BS ISO 16242:2011

BSI Standards Publication

Surface chemical analysis — Recording and reporting data in Auger electron spectroscopy (AES)

National foreword

This British Standard is the UK implementation of ISO 16242:2011.

The UK participation in its preparation was entrusted to Technical Committee CII/60, Surface chemical analysis.

A list of organizations represented on this committee can be obtained on request to its secretary.

ISBN 978 0 580 66875 3

ICS 71.040.40

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 31 December 2011.

Amendments issued since publication

Date	Text affected

INTERNATIONAL
STANDARD

ISO
16242

First edition
2011-12-01

Surface chemical analysis — Recording and reporting data in Auger electron spectroscopy (AES)

Analyse chimique des surfaces — Enregistrement et notification des données en spectroscopie des électrons Auger (AES)

Reference number
ISO 16242:2011(E)

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Published in Switzerland

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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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 16242 was prepared by Technical Committee ISO/TC 201, *Surface chemical analysis*, Subcommittee SC 2, *General procedures*.

Introduction

Auger electron spectroscopy is used for experimental and routine surface analysis of a wide range of test specimen types. The results of the analysis should be recorded in a standard format that should include sufficient detail to allow the experiment to be repeated. This material should be available for reporting, as required.

Experimental conditions and data acquisition parameters should be included so that the quality of the data can be assessed.

Surface chemical analysis — Recording and reporting data in Auger electron spectroscopy (AES)

1 Scope

This International Standard specifies the minimum level of information to be reported by the analyst following the analysis of a test specimen using Auger electron spectroscopy (AES). It includes information that is to be recorded on or in the analytical record.

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 17973, *Surface chemical analysis — Medium-resolution Auger electron spectrometers — Calibration of energy scales for elemental analysis*

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

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 and the following apply.

3.1
ex situ
outside the analytical system

3.2
in situ
inside the analytical system

4 Symbols and abbreviated terms

AES	Auger electron spectroscopy
KLL	shorthand notation for an Auger line resulting from an electron hole in the K shell, electron transfer from the L shell to the K shell vacancy and simultaneous emission of the Auger electron from the L shell
KVV	shorthand notation for an Auger line resulting from an electron hole in the K shell, electron transfer from a valence band (“V”) to the K shell vacancy and simultaneous emission of the Auger electron from the valence band

Similar notation is used for the L, M and further Auger series.

5 Levels of recording and reporting

5.1 General

This International Standard defines the minimum level of information that shall be recorded and reported by an analyst following the analysis of a test specimen using AES. The levels of recording and reporting are separated into six main areas:

a) the analyst's record book or electronic log (e.g. computer data-storage system);

b) spectra;

c) quantitative analysis of the specimen;

d) compositional depth profiles;

e) element maps, linescans or images;

f) chemical information obtained from the specimen.

5.2 Analyst's record

5.2.1 Specimen identification and preparation

For each individual specimen, the record book or electronic log shall contain the following information (sufficient information shall be recorded to allow the measurements to be repeated at a later date):

a) the name and contact address of the laboratory or the individual providing the specimen, with electronic address and telephone number when available;

b) a unique specimen number;

c) a description of the specimen before and after analysis (including details of its physical appearance, its roughness, its colour and any other distinguishing features);

d) the date of the measurement(s);

e) the name of the analyst, and the analyst's department and affiliation;

f) all details concerning *ex situ* specimen preparation before analysis (including details of the method of mounting by tape or clip, the orientation on the specimen holder with respect to any specific surface features, whether the specimen was cut and, if so, how, details of any solvent-cleaning, etc.) (see NOTE 1);

g) all details concerning *in situ* specimen preparation before analysis (relevant details include inert ion cleaning, specimen heating, fracture, etc.) (see NOTE 2).

NOTE 1 Guidelines for preparation and mounting of specimens are given in ISO 18116.

NOTE 2 Handling of specimens prior to analysis is described in ISO 18117.

