
**Surface chemical analysis — Auger
electron spectroscopy and X-ray
photoelectron spectroscopy —
Determination of lateral resolution,
analysis area, and sample area viewed by
the analyser**

*Analyse chimique des surfaces — Spectroscopie des électrons Auger
et spectroscopie de photo-électrons — Détermination de la résolution
latérale, de l'aire de la surface d'analyse et de l'aire de la surface de
l'échantillon contribuant au signal détecté*



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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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.

In exceptional circumstances, when a technical committee has collected data of a different kind from that which is normally published as an International Standard ("state of the art", for example), it may decide by a simple majority vote of its participating members to publish a Technical Report. A Technical Report is entirely informative in nature and does not have to be reviewed until the data it provides are considered to be no longer valid or useful.

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ISO/TR 19319 was prepared by Technical Committee ISO/TC 201, *Surface chemical analysis*, Subcommittee SC 5, *Auger electron spectroscopy*.

Introduction

This Technical Report is intended to be used as follows:

- a) To provide guidance on the determination of lateral resolution in Auger electron spectroscopy and X-ray photoelectron spectroscopy where measurements are made of Auger electron or X-ray photoelectron peak intensities as a function of position on a sample surface.
- b) To provide guidance on the determination of analysis area in similar applications of Auger electron spectroscopy and X-ray photoelectron spectroscopy.
- c) To provide guidance on the determination of sample area viewed by the analyser in applications of Auger electron spectroscopy and X-ray photoelectron spectroscopy.
- d) To serve as a basis for the development of International Standards for measurements of lateral resolution, analysis area, and sample area viewed by the analyser in Auger electron spectroscopy and X-ray photoelectron spectroscopy.

Surface chemical analysis — Auger electron spectroscopy and X-ray photoelectron spectroscopy — Determination of lateral resolution, analysis area, and sample area viewed by the analyser

1 Scope

This Technical Report provides information for measuring (1) the lateral resolution, (2) the analysis area, and (3) the sample area viewed by the analyser in Auger electron spectroscopy and X-ray photoelectron spectroscopy.

2 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 18115 [1] apply. The definitions of “analysis area <sample>” and “lateral resolution” from ISO 18115 are given for convenience here. A definition of “sample area viewed by the analyser” is proposed. This definition is similar to the definition of “analysis area <spectrometer>” in ISO 18115. The term “sample area viewed by the analyser” is preferred in this Technical Report to distinguish this area from the corresponding area when the sample is set in a plane at right angles to the spectrometer axis.

2.1

analysis area

<sample> two-dimensional region of a sample surface measured in the plane of that surface from which the entire analytical signal or a specified percentage of that signal is detected

2.2

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 realised 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.

2.3

sample area viewed by the analyser

two-dimensional region of a sample surface measured in the plane of that surface from which the analyser can collect an analytical signal from the sample or a specified percentage of that signal

3 Symbols and abbreviated terms

AES	Auger electron spectroscopy
erf	error function
FWHM	full width at half maximum
I	Auger electron intensity
I_i	incident beam current (in AES)
I_{max}	maximum Auger electron intensity
$J_A(r)$	intensity distribution of detected Auger electrons as a function of the radius r
$J_{Ab}(r)$	intensity distribution of detected Auger electrons that were created by backscattered electrons as a function of the radius r
$J_{Ai}(r)$	intensity distribution of detected Auger electrons that were created by the incident beam as a function of the radius r
R	backscattering factor (in AES)
r	radius from the centre of the incident electron beam on the sample surface (in AES)
r_{max}	upper limit of integration in equation (5)
XPS	X-ray photoelectron spectroscopy
δr	lateral resolution
$\delta r(50)$	lateral resolution determined from a 25% to 75% intensity change in a line profile
σ_b	Gaussian parameter describing the radial distribution of backscattered electrons (in AES)
σ_i	Gaussian parameter describing the radial distribution of the incident electron beam (in AES)

4 Background information on lateral resolution, analysis area, and sample area viewed by the analyser

4.1 General 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 micrometer and nanometer scales, particularly in the semiconductor industry [2] and for emerging nanotechnology applications, there is an increasing need to characterize materials using tools with lateral resolutions and dimensions of analysis areas 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 and the analysis area are important and related parameters in the application of characterization techniques such as AES and

XPS for the surface characterization of materials containing features with micrometer and nanometer dimensions. Another parameter that is important in some measurements is the sample area viewed by the electron energy analyser. The needs for measurements of lateral resolution, analysis area, and sample area viewed by the analyser are described in the following sections.

As in optical [3-6] and various forms of electron microscopy [7-9], the achievable lateral resolution is related to the contrast found in a measured image. A discussion of contrast mechanisms, various definitions of lateral resolution, and image quality is beyond the scope of this report, and the reader is referred to detailed analyses available elsewhere [3-9]. It is pointed out, however, that the contrast transfer function is a useful means for describing the contrast in an image as a function of spatial frequency [3-9]. At the highest detectable spatial frequency, the contrast approaches zero. The achievable lateral resolution in a particular AES or XPS measurement will therefore depend not only on the instrumental characteristics but on the available contrast (e.g., from the signals associated with two neighbouring chemical phases for a particular measurement time).

