## BS ISO 14606:2015



## **BSI Standards Publication**

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



BS ISO 14606:2015 BRITISH STANDARD

#### National foreword

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A list of organizations represented on this committee can be obtained on request to its secretary.

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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



BS ISO 14606:2015 **ISO 14606:2015(E)** 



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#### Foreword

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

This second edition cancels and replaces the first edition (ISO 14606:2000), of which it constitutes a minor revision to update the content of <a href="Table C.1">Table C.1</a>

#### 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]

## 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 document, 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 1 to entry: 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. [Z]

#### 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

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#### 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 1 to entry: 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 1 to entry: 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

$\Delta z$	depth resolution
I	signal intensity
$\overline{Z}$	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 <u>Table 1</u> and <u>Table 2</u>. 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 — Typica	l probing param	eters for sputte	er depth profiling

	AES	XPS	SIMS
Probing species	Electrons	Photons:	Primary ions: Cs+, O-, O <sub>2</sub> +,
		Mg Kα, Al Kα	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 <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° ≤ 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 <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+, Kr+, Xe+, O-, O <sub>2</sub> +, 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 <sup>4</sup> nA	Faraday cup	
Angle of incidence	0° ≤ θ < 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 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. [3]

- 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 [9], 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).<sup>1)</sup>

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]

<sup>1)</sup> 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 (for example, the cylindrical mirror analyser), Auger electron intensities are presented in EN(E) and dEN(E)/dE formats, in which the spectrum approximates E times the true spectrum.

#### 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  $\Delta z$  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  $\Delta z$  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.

NOTE 2 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.

#### 5.2 Average sputtering rate

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

$$\overline{z}_{\rm av} = z_{\rm tot}/t_{\rm tot} \tag{1}$$

where

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

 $t_{\rm tot}$  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** $\Delta z$

 $\Delta z$  is given by the following expression:

$$\Delta z = \overline{z}_{av} \times \Delta t \tag{2}$$

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).

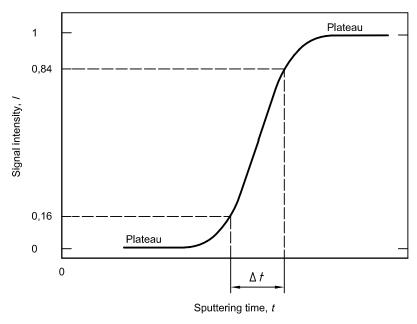


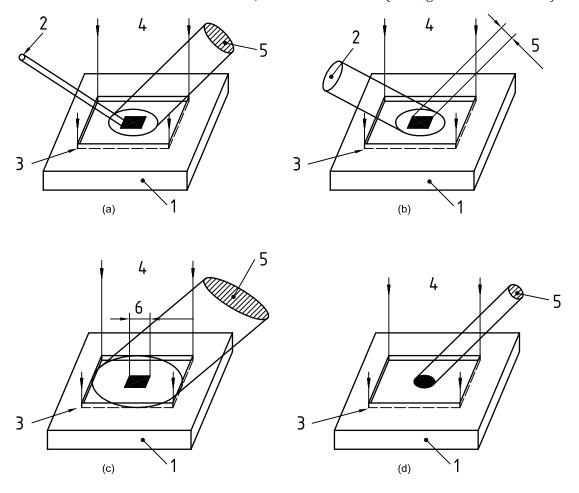
Figure 1 — Diagram of the measurement of  $\Delta t$  at an ideally sharp interface in a sputter depth profile

#### 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 <u>Figure 2</u> and by "X" in <u>Table 3</u> whereas the third area is given by "Y" in <u>Table 3</u>.

