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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

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*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

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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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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

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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.

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

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## 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

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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

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$\theta$ Angle of incidence of ion beam with respect to sample surface normal

$\theta_a$ Angle of incidence of ion beam with respect to Faraday cup surface normal in usual position

$\theta_b$ 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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### 4.2 Limitations

This document is an important part of the setting up of depth profiling generally; nevertheless, depending on the material of the sample and its structure, there are several depth profiling procedures that may be applied to achieve the best depth resolution, not all of which are aided by this document. Some of the most popular procedures are:

a) ion bombardment of fixed position samples at angles of incidence in the range 0° to 60° with respect to the surface normal;

b) ion bombardment at grazing angles of incidence;

c) sample rotation during ion bombardment;

d) simultaneous ion bombardment applying two ion guns;

e) sample rotation and grazing angle of incidence for ion bombardment.

This document will assist in the use of procedure a). Some aspects could relate to the other procedures but further considerations might be required that are not necessarily included in this document.

## 5 Ion beam alignment methods

### 5.1 General

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This document describes six simple methods for ion beam alignment, all easily applied. These methods and a summary of their advantages are set out in Table 1. Also indicated are which methods are best for ion beam current or current density measurement.

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Each method has different advantages and requires different instrumental capabilities. The analyst needs to select the method based on requirements and equipment capabilities. Some issues depend on the raster size of the ion beam. A small raster is good, since little material is consumed or sputter deposited in the spectrometer. Additionally, for industrial samples, the material to be profiled may only occupy a small area. A very small raster is possible in AES where the electron beam is small and some users may deliberately use higher ion beam energies where ion beams tend to be better focused to obtain small sputtered areas with a faster sputtering rate. In these cases, and for systems with small-area XPS analysis, particular care needs to be taken with alignment. For broader ion beams, such as for some XPS instruments, the alignment accuracy may be more relaxed. If more than one method is suitable, tests with each will show which is most convenient for the sputtering conditions intended.

The effects of good and poor ion beam alignment in sputter depth profiling are illustrated in Annex A.

General precautions are given in 5.2. If analysts wish to align the beam and measure the ion beam current or current density, or change the ion beam energy, they can choose one of the two methods that use a Faraday cup. The alignment methods specified in 5.3 and 5.4 are those using Faraday cups with a circular aperture and an elliptical aperture, respectively. Annex B introduces a method using co-axial electrodes giving measurements proportional to the ion current or current density. If analysts wish to align the beam and not measure the ion current or current density, they can align the beam using images from secondary electrons or ions excited by ions or primary electrons, an optical image or by ion-induced luminescence, using the methods specified in 5.5, 5.6, 5.7 and 5.8, respectively. The method chosen depends on the capability and facility of the instrument used.

Clause 6 describes when to conduct the ion beam alignment.

### 5.2 Important issues to be considered prior to ion beam alignment

**5.2.1** For consistent, high-quality analysis, the analytical probe beam, whether stationary or rastered over an area, and the electron energy analyser axis shall be aligned at the analysis position. The

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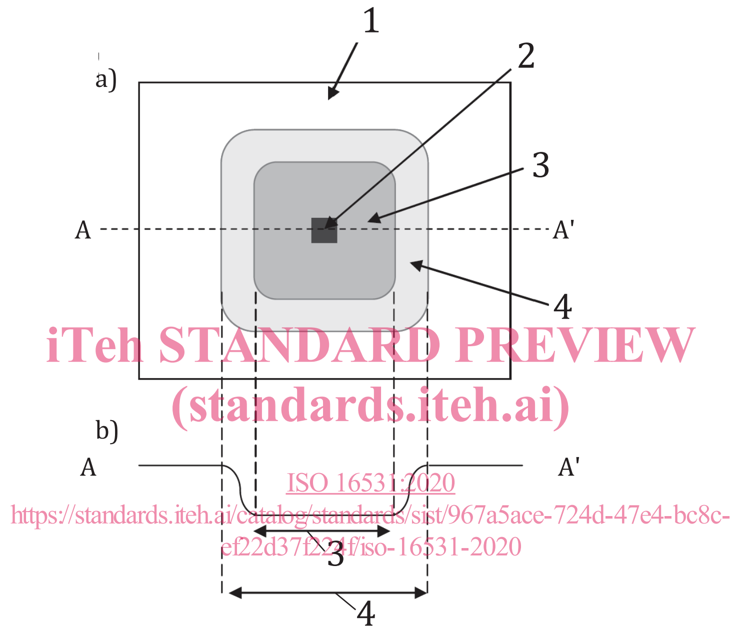
intersection of these two axes with the sample surface shall also define the centre for the sputtered area for sputter depth profiling.

