Designation: E1217 – 11

Standard Practice for
Determination of the Specimen Area Contributing to the Detected Signal in Auger Electron Spectrometers and Some X-Ray Photoelectron Spectrometers[1]

This standard is issued under the fixed designation E1217; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice describes methods for determining the specimen area contributing to the detected signal in Auger electron spectrometers and some types of X-ray photoelectron spectrometers (spectrometer analysis area) when this area is defined by the electron collection lens and aperture system of the electron energy analyzer. The practice is applicable only to those X-ray photoelectron spectrometers in which the specimen area excited by the incident X-ray beam is larger than the specimen area viewed by the analyzer, in which the photoelectrons travel in a field-free region from the specimen to the analyzer entrance. Some of the methods described here require an auxiliary electron gun mounted to produce an electron beam of variable energy on the specimen ("electron-gun method"). Other experiments require a sample with a sharp edge, such as a wafer covered with a uniform clean layer (for example, gold (Au) or silver (Ag)) and cleaved to obtain a long side ("sharp-edge method").

1.2 This practice is recommended as a useful means for determining the specimen area viewed by the analyzer for different conditions of spectrometer operation, for verifying adequate specimen and beam alignment, and for characterizing the imaging properties of the electron energy analyzer.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E673 Terminology Relating to Surface Analysis (Withdrawn 2012)[3]

E1016 Guide for Literature Describing Properties of Electrostatic Electron Spectrometers

2.2 *ISO Standards:*[4]

ISO 18115:2001 Surface Chemical Analysis—Vocabulary

ISO 18516:2006 Surface Chemical Analysis – Auger Electron Spectroscopy and X-ray Photoelectron Spectroscopy – Determination of Lateral Resolution

3. Terminology

3.1 *Definitions*—See Terminology E673 and ISO 18115:2001 for terms used in Auger electron spectroscopy and X-ray photoelectron spectroscopy.

4. Summary of Practice

4.1 *Electron-Gun Method*—An electron beam with a selected energy is scanned across the surface of a test specimen. The beam may be scanned once, that is, a line scan, or in a pattern, that is, rastered. As the electron beam is deflected across the specimen surface, measurements are made of the intensities detected by the electron energy analyzer as a function of the beam position for selected conditions of analyzer operation. The measured intensities may be due to electrons elastically scattered by the specimen surface, to electrons inelastically scattered by the specimen, or to Auger electrons emitted by the specimen. The intensity distributions for particular detected electron energy can be plotted as a function of beam position in several ways and can be utilized to obtain information on the specimen area contributing to the detected signal and on analyzer performance for the particular

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[1] This practice is under the jurisdiction of ASTM Committee E42 on Surface Analysis and is the direct responsibility of Subcommittee E42.03 on Auger Electron Spectroscopy and X-Ray Photoelectron Spectroscopy.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The last approved version of this historical standard is referenced on www.astm.org.

[4] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

conditions of operation. This information can be used to determine the analysis area (see Terminology E673 or ISO 18115:2001).

4.2 *Sharp-Edge Method*—A sample with a sharp edge is scanned through the focal area of the analyzer with its sharp edge perpendicular to the scanning direction (*knife edge* experiments). As the sample is moved to different positions, measurements are made of the intensity of a characteristic photoelectron peak of the sample surface (for example, Au 4f peak if the sample was covered with gold) for selected conditions of the analyzer operation. The measured intensity is maximum when the sampled area is completely contained by the sample surface, and minimum when there is no overlap between the analysis volume of the analyzer and the sample surface. The length of the intermediate region will depend on the size of the analysis area. The area of the photoelectron peak can be plotted as a function of sample position. The behavior of this curve can be used to assess the width of the analysis area in the scanning direction.

5. Significance and Use

5.1 Auger electron spectroscopy and X-ray photoelectron spectroscopy are used extensively for the surface analysis of materials. This practice summarizes methods for determining the specimen area contributing to the detected signal (*a*) for instruments in which a focused electron beam can be scanned over a region with dimensions greater than the dimensions of the specimen area viewed by the analyzer, and (*b*) by employing a sample with a sharp edge.

5.2 This practice is intended as a means for determining the observed specimen area for selected conditions of operation of the electron energy analyzer. The observed specimen area depends on whether or not the electrons are retarded before energy analysis, the analyzer pass energy or retarding ratio if the electrons are retarded before energy analysis, the size of selected slits or apertures, and the value of the electron energy to be measured. The observed specimen area depends on these selected conditions of operation and also can depend on the adequacy of alignment of the specimen with respect to the electron energy analyzer.

5.3 Any changes in the observed specimen area as a function of measurement conditions, for example, electron energy or analyzer pass energy, may need to be known if the specimen materials in regular use have lateral inhomogeneities with dimensions comparable to the dimensions of the specimen area viewed by the analyzer.

5.4 This practice can give useful information on the imaging properties of the electron energy analyzer for particular conditions of operation. This information can be helpful in comparing analyzer performance with manufacturer's specifications.

5.5 Information about the shape and size of the area viewed by the analyzer can also be employed to predict the signal intensity in XPS experiments when the sample is rotated and to assess the axis of rotation of the sample manipulator.

5.6 Examples of the application of the methods described in this practice have been published (**1-7**).[5]

5.7 There are different ways to define the spectrometer analysis area. An ISO Technical