INTERNATIONAL STANDARD

ISO

18516

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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Foreword

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

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.

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

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.

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.

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].

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

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:

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.
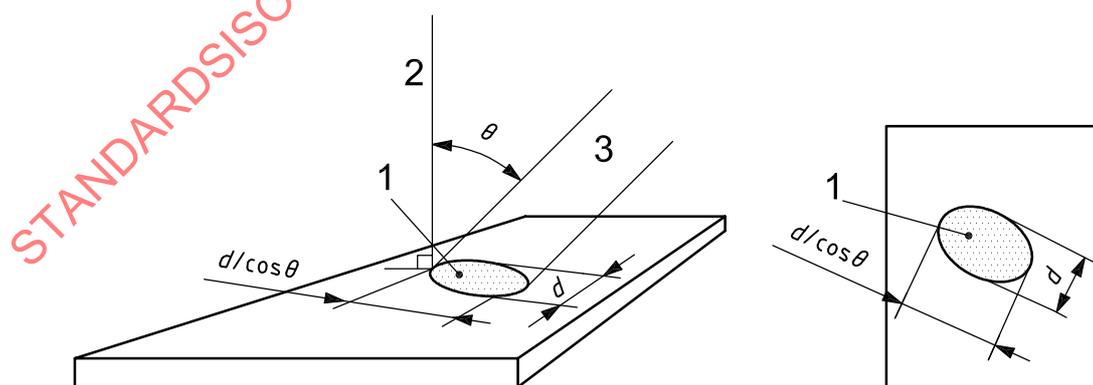
Key

1   analysed area   3   electron beam

2   surface normal 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

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).

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.

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.

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)]

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 μA·min·cm^{-2} will be found to be sufficient. The contaminants most commonly observed are oxygen and carbon.

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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.

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.

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.
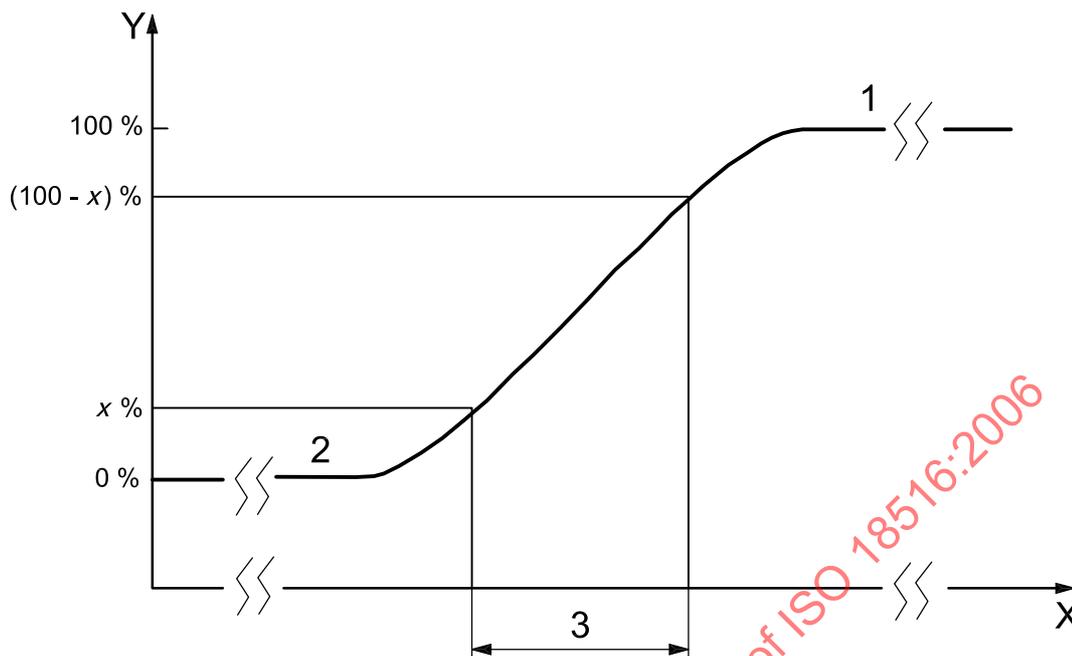
Key

X   distance

Y   peak area, %

1   upper plateau

2   lower plateau

3   resolution

Figure 2 — Schematic diagram illustrating the variation of peak area as a function of distance along a line scan

The data-acquisition time for the spectra of the line scan will depend upon the conditions used and there will be statistical scatter in the peak intensities for the upper plateau (where the signal intensity is a maximum). The data-acquisition time shall be such that the relative standard deviation of the statistical scatter of the signal intensity in the upper plateau region is less than 2 %.

5.7.2   Data collection for method 4

Method 4 of 5.2 is for the parallel-imaging mode of data collection. The straight-edge specimen shall be positioned in the spectrometer so that a signal-electron image of the straight-edge material will appear close to the middle of the imaged area. The spectrometer shall be set and operated in accordance with the manufacturer's instructions. The number of adjacent pixels in the image shall be chosen so that there are at least five pixels over an image distance corresponding to the lateral resolution expected for the value of the parameter x (see 5.8) to be used in the data analysis. The magnification of the spectrometer in its imaging mode shall be chosen so that the distances from the edges of the image 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 selected value of the parameter x. The magnification of the spectrometer shall be calibrated using a method provided by the manufacturer or other relevant traceable method.