**5.2.2** It is important that the analysis area be located in the central, uniform region of the ion beam irradiation area. This is shown in Figure 1[4]. It is useful to know the sputtering rate for the ion gun and sample as a function of sputtering parameters such as the ion beam energy, beam current, raster size and so on or their equivalent instrumental control settings in order to choose the best settings for the alignment. The two most important aspects for the analyst are to ensure that, through alignment of the ion beam, the analysis area coincides with the central uniform region of the ion beam irradiation area and also that an appropriate ion beam current density and raster size can be set. Ion beam currents and current densities may be measured using a Faraday cup using the methods specified in 5.3 and 5.4, as summarized in Table 1. Some design details and the accurate measurement of both electron and ion beam currents using Faraday cups are given in References [5] and [6].

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**Key**

1 specimen
2 analysis area
3 flat area
4 sputtered area
a) top view
b) cross-section view along line A–A′

SOURCE Urushihara N., Sanada N., Paul D., Suzuki M. Ion Beam Alignment Procedures using a Faraday Cup or a Silicon Dioxide Film on Silicon Substrate with Auger Electron Microscope. *J. Surf. Anal*. 2007, 14 (2) pp. 124–130[4], reproduced with the permission of the Surface Analysis Society of Japan.

**Figure 1 — Configuration of sputtered flat and analysis areas**

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**Table 1 — Detected signals when aligning ion beam — Summary of methods**

| Detected signal | Subclause: method | Feature | Minimum ion energy[a] eV | Measurement of current and current density | Equipment required |
|---|---|---|---|---|---|
| Ion current | 5.3: FC with circular aperture | Good for alignment. Gives the best measure of C and CD for quantitative sputtering rates but, for this, may require a FC that can be set normal to the ion beam at the analytical position. If the FC is in the sample plane, CD measurement may be poor at incidence angles greater than that for which the FC is designed, often ~45°. | ~50 | C: good<br>CD: good | FC may be orientated towards the ion gun or in the sample plane |
| | 5.4: FC with elliptical aperture | Good for alignment. This modification can allow greater angles to be used than those given by 5.3.<br>Some manufactures have such automated routines based on this standard incorporated as default. When using the automated routines, follow the manufacturer's instructions. | ~50 | C: good<br>CD: good | FC with elliptical aperture may be orientated towards the ion gun or in the sample plane |
| Excited secondary signal | 5.5: Ion-induced secondary electrons | Allows rastered ion beam to be aligned to within a fraction of the beam size and the raster size to be determined, but quantitative C and CD measurements are poor or shall be conducted separately. | ~50 | C: poor<br>CD: poor | Raster for ion beam |
| | 5.6: Ion-induced secondary emission imaging | Allows unrastered ion beam to be aligned to within a fraction of the beam size, but quantitative C and CD measurements are poor or shall be conducted separately. | ~50 | C: poor<br>CD: poor | Imaging for secondary electrons or ions |
| | 5.7: Ion spot image in SEI or OMI | Allows an unscanned ion beam to be focused and aligned in a system with i) an electron beam raster either during d) or after a) sputtering or ii) an optical microscope after a) sputtering. C and CD measurements shall be conducted separately. After sputtering, methods are very slow. | ~2 000 [i), d)]<br>~50 [i), a)]<br>~1 000 [ii), a)] | C: no<br>CD: poor | Raster for electron beam or optical microscope aligned at analytical point |
| | 5.8: Ion-induced luminescence | Allows an unscanned ion beam to be focused and aligned in a system, but the ion beam energy range available is limited. C and CD measurements shall be conducted separately. Most, if not all, phosphorescent materials are also electrical insulators and not stable under irradiation from either ions or electrons. | ~2 000 | C: no<br>CD: no | Phosphor screen for ion detection |

**Key**

C current

CD current density

FC Faraday cup

[a] The minimum energy is the energy for which the beam size is below ~1 mm and which is rarely below 50 eV.

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