INTERNATIONAL STANDARD

ISO 18516

First edition
2006-11-01

Surface chemical analysis — Auger electron spectroscopy and X-ray photoelectron spectroscopy — Determination of lateral resolution

Analyse chimique des surfaces — Spectroscopie d'électrons Auger et spectroscopie de photoélectrons de rayons X — Détermination de la résolution latérale

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Reference number
ISO 18516:2006(E)

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Published in Switzerland

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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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 document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 18516 was prepared by Technical Committee ISO/TC 201, *Surface chemical analysis*, Subcommittee SC 5, *Auger electron spectroscopy*.

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Introduction

Auger electron spectroscopy (AES) and X-ray photoelectron spectroscopy (XPS) are surface-analytical techniques that are used to generate chemical maps and line scans of surfaces, and to provide spectroscopic analyses from defined areas. These techniques can have lateral resolutions as good as 10 nm for AES and can cover areas as large as many square centimetres in XPS. Different instruments generate images or define spectroscopic areas with different lateral resolutions, so inter-comparisons of image quality are poorly defined without clearly defined methods and terms with which to express the results. Different settings of an instrument may also change the lateral resolution. An analyst needs to have a suitable method to measure the lateral resolution of an instrument for any given settings. In this way, analysts can obtain the optimum lateral resolution from a given instrument, appropriate to the analytical requirements, in a consistent and clear way. The resolution actually achieved in subsequent analyses will approach these values in XPS but, generally, the resolution in AES may be degraded by the effects of electron backscattering. The ability of the analyst to realise these resolutions in an effective way will, of course, also depend on the quality of the signal levels obtained.

This International Standard describes three methods for the determination of lateral resolution in AES and XPS. The method chosen for use depends on the expected value of the lateral resolution. Annexes A, B and C provide illustrative examples of the measurement of lateral resolution.

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Surface chemical analysis — Auger electron spectroscopy and X-ray photoelectron spectroscopy — Determination of lateral resolution

1 Scope

This International Standard describes three methods for measuring the lateral resolution achievable in Auger electron spectrometers and X-ray photoelectron spectrometers under defined settings. The straight-edge method is suitable for instruments where the lateral resolution is expected to be larger than 1 µm. The grid method is suitable if the lateral resolution is expected to be less than 1 µm but more than 20 nm. The gold-island method is suitable for instruments where the lateral resolution is expected to be smaller than 50 nm.

Annexes A, B and C provide illustrative examples of the measurement of lateral resolution.

2 Normative references

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The following referenced documents are indispensable for the application 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 18115:2001, *Surface chemical analysis — Vocabulary*

3 Terms, definitions, symbols and abbreviated terms

3.1 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 18115 apply. The definition of lateral resolution is repeated here for convenience.

3.1.1
resolution, lateral
distance measured either in the plane of the sample surface or in a plane at right angles to the axis of the image-forming optics over which changes in composition can be separately established with confidence

NOTE 1 The choice of plane should be stated.

NOTE 2 In practice, the lateral resolution may be realized as either (i) the FWHM of the intensity distribution from a very small emitting point on the sample, or (ii) the distance between the 12 % and 88 % intensity points in a line scan across a part of the sample containing a well-defined step function for the signal relating to the property being resolved. These two values are equivalent for a Gaussian intensity distribution. For other distributions, other parameters may be more appropriately chosen. Often, for a step function, the distance between the 20 % and 80 % intensity points or the 16 % and 84 % intensity points in the line scan are used. The latter pair gives the two-sigma width for a Gaussian resolution function.

ISO 18115:2001, definition 5.255

NOTE 3 For the purposes of this International Standard, measurement in the plane of the sample is preferred.

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3.2 Symbols and abbreviated terms

AES Auger electron spectroscopy

d diameter of an electron beam (of axial symmetry) incident on a sample surface

FWHM full width at half maximum

XPS X-ray photoelectron spectroscopy

x parameter needed for the determination of lateral resolution; the measurement of lateral resolution begins when the signal intensity is x % of the maximum intensity and ends when the intensity is $(100 - x)$ % of the maximum. In the case of $\delta r(50)$, x is 25

θ angle of incidence of an electron beam or an X-ray beam on a sample surface measured with respect to the surface normal

4 General information

4.1 Background information

A common need in AES and XPS is the measurement of composition as a function of position on the sample surface. Typically, an analyst wishes to determine the local surface composition of some identified region of interest. This region of interest could be a feature on a semiconductor wafer (such as an unwanted defect particle or contamination stain), a corrosion pit, a fibre or an exposed surface of a composite material. With growing industrial fabrication of devices with dimensions on the micrometre and nanometre scales, particularly in the semiconductor industry and for emerging nanotechnology applications, there is an increasing need to characterize materials using tools with lateral resolutions that are smaller than those of the features of interest. It is generally necessary in these applications to be able to determine that devices have been fabricated as intended (quality control), to evaluate new or current fabrication methods (process development and process control), and to identify failure mechanisms (failure analysis) of a device during its service life or after exposure to different ambient conditions. The lateral resolution is an important parameter in the application of characterization techniques such as AES and XPS for the surface characterization of materials containing features with micrometre and nanometre dimensions.