The image shall show three areas, one having a close-to-zero signal, one showing a fairly uniform signal, and the third having a transition region between the other two, as indicated in the idealized image of Figure 3. The proportion of the image displaying the transition region will depend on the magnification for the image and on the lateral resolution. Depending on the orientation of the mounted specimen, the acquired image may be rotated with respect to the image of Figure 3.
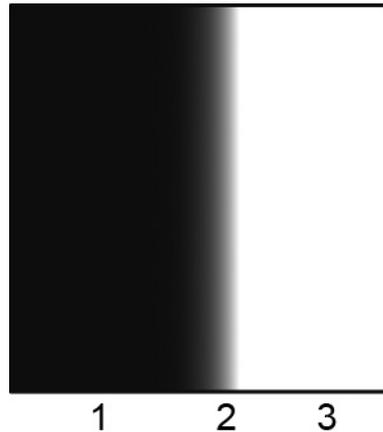
Key

1   close-to-zero signal

2   transition region

3   uniform signal

NOTE   White represents a large signal, black a close-to-zero signal, and grey an intermediate signal between the other two.

Figure 3 — Appearance of an idealized image of a straight edge

Obtain a line scan from the acquired image. The line scan shall be in a direction perpendicular to that of the straight edge and shall extend over as much of the image as possible. In this case, the line scan is a graph showing pixel intensity as a function of distance normal to the straight edge. The line scans shall be generated from an image in such a way that the values plotted on the ordinate scale are directly proportional to the acquired signal intensities. The data-acquisition time for the image will depend upon the conditions used and there will be statistical scatter in the pixel intensities for the upper plateau (where the signal intensity is a maximum). The data-acquisition time shall be such that the relative standard deviation of the statistical scatter of the signal intensity is less than 2 % of the maximum signal in the upper plateau obtained from the line scan. 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 the requirements specified earlier in this subclause, the measurement shall be repeated with modified start and end positions or a modified step size.

To reduce the statistical noise in the graphical line scan, the signals present in a number of pixels in a direction perpendicular to the direction of the line scan can be added; this scan is often referred to as a "band scan". A band scan can be regarded as being equivalent to a line scan for the purposes of determining the lateral resolution.

5.8   Data analysis

5.8.1   General information

The lateral resolution is obtained from the line scans plotted in 5.6. These line scans should resemble Figure 2 in which there is a lower plateau and an upper plateau; in each plateau region, the signal intensity should be approximately constant (within expected statistical variations). The difference between the average signal intensities for the upper and lower plateaux is 100 % of the signal change. The lateral resolution can be obtained from the line scan by determining the distance along the scan direction corresponding to a change in signal intensity from x % to $(100 - x)$ %, as shown in Figure 2. The value of x may be chosen by the analyst to satisfy the requirements of the measurement, a value recommended by the instrument manufacturer, or a value recommended as being appropriate for AES (x = 20) [5] or for XPS (x = 12) [6]. Common values of x are 25, 20, 16 and 12. These values of x may have significance as noted in the following subclauses. Alternatively, the lateral resolution can be obtained from the FWHM of the line spread function, as described in 5.8.7.

NOTE   Signal intensities in the upper-plateau region of a line scan can vary with position if the primary beam is incident on a contamination film of varying thickness or if the beam is incident on grains of different crystalline orientation. If such variations are observed, clean the sample or select another region on the sample for the line scan.

5.8.2   When x = 25

If x is 25, the derived lateral resolution corresponds to a 50 % signal change (25 % to 75 % of the total signal intensity in the line scan). This choice is convenient because the measurement is only minimally affected by the tails of the line scan that are mainly due to backscattered electrons in AES [1]. There is no physical significance to this value of x.

5.8.3   When x = 20

If the response of the spectrometer is uniform (100 %) within a circular region on the sample and zero outside this region (a "top hat" distribution), and if the incident beam is relatively broad, it can be shown that the lateral resolution corresponding to x = 20 (20 % to 80 % of the total signal intensity in the line scan) is approximately equal to the radius of the analysis area (more precisely, the lateral resolution is 0,984 of the radius of that area) [7]. The same result is obtained if the incident beam has a top-hat distribution and the spectrometer response is relatively broad.

5.8.4   When x = 16

If the response of the spectrometer can be represented by a Gaussian function to describe measured intensity as a function of radial distance on the sample surface and the incident beam is relatively broad, it can be shown that the lateral resolution corresponding to x = 16 (16 % to 84 % of the total signal intensity in the line scan) corresponds to twice the value of the standard deviation in the Gaussian function [7]. The same result is obtained if the incident beam has a Gaussian distribution and the spectrometer response is relatively broad.

