TECHNICAL REPORT



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

iTeh STANDARD PREVIEW Analyse chimique des surfaces — Spectroscopie des électrons Auger

Sectroscopie 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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Contents

Forev	vord	iv
Introd	Juction	v
1	Scope	1
2	Terms and definitions	1
3	Symbols and abbreviated terms	2
4	Background information on lateral resolution, analysis area, and sample area viewed by the analyser	2
4.1 4.2	General information	2 3
4.3 4.4	Analysis area Sample area viewed by the analyser	10 12
5	Measurements of lateral resolution, analysis area, and sample area viewed by the analyser	12
5.1 5.2	General information	12 13
5.3	Analysis area ITED STANDARD PREVIEW	14
5.4 Biblic	Sample area viewed by the analyser. (standards.iteh.ai)	14 16

<u>ISO/TR 19319:2003</u> https://standards.iteh.ai/catalog/standards/sist/83099b06-c4da-444e-92b6d7f75ce7e41e/iso-tr-19319-2003

Foreword

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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.

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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 <samples" 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 <spectrometers" 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.

ISO/TR 19319:2003

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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

ISO/TR 19319:2003(E)

Symbols and abbreviated terms 3 AES Auger electron spectroscopy erf error function FWHM full width at half maximum Auger electron intensity 1 incident beam current (in AES) I_i maximum Auger electron intensity I_{max} intensity distribution of detected Auger electrons as a function of the radius r $J_A(r)$ intensity distribution of detected Auger electrons that were created by backscattered electrons as a $J_{Ab}(r)$ function of the radius r intensity distribution of detected Auger electrons that were created by the incident beam as a function $J_{Ai}(r)$ of the radius r R backscattering factor (in AES) radius from the centre of the incident electron beam on the sample surface (in AES) r upper limit of integration in equation (5) r_{max} ISO/TR 19319:2003 XPS X-ray photoelectron spectroscopy https://standards.iteh.ai/catalog/standards/sist/83099b06-c4da-444e-92b6d7f75ce7e41e/iso-tr-19319-2003 δr lateral resolution $\delta r(50)$ lateral resolution determined from a 25% to 75% intensity change in a line profile Gaussian parameter describing the radial distribution of backscattered electrons (in AES) $\sigma_{\rm b}$ Gaussian parameter describing the radial distribution of the incident electron beam (in AES) σ_i

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. DARD PREVIEW

4.2 Lateral resolution

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4.2.1 Introduction

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It is clearly desirable that the lateral **resolution of the technique** be smaller than the lateral dimensions of the feature of interest in order that the feature can be 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 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 μ m. 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 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 FHWM 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 μ m. 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.



a) XPS with single-point analysis



b) XPS with multi-point analysis

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.