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It is clearly desirable that the lateral resolution of the technique be smaller than the lateral dimensions of the feature of interest in order that the feature can be readily imaged. The feature of interest in an AES instrument might typically be initially detected in a scanning electron micrograph. The primary electron beam could then be positioned on the feature and an Auger spectrum recorded. In XPS instruments, the feature of interest must generally be detected from an image or a line scan in which a particular signal (often the intensity of a selected photoelectron peak) is displayed as a function of position on the sample surface.

In practice, the detectability of a feature in AES and XPS measurements depends not only on the lateral resolution but also the difference in signal intensities for measurements made on and off the possible feature (materials contrast) and the observation time (through the statistical variations in the signal intensities). The detectability of a feature thus depends on an instrumental characteristic (the lateral resolution), the particular constituents of the sample, and the measurement time. Reliable detection of a feature will also depend on instrumental stability (particularly the stability of the incident electron beam current in AES, the X-ray flux in XPS, and the positional stability of the sample stage with respect to the electron or X-ray beam) and the chemical stability of the sample during the time needed for acquisition of AES or XPS data.

Many authors have described and discussed the lateral resolution (often referred to as spatial resolution) of AES and XPS instruments. Useful information can be found in Reference [1] for AES and in Reference [2] for XPS. ISO/TR 19319 gives guidance on the determination of lateral resolution and related parameters in AES and XPS [3].

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4.2 Measurement of lateral resolution in AES and XPS

The lateral resolution for AES and XPS measurements typically depends on either the characteristics of the incident radiation or the characteristics of the lens-analyser-detector system used in the spectrometer. In the former case, the lateral resolution will depend mainly on the cross-sectional dimensions (e.g. the beam diameter) of the incident radiation (electron beam in AES or the X-ray beam in XPS) at the sample surface, and will improve as the beam diameter decreases. In the latter case, the lateral resolution will depend mainly on the electron-optical design of the lens-analyser-detector system together with any apertures that may be positioned in the electron-optical path. This is the situation

a) when the spectrometer is used for lens-defined small-area XPS,

b) when images are produced by scanning the acceptance area of the lens, or

c) when the spectrometer produces parallel images by projecting photoelectrons of the appropriate energy through the lens-analyser system to the detector.

The methods described in Clause 5 involve measurements of the intensity of a selected AES or XPS spectral feature while a sufficiently sharp chemical gradient (a chemical edge) on the sample is translated through the analysis position (defined by the incident beam) or the analysis position is translated across a chemical edge. The measured lateral resolution will depend on the instrumental design (i.e. the beam diameter or the electron-optical design of the spectrometer), the intrinsic sharpness of the chemical edge used for the measurements and, for AES, the magnitude and width of the Auger signal excited by back-scattered electrons [1].

4.3 Dependence of lateral resolution on scan direction

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The measured lateral resolution can depend upon the direction in which the translation of the sample with respect to the incident beam or the spectrometer is made. This variation can arise in any of the following three situations:

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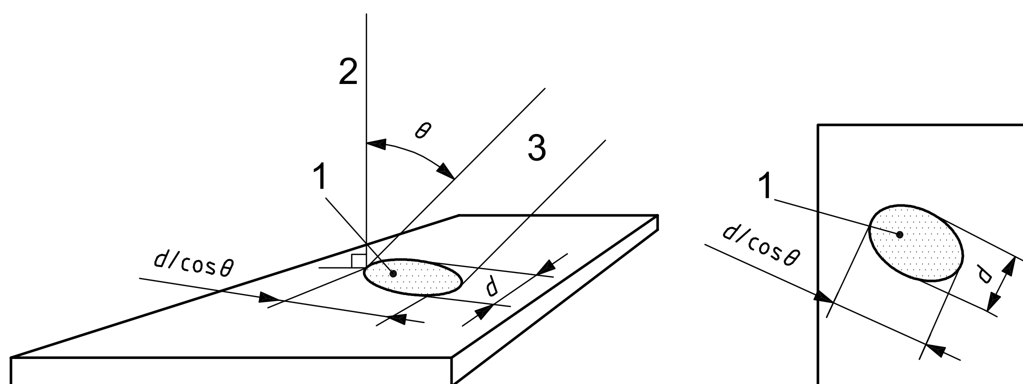
a) if an X-ray or electron beam of circular cross section (i.e. the beam has axial symmetry) is incident on the sample at a non-zero angle relative to the surface normal, the beam-intensity profile on the sample will then be an ellipse, as shown in Figure 1 for the case of an incident electron beam;

b) if the lateral resolution is defined by the analyser or lens, and the sample normal is not parallel with the entrance axis of the analyser; or

c) if the incident beam is astigmatic.



Key

1 analysed area

2 surface normal

3 electron beam

d is the diameter of the beam

NOTE The elliptical intensity profile on the sample surface is shown in the plan view on the right.

Figure 1 — Example of an electron beam striking the sample at an angle θ relative to the surface normal

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Lateral resolution should therefore be measured in at least two directions. In the case of a circular beam incident on a sample at some angle with respect to the surface normal, the measurements should be made along the directions of the short and long axes of the ellipse shown in the plan view of Figure 1. In the case of an astigmatic beam, the measurements should be made in at least two directions; normally, these directions should be orthogonal to each other. If possible, these directions should be chosen to show the smallest and the largest values of the lateral resolution.

4.4 Methods for the measurement of lateral resolution in AES and XPS

The method to be used for the measurement of lateral resolution in AES and XPS depends on the magnitude of the lateral resolution to be measured and on the experimental configuration. Three alternative methods are described.

a) The straight-edge method in Clause 5 will generally be satisfactory if the lateral resolution is expected to be larger than 1 µm. Four variants of this method may be used depending on the particular experimental configuration. This method is typically used to measure lateral resolution in XPS instruments.

b) The grid method of Clause 6 is suitable if the lateral resolution is expected to be less than 5 µm but more than 20 nm. The grid method is typically used for scanning Auger microscopy on instruments where the incident beam may have a diameter of about 100 nm. This method may also be used for XPS instruments.

c) The gold-island method in Clause 7 will be satisfactory if the lateral resolution is expected to be less than 50 nm. The gold-island method is typically used in scanning Auger microscopes where the incident electron beam may have a diameter of about 10 nm.

The straight-edge or grid methods should not be used in high-lateral-resolution scanning Auger microscopes because imperfections of the straight edge or of bars in the grid structure may have dimensions comparable to those of the electron beam on the sample (see Figure 1).

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Note that the resolution, if defined by the spectrometer, may or may not depend on the measured electron energy or any changes in the operating conditions.

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5 Measurement of lateral resolution with the straight-edge method

5.1 Introduction

The straight-edge method may be used for the measurement of lateral resolution in AES and XPS instruments if the lateral resolution is expected to be larger than 1 µm. In all variants of the straight-edge method, the scanning increment or the distance between the pixels in the image shall be less than 20 % of the expected lateral resolution.

5.2 Variants of the straight-edge method

Four variants of the straight-edge method can be used depending on the experimental configuration.

5.2.1 Method 1

A straight edge is translated through a stationary analysis area. If this method is applied, the sample manipulator or sample stage shall have a precision of position that is at least five times smaller than the lateral resolution to be measured.

5.2.2 Method 2

The analyser acceptance area is scanned over a stationary straight edge.

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5.2.3 Method 3

The primary beam (electrons or X-rays) is scanned over a stationary straight edge.

5.2.4 Method 4

An image of the straight edge is formed at a known magnification using electrons of a selected energy, and the lateral resolution is determined from that image.

5.3 Selection of the straight-edge specimen

The straight-edge specimen shall have a straight, sharp edge whose length is at least ten times larger than the lateral resolution to be measured. The material should be as thin as possible to minimize the detection of any signal arising from the vertical plane of its edge. It is an advantage if the material has a large cross section for photoelectron or Auger-electron emission because this minimizes the time needed to produce a signal of sufficient intensity. The material should be a metallic conductor to eliminate signal variations that may be brought about by changes in charge-compensation conditions needed for a non-conductor. The specimen surface should preferably consist of a single element to avoid complications introduced by any variations of composition across the surface (such as could occur by preferential sputtering during sputter cleaning of the surface). A specimen consisting of a noble metal is recommended because surface contamination during the measurements will occur at a slower rate than for other metals, and there will thus be smaller changes of an unwanted nature occurring in the signal-electron intensities. The specimen should also be as smooth as possible so that signal variations due to the changing topography of the specimen are minimized.

NOTE A suitable specimen for this measurement is a silver-coated specimen with a slot, available as specimen supports in scanning electron microscopy. Such a specimen is available as catalogue number G220-S6 from Agar Scientific Limited, 66a Cambridge Road, Stansted, Essex CM24 8DA, UK. This specimen consists of a disc 3,05 mm in diameter having a slot 500 µm wide and 2 mm long. The supplier reports that the thickness of this material is in the range 12 µm to 15 µm. Specimens having slots of different widths are also available. Slot grids are also available from Structure Probe Inc., P.O. Box 656, West Chester, PA 19381-0656, USA, and Ted Pella Inc., P.O. Box 492477, Redding, CA 96049-2477, USA.[1)]

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5.4 Mounting the straight-edge specimen

The straight-edge specimen shall be mounted on the sample stage or manipulator of the AES or XPS instrument in such a way that good electrical contact can be established between the specimen and the spectrometer. If possible, the specimen should be mounted so that the straight edge overhangs the specimen holder and so that the spectrometer does not receive any signal when the specimen is not within the analysis area. The specimen should then be loaded into the spectrometer using the manufacturer's recommended procedure.

5.5 Cleaning the straight-edge specimen

If the straight-edge specimen has appreciable surface contamination, the required data-acquisition time will become very long. Sample cleaning using the following procedure is recommended for this situation. The straight-edge sample should be washed in research grade alcohol and dried by passing dry argon over the surface. The region of the straight-edge specimen where the AES or XPS measurements are to be made should be cleaned by ion etching (e.g. by sputtering with noble-gas ions having an energy of less than 3 keV). The ion dose should be sufficient to reduce the intensities of any contaminant peak to less 2 % of the most intense metal peak in an AES or XPS survey spectrum. Typically, an argon-ion dose of about 10 $\mu A \cdot min \cdot cm^{-2}$ will be found to be sufficient. The contaminants most commonly observed are oxygen and carbon.

1) These are examples of suitable products available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products.

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5.6 Operating the instrument

The AES or XPS instrument shall be operated in accordance with the manufacturer's documented instructions. Choose settings for the incident beam (beam energy and beam current for AES, and X-ray source and X-ray source power for XPS). Choose spectrometer settings (e.g. analyser operating mode, analyser pass energy or retarding ratio, apertures, lens settings, detector multiplier settings) required for or appropriate to the needed determination of the lateral resolution. Ensure that the count rates for the selected signal are within the linear operating range for the detector and associated electronic systems [4].

5.7 Data collection

5.7.1 Data collection for method 1, method 2 and method 3

A line scan shall be performed in a direction perpendicular to the straight edge of the straight-edge specimen using method 1, method 2 or method 3 of 5.2. The line scan shall be performed in accordance with the manufacturer's instructions.

The line scan shall start and end with analysis positions that are wholly on the specimen and wholly off the specimen. The distances from the start or end positions to the straight edge (as judged by the position at which the signal is the average of the signals at the start and end positions) shall each be at least three times larger than the lateral resolution expected for the value of the parameter x (see 5.8) to be used in the data analysis. This requirement ensures that there is an adequate region of near-constant signal intensity at each end of the line scan. A region of near-constant signal intensity at the end of a line scan will be referred to as a “plateau”. The step size for the line scan shall be at least five times smaller than the expected lateral resolution. This requirement ensures that an adequate number of measurements will be made in the region where the straight edge is passing through the analysis area. If, after the line scan is measured, the positions of the start and end points or the value of the step size do not fulfil these conditions, the measurement shall be repeated with modified start and end positions or a modified step size.

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If the spectrometer transfer lens is used to define the lateral resolution of the instrument, the lateral resolution may depend upon the kinetic energy of the electrons being measured. Under these circumstances, the lateral resolution shall be measured using at least two electron energies. The two electron energies should ideally be chosen to be as near as possible to the extremes of the energy range normally measured. In practice, a test specimen should be chosen that provides photoelectron or Auger-electron signals of sufficient intensity for the measurements and with energies as near as possible to the energy extremes.

A spectrum for the selected specimen-signal electrons shall be acquired at each point of the line scan. These spectra shall include at least one major peak from the element comprising the straight-edge specimen (e.g. Ag $3d_{5/2}$ for XPS). The spectra shall be recorded for an energy range that is sufficient to obtain peak intensities by subsequent data processing. The peak intensity for a spectrum will normally be a peak area (after subtraction of a suitable background), although it could be a differential intensity for an Auger spectrum. The peak intensity shall be computed at each point on the line scan. A graph can then be constructed to show the measured intensity as a function of position on the specimen. Figure 2 shows a schematic form of this graph.

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