5.8.5   When x = 12

If the response of the spectrometer can be represented by a Gaussian function and the incident beam is relatively broad, it can be shown that the lateral resolution corresponding to x = 12 (12 % to 88 % of the total signal intensity in the line scan) corresponds to the FWHM of the Gaussian function [7]. The same result is obtained if the incident beam has a Gaussian distribution and the spectrometer response is relatively broad.

5.8.6   Choice of x

The spectrometer response of actual AES and XPS instruments cannot generally be represented by a top-hat or a Gaussian distribution [1]. A particular value of x (as in 5.8.2, 5.8.3, 5.8.4 and 5.8.5) is therefore unlikely to have physical significance for a practical instrument.

If there is no clear reason for preferring one value of x over another, choose x = 20 for AES [5] or x = 12 for XPS [6]. The value of x shall be specified in a report of the lateral resolution measurement.

5.8.7   Line spread function

The line scan in Figure 2 can be differentiated to generate the line spread function. The FWHM of the line spread function is an alternative measure of lateral resolution [1], [4]. The advantage of this measure is two-fold. First, the derived lateral resolution does not depend on an empirical choice of the parameter x. Secondly, this FWHM is insensitive to the effects of backscattered electrons in AES [1]. The numerical methods used to obtain the line spread function and the FWHM shall be specified in a report of the lateral-resolution measurement.

6 Measurement of lateral resolution with the grid method

6.1 Introduction

The grid method can be used for the measurement of lateral resolution in AES and XPS instruments if the lateral resolution is expected to be smaller than 1 μm but larger than 20 nm. This method is expected to be most useful for scanning Auger microscopes, and the procedure is described for this application.

6.2 Selection of the grid specimen

The recommended specimen for this method consists of a fine copper grid. The mesh bars are regularly spaced and arranged in two orthogonal directions. This type of specimen is suitable for the lateral-resolution measurements because specimens are readily available from commercial sources, there exist a range of mesh sizes, the bars on the mesh have well-defined edges, the bars of the mesh can be positioned over a hole in the specimen holder and will show high contrast in both secondary-electron and Auger-electron images, and the specimen has sufficient electrical conductivity. The widths or diameters of the bars and the spacing between the bars shall be at least ten times larger than the expected lateral resolution.

NOTE 1   Suitable copper grid specimens are available from companies providing supplies for scanning electron microscopes. One possible source is Agar Scientific Limited, 66a Cambridge Road, Stansted, Essex CM24 8DA, UK. Their catalogue number G2785C is a 1 500 mesh (equivalent to a pitch of about 16,9 μm). This specimen can be used for measuring a wide range of lateral resolutions. Other specimens with different pitches are available from Agar Scientific Ltd.; the most appropriate of these specimens for the expected lateral resolution should be selected. These mesh samples are also available with gold or silver coatings; if preferred, these coated mesh specimens may be used in place of the uncoated copper mesh. 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.[2]

NOTE 2   Alternatives to the copper grid specimen are the specimens MRS-3 and MRS-4 available from Geller Analytical Laboratory, 426e Boston St., Topsfield, MA 01983-1216, USA.[2] These are magnification reference standards, traceable to the National Institute of Standards and Technology in the USA and to the National Physical Laboratory in the UK. They consist of patterns of known dimensions that provide contrast in AES, XPS, and secondary-electron images and line scans.

6.3 Mounting the grid specimen

The grid specimen shall be mounted on the sample stage or manipulator of the AES instrument in such a way that good electrical contact can be established between the specimen and the spectrometer. The specimen should then be loaded into the spectrometer using the manufacturer's recommended procedure.

6.4 Cleaning the grid specimen

If lateral resolution is to be measured using the secondary-electron signal, it will not be necessary to clean the grid specimen. If the Auger-electron signal is to be used for this measurement, sample cleaning will be required if there is appreciable surface contamination (in which case the required data-acquisition time will be very long). Sample cleaning using the following procedure is recommended for this situation. The grid sample should be washed in research grade alcohol and dried by passing dry argon over the surface. The region of the grid 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 μA·min·cm^{-2} will be found to be sufficient. The contaminants most commonly observed are oxygen and carbon.

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2) 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.

6.5   Operating the instrument

The AES instrument shall be operated in accordance with the manufacturer's documented instructions. Choose settings for the incident beam (beam energy and beam current). 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]. Measure the magnification for the display, using a traceable standard, of the selected signal in accordance with the manufacturer's instructions.