An overview is given in this report of certain instrumental and measured properties that are described in terms of Gaussian functions. This approach is believed to be a useful guide but it should be emphasized that the properties of real instruments and of real measurements can depart from the Gaussian model considered here. In addition, the detectability of a feature in AES and XPS measurements depends in part on the measure of lateral resolution of the instrument and in part on the difference in signal intensities for measurements made on and off the possible feature and the observation time (through the statistical variations in the signal intensities). The detectability of a feature thus depends on the contrast transfer function for the measurement and the measurement time. The specific results will thus be a function of both instrumental and sample properties. Reliable detection of a feature will also depend on instrumental stability (particularly the stability of the incident electron beam current in AES and 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.

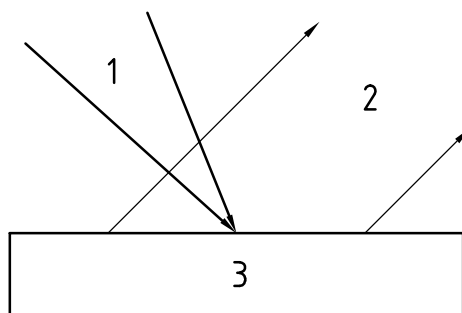
4.2 Lateral resolution

4.2.1 Introduction

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 reliably analysed. 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 electron 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.

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 a review by Cazaux [9] for AES and in a review by Drummond [10] for XPS.

Figures 1 to 3 show schematic diagrams of typical experimental configurations for AES and XPS. These Figures show the exciting radiation incident on the sample surface. For AES (Figure 1), an electron beam with an energy between 3 keV and 25 keV is focused to a "spot" on the sample surface. With a field-emission electron source, the full width at half maximum (FWHM) intensity of the focused spot may be between 5 nm (or even lower) and 50 nm depending on the beam energy and the beam current. The beam is scanned across a region of interest on the sample surface, and various signals collected (such as secondary-electron and Auger electron signals). The Auger electron signal arises from inner-shell ionisations caused in part by the incident beam and in part by backscattered electrons [9]. The lateral resolution in AES is mainly determined by the FWHM of the focused spot [9]; further details are given in 4.2.2.

**Key**

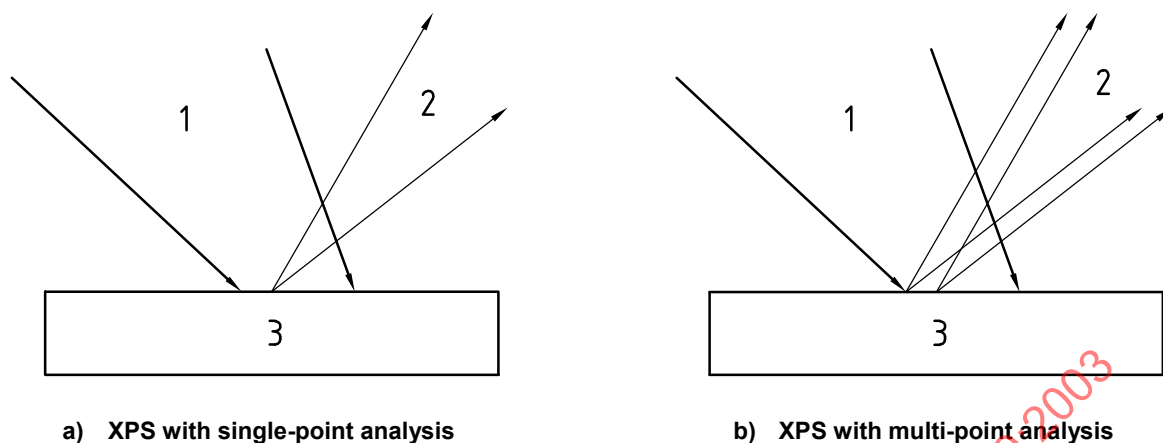
- 1 focused electron beam for AES
- 2 focused X-ray beam for XPS
- 2 to analyser
- 3 sample

Figure 1 — Schematic outlines of experimental configurations for AES and XPS with a focused incident beam

Figure 1 also indicates an XPS configuration in which the incident X-ray beam is focused to a spot on the sample surface. With a conventional X-ray source and a bent-crystal focusing X-ray monochromator, the FWHM of the focused spot can be less than 10 μm . With a synchrotron source of X-rays and a zone-plate, the FWHM of the focused spot can be less than 100 nm [11]. The lateral resolution is determined by the FWHM of the focused spot. The experimental configurations for AES and XPS in Figure 1 are thus similar in that an incident beam is focused to a small area on the sample surface. Lateral variations of surface composition can thus be detected as the beam is positioned on different regions of interest, is linearly scanned across a selected region, or is rastered to obtain information from a selected area. If the incident beam in Figure 1 is not normally incident on the sample surface, the beam profile will be elliptical instead of circular. In such cases, the lateral resolution will be given by the FWHM of the beam profile in two orthogonal directions (parallel and perpendicular to the plane of incidence).

