E)$, or differential mode, $dN(E)/dE$ or $dEN(E)/dE$ (important for analysis).¹⁾

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 analyzer (for example, the cylindrical mirror analyzer), Auger electron intensities are presented in $EN(E)$ and $dEN(E)/dE$ formats, in which the spectrum approximates E times the true spectrum.

NOTE Usually 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. The problem of ion-induced Auger electrons seems only significant for Auger electron peaks below 100 eV ^{[10][11]}.

4.3 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.

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

4.4 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 may be 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.

5 Depth resolution at an ideally sharp interface in sputter depth profiles

5.1 Measurement of depth resolution

For the purposes of this International Standard, the measurement of the depth resolution Δz of sputter depth profiles of a single layered or an A/B/A/B/... multilayered system is as follows ^{[7][12][13]}.

NOTE 1 The definition of depth resolution Δz 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.

NOTE 2 For SIMS, where matrix effects are significantly different between the two layers, Δz may still be used for optimization but may not relate closely to the real depth resolution of the underlying chemical composition.

5.2 Average sputtering rate $\bar{z}_{av}$

$\bar{z}_{av}$ is given by the following expression:

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

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.

5.3 Depth resolution Δz

Δz is given by the following expression:

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

where Δt 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 Δt is only applicable where plateau regions have been obtained for both maximum and minimum intensities (see Figure 1).