5.2.2 Analytical conditions

A detailed list of the analytical conditions shall be recorded in the record book and/or the electronic log (sufficient information shall be recorded to allow the measurements to be repeated at a later date). The information shall include:

a) the name or identification of the equipment used;

b) the primary electron beam energy and the angle of incidence;

c) the specimen current or beam current;

d) the type of detector, the number of detectors and, in the case of multichannel detectors, the working mode;

e) the analyser energy resolution;

f) the spectrometer retardation ratio or pass energy, and slit widths, if applicable;

g) the take-off angle used for the measurement;

h) the pressure of the analysis chamber before and during the analysis;

i) the beam diameter under the analytical conditions if pertinent to the analysis, e.g. for imaging or mapping (if the beam is scanned during acquisition, state over what distance);

j) the start energy;

k) the end energy or scan width and the number of cycles;

l) the number of data points, expressed as an integer or as volts/step, and the width of the energy channel;

m) the acquisition time, expressed as the time/step or as a total time indicating the measurement time and the beam exposure time;

n) when data are collected in an analogue differential mode, the analogue modulation.

All the above information shall be subsequently given to the customer, if requested, along with the analysis of the AES data by the instrument operator. The customer and the analyst will define the format used to transfer this information. For example, the experimental information may be contained in the appendix or the experimental section of a report.

The kinetic-energy scale of the Auger electron spectrometer shall be calibrated in accordance with either ISO 17973 or ISO 17974 or in accordance with the manufacturer's documented calibration procedure.

5.3 Spectra

All AES spectra supplied to a customer shall include the following minimum information:

a) peak or region labels, e.g. Cu KLL;

b) an abscissa label, e.g. kinetic energy, E (eV);

c) abscissa tic marks showing the energy as width of scan, e.g. 0 eV to 1 200 eV, or as energy/division;

d) an ordinate label which, for direct spectra, might be e.g. counts/s or counts and, for differentiated data, $\mathrm{d}C/\mathrm{d}E$, where C = counts, with a note if a transmission function correction has been applied;

e) for direct spectra, ordinate tic marks, showing intensity as counts/s or counts per division (see the Note);

f) details of all the data-processing functions applied to the raw spectrum, for example differentiation, smoothing, transmission function correction, spike removal.

NOTE For differentiated spectra, tic marks are appropriate, but the axis is, nominally, unitless.

Further information may be included at the discretion of the analyst or at the request of the customer. Examples of spectra are shown in Annex A (see Figures A.1 to A.4).

5.4 Quantitative information

When the AES data are processed and supplied to a customer as quantitative data, the following information on the method of quantification shall also be available to the customer, if required:

a) the quantification model, e.g. homogeneous solid, homogeneous solid under a contamination layer, layered solid;

b) the intensity parameter chosen, e.g. peak area, peak height, differential peak-to-peak height, differential peak-to-background height (see the Note);

c) details of the elements included in the quantification and those deliberately excluded;

d) the type of background fitted to the data, with start and end points, if appropriate;

e) the analyser transmission function and sensitivity factors used and their source, e.g. manufacturers', in-house standards, theory);

f) any other correction terms used and their justification, e.g. dead time, spectrometer energy window, specimen roughness, matrix effects);

g) details of data-processing techniques applied, such as factor analysis or least-squares fitting;

h) an estimate of the errors in quantification, as discussed for example in ISO 20903.

NOTE Methods used to determine peak intensities and information required when reporting results are described in ISO 20903.

5.5 Compositional depth profiles

Compositional depth profiles may be presented as a montage of spectra or as depth profiles derived from peak area or peak-to-peak measurements on derivative spectra. If the data are quantified, the factors listed in 5.4 shall be considered and noted where appropriate. In addition, the method of quantification of the depth of analysis of ion-sputtered depth profiles (which may be estimated with respect to the sputter rate of a standard material, such as thin films of Ta_2O_5 on Ta or SiO_2 on silicon, under the same conditions or measured directly by profilometer) shall be recorded (see Annex A, Figures A.5 and A.6).

NOTE The measurement of sputtered depth in depth profiling is described in ISO/TR 15969.

Where depth profiles are acquired from taper sections or dimpled craters, the depth can be estimated by geometrical calculation.

The following information shall be available to be supplied with the compositional profiles, if required:

a) an ordinate label, e.g. the atomic fraction (together with details of quantification as outlined in 5.4), or nominal counts if direct spectra are used;

b) ordinate tic marks showing the atomic fraction or, for direct spectra, counts or simply a scale in arbitrary units;

c) an abscissa label, i.e. sputtering time or estimated depth;

d) abscissa tic marks showing time or depth;

e) for ion-sputtered profiles, the sputtering rate and the sputtering-rate calibration material used;

f) details of the ion beam energy and current;

g) details of the specimen area sputtered and the dimensions scanned, where appropriate.