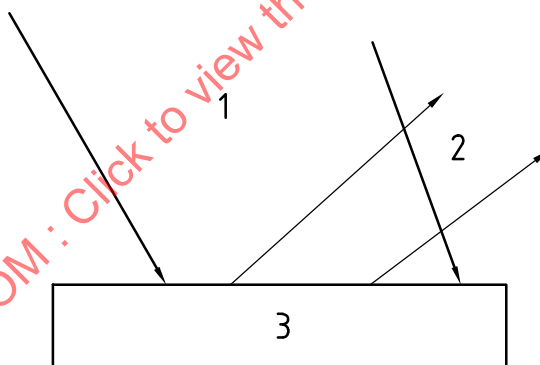
Figure 2a) illustrates an XPS configuration in which the electron energy analyser is part of an electron-optical configuration that views a selected single small area on the sample surface. The lateral resolution for this configuration depends on the electron-optical design and can be less than 10 μm . Figure 2b) shows an XPS configuration in which the electron-optical system produces an image of a selected region of the surface. In this mode, different pixels of the image correspond to particular regions of the surface; information from multiple points on the surface can be recorded in parallel. Figures 2a) and 2b) are similar in that the regions of interest are selected by the electron-optical system. Lateral variations of surface composition can be detected, in principle, by mechanically moving the sample with respect to the analyser or, usually, by adjustment of the electron-optical system to select the particular regions of interest on the sample surface from which photoelectrons are detected. As for Figure 1, photoelectron signals can be obtained from a selected region, from multiple regions along a line, or from multiple regions within a selected area.

Figure 3 shows a simpler XPS configuration in which the sample is irradiated by X-rays from a nearby X-ray source and photoelectrons are detected as in Figure 2 from an area defined by the electron-optical properties of the analyser. Unlike the configurations of Figure 2, however, the instruments represented by Figure 3 were not designed to detect lateral variations of surface composition except by movement of the sample with respect to the analyser. In this way, a lateral resolution of about 0,1 mm to 1 mm could be achieved.

**Key**

- 1 X-ray beam
- 2 to analyser
- 3 sample

Figure 2 — Schematic outlines of XPS configurations in which a) the analyser accepts photoelectrons from a selected area on the sample surface (single-point analysis) or b) the analyser accepts photoelectrons from multiple regions on the sample surface to create an image of the surface (multi-point analysis)

**Key**

- 1 X-ray beam
- 2 to analyser
- 3 sample

Figure 3 — Schematic XPS configuration in which the sample is irradiated by a broad X-ray beam and in which photoelectrons are accepted by the analyser from a larger area of the sample surface than for Figure 2

4.2.2 Lateral resolution for AES

For simplicity in the following discussion, it will be assumed that the sample has a plane surface and that the primary electron beam is normally incident on the sample. It is also assumed that the analysis area is smaller than the sample area viewed by the analyser and that the detection efficiency of the analyser is uniform within the analysis area.

Although the incident electron beam in AES can be focused to a spot with FWHM less than 50 nm, detected Auger electrons originate from ionisations caused by the incident beam and by backscattered electrons [9,12]. Due to multiple elastic- and inelastic-electron scattering, the backscattered electrons can cause inner-shell ionisations that lead to detected Auger electrons from sample regions of up to about 1 µm from the incident-beam spot. The intensity distribution $J_A(r)$ of detected Auger electrons as a function of radius r can be described by the sum of two Gaussian functions [9,12,13]:

$$J_A(r) = (I_i / 2\pi\sigma_i^2) \exp(-r^2 / 2\sigma_i^2) + [(R-1)I_i / 2\pi\sigma_b^2] \exp(-r^2 / 2\sigma_b^2) \quad (1)$$

or

$$J_A(r) = J_{Ai}(r) + J_{Ab}(r) \quad (2)$$

where I_i is the incident beam current, σ_i is the Gaussian parameter describing the radial distribution of the incident electron beam, σ_b is the Gaussian parameter describing the radial distribution of backscattered electrons, R is the backscattering factor, and $J_{Ai}(r)$ and $J_{Ab}(r)$ are the radial intensity distributions for Auger electrons created by the incident beam and by backscattered electrons, respectively. The FWHM values for these two Gaussian functions are $2,35\sigma_i$ and $2,35\sigma_b$, respectively.

Seah [14] has shown that, with 20 keV incident electrons, the FWHM values for backscattered electrons vary between about 0,2 µm and 3,0 µm for different elements; values of σ_b thus range from about 0,085 µm to about 1,3 µm. As an illustrative example, Figure 4 shows a plot of $J_A(r)$ with $\sigma_i = 10$ nm, $\sigma_b = 200$ nm, and $R = 1,5$; for simplicity, $J_A(r)$ has been normalised to unity at $r = 0$. Because $\sigma_b \gg \sigma_i$, $J_{Ai}(r=0)$ is about three orders of magnitude greater than $J_{Ab}(r=0)$ in this example. It is thus possible for the lateral resolution to be determined mainly by the value of σ_i although, as will be shown, the magnitude of $J_{Ab}(r)$ in the vicinity of $r = 0$ also affects the lateral resolution.