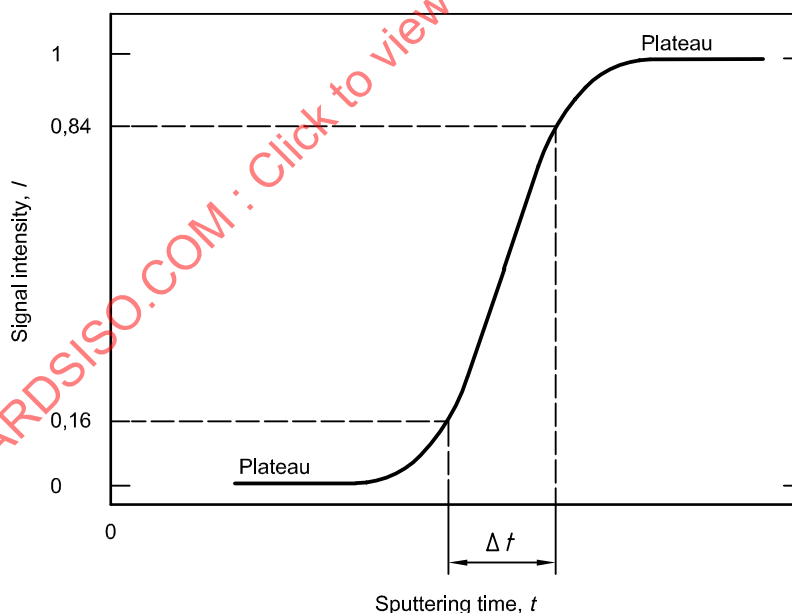


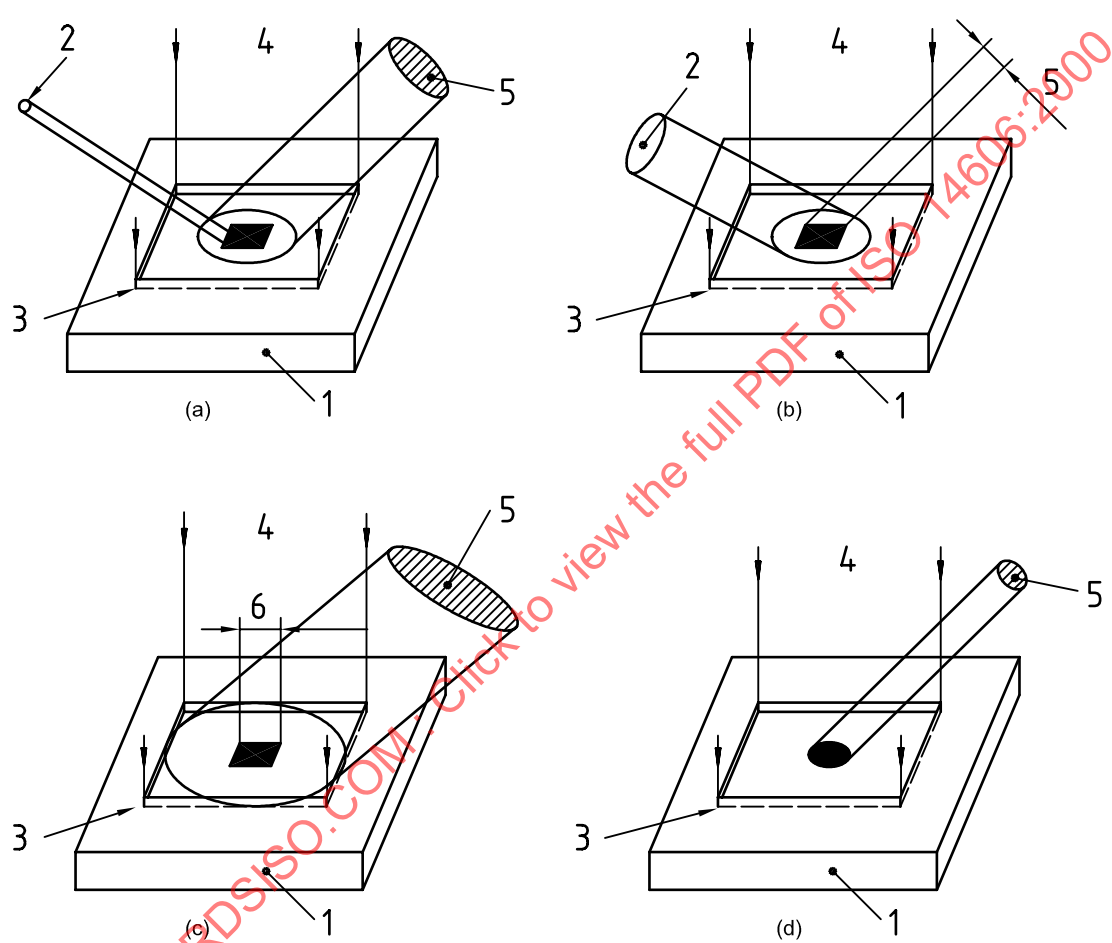
Figure 1 — Diagram of the measurement of Δt at an ideally sharp interface in a sputter depth profile

6 Procedures for optimization of parameter settings

6.1 Alignment of sputtered area with a smaller analysis area

6.1.1 General

The centre of a sputtered area shall be aligned with a smaller analysis area using an appropriate method. A number of different situations exist, as discussed below (see Figure 2 and the note).



- Key**
- 1 Sample
 - 2 Probe
 - 3 Sputtered area
 - 4 Direction of ions
 - 5 Spectrometer analysis area
 - 6 Electronic gate

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

NOTE In some cases a third area, a broader area at the sample surface is used in alignment. For each example, the smaller area is given as a black shaded area in Figure 2 and by "X" in Table 3 whereas the third area is given by "Y" in Table 3.

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
2 c)	Electronic gate	Spectrometer analysis area	Dynamic SIMS with electronic gate
2 d)	Optical aperture	Not appropriate	Dynamic SIMS with optical aperture

6.1.2 AES

6.1.2.1 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).

6.1.2.2 Correct alignment may 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)^[14]]. If necessary, alignment and post-profile crater observation may be repeated.

6.1.2.3 If an instrument is available with SEM imaging, post-profile crater observation may be performed using a monitor display^[14].

6.1.3 XPS with a small probe (for example monochromator)

6.1.3.1 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)].

6.1.3.2 Correct alignment may 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)^[14]]. If necessary, alignment and post-profile crater observation may be repeated.