5.6 Maps and linescans

When Auger maps or linescans (see Annex A, Figure A.7) are provided, the following additional information shall be supplied to the customer, as required (if the information is provided as part of a depth profile, also include the details requested in 5.5):

a) the identity of the mapped AES peak and its energy, together with details of any background removal procedure;

b) details of the map field of view or any scale marker included (note that it is preferable to include information for both X and Y directions as these are frequently not equivalent; many current systems do not include Y direction information and this shall be reported);

c) details of the map field of view calibration in the X and Y directions;

d) details of the property mapped, e.g. atomic percentage, together with a scale indicating the colour, if appropriate.

5.7 Chemical-state data

If the Auger electron spectra are analysed for chemical-state information, the following experimental information shall be recorded and be available to the customer, if required:

a) the scan width, in eV;

b) the eV per channel;

c) the FWHM (full width at half maximum) of the peaks, if measured;

d) the energy reference used;

e) details of the FWHM of peaks in the spectra of the reference materials analysed during the calibration process using the same analytical conditions;

f) details of the method of peak synthesis, the algorithm used, the method used to define the synthetic envelope and clearly labelled peak components.

NOTE Derivation of chemical information is described in ISO/TR 18394.

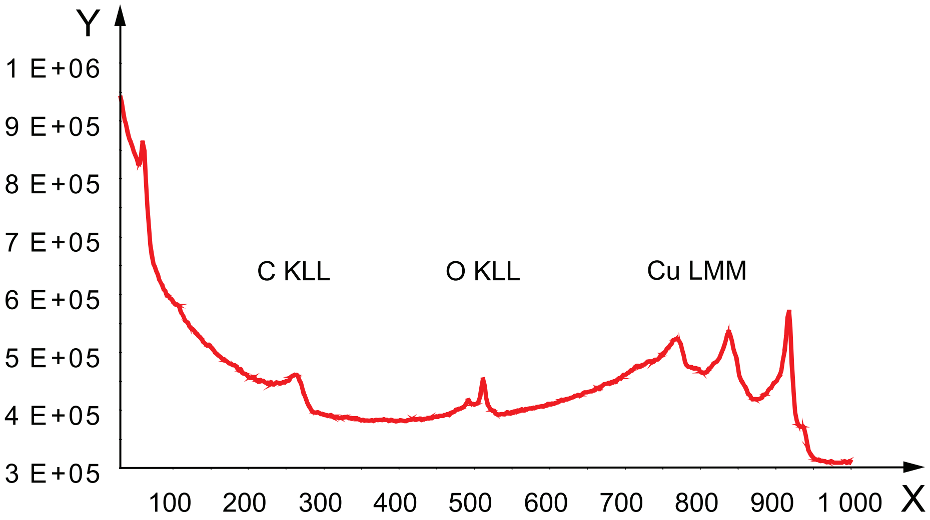
6 Release of data to the customer

This will be covered by the relevant International Standard on reporting results or suitable in-house procedures, but shall include, as a minimum, the name and signature (or other identifying mark or stamp) of the analyst and the date of issue.

Annex A
(informative)

Examples of spectra

Figures A.1 to A.4 are examples of spectra showing data acquired from copper before and after ion sputtering. Figures A.5 and A.6 are examples of an Auger depth profile, while Figure A.7 is an example of a set of Auger composition maps.