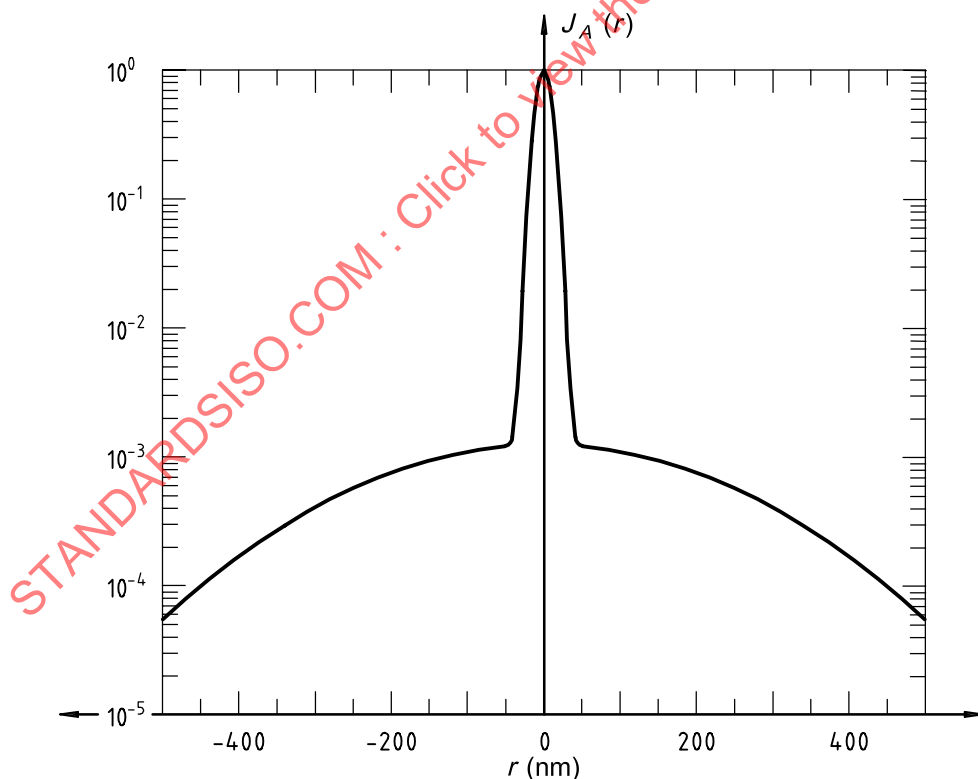
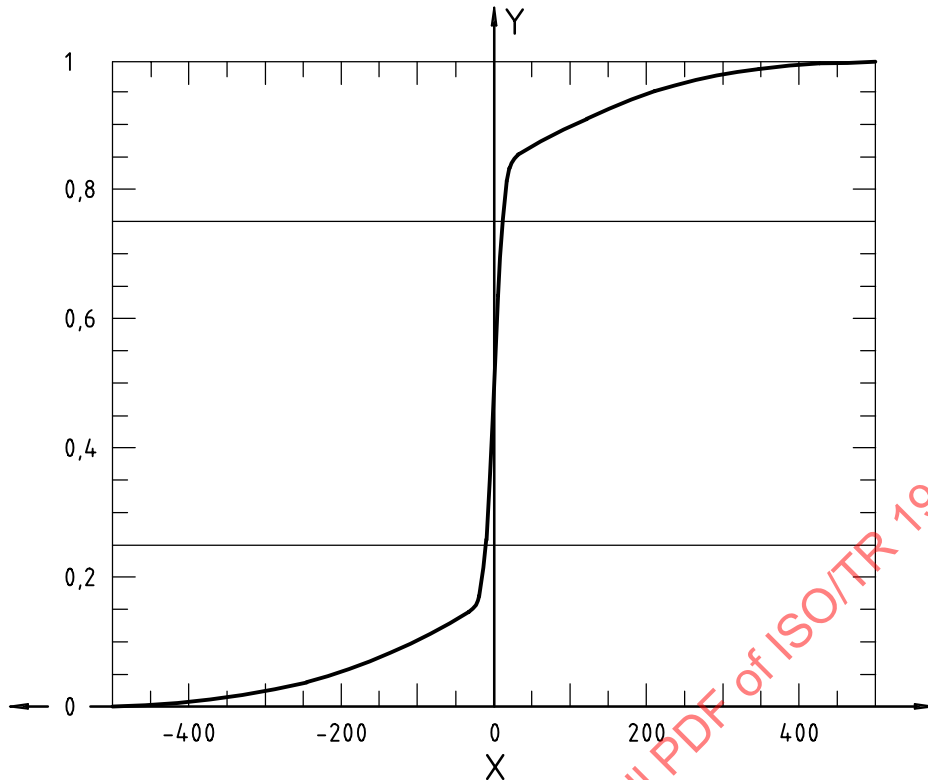


Figure 4 — Plot of the total Auger electron intensity distribution $J_A(r)$ (normalised to unity at $r = 0$) from equation (1) as a function of r with $\sigma_i = 10$ nm, $\sigma_b = 200$ nm, and $R = 1,5$

The lateral resolution in AES has often been determined by scanning the primary electron beam across a sufficiently sharp chemical gradient (a chemical edge) in the plane of the sample; the scan direction is normal to the chemical edge in such measurements. The Auger electron intensity, I , for one of the materials is then measured as a function of beam position on the sample. The lateral resolution, δr , has been variously defined as the distance over which the intensity I changes from 25% to 75% of its maximum value, I_{max} , the distance over which I changes from 20% to 80% of I_{max} , the distance over which I changes from 16% to 84% of I_{max} , the distance over which I changes from 12% to 88% of I_{max} , and the distance over which I changes from 10% to 90% of I_{max} [1, 12].

