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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.

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.

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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 (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*.

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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

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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 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.

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3 Symbols and abbreviated terms

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Δz 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 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.

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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	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	$0° \leqslant \theta < 90°$	$0° \leqslant \theta < 90°$	$0° \leqslant \theta < 90°$
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	0 keV to 3 keV	0 keV to 1,5 keV	0 keV to 0,125 keV
Angle of emission	$0° \leqslant \theta \leqslant 90°$	$0° \leqslant \theta \leqslant 90°$	$0° \leqslant \theta \leqslant 90°$
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	$0° \leqslant \theta \leqslant 90°$	
Sputtered area	10^{-2} mm^2 to 10^2 mm^2	Raster scan of focused ion beam

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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.

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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].

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, d$N(E)$/dE or d$EN(E)$/dE (important for analysis).[1)]

1) $N(E)$, $EN(E)$, d$N(E)$/dE and d$EN(E)$/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 d$N(E)$/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 d$EN(E)$/dE formats, in which the spectrum approximates E times the true spectrum.

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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.

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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.

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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.

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

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

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

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:

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

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).

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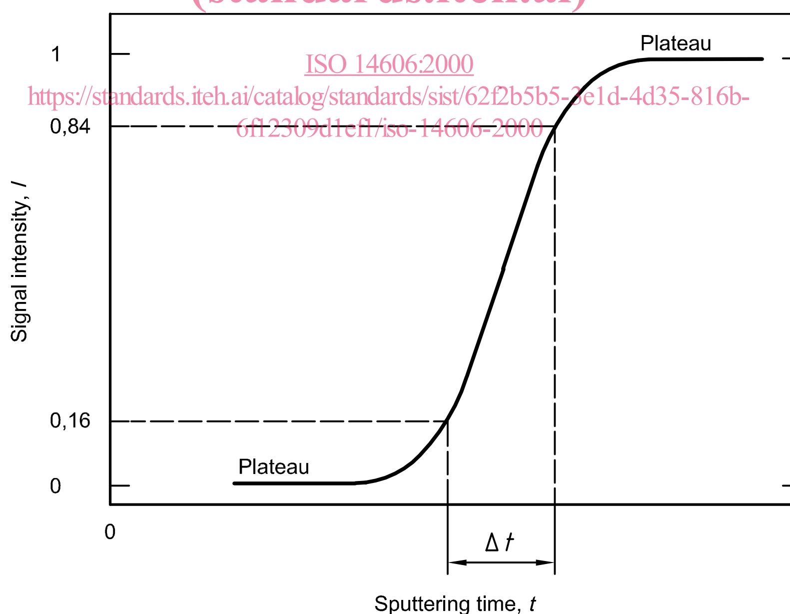

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Figure 1 — Diagram of the measurement of Δt at an ideally sharp interface in a sputter depth profile

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