6.6   Data collection

6.6.1   General requirements

The lateral resolution shall be determined from a line scan of measured secondary-electron or Auger-electron intensities. In each case, a line scan of signal-electron intensity versus distance is made over at least one edge of a selected bar on the grid.
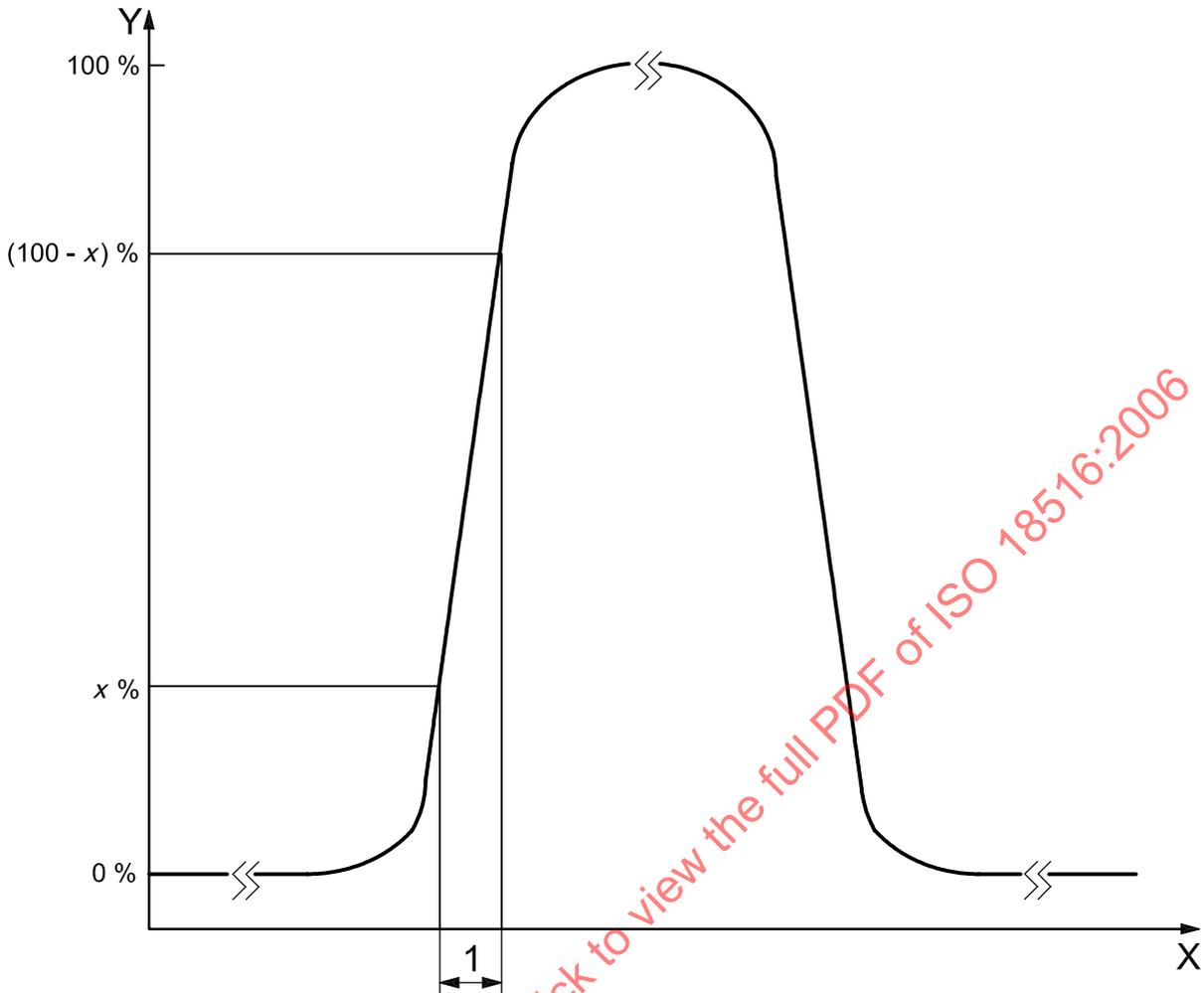
6.6.2   Secondary-electron measurements

A secondary-electron image shall be recorded in accordance with the manufacturer's instructions. The image shall be collected such that the brightest areas do not saturate the detector and the darkest areas contain some signal. This condition is achieved using the gain and offset controls. The image should be optimized in accordance with the manufacturer's instructions using the focusing and alignment controls.

Select a bar on the grid for the line scan. The secondary-electron image shall have at least five pixels (in the direction of the planned line scan normal to the edge) over the expected resolution of the selected bar for the selected value of the parameter x (see 5.8).

Obtain a line scan by measuring the signal intensity as a function of distance on the image. Figure 4 illustrates schematically the appearance of a line scan over a grid bar. The plateau regions (see 5.7.1) shall extend a distance from the edge of the grid bar (as judged by the position at which the signal is the average of the signals at the start and end regions) of at least three times the expected lateral resolution for the selected value of x. 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 the specified requirements conditions, the measurement shall be repeated with modified start and end positions or a modified step size.

As an alternative to obtaining the line scan from a secondary-electron image, the image can be used to define the position and direction of a directly acquired line scan. This method may be preferred if long pixel dwell times are required in order to obtain a line scan with sufficiently small values of statistical noise.

Proceed to 6.6.3 if it is desired to measure lateral resolution from an Auger-electron line scan; otherwise, proceed to 6.7.