The intensity distribution of detected Auger electrons shown in equation (1) can also be written in Cartesian coordinates [12,13]. It is then possible to calculate the change in detected Auger intensity as the primary beam is scanned across an abrupt chemical interface as in the experiments. Figure 5 shows a plot of III_{max} as a function of scan distance for the same parameters used in Figure 4. While there is a steep increase in the value of III_{max} in the vicinity of the origin in Figure 5 (corresponding to the primary-beam component $J_{Ai}(r)$ in Figure 4), there are significant tails in the plotted III_{max} due to the backscattered electron component $J_{Ab}(r)$ in Figure 4.

In the example of Figure 5, the measures of the lateral resolution are about 15 nm, 22 nm, 32 nm, 102 nm, and 150 nm for the 25 % to 75 %, 20 % to 80 %, 16 % to 84 %, 12 % to 88 %, and 10 % to 90 % Auger intensity changes, respectively. It is clear that the measure of lateral resolution is mainly determined by the FWHM of the primary beam [that is, the parameter σ_i in equation (1)] if in this example, the measure of lateral resolution is found from the distances corresponding to the 25 % to 75 %, 20 % to 80 %, and 16 % to 84 % Auger electron intensity changes (although, as will be shown shortly, these measures of lateral resolution also depend weakly on σ_b and R). In contrast, the measure of lateral resolution is a strong function of all three parameters in equation (1) (σ_i , σ_b , and R) if the measure of lateral resolution is found from the distances corresponding to the 12% to 88% and 10% to 90% Auger electron intensity changes. Since the values of σ_b and R depend on the sample and the primary electron energy [7],[15], it is desirable for the measure of lateral resolution in AES to be determined in a way that is least dependent on the sample properties. It is therefore recommended that the lateral resolution be obtained from the distances corresponding to the 25% to 75% Auger electron intensity changes in a scan such as that shown in Figure 5.

**Key**

X scan distance (nm)

Y $I/I_{\max}$

Figure 5 — Plot of the ratio of the calculated Auger electron intensity, I , to the maximum Auger electron intensity, $I_{\max}$, as a function of scan distance as the primary beam is scanned across a sharp chemical boundary located at the origin
(with the beam and backscattering parameters used in Figure 4)

(In this example, $I/I_{\max}$ is plotted for the material on the right-hand side of the edge. The horizontal lines show $I/I_{\max} = 0,25$ and $I/I_{\max} = 0,75$. The lateral resolution can be determined from the difference between the scan distances for these values of $I/I_{\max}$; in this example, the lateral resolution is about 15 nm.)

Seah [13] has shown that the measure of the lateral resolution, $\delta r(50)$, corresponding to the 25 % to 75 % change in Auger electron intensity across an abrupt chemical edge can be determined from the relation:

$$0,5R = \text{erf}[z(\sigma_i)] + (R-1)\text{erf}[z(\sigma_b)] \quad (3)$$

where $\text{erf}(z)$ is the error function defined by:

$$\text{erf}(z) = (2/\sqrt{\pi}) \int_0^z \exp(-t^2) dt \quad (4)$$

and where $t = \delta r(50)/\sqrt{2}\sigma_i$ and $t = \delta r(50)/\sqrt{2}\sigma_b$ for the first and second terms in equation (3), respectively. Figure 6 shows plots of $\delta r(50)/\sigma_i$ versus σ_b/σ_i for four values of the backscattering factor R . These plots show that the value of $\delta r(50)/\sigma_i$ does not vary appreciably with σ_b/σ_i when the latter ratio is greater than about 20. The value of $\delta r(50)/\sigma_i$ does, however, depend on R although $\delta r(50)/\sigma_i$ is between 1,35 (when $R = 1$) and about 3,1 (when $R = 1,8$). If, however, the measure of the lateral resolution was determined from the 10% to 90% changes in Auger electron intensity across an edge, Cazaux has shown that this measure changes almost linearly with σ_b/σ_i and with a slope that depends on the value of R [12].

The results shown in Figures 4 to 6 were for normal incidence of the primary electron beam. Cazaux [12] has made similar analyses for primary beams at non-normal incidence. The results of these model calculations agree well with experimental measurements and with Monte Carlo simulations of Auger electron production by backscattered electrons [12,16]. Cazaux [9] has also considered the detectability of features in the form of

stripes in the plane of the surface and has examined Auger electron intensity profiles for chemically non-abrupt edges. El Gomati *et al.* [17] have shown the importance of edge effects in Auger electron line profiles when the primary electron beam was scanned across metal lines with sharp rectangular cross sections; good agreement was found between the experimental profiles and those obtained from Monte Carlo simulations.