Table 3 — I	Description	of sputtering a	alignment methods
-------------	-------------	-----------------	-------------------

Figure	ure Smaller area Larger area		Example	
	X	Y		
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 2a).
- **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 2a)[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 2a)].
- **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 <u>Figure 2</u>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 2b)].
- **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 <u>Figure 2</u>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 2c)[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 2d)], 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 2a)].
- **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 <u>Table 1</u> and <u>Table 2</u> 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 <u>Table 1</u> and <u>Table 2</u>).
- **6.2.3** Determine the depth resolution  $\Delta z$  from the data sets of depth profiles using Formulae (1) and (2) (see <u>Clause 5</u>).
- **6.2.4** Choose the parameter settings so as to obtain the minimum depth resolution  $\Delta z$ .
- 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  $SiO_2$  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.
- 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 <u>Annex B</u> and <u>Annex C</u>.

#### 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  $\Delta z$ [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.  $0^-$ ,  $0_2^+$ ) or metal ions (e.g.  $6a^+$ ,  $6a^+$ ).

#### 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  $\Delta z$  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[ $\frac{37}{38}$ ] ( $E_k > 30 \text{ eV}$ ).

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  $\lambda$  is the attenuation length,  $\theta$  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 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.[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][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 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.

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

NOTE 1  $Ta_2O_5$  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 SiO<sub>2</sub> 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:

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

## 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 num- ber of layers	Fabrication meth- od	Crystalline state	Reference
Ni/Cr multilay-	63/53	9	Sputter deposition	Polycrystalline	NIST SRM
ered	03/33		spaceer acposition	1 oryer ystamme	2135c, d, e
Ta <sub>2</sub> O <sub>5</sub> /Ta multi- layered	30/30	6	Sputter deposition	Amorphous Ta <sub>2</sub> O <sub>5</sub> / polycrystalline Ta	KRISS 03-04-101
SiO <sub>2</sub> /Si multilay- ered	20/20	5	Sputter deposition	Amorphous SiO <sub>2</sub> / polycrystalline Si	NMIJ CRM 5202-a
AlAs/GaAs super- lattice	10/10	6	MBE	Single crystal	NMIJ CRM 5203-a

NOTE 1 Abbreviated term: MBE molecular beam epitaxy.

NOTE 2 Standard Reference Material (SRM) 2135c, d, e[19][35][41] is available from the National Institute of Standards and Technology (NIST), 100 Bureau Drive, Gaithersburg, MD 20899, USA.

NOTE 3 KRISS 03-04-101 is available from the Korea Research Institute of Standards and Science (KRISS), Yusoung PO No. 102, Taejon 305-606, Republic of Korea. The report (KRISS-94-053-IR) was issued with the title "Development of chemical composition SRM for advanced industries."

NOTE 4 SiO<sub>2</sub>/Si multilayered and AlAs/GaAs superlattices[[22][24][25][26] are available from the National Metrology Institute of Japan (NMIJ), 3–9 Tsukuba Central, 1–1-1 Umezono, Tsukuba, Ibaraki 305–8563, Japan.

## Annex D

(informative)

## Uses of multilayered systems

#### D.1 Relative sputtering rate

The relative sputtering rates of the layers A and B of an A/B/A/B/... multilayered system given in Annex C can be determined accurately by using the ratio of known layer thicknesses and the sputtering times between 50 % intensity values under those setting parameters.[22][41]

#### D.2 Dependence of depth resolution

The dependence of depth resolution  $\Delta z$  for the signal of element A or B on the sputtered depth z can be evaluated by using the values of depth resolution  $\Delta z$  at individual interfaces of an A/B/A/B/... multilayered system[19][26][28][35][36]

NOTE 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). Furthermore, the depth resolution depends on the details of the elemental distribution in the atomic mixing zone (i.e. the "altered layer"). The same  $\Delta z$  for elements A and B may be expected in cases where the elemental distribution in the atomic mixing zone is homogeneous.