Key

X   distance
Y   signal intensity, %
1   lateral resolution

Figure 4 — Schematic diagram of a line scan (showing secondary-electron or Auger-electron intensity as a function of distance) over a grid bar or a gold island

6.6.3 Auger-electron measurements

The secondary-electron image from 6.6.2 is used to select a suitable position and direction for the line scan. An Auger-electron line-scan experiment shall be set up using a suitable Auger transition; the copper L_3VV peak at approximately 919 eV for spectra measured in the direct mode and referred to the Fermi energy is recommended for a copper grid [8]. The silver M_4NN transition or the gold $N_{6,7}VV$ transition would be appropriate if grids coated with Ag or Au were used [8].

Acquire an Auger-electron line scan in accordance with the manufacturer's instructions. The line scan may be acquired by measuring either the peak and nearby background intensities at each point of the line scan or a spectrum at each point of the line scan (from which the peak and nearby background intensities can be obtained by subsequent data analysis). The data-acquisition time shall be such that the peak-to-peak statistical variation of the signal near the centre of the grid bar is less than 5 % of the maximum signal. The line scan shall be recorded with the start and end positions and the step size set as described in 6.6.2.

Plot an Auger-electron line scan showing Auger-electron intensity on the ordinate versus distance on the abscissa. The Auger-electron intensity on the ordinate scale may be a difference between the measured peak

and background intensities divided by the background signal, a peak area obtained from a direct spectrum, or a peak-to-peak amplitude from a differential spectrum. If the background signal is used in the determination of Auger signal, this signal should be measured at a kinetic energy of 30 eV larger than that of the peak. The line scan should have an appearance similar to that shown in Figure 4.

6.7 Data analysis

The lateral resolution shall be measured from a line scan obtained from 6.6 in a manner analogous to that described in 5.8 for the straight-edge specimen. As indicated in Figure 4, the secondary-electron or Auger-electron signal varies from an average minimum value of 0 % to an average maximum value of 100 %. The lateral resolution can be obtained from the line scan by determining the distance along the scan direction corresponding to a change in signal intensity from x % to $(100 - x)$ %, as shown in Figure 4. The value of x may be chosen by the analyst to satisfy the requirements of the measurement, may be a value recommended by the instrument manufacturer, or may be the value $x = 20$ recommended as appropriate for AES [5]. Common values of x are 25, 20, 16 and 12, as described in 5.8. If there is no clear reason for preferring one value of x over another, choose $x = 20$ for AES [5] or $x = 12$ for XPS [6]. The value of x shall be specified in a report of the lateral-resolution measurement.

The line spread function can be obtained, if desired, by differentiating the line scan shown in Figure 4. The FWHM of the line spread function, as described in 5.8.7, is an alternative measure of the lateral resolution. The numerical methods used to obtain the line spread function and the FWHM shall be specified in a report of the lateral-resolution measurement.

7 Measurement of lateral resolution with the gold-island method

7.1 Introduction

The gold-island method may be used for the measurement of lateral resolution in AES instruments if the lateral resolution is expected to be smaller than 50 nm. This method is expected to be most useful for scanning Auger microscopes, and the procedure is described for this application.

7.2 Selection of the gold-island specimen

The specimen for this method consists of small gold islands dispersed on a carbon substrate. This type of specimen is suitable for the lateral-resolution measurements because specimens are readily available from commercial sources, the gold islands exist in a range of sizes, the gold islands have well-defined edges, gold and carbon are materials that show high contrast in both secondary-electron and Auger-electron images, and the specimen has sufficient electrical conductivity. The average diameters of the gold islands shall be at least ten times larger than the expected lateral resolution.

NOTE   Suitable gold-island on carbon specimens are available from companies providing supplies for scanning electron microscopes. One possible source is Agar Scientific Limited, 66a Cambridge Road, Stansted, Essex CM24 8DA, UK. Their catalogue number S168 has gold islands ranging in size from 5 nm to 150 nm. This specimen can be used for measuring a wide range of lateral resolutions. Other specimens with different ranges of gold-island sizes are available from Agar Scientific Ltd. that have gold islands with different size ranges; the most appropriate of these specimens for the expected lateral resolution should be selected. Gold islands are also available from Structure Probe Inc., P.O. Box 656, West Chester, PA 19381-0656, USA, and from Ted Pella Inc., P.O. Box 492477, Redding, CA 96049-2477, USA.[3]

7.3 Mounting the gold-island specimen

The gold-island specimen shall be mounted on the sample stage or manipulator of the AES instrument in such a way that good electrical contact can be established between the specimen and the spectrometer. The specimen should then be loaded into the spectrometer using the manufacturer's recommended procedure.

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3)   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.

7.4   Cleaning the gold-island specimen

If lateral resolution is to be measured using the secondary-electron signal, sample cleaning will be unnecessary. If the Auger-electron signal is to be used for this measurement, sample cleaning may be required.

