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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

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

Introduction

In surface chemical analysis with AES (Auger electron spectroscopy) and XPS (X-ray photoelectron spectroscopy), 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, for example, from replacement of ion-gun filaments or from an instrument bake-out. During the beam alignment, care must be taken not to sputter or otherwise affect specimens for analysis on the sample holder. Instruments have different facilities to conduct alignment and seven 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 may not be necessary but may help to validate that method.

ISO 14606[1] 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.

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 International Standard 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 and X-ray photoelectron spectroscopy. 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 below ~1 mm in diameter. The methods do not include depth resolution optimization.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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 and the following symbols and abbreviated terms apply.

A	Area of Faraday cup aperture
A_0	Area of ion beam raster in sample plane
A_R	Raster area at a known orientation to the ion beam
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
FC	Faraday cup
FWHM	Full width at the half maximum
I	Rastered ion beam current measured in aperture of Faraday cup
I_0	Stationary, small diameter 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
J	Current density in ion beam measured per unit area of sample surface
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
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
AES	Auger electron spectroscopy
OMI	Optical microscope image
SEI	Secondary electron image
SEM	Secondary electron microscope
XPS	X-ray photoelectron spectroscopy

4 System requirements

4.1 General

This International Standard is applicable to the focusable ion gun for sputtering with inert gases that is usually supplied with most of 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. Seven alternative methods of ion beam alignment are described that require the equipment to have provision for the measurement of the ion current, or for detecting excited secondary signals, or 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, 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 International Standard, 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.

4.2 Limitations

This International Standard 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 International Standard. Some of the most popular procedures are

a) ion bombardment of fixed position samples at angles of incidence in the range of 0°–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, and

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

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

5 Ion beam alignment methods

5.1 General

This International Standard describes not all but seven simple methods for ion beam alignment, 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.

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; whereas 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, or 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 intersection of these two axes with the specimen 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].

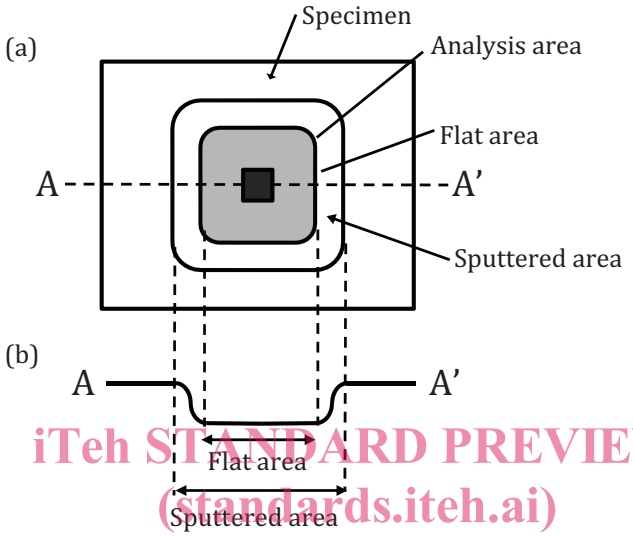

Figure 1 — Configuration of sputtered flat and analysis areas — (a) Top view (b) cross-section view along line A–A'[4]

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 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.	~50	C: good 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 must be conducted separately.	~50	C: poor 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 must be conducted separately.	~50	C: poor 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 must be conducted separately. After sputtering methods are very slow.	~2 000 [i), d)] ~50 [i), a)] ~1 000 [ii), a)]	C: no 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 must 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 CD: no	Phosphor screen for ion detection

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.

5.2.3 In general, the components on the sample stage used in the methods given in this International Standard do not all lie in a single plane, for essential mechanical reasons, and this may cause errors in