#### D.3 Drift of ion beam current

The drift of the ion beam current during sputtering can be checked for proper behaviour by using the known layer thicknesses of the periodically layered structure.[19]

## **Bibliography**

- [1] ISO Guide 30:2015, Reference materials Selected terms and definitions
- [2] ISO Guide 31:2000, Reference materials Contents of certificates and labels
- [3] ISO Guide 33:2015, Reference materials Good practice in using reference materials
- [4] ISO Guide 34:2009, General requirements for the competence of reference material producers
- [5] ISO Guide 35:2006, Reference materials General and statistical principles for certification
- [6] ASTM E 673-97, Standard Terminology Relating to Surface Analysis
- [7] ASTM E 1438-91 ( 1996), Standard Guide for Measuring Widths of Interfaces in Sputter Depth Profiling Using SIMS
- [8] ASTM E 1127-91 (1997), Standard Guide for Depth Profiling in Auger Electron Spectroscopy
- [9] ASTM E 684-95, Standard Practice for Approximate Determination of Current Density of Large-Diameter Ion Beams for Sputter Depth Profiling of Solid Surfaces
- [10] POWELL R.A. Perturbing Effects of Ion-exited Auger Emission during Auger-Sputter Profiling Studies. J. Vac. Sci. Technol. 1978, **15** pp. 125–127
- [11] ZALAR A., HOFMANN S., PANJAN P., KRASEVEC V. Auger Electron Spectroscopy Depth Profiling Studies on Stationary and Rotated Samples of a New Model Metal/Semiconductor Multilayer Structure. Thin Solid Films. 1992, **220** pp. 91–196
- [12] SEAH M.P., & HUNT C.P. The Depth Dependence of the Depth Resolution in Composition-Depth Profiling with Auger Electron Spectroscopy. Surf. Interface Anal. 1983, 5 pp. 33–37
- [13] HOFMANN S. Determination of Depth Resolution from Measured Sputtering Profiles of Multilayer Structures: Equations and Approximations. Surf. Interface Anal. 1986, 8 pp. 87–89
- [14] HUNT C.P., & SEAH M.P. Method for the Alignment of Samples and the Attainment of Ultrahigh-resolution Depth Profiles in Auger Electron Spectroscopy. Surf. Interface Anal. 1990, 15 pp. 254–258
- [15] WITTMAACK K. Raster Scanning Depth Profiling of Layer Structures. *Appl. Phys.*, **12**, 1977, pp. 149-156.
- [16] HOFMANN S. Depth Resolution in Sputter Profiling. Appl. Phys. (Berl.). 1977, 13 pp. 205–207
- [17] Hunt C.P., & Seah M.P. Characterization of a High Depth-Resolution Tantalum Pentoxide Sputter Profiling Reference Material. Surf. Interface Anal. 1983, **5** pp. 199–209
- [18] SEAH M.P., MATHIEU H.J., HUNT C.P. The Ultra-high Resolution Depth Profiling Reference Material Ta<sub>2</sub>O<sub>5</sub> Anodically Grown on Ta. Surf. Sci. 1984, **139** pp. 549–557
- [19] Fine J., Lindfors P.A., Gorman M.E., Gerlach R.L., Navinsek B., Mitchel D.F. Interface Depth Resolution of Auger Sputter Profiled Ni/Cr Interfaces: Dependence on Ion Bombardment Parameters. J. Vac. Sci. Technol. A. 1985, 3 pp. 1413–1417
- [20] HOFMANN S. Compositional Depth Profiling by Sputtering. *Progress in Surface* Science, 36, 1991, pp. 35-87.
- [21] BARNA A. and MENYHARD M. Auger Depth Profile Analysis of Deeply Buried Interfaces. Phys. Status Solidi. 1994, 145 pp. 263–274