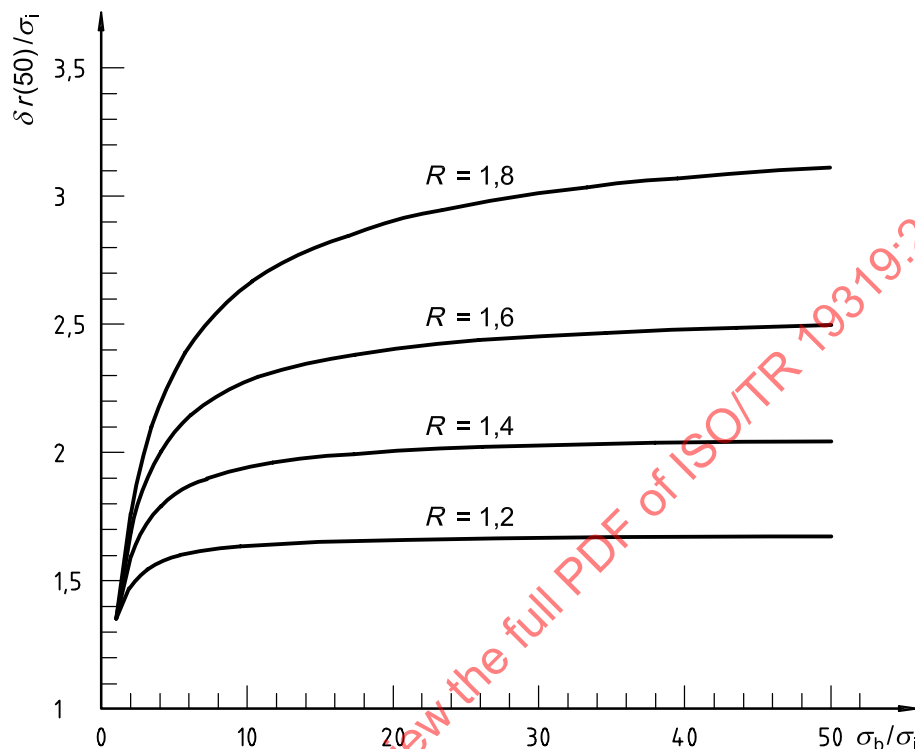


Figure 6 — Plot of $\delta r(50)/\sigma_i$ versus σ_b/σ_i for the indicated values of the backscattering factor R

4.2.3 Lateral resolution for XPS

The lateral resolution for XPS can be described in a similar way as for AES, except there is no backscattering effect. A single Gaussian function can be used to describe the intensity-position distribution of a focused X-ray beam on the sample surface for an XPS instrument of the type illustrated in Figure 1 or the intensity-position response of the electron-optical system for an XPS instrument of the types shown in Figure 2.

Baer and Engelhard [18] reported measurements of analysis areas (discussed in 4.3.3) for two XPS instruments, one having a focused X-ray beam incident on the sample surface as in Figure 1 and the other having an electron-optical system to select a small area of interest on the sample surface as in Figure 2a). They measured the lateral resolution δr of their instruments to be between 9 μm and 200 μm for different instrumental settings from the distances between the 16% and 84% intensity points across an edge between two materials of different compositions. For these instruments, the lateral resolution could be described by a Gaussian function although other functions [a Lorentzian function, a function with a constant central intensity and sharp edges, a function with a $1/(1+|r|^3)$ intensity distribution, and a function with a constant intensity for small radii and $1/(1+|r|^3)$ tails] gave essentially similar results.

4.3 Analysis area

4.3.1 Introduction

After detection of a feature of interest in an AES or XPS instrument, it is often desired to analyse the AES or XPS data in order to obtain elemental and chemical information on the feature. For such data, it is important to know the analysis area so that the AES or XPS data can be reliably analysed. We now describe the factors that affect the analysis area for the AES and XPS configurations shown in Figures 1 and 2. It will again be assumed that the sample has a plane surface and that the analysis area is smaller than the sample area viewed by the analyser.

4.3.2 Analysis area for AES

Consideration is again given to the Auger electron intensity distribution of equation (1) and the illustrative example in Figure 4 showing $J_A(r)$ versus r for $\sigma_i = 10$ nm, $\sigma_b = 200$ nm, and $R = 1.5$. The ratio of the total Auger electron intensity, I , from a circular area of radius r_{max} to the total Auger electron intensity, I_{max} , from a circular area of infinite radius can be found by integrations of equation (1):

$$\frac{I}{I_{max}} = \frac{\int_0^{r_{max}} r J_A(r) dr}{\int_0^{\infty} r J_A(r) dr} = \{[1 - \exp(-r_{max}^2 / 2\sigma_i^2)] + (R - 1)[1 - \exp(-r_{max}^2 / 2\sigma_b^2)]\} / R \quad (5)$$

Figure 7 shows a plot of I/I_{max} from equation (5) as a function of r_{max} for the same parameter values selected for Figure 4. As expected, the intensity distribution in Figure 7 consists of two regions. Two-thirds of the total intensity is due to Auger electrons created by the primary beam while the remaining one-third is due to backscattered electrons. Approximately 28% of the total intensity comes from an area of radius 10 nm (the value of σ_i in this example), about 59% from an area of radius 20 nm, and about 66% from an area of radius 30 nm. The remaining intensity comes from a much larger area, with 90% from an area of radius about 310 nm, 95% from an area of radius about 390 nm, and 99% from an area of radius about 530 nm. Thus, while the lateral resolution $\delta r(50)$ is about 15 nm for this example, about two-thirds of the total Auger intensity comes from an area of radius 30 nm (double the lateral resolution) while 95% of the total intensity comes from an area of radius 390 nm (26 times the lateral resolution). The intensity from this larger area needs to be considered in interpretations of line scans and of "point" analyses (with the incident beam at a fixed location on the sample surface). In general, the analysis area will depend on the relevant material parameters (σ_b and R) and on the particular percentage chosen in the operational definition for the analysis area (percentages of 90%, 95% and 99% of the total Auger intensity were used as examples here). It should also be emphasised that equation (1) is only expected to be a useful guide when the primary beam is normally incident on the sample surface. For other angles of incidence, analytical expressions [12] can be utilised or Monte Carlo simulations [15-17] can be performed to determine the analysis area. Monte Carlo calculations would be required if the sample of interest consisted of materials with significantly different values of σ_b and R [17]. Finally, the Gaussian expression for the incident-beam profile in equation (1) may not be realistic for some instruments [2].

