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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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Contents

Page

Foreword .... **iv**

Introduction .... **v**

1 Scope **1**

2 Normative references **1**

3 Terms and definitions **1**

4 Symbols and abbreviated terms **1**

5 Setting parameters for sputter depth profiling **2**

5.1 General 2

5.2 Auger electron spectroscopy 3

5.3 X-ray photoelectron spectroscopy 3

5.4 Secondary ion mass spectrometry 4

6 Depth resolution at an ideally sharp interface in sputter depth profiles **4**

6.1 Measurement of depth resolution 4

6.2 Average sputtering rate $\bar{z}_{av}$ 4

6.3 Depth resolution Δz 5

7 Procedures for optimization of parameter settings **5**

7.1 Alignment of sputtered area with a smaller analysis area 5

7.1.1 General 5

7.1.2 AES 7

7.1.3 XPS with a small probe (e.g. monochromator) 7

7.1.4 XPS with a large area source (e.g. without monochromator) 7

7.1.5 SIMS 7

7.2 Optimization of parameter settings 8

Annex A (informative) **Factors influencing the depth resolution** **9**

Annex B (informative) **Typical single-layered systems as reference materials** **12**

Annex C (informative) **Typical multilayered systems used as reference materials** **13**

Annex D (informative) **Uses of multilayered systems** **14**

Bibliography **15**

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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).

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

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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.

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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[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 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*

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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

Δz depth resolution

I signal intensity

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$\bar{z}$ 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 Kα, Al Kα	Primary ions: Cs^+, O^-, O_2^+, Ga^+
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 10^3nA (Faraday cup)	1 W to 10^4 W (Source power)	1 nA to 10^4 nA (Faraday cup)
Angle of incidence	$0° \leq \theta < 90°$	$0° \leq \theta < 90°$	$0° \leq \theta < 90°$
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	$0° \leq \theta < 90°$	$0° \leq \theta < 90°$	$0° \leq \theta < 90°$
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	$0° \leq \theta < 90°$	
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 can also affect the depth resolution. For example, the gas pressure in the ion source can change during the course of the analysis.

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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[6].

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[7], 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, $\mathrm{d}N(E)/\mathrm{d}E$ or $\mathrm{d}EN(E)/\mathrm{d}E$ (important for analysis).

   NOTE 1 $N(E)$, $EN(E)$, $\mathrm{d}N(E)/\mathrm{dE}$ and $\mathrm{d}EN(E)/\mathrm{d}E$ 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 $\mathrm{d}N(E)/\mathrm{dE}$ 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 $\mathrm{d}EN(E)/\mathrm{d}E$ 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[8][9].

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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.

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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.

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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 Δz of sputter depth profiles of a single layered or an A/B/A/B/... multilayered system is as follows[10][11][12].

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.

NOTE 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.

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 $\bar{z}_{av}$.

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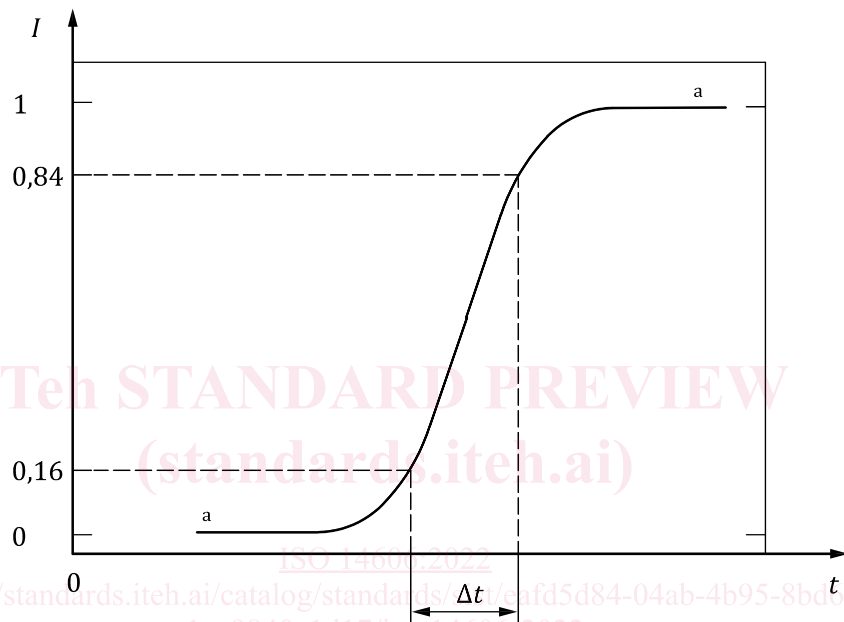
6.3 Depth resolution Δz

Depth resolution, Δz, is given by Formula (2):

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

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).



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Key

I signal intensity

t sputtering time

a Plateau region.

Figure 1 — Diagram of the measurement of Δt 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[15]. A number of different situations exist, as shown in Figure 2.

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