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Surface chemical analysis — Depth profiling — Methods for ion beam alignment and the associated measurement of current or current density for depth profiling in AES and XPS

Analyse chimique des surfaces — Profilage d'épaisseur — Méthodes d'alignement du faisceau d'ions et la mesure associée de densité de courant ou de courant pour le profilage d'épaisseur en AES et XPS

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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.

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

This second edition cancels and replaces the first edition (ISO 16531:2013), which has been technically revised.

The main changes to the previous edition are as follows:

— Table 1, in reference to 5.4: a comment has been added to mention the use of automated alignment routine.

— 5.3.2, 5.3.3 and 5.5.4: some descriptions in notes have been changed to body text.

— minor editorial changes have been introduced for clarity.

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

In surface chemical analysis with Auger electron spectroscopy (AES) and X-ray photoelectron spectroscopy (XPS), ion sputtering has been extensively incorporated for surface cleaning and for the in-depth characterization of layered structures in many devices and materials. Currently, ultra-thin films of < 10 nm thickness are increasingly used in modern devices and so lower energy ions are becoming more important for depth profiling. For reproducible sputtering rates and for good depth resolution, it is important to align the ion beam at the optimal position. This optimization becomes increasingly critical as better and better depth resolutions are required. It is not necessary to conduct a beam alignment routinely but it is necessary to align the beam when instrument parameters change as a result of, for example, replacement of ion-gun filaments or an instrument bake-out. During the beam alignment, care must be taken not to sputter or otherwise affect samples for analysis on the sample holder. Instruments have different facilities to conduct alignment and six methods are described to ensure that most analysts can conduct at least one method. Two of these methods are also useful for measuring the ion beam current or the current density – important when measuring sputtering yields and for measuring sputtering rate consistency. With commercial instruments, the manufacturer may provide a method and equipment to conduct the beam alignment. If this is adequate, the methods described here might not be necessary but could help to validate that method.

ISO 14606 describes how the depth resolution may be measured from a layered sample and used to monitor whether the depth profiling is adequate, properly optimized or behaving as intended. That method, from the instrumental setup to the depth resolution evaluation via in-depth measurement, is, however, time-consuming and so the present, quicker procedure is provided to ensure that the ion beam is properly aligned as the first step to using ISO 14606 or for more routine checking.

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Surface chemical analysis — Depth profiling — Methods for ion beam alignment and the associated measurement of current or current density for depth profiling in AES and XPS

1 Scope

This document specifies methods for the alignment of the ion beam to ensure good depth resolution in sputter depth profiling and optimal cleaning of surfaces when using inert gas ions in Auger electron spectroscopy (AES) and X-ray photoelectron spectroscopy (XPS). These methods are of two types: one involves a Faraday cup to measure the ion current; the other involves imaging methods. The Faraday cup method also specifies the measurements of current density and current distributions in ion beams. The methods are applicable for ion guns with beams with a spot size less than or equal to 1 mm in diameter. The methods do not include depth resolution optimization.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

3 Terms, definitions, symbols and abbreviated terms

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

For the purposes of this document, the following symbols and abbreviated terms apply.

A	Area of Faraday cup aperture
A_R	Raster area at a known orientation to the ion beam
A_0	Area of ion beam raster in sample plane
AES	Auger electron spectroscopy
B	Ion beam broadening parameter equal to ratio I_{outer}/I_{inner}
C	Current
CD	Current density
D'	Ion dose rate at the sample
F'	Ion fluence rate delivered by ion gun

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FC	Faraday cup
FWHM	Full width at the half maximum
I	Rastered ion beam current measured in aperture of Faraday cup
I_{inner}	Ion current measured at inner electrode of co-axial cup
I_{outer}	Ion current measured at outer electrode of co-axial cup
I_S	Beam current as measured into dark region in the method specified in 5.5
I_0	Stationary, small diameter ion beam current measured in aperture of Faraday cup
J	Current density in ion beam measured per unit area of sample surface
OMI	Optical microscope image
SEI	Secondary electron image
SEM	Secondary electron microscope
X	Position of ion beam on x-axis set by ion gun controller
X_0	Aligned position on x-axis of ion beam set by ion gun controller
XPS	X-ray photoelectron spectroscopy
Y	Position of ion beam on y-axis set by ion gun controller
Y_0	Aligned position on y-axis of ion beam set by ion gun controller
θ	Angle of incidence of ion beam with respect to sample surface normal
θ_a	Angle of incidence of ion beam with respect to Faraday cup surface normal in usual position
θ_b	Minimized angle of incidence of ion beam with respect to Faraday cup surface normal

4 System requirements

4.1 General

This document is applicable to the focusable ion gun for sputtering with inert gases that is usually supplied with most AES and XPS instruments or available from after-market suppliers. The beam size or raster area of the ion beam shall be larger than and uniform over the analysis area. Six alternative methods of ion beam alignment are described that require the equipment to have provision for the measurement of the ion current or the detection of excited secondary signals or to have an optical microscope aligned at the analytical point. Depending on the equipment available, measurements of increasing sophistication may be made. The methods for measuring the ion beam current involve measurement by a circular-aperture Faraday cup, an elliptical-aperture Faraday cup or a co-axial electrode cup. The methods involving the excited secondary signals are categorized by ion/electron-induced secondary electrons or emitted photons that are detected with a secondary electron detector, an optical microscope or a phosphor screen.

To conduct the relevant surface analysis, the electron energy analyser, the analysis probe beam and the ion beam need to be focused and aligned correctly on the same analysis point or area to be analysed. To apply this document, the electron energy analyser and the analysis probe beam shall already be aligned to the optimum position using the manufacturer's or in-house documented procedure.

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