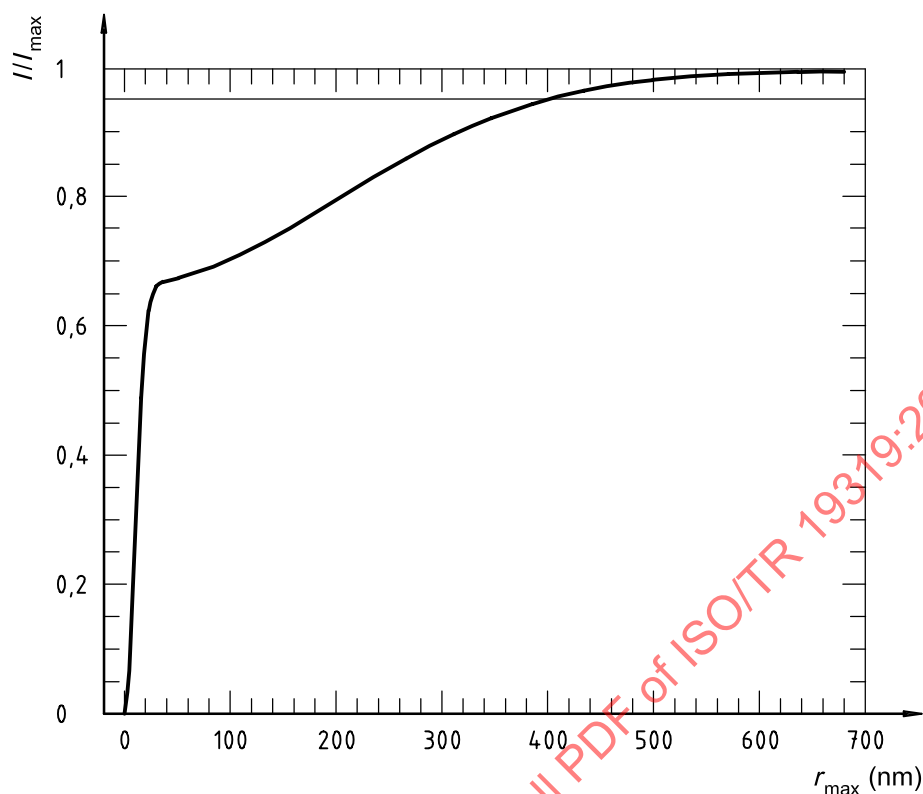


Figure 7 — Plot of $I/I_{\max}$ versus $r_{\max}$ from equations (1) and (5) with the same parameter values as for Figure 4

(The horizontal line shows $I/I_{\max} = 0,95$, for which the corresponding value of $r_{\max}$ is 390 nm.)

4.3.3 Analysis area for XPS

Baer and Engelhard [18] described measurements made with a test sample that had a series of circular spots with diameters between 2 μm and 100 μm . The spots consisted of an indium-tin-oxide coating while the surrounding material was a chromium-containing compound. If the XPS instruments were adjusted to obtain data from the centre of a spot, Baer and Engelhard found that the spot radius had to be about four times δr to obtain 80% of the maximum signal for the spot material that could be measured for much larger spot radii. These results were interpreted in terms of a function describing the spatial distribution of X-ray intensity on the sample surface (for the instrument represented by Figure 1) and a similar function describing the spatial selectivity for the detection of photoelectrons emitted from the sample surface [for the instrument represented by Figure 2a)]. While a Gaussian function has been conventionally used to describe the intensity-position functions for these two types of XPS instruments, Baer and Engelhard found that such a function was inadequate for their instruments. Instead, they were able to describe their spot-intensity measurements with either an $1/(1+|r|^3)$ intensity-position function or a function consisting of a constant intensity for small radii and $1/(1+|r|^3)$ tails. These functions had higher intensities in the tail regions (that is, for $r \gg \delta r$) than a Gaussian function representing the same value of δr . It is thus clear that the analysis area for these instruments would be about $10\pi(\delta r)^2$ if the analysis area was defined to include 94% of the total photoelectron signal. Baer and Engelhard pointed out that the extent of non-Gaussian behaviour (that is, the intensity of the tails in the intensity-position function for an XPS instrument) could be highly dependent upon lens operation and set-up parameters [18].

4.4 Sample area viewed by the analyser

For the instruments represented by Figure 1, the analysis area is defined by the incident electron beam or the incident X-ray beam and, for AES, by sample properties as described in 4.3.1. The electron energy analyser in these instruments is designed to view a larger area of the sample surface so that particular regions of interest of different areas, up to some maximum area given by the analyser design and settings, can be viewed in the imaging or line-scan modes of the instruments. It may be necessary for some applications to measure the sample area viewed by the analyser that can depend on experimental conditions such as electron energy, analyser pass energy, choice of apertures, and sample alignment in the instrument.

The sample area viewed by the analyser is particularly important for XPS instruments represented by Figure 3. The sample surface here is irradiated by a broad X-ray beam (often of about 1 cm diameter), and photoelectrons are detected from a sample area defined by the analyser design, the analyser settings, and the extent of any sample misalignment. For such instruments, the analysis area is the sample area viewed by the analyser.