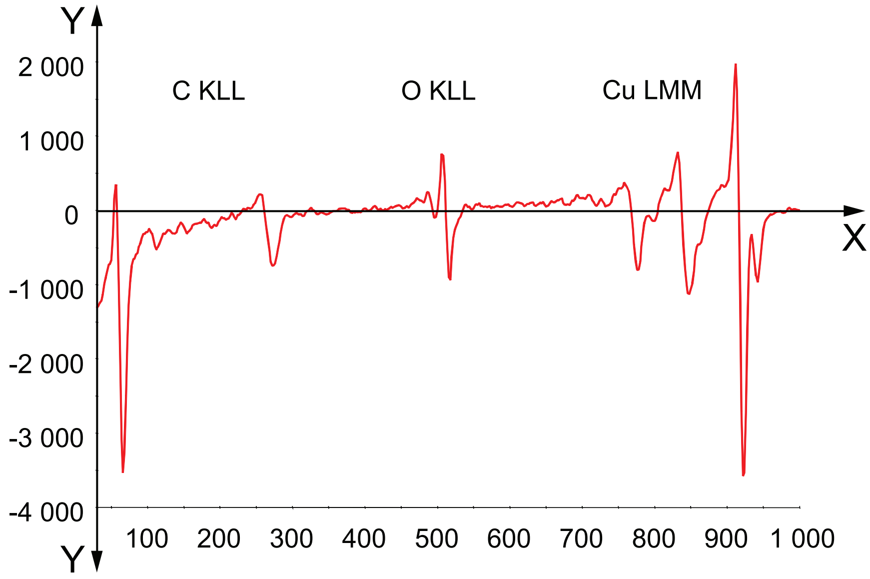


Key

X kinetic energy (eV)

Y counts/s

Figure A.1 — Before ion beam sputtering — Direct spectrum

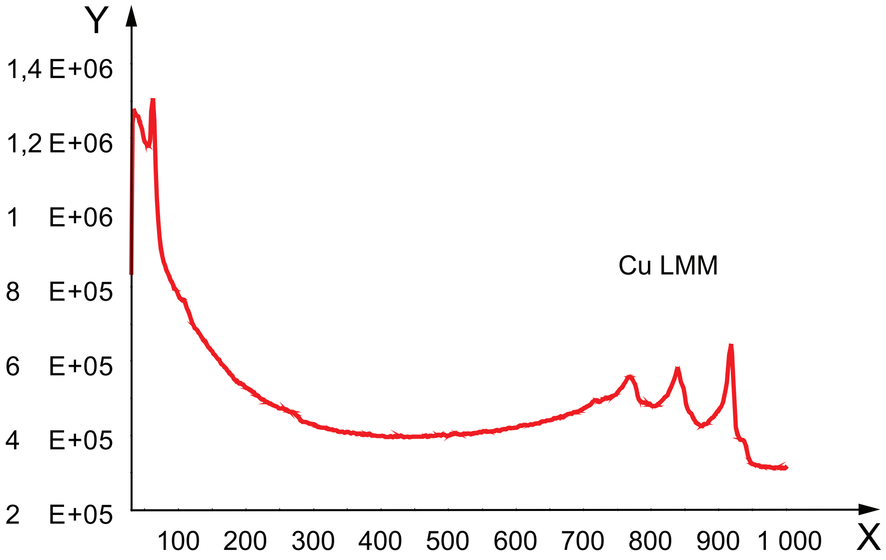


Key

X kinetic energy, E (eV)

Y dC/dE, where C = counts

Figure A.2 — Before ion beam sputtering — Differentiated spectrum

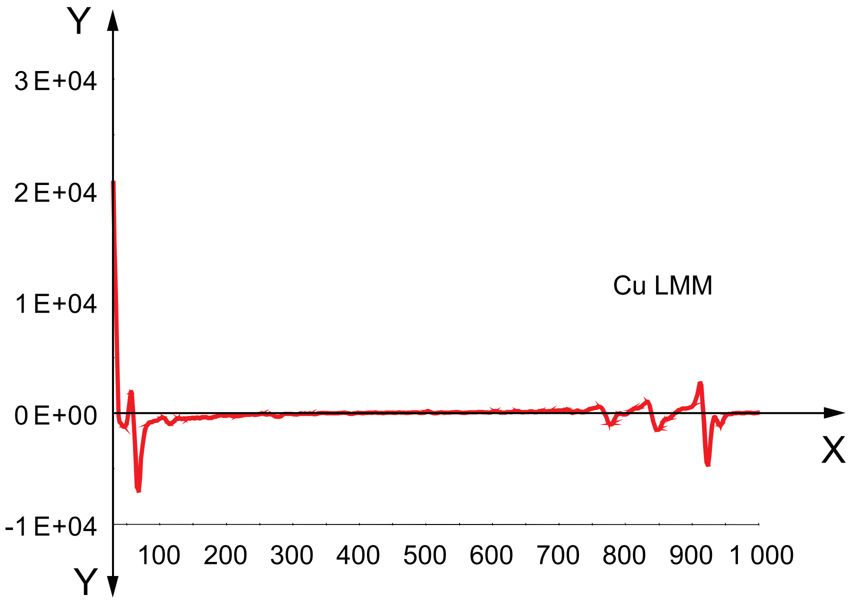


Key

X kinetic energy (eV)

