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Surface chemical analysis — Auger electron spectroscopy — Description of selected instrumental performance parameters

Analyse chimique des surfaces — Spectroscopie d'électrons Auger — Description de certains paramètres relatifs à la performance instrumentale

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ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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The committee responsible for this document is ISO/TC 201, *Surface chemical analysis*, Subcommittee SC 7, *Electron spectroscopies*.

This second edition cancels and replaces the first edition (ISO 15471:2004), of which it constitutes a minor revision with the following modifications:

- addition of a Bibliography;
- minor editorial changes to the text.

Introduction

Auger electron spectrometers (AESs) and scanning Auger electron microscopes (SAMs) are produced by many manufacturers throughout the world. While the basic principles of the AES analytical method in each instrument are the same, the specific designs of the instruments and the way that performance specifications are provided differ widely. As a result, it is often difficult to compare the performance of instruments from one manufacturer with those from another. This International Standard provides a basic list of items devised to enable all Auger electron spectrometers to be described in a common manner. This International Standard is not intended to replace the manufacturer's specification, which may extend to 30 or more pages. It is intended that, where certain items are contained in that specification, there are agreed and defined meanings to those items.

Surface chemical analysis — Auger electron spectroscopy — Description of selected instrumental performance parameters

1 Scope

This International Standard specifies the requirements for the description of specific aspects of the performance of an Auger electron spectrometer.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 18115-1, *Surface chemical analysis — Vocabulary — Part 1: General terms and terms used in spectroscopy*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 18115-1 apply.

4 Symbols and abbreviated terms

AES	Auger electron spectroscopy (also Auger electron spectrometer)
FWHM	full width at half maximum
rms	root mean square
SAM	scanning Auger electron microscope (also scanning Auger electron microscopy)
SEM	scanning electron microscope
FL	Fermi level
VL	vacuum level

NOTE Historically, the kinetic-energy scales of AES instruments have been referred to the VL while XPS or combined AES/XPS instruments have been referred to the FL. Conversion from FL to VL referencing is accomplished by subtracting the spectrometer work function from the electron kinetic energies; an approximate means for doing this, satisfactory for most practical AES and SAM applications, is to subtract 4,5 eV from kinetic energies referred to the FL.

5 Description of selected instrumental performance parameters

5.1 Method of analysis

A short description of the methods used to obtain information from the sample shall be given and the availability (as an option) of other analytical techniques in the system under consideration shall be stated.

5.2 Samples

The size and shape of samples that may be analysed with the instrument performing to specification shall be given. If the size or shape is restricted for particular modes of analysis, e.g. angle-resolved measurements, measurements for insulators, etc., this shall be specified.

5.3 System configuration

The designed geometric configuration of the significant analytical components of the system and their tolerances shall be described.

EXAMPLE Tolerances for angles are often given as ±1°.

5.4 Electron gun cathode

5.4.1 Cathode type

The cathode system shall be specified.

EXAMPLE Thermionic tungsten, lanthanum hexaboride (LaB_6), cold field emission tungsten (110), or Schottky.

5.4.2 Cathode lifetime

The expected lifetime of the cathode under the operating conditions specified in 5.5 shall be stated. The emission currents at chosen source potentials in 5.5 shall be stated. This would normally be a guarantee of lifetime operation but could, alternatively, be a mean historical lifetime. The type of lifetime shall be specified.

5.5 Lateral resolution and beam current

5.5.1 General

Lateral resolutions shall be specified for the following:

a) SEM acquired at optimum operating conditions;

b) AES at specified conditions for each defined beam energy.

The measured value of the spatial resolution shall be obtained by one of the methods in 5.5.2, 5.5.3 or 5.5.4. Curves giving the typical lateral resolution as a function of beam current at 5 keV and/or 10 keV and any other appropriate beam energy shall be given.

NOTE If an instrument has a lateral resolution function that can be represented by a Gaussian function, then the FWHM of such a function corresponds to the distance over which the measured signal changes from 12 % to 88 % of its maximum value. In AES, the point spread function for the emitted Auger electrons is the Gaussian distribution of the beam superimposed on a backscattered halo. For this reason, it is convenient to define the lateral resolution as the distance corresponding to a 20 % to 80 % change in the Auger signal across the step edge, which is equivalent to 71,5 % of the Gaussian resolution function. Although there is no physical basis for this choice, it has been widely used. Lateral resolution methods are reported in ISO/TR 19319.

5.5.2 Method 1

A sample shall be analysed which has an isolated feature whose size is smaller than 30 % of the instrument's stated spatial resolution. The measured FWHM of a line trace for an Auger electron signal characteristic of that feature defines the spatial resolution. The distance for the feature signal to rise from 50 % of the maximum to 100 % and then fall again to 50 % defines the measured spatial resolution.

NOTE 1 If the width of the isolated feature is greater than 30 % of the spatial resolution, the measured spatial resolution will be greater than the true spatial resolution.

NOTE 2 The use of a small sample allows easy confirmation of the system astigmatism.

5.5.3 Method 2

A sample shall be analysed which is comprised of two materials with their surfaces in the same plane and joined along a common straight edge. A line trace for an Auger electron intensity, characteristic of one of the two materials, measured at 90° to the edge, is used to define spatial resolution. The distance for the Auger electron intensity to change from 20 % to 80 % of the difference in the intensities in the plateau regions away from the edge defines the spatial resolution in the direction of the scan.