If the gold-island 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 region of the gold-island specimen where the AES measurements are to be made should be cleaned by ion etching (e.g. by sputtering with argon ions having an energy of less than 1 keV). The ion dose should be sufficient to reduce the intensities of any contaminant peak to less than 2 % of the most intense metal peak in an AES survey spectrum taken from the centre of a gold island. Typically, an argon-ion dose of about 10 µA·min·cm^{-2} will be found to be sufficient. The contaminants most commonly observed are oxygen and carbon.

7.5   Operating the instrument

The AES instrument shall be operated in accordance with the manufacturer's documented instructions. Choose settings for the incident beam (beam energy and beam current). 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 spatial resolution. Ensure that the count rates for the selected signal are within the linear operating range for the detector and associated electronic systems [4]. Measure the magnification for the display, using a traceable standard, of the selected signal in accordance with the manufacturer's instructions.

7.6   Data collection

7.6.1   General requirements

The lateral resolution shall be determined from a line scan of measured secondary-electron or Auger-electron intensities. In each case, a line scan of signal-electron intensity versus distance is made over at least one edge of a selected gold island.

7.6.2   Secondary-electron measurements

7.6.2.1   Recording a secondary-electron image

A secondary-electron image shall be recorded in accordance with the manufacturer's instructions. The image shall be collected such that the brightest areas do not saturate the detector and the darkest areas contain some signal. This condition is achieved using the gain and offset controls. The image should be optimized in the normal way using the focusing and alignment controls.

7.6.2.2   Selection of a gold island

Select a gold island for the line scan. The dimensions of this island shall be at least ten times larger than the expected lateral resolution and separated from other islands by at least five times the expected lateral resolution; if the line scan is made over a smaller island or with one having a second island too close to the selected island, the lateral resolution will be underestimated. The step size for the line scan shall be at least five times smaller than the expected lateral resolution for the selected value of the parameter x (see 5.8).

7.6.2.3   Collecting a line scan from an image

Obtain a line scan by measuring the signal intensity as a function of distance on the image. The line scan shall include at least two gold/carbon edges. Figure 4 is also a schematic illustration of the appearance of a line scan over a single island. The line scan shall have at least five pixels (in the direction of the planned line scan normal to the island edge) over the expected resolution of the island for the selected value of the parameter x (see 5.8). The plateau regions (see 5.7.1) shall extend a distance from the edge of the island (as judged by

the position at which the signal is the average of the signals at the start and end regions) of at least three times the expected lateral resolution for the selected value of x. 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 the specified requirements conditions, the measurement shall be repeated with modified start and end positions or a modified step size.

If the measurements of lateral resolution from the two gold/carbon edges differ, then the larger of the two numbers obtained shall be used in a report of the lateral resolution.

7.6.2.4 Direct collection of a line scan

As an alternative to obtaining the line scan from a secondary-electron image, the image may be used to define the position and direction of a directly acquired line scan. The line scan shall be measured as described in 7.6.2.3. This method may be preferred if long pixel dwell times are required in order to obtain a line scan with sufficiently small values of statistical noise.

Proceed to 7.6.3 if it is desired to measure lateral resolution from an Auger-electron line scan; otherwise, proceed to 7.7.

7.6.3 Auger-electron measurements

7.6.3.1 Selection of the Auger transition

The secondary-electron image from 7.6.2 is used to select a suitable position and direction for the line scan. An Auger-electron line-scan experiment shall be set up using a suitable Auger transition; the Au $N_{6,7}VV$ peak at approximately 71 eV for spectra measured in the direct mode and referred to the Fermi energy is one that may be selected [8]. An alternative transition is the Au $M_5N_{67}N_{67}$ transition at approximately 2016 eV measured in the same way [8]. Figure C.2 shows a comparison of line scans acquired using each of these two transitions. This example indicates that there is no significant difference in the lateral resolution measured using these transitions. A measurement using both transitions should be made to determine whether the lateral resolution depends on electron energy. If, as in the example of Figure C.2, there is no such influence, the analyst may choose either transition and shall report the transition used. If the lateral resolution does depend significantly on electron energy, measurements for both Auger transitions shall be made and the results reported.

7.6.3.2 Collection of the Auger line scan

Acquire an Auger-electron line scan in accordance with the manufacturer's instructions. The line scan may be acquired by measuring either the peak and the nearby background intensities at each point of the line scan or a spectrum at each point of the line scan (from which the peak and nearby background intensities can be obtained by subsequent data analysis). The data-acquisition time shall be such that the peak-to-peak statistical variation of the signal near the centre of the gold island is less than 5 % of the maximum signal. If image tracking and registration software is available, this can be used according to the manufacturer's instructions provided that it is also used in the measurements for which the lateral resolution, derived in this way, is quoted.