Three groups have reported measurements of the sample area viewed by the analyser for XPS instruments [19-23]. A focused electron beam from an available electron gun was rastered across the sample surface and measurements were concurrently made of a selected analyser signal, generally the intensity of elastically scattered electrons, as a function of the position of the electron beam on the surface. Measurements of this type have been reported for different types of electron energy analysers, for various analyser settings, and for particular sample misalignments [19-23]. As an example, Figure 8 shows illustrative elastic-peak images for a double-pass cylindrical-mirror analyser operated at electron energies of 100 eV, 500 eV, and 1000 eV [20]. The sample area viewed by the analyser can be determined from these images for a specified percentage of the total analytical signal.

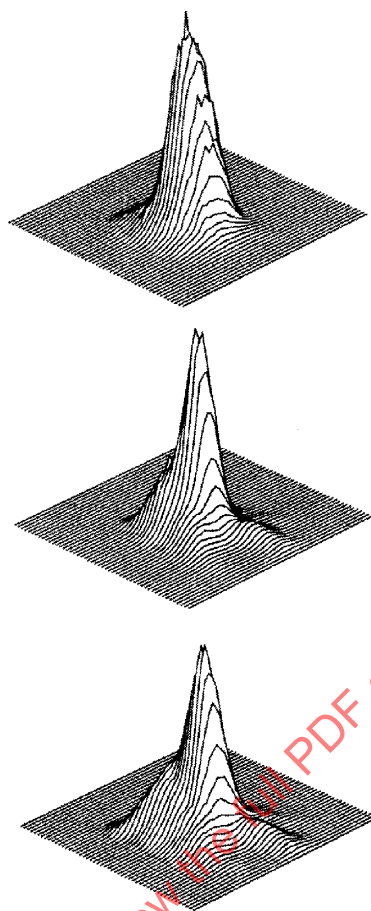
The importance of adequate alignment of the sample surface with respect to the X-ray source and electron energy analyser of an XPS instrument has been pointed out by Seah *et al.* [24]. For some XPS instruments, the sample area viewed by the analyser is independent of the electron energy while for other instruments this area depends on electron energy. In the latter class of instruments, it is important that the sample be aligned correctly at the smallest sample area viewed by the analyser. This condition generally corresponds to the highest electron energy that is to be measured.

5 Measurements of lateral resolution, analysis area, and sample area viewed by the analyser

5.1 General information

As noted in 4.1, the contrast transfer function gives information on the contrast in an image as a function of spatial frequency. In AES and XPS, the contrast transfer function depends in part on instrumental characteristics (e.g., the distribution of incident electron or X-ray intensity as a function of position on the sample in the focused-incident-beam instruments shown in Figure 1) and in part on properties of a particular specimen that give rise to detectable contrast. Although knowledge of the contrast transfer function would be useful in AES and XPS, information is given here on measurements that give more limited information. These measurements are simpler but nevertheless provide a convenient means for describing instrumental performance as determined with a particular specimen material.

Reimer [7] has discussed aspects of resolution tests in scanning electron microscopy and Cazaux [9,12,25] has described corresponding tests in scanning Auger-electron microscopy. Cazaux [8] also points out that fluctuations in Auger-electron intensity due to variations in sample topography can complicate determinations of lateral resolution. Postek *et al.* [26,27] have developed an objective procedure for determining the "sharpness" of images obtained by scanning electron microscopy. Briefly, a two-dimensional Fourier transform is made of an image, and an evaluation is made of the resulting frequency components. This approach can also be used to check and optimise the focus and astigmatism of the incident electron beam.



NOTE Elastic-peak images were recorded for an analyser pass energy of 50 eV and for electron energies of 100 eV (top), 500 eV (centre), and 1 000 eV (bottom). The horizontal distance scanned by the electron beam on the sample surface (corresponding to the bottom left to right line scan in each image) was 13 mm and the vertical distance was 15 mm.

Figure 8 — Examples of electron elastic-peak images obtained with a double-pass cylindrical-mirror electron energy analyser in an XPS instrument [20]

5.2 Lateral resolution

Measurements of lateral resolution on AES and XPS instruments represented by Figures 1 and 2 can be made with test samples having known lateral dimensions such as electron microscope grids. Other suitable test samples are gold islands on a carbon substrate or distinct edges or steps between two different materials. The gold islands/carbon substrate test sample is attractive for AES because the effects of backscattered electrons on the lateral resolution should be negligible with a substrate of low atomic number. Compositional gradients (in the plane of the sample surface) of the test samples should occur over lateral distances much smaller than the expected lateral resolution.

Typically, measurements of particular Auger electron or photoelectron intensities are made as a function of lateral position in the line-scan mode. The lateral resolution can then be determined from the selected definition of lateral resolution. It should be remembered that the measured line scans are the result of a convolution of an instrumental property (the function describing the intensity-position response for the selected conditions) and a material property (the function describing composition change with respect to position on the test sample). The measured lateral resolution will therefore give useful information on the instrumental function only if the composition change in the test sample occurs over distances much smaller than δr . It should also be remembered that an AES or XPS instrument may have aberrations (for example, astigmatism) or other conditions (associated with, for example, inadequate alignment, stray fields, or manufacturing tolerances) that lead to axial asymmetry in its intensity-position response function and to deviations of this