NOTE 1 If an instrument has a spatial resolution function that can be represented by a Gaussian function, then such an intensity distance distribution is equivalent to 71,5 % of the FWHM of the spatial resolution function of the instrument.

NOTE 2 Close to the limit of resolution, astigmatism can be observed and so the spatial resolution might need determination in more than one azimuth.

5.5.4 Method 3

A sample shall be analysed which is composed of a knife edge of one material, in the sample plane, over a hole with a depth more than five times its diameter. A line trace for an Auger electron intensity, characteristic of the knife edge material, measured at 90° to the edge, is used to define spatial resolution. The distance for the Auger electron intensity to change from 20 % to 80 % of the difference in the intensities in the plateau regions away from the edge defines the spatial resolution in the direction of the scan.

NOTE 1 If an instrument has a spatial resolution function that can be represented by a Gaussian function, then such an intensity distance distribution is equivalent to 71,5 % of the FWHM of the spatial resolution function of the instrument.

NOTE 2 Close to the limit of resolution, astigmatism can be observed and so the spatial resolution might need determination in more than one azimuth.

5.6 Spectrometer intensity performance and energy resolution

5.6.1 General

The spectrometer intensity performance is determined from the difference between the intensity (counting rate) of the Cu L_3VV peak at 918 eV and the background intensity (counting rate) at 950 eV (both measured in the direct mode). Performance shall be specified in pulse-counting systems as the difference in counting rates per nA of beam current or, alternatively, as the difference in counting rates at specified beam energy and beam current for (a) optimum energy resolution and (b) optimum sensitivity. If the spectrometer can be operated at different energy resolutions, the performance, the background intensity and the FWHM of the peak above background for each energy resolution shall be given at at least one beam energy. The beam current shall be given for each beam energy. The signal-to-noise ratio shall be defined as the ratio of the spectrometer intensity performance, obtained using data-acquisition time(s) of 1 s at 918 eV and 950 eV, to the noise obtained from 5.6.2 or 5.6.3. The method by which the noise is measured shall be stated.

5.6.2 Method 1

The noise is defined as the rms deviation of 121 separate measurements of the background intensity measured at regularly spaced energies between 970 eV and 994 eV using data-acquisition time(s) of 1 s for each measurement (channel) and approximately 0,2 eV channel intervals. The rms deviation is calculated from the deviations of the measured intensities from a least-squares fit of a straight line to the measured intensities.

5.6.3 Method 2

The noise is defined as the rms deviation of 121 samples of the background intensity at 975 eV, each measured using a 1 s data-acquisition time, from the mean value of those measurements.

5.7 Spectrometer energy scale

The energy scale shall be specified with reference to the Fermi level and shall include specifications for the scale linearity, repeatability of the standard deviation of the measured energy of the Cu L_3VV peak for samples re-positioned by the positioning procedure described in the instrument manual and the accuracy of the calibration as a function of time at the energy of the Cu L_3VV peak.

NOTE A method for calibrating the energy scale of medium-energy resolution Auger electron spectrometers is available as ISO 17973, while a method for calibrating the energy scale of high-energy resolution spectrometers is available as ISO 17974.

5.8 Spectrometer intensity linearity

The maximum useful count rate and the maximum count rate for a defined limit of count rate linearity, such as ±2 %, shall be stated.

5.9 Spectrometer response function

The analyser response function shall be provided for the relevant operating modes for which intensity performance is given. The extent to which these functions remain constant with time shall be stated.

5.10 Spectrometer parameters

5.10.1 Spectrometer aberration

The effects of the unwanted spectrometer dispersion (aberration) expressed as the measured shift of the Cu L_3VV peak at approximately 918 eV in eV per mm movement of the beam impact point on the sample in the X, Y and Z directions shall be specified.

5.10.2 Analytical area

The analytical area, which may be determined by either the raster area of the incident beam on the sample surface or the spectrometer field of view, shall be specified.

NOTE The analytical area is here defined as the area over which the intensity remains above 95 % of the peak intensity for both the Cu L_3VV peak, at approximately 918 eV, and the Cu $M_{23}VV$ peak at approximately 62 eV.

5.11 Image drift

For imaging systems, the image drift rates shall be specified for a specified installation environment and if software driven drift correction is used.

5.12 Vacuum environment

The base pressure attainable 24 h after a 12 h bake at a specified temperature or the time to reach the specified base pressure shall be given. The sample introduction time for analysis at a specified pressure shall also be given.

Bibliography

[1] ISO 17973, *Surface chemical analysis—Calibration of the energy scales for elemental analysis*

[2] ISO 17974, *Surface chemical analysis — High-resolution Auger electron spectrometers — Calibration of energy scales for elemental and chemical-state analysis*

[3] ISO/TR 19319, *Surface chemical analysis — Fundamental approaches to determintaion of lateral resolution and sharpness in beam-based methods*

[4] ISO 21270, *Surface chemical analysis — X-ray photoelectron and Auger electron spectrometers — Linearity of intensity scale*

[5] ISO 24236, *Surface chemical analysis — Auger electron spectroscopy — Repeatability and constancy of intensity scale*

[6] ISO 29081, *Surface chemical analysis — Auger electron spectroscopy — Reporting of methods used for charge control and charge correction*

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