Y counts/s

Figure A.3 — After ion beam sputtering — Direct spectrum

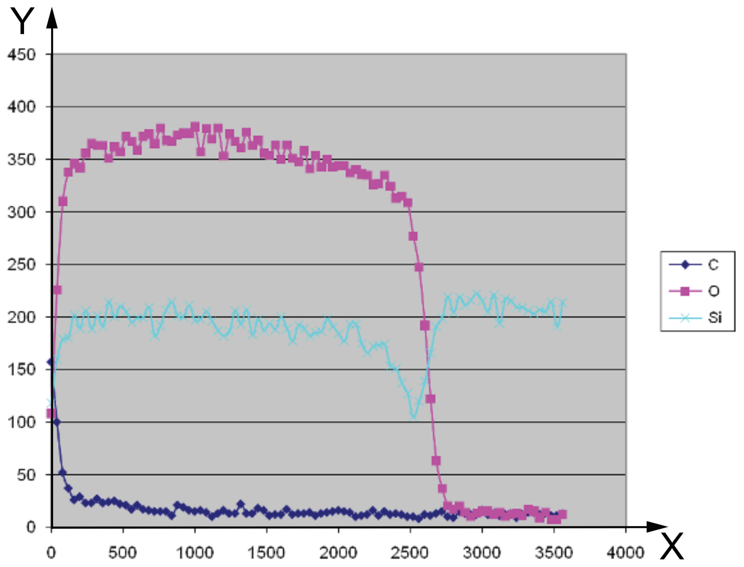


Key

X kinetic energy, E (eV)

Y dC/dE, where C = counts

Figure A.4 — After ion beam sputtering — Differentiated spectrum

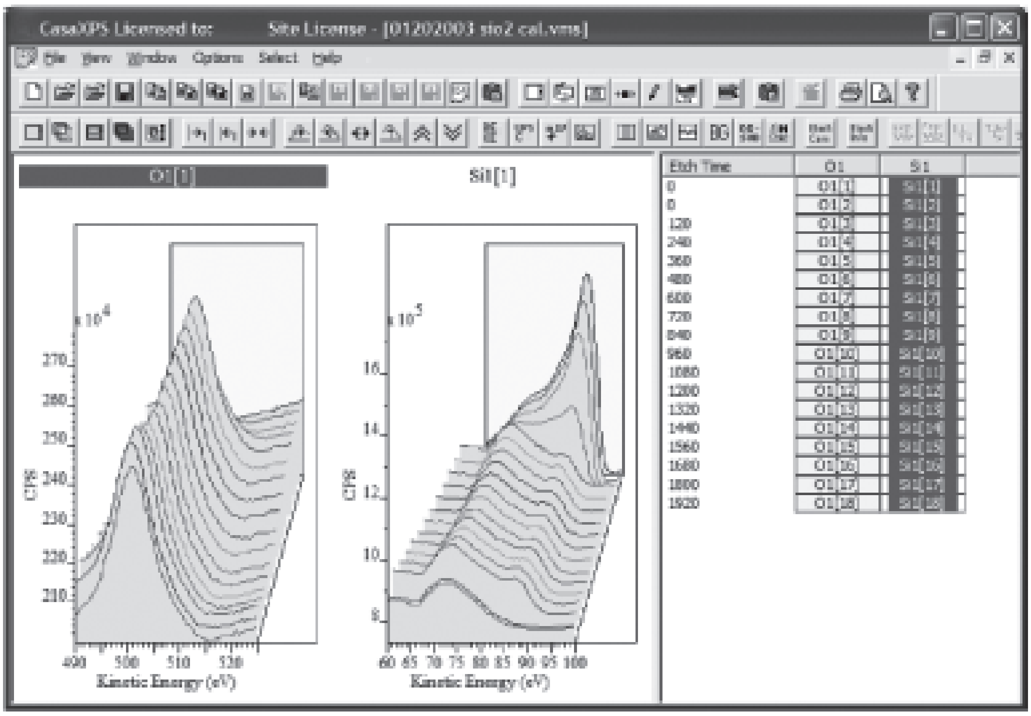


Key

X time (s)