6.1.4 XPS with a large area source (for example without monochromator)

6.1.4.1 The centre of the sputtered area shall be aligned with the analysis area as defined by the electron spectrometer [see Figure 2 b)].

6.1.4.2 Correct alignment may 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 b)^[14]]. If necessary, alignment and post-profile crater observation may be repeated.

6.1.5 SIMS

6.1.5.1 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)^[15]].

6.1.5.2 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.

6.1.5.3 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)].

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

6.2 Optimization of parameter settings

6.2.1 See Table 1 and Table 2 for probing and sputtering parameters which shall be optimized as follows.

6.2.2 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).

6.2.3 Determine the depth resolution Δz from the data sets of depth profiles using equations (1) and (2) (see clause 5).

6.2.4 Choose the parameter settings so as to obtain the minimum depth resolution Δz .

NOTE 1 Useful information for a number of important parameters is given in annex A.

NOTE 2 In general, the ratio of the analysis area to the sputtered area is 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^[15].

NOTE 3 If the analysis area is too small, the measured resolution may 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 SiO₂ 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.

NOTE 4 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.

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 Δz ^{[12][16][17][18][19][20][21][22]}.

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^{[23][24]}. Typical ion species used for sputtering are inert gas ions (e.g. Ar⁺, Kr⁺, Xe⁺), reactive gas ions (e.g. O⁻, O₂⁺) or metal ions (e.g. Ga⁺, Cs⁺).

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^{[25][26][27]}. 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^{[26][28][29]}. 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^{[29][30][31][32]}.

A.2.4 Sample stage

The rotational mode is generally preferable, in particular, for polycrystalline metallic materials so as to reduce sputter-induced roughness^{[28][29][33][34][35][36]}. The speed of rotation is generally chosen to be greater than a critical value so as to ensure an improvement of Δz when compared to stationary conditions^[29].

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^{[37][38]} ($E_k > 30$ 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 may 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 λ is the attenuation length, θ is the angle between surface normal and optical axis of the analyser.

NOTE Usually molecular ions such as dimers (i.e. A_2^+) or trimers (i.e. A_3^+) used in SIMS secondary ion signals have non-linear 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 non-linear due to topography development when sputtering films are not rotated^[39].

A.4.2 The sputtering rate may be affected in AES by electron irradiation during the sputtering process. The electron beam can be unfocused to avoid this effect which can double the sputtering rate (e.g. on SiO_2 thin films). 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^{[10][11]}. 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^[40] and by rotating the sample under ion beam irradiation^{[28][33][35][39][40]}.

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 analyzer.

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.

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.

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

Reference material	Thickness nm	Fabrication method	Crystalline state	Reference
Ta ₂ O ₅ /Ta	30, 100	Anodic oxidation	Amorphous	BCR No. 261
SiO ₂ /Si	10 to 200	Thermal oxidation	Amorphous	NIST SRM 2531-2536

NOTE 1 Ta₂O₅ films on tantalum at thicknesses of 30 nm and 100 nm^{[17][18]} are available as BCR No. 261 from the EC Institute for Reference Materials and Measurements, Geel, Belgium.

NOTE 2 SiO₂ films on silicon at thicknesses from 10 nm to 200 nm are available as SRM 2531-2536 from the National Institute of Standards and Technology (NIST), Gaithersburg, MD 20899, USA.

NOTE 3 Reference materials are also available from the following commercial suppliers:

- JEOL Datum Ltd., Akishima, Tokyo 196-0022, Japan
- ULVAC-PHI, Inc., 370 Enzo, Chigasaki, Kanagawa 253-0084, Japan
- Geller MicroAnalytical Laboratory, 426E Boston Street, Topsfield, MA 01983-1216, USA
- Physical Electronics, 6509 Flying Cloud Drive, Eden Prairie, MN 55344, USA
- Surface/Interface Laboratory, Mountain View, CA 94043, USA