- [22] HOFMANN S. Atomic Mixing, Surface Roughness and Information Depth in High-resolution AES Depth Profiling of a GaAs/AlAs Superlattice Structure. Surf. Interface Anal. 1994, **21** pp. 673–678
- [23] GRIES W.H. Radiation-induced Sample Modification in Surface Analysis: InP as an Extreme Example. Surf. Interface Anal. 1989, **14** pp. 611–618
- [24] KAJIWARA K., & SHIMIZU R. Resolution in Sputter Depth Profiling Assessed by AlAs/GaAs Superlattices. J. Vac. Sci. Technol. A. 1995, **13** pp. 1316–1320
- [25] KAJIWARA K., & KAWAI H. Analysis of AlGaAs/GaAs Superlattices by Means of Sputter-assisted AES, SEM and TEM. Surf. Interface Anal. 1990, **15** pp. 433–439
- [26] YOSHIHARA K., MOON D.W., FUJITA D., KIM K.J., KAJIWARA K. GaAs/AlAs Superlattice as a Proposed New Reference Material for Sputter Depth Profiling. Surf. Interface Anal. 1993, **20** pp. 1061–1066
- [27] MENYHARD M., BARNA A., BIERSACK J.P., JARRENDAHL K., SUNDGREN J.-E. Study of ion mixing during Auger depth profiling of Ge-Si multilayer system. II. Low ion energy (0,2-2 keV) range. J. Vac. Sci. Technol. A. 1995, **13** pp. 1999–2004
- [28] ZALAR A. Significance of Sample Rotation in Auger Electron Spectroscopy Sputter Depth Profiling of Thin Films. Thin Solid Films. 1990, **193/194** pp. 258–269
- [29] HOFMANN S., & ZALAR A. Depth Profiling with Sample Rotation: Capabilities and Limitations. Surf. Interface Anal. 1994, **21** pp. 304–309
- [30] KIM K.J., & Moon D.W. Significant Improvement in Depth Resolution of Cr/Ni Interfaces by Secondary Ion Mass Spectrometry Profiling under Normal O<sub>2</sub>+ Ion Bombardment. Appl. Phys. Lett. 1992, **60** pp. 1178–1180
- [31] Moon D.W., & Kim K.J. Surface Topography Development on Ion Beam Sputtered Surfaces: Role of Surface Inhomogeneity Induced by Ion Beam Bombardment. J. Vac. Sci. Technol. A. 1996, **14** pp. 2744–2756 [</jrn>]
- [32] JIANG Z.X., ALKEMADE P.F.A., ALGRA E., RADELAAR S. High Depth Resolution SIMS Analysis with Low-energy Grazing O<sub>2</sub>+ Beams. Surf. Interface Anal. 1997, **25** pp. 285–291
- [33] ZALAR A. Improved Depth Resolution by Sample Rotation during Auger Electron Spectroscopy Depth Profiling. Thin Solid Films. 1985, **124** pp. 223–230
- [34] MARTON D., & FINE J. On The Development of Increasing Surface Roughness during Ion Sputtering. Thin Solid Films. 1987, **151** pp. 433–439
- [35] HOFMANN S., ZALAR A., CIRLIN E.-H., VAJO J.J., MATHIEU H.J., PANJAN P. Interlaboratory Comparison of the Depth Resolution in Sputter Depth Profiling of Ni/Cr Multilayers with and without Sample Rotation using AES, XPS and SIMS. Surf. Interface Anal. 1993, **20** pp. 621–626
- [36] KAJIWARA K. Crystalline Effects on Depth Resolution in AES Depth Profiling. Surf. Interface Anal. 1994, **22** pp. 22–26
- [37] SEAH M.P., & DENCH W.A. Quantitative Electron Spectroscopy of Surfaces: A Standard Data Base for Electron Inelastic Mean Free Paths in Solids. Surf. Interface Anal. 1979, 1 pp. 2–11
- [38] TANUMA S., POWELL C.J., PENN D.R. Calculations of Electron Inelastic Mean Free Paths (IMFPs). Surf. Interface Anal. 1993, **20** pp. 77–89
- [39] Veisfeld N., & Geller J.D. Ion Sputtering Yield Measurements for Submicrometer Thin Films. J. Vac. Sci. Technol. A. 1988, 6 pp. 2077–2081
- [40] SYKES D.E., HALL D.D., THURSTANS R.E., WALLS J.M. Improved Sputter-Depth Profiles Using Two Ion Guns. Appl. Surf. Sci. 1980, **5** pp. 103–106

## BS ISO 14606:2015 **ISO 14606:2015(E)**

[41] Fine J., & Navinsek B. Characterization of NBS Standard Reference Material 2135 for Sputter Depth Profile Analysis. J. Vac. Sci. Technol. A. 1985, **3** pp. 1408–1412





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