Y counts

Figure A.5 — Auger depth profile through layer of SiO_2 used for calibrating the Ar ion sputtering rate
(The counts are taken from the peak heights in the derivative spectrum.)
(Image reproduced with permission of S. Wight, NIST)



Etch Time	O1	Si1
0	O1[1]	Si1[1]
0	O1[2]	Si1[2]
120	O1[3]	Si1[3]
240	O1[4]	Si1[4]
360	O1[5]	Si1[5]
480	O1[6]	Si1[6]
600	O1[7]	Si1[7]
720	O1[8]	Si1[8]
840	O1[9]	Si1[9]
960	O1[10]	Si1[10]
1080	O1[11]	Si1[11]
1200	O1[12]	Si1[12]
1320	O1[13]	Si1[13]
1440	O1[14]	Si1[14]
1560	O1[15]	Si1[15]
1680	O1[16]	Si1[16]
1800	O1[17]	Si1[17]
1920	O1[18]	Si1[18]

Figure A.6 — Auger depth profile montage of O KLL and Si LVV from SiO_2 on Si
(Image reproduced from the *The CASA Cookbook*[6] with permission of A. Carrick)

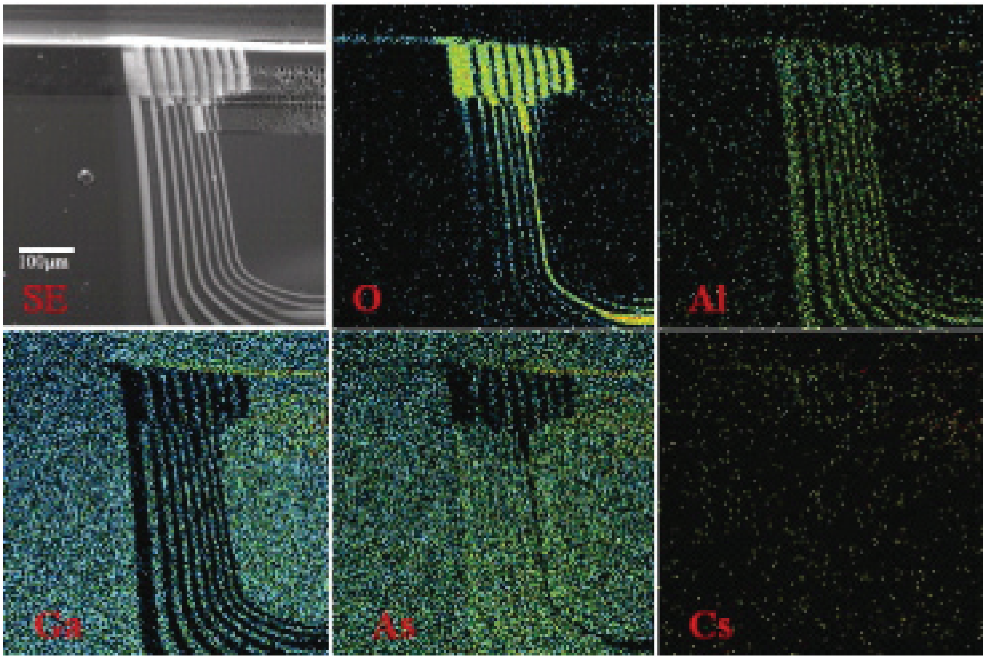


Figure A.7 — Auger maps and secondary-electron (SE) image recorded from an ion-etched crater at the edge of an oxidation trench in a GaAs/AlGaAs multilayer
(Image reproduced with permission of S. Wight, NIST)

Bibliography

[1] ISO/TR 15969, *Surface chemical analysis — Depth profiling — Measurement of sputtered depth*

[2] ISO 18116, *Surface chemical analysis — Guidelines for preparation and mounting of specimens for analysis*

[3] ISO 18117, *Surface chemical analysis — Handling of specimens prior to analysis*

[4] ISO/TR 18394, *Surface chemical analysis — Auger electron spectroscopy — Derivation of chemical information*

[5] ISO 20903, *Surface chemical analysis — Auger electron spectroscopy and X-ray photoelectron spectroscopy — Methods used to determine peak intensities and information required when reporting results*

[6] FAIRLEY, N., and CARRICK, A., *The CASA Cookbook — Part 1: Recipes for XPS Data Processing*, Acolyte Science, Knutsford, UK, 2005, p. 313, ISBN 9780954953300

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