Plot an Auger-electron line scan showing Auger-electron intensity on the ordinate versus distance on the abscissa. The Auger-electron intensity on the ordinate scale can be a difference between the measured peak and the background intensities divided by the background signal, a peak area obtained from a direct spectrum, or a peak-to-peak amplitude from a differential spectrum. If a background intensity is used in the determination of Auger-electron intensity, the background intensity should be measured at a kinetic energy of 30 eV larger than that of the Auger-electron peak. The line scan shall have at least five pixels (in the direction of the planned line scan normal to the island edge) over the expected resolution of the island for the selected value of the parameter x (see 5.8). The plateau regions (see 5.7.1) shall extend a distance from the edge of the island (as judged by the position at which the signal is the average of the signals at the start and end regions) of at least three times the expected lateral resolution for the selected value of x. 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 the specified requirements conditions, the measurement shall be repeated with modified start and end positions or a modified step size. The line scan should have the appearance of that shown in Figure 4.

7.6.3.3 Lateral drift

Significant lateral drift may occur in the instrument during the measurement. That is, the specimen stage may move with respect to the position of the electron beam on the specimen, and the measured line profiles will be distorted compared to those that would be measured in the absence of drift. Drift rates can be conveniently determined from repeated measurements of secondary-electron images (see 7.6.2) or of Auger-electron line scans. Reports of measurements of lateral resolution shall include a statement of either of the measured drift rate or of the magnitude of the lateral drift that was observed during a time interval similar to that required for measurement of the Auger-electron line scan.

If the lateral drift is larger than a stated amount (e.g. 10 %) of the expected lateral resolution (e.g. from the manufacturer's specifications) during the measurement time for the Auger-electron line scan, the measurement of lateral resolution will be invalid. If the cause of the drift can be established and removed, or minimized, then repeated measurements of Auger-electron line scans may be used to derive multiple measures of lateral resolution. The average lateral resolution may then be reported together with the corresponding standard deviation or a statement indicating the range of lateral resolutions obtained. Repeated line-scan measurements will be necessary if there is no prior knowledge of the expected lateral resolution. If the drift cannot be reduced to a satisfactory level, the derived lateral resolution will depend on the drift rate and the time required for the line-scan measurement.

7.7 Data analysis

The lateral resolution shall be measured from the line scan obtained from 7.5 in a manner analogous to that described in 5.8 for the straight-edge specimen. As indicated in Figure 4, the secondary-electron or Auger-electron signal varies from an average minimum value of 0 % to an average maximum value of 100 %. The lateral resolution can be obtained from the line scan by determining the distance along the scan direction corresponding to a change in signal intensity from x % to $(100 - x)$ %, as shown in Figure 4. The value of x may be chosen by the analyst to satisfy the requirements of the measurement, may be a value recommended by the instrument manufacturer, or may be the value $x = 20$ recommended as appropriate for AES [5]. Common values of x are 25, 20, 16 and 12, as described in Section 5.8. If there is no clear reason for preferring one value of x over another, choose $x = 20$ for AES [5]. The value of x shall be specified in a report of the lateral resolution measurement.

The line spread function can be obtained, if desired, by differentiating the line scan shown in Figure 4. The FWHM of the line spread function, as described in 5.8.7, is an alternative measure of the lateral resolution. The numerical methods used to obtain the line spread function and the FWHM shall be specified in a report of the lateral resolution measurement.

Annex A
(informative)

Determination of lateral resolution of an XPS instrument with a focused X-ray spot

A.1 Collection of XPS line-scan data

This Annex provides an example of a measurement of lateral resolution from an XPS line scan of a silver straight edge acquired with commercially available XPS instrument providing a focused X-ray spot on the sample (see 5.7.1). In this example, the lateral resolution is determined by the size of the X-ray spot. The silver straight edge was stepped through the X-ray beam, and the distance between successive measurements was 40 μm. The Ag 3d region of the XPS spectrum was collected at each point in the line scan and, following subtraction of a suitable background, the areas of the Ag $3d_{5/2}$ and the Ag $3d_{3/2}$ peaks were measured and summed at each position of the line scan. A plot was then made of sum of the Ag peak areas versus the position of the straight edge, as shown in Figure A.1.

Key
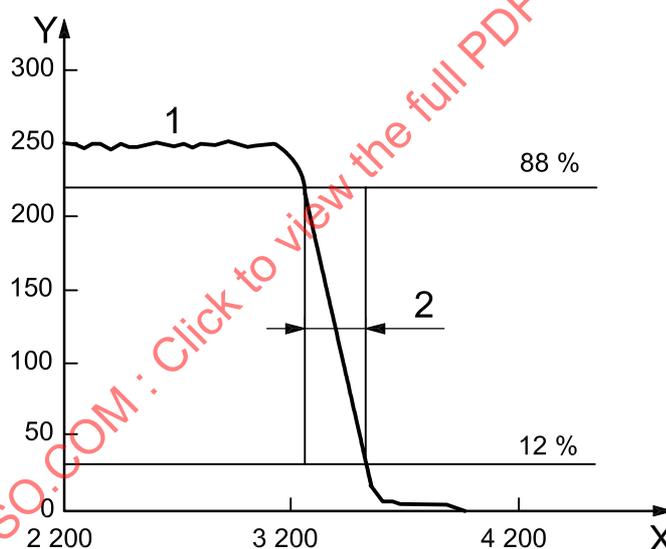
X   distance, μm

Y   peak area, counts eV/1 000

1   upper plateau (= 100 %)

2   lateral resolution (= 270 μm)

Figure A.1 — XPS line scan showing total silver 3d peak intensity versus position of a silver straight edge

A.2 Determination of lateral resolution

The mean intensity of the upper plateau in Figure A.1 was determined. Horizontal lines were drawn on the plot of Figure A.1 corresponding to 12 % and 88 % of the mean upper-plateau intensity (i.e. for $x = 12$). The positions of the sample when the signal intensity was equal to these values were determined (vertical lines in Figure A.1) and the distance between these positions was calculated. In the example of Figure A.1, the lateral resolution was found to be 270 μm with $x = 12$.

A.3 Further information

The lateral resolution for the example data in Figure A.1 may be determined for other values of x using an analogous procedure to that given in A.2 for x = 12 %. The resulting values of lateral resolution are shown in Table A.1.

**Table A.1 — Values of lateral resolution determined from the data
in Figure A.1 for different values of x**

x %	Lateral resolution µm
12	270
16	237
20	204
25	171

Annex B
(informative)

Determination of lateral resolution from a secondary-electron line scan

B.1 Collection of secondary-electron line scan

This Annex provides an illustration of a measurement of lateral resolution from a secondary-electron micrograph acquired with a commercially available Auger electron spectrometer and a gold-island sample (see 7.6.2). The electron beam was stepped across a selected gold island. The distance between successive measurements was about 0,63 nm. The secondary-electron signal was collected at each point in the line scan. A plot was then made of the secondary-electron intensity as a function of the position of the electron beam on the sample, as shown in Figure B.1. In this example, the energy of the primary beam was 20 keV and the beam current was 1,3 nA.
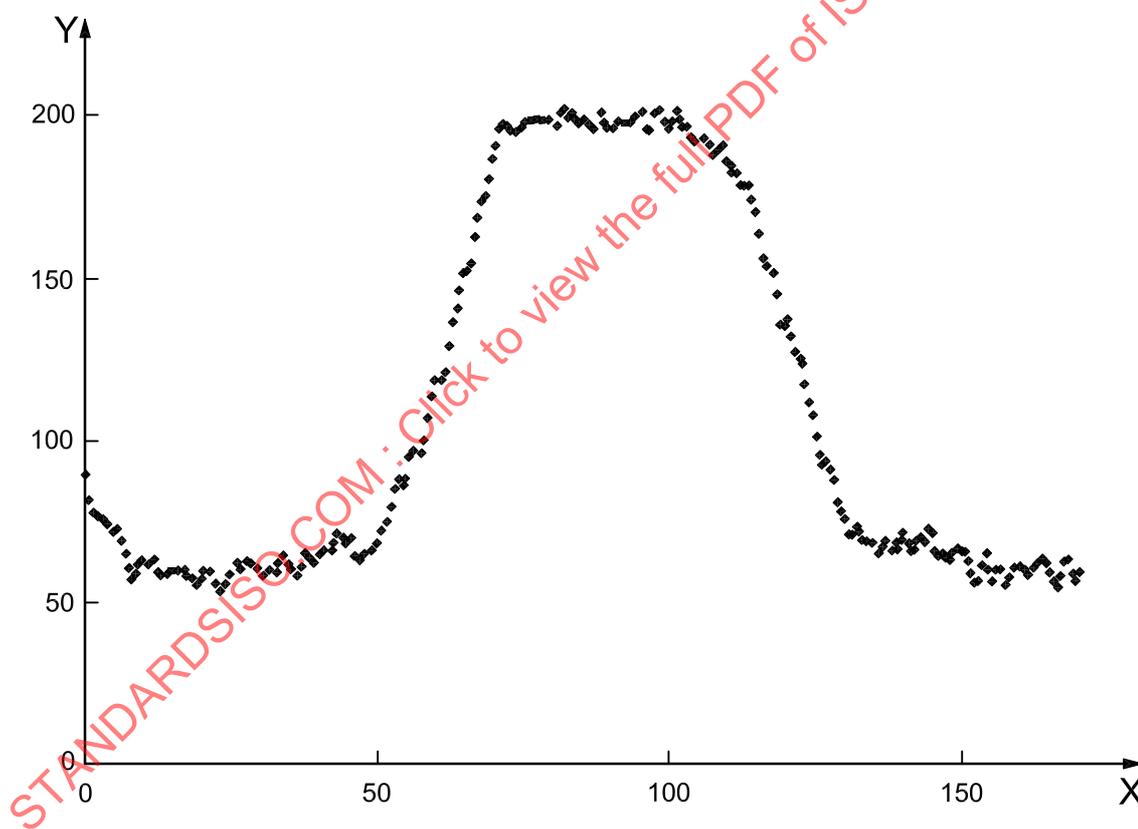
Key

X   distance, nm

Y   SEM intensity (in arbitrary units)

Figure B.1 — Secondary-electron signal as a function of position of the electron beam on the sample as the beam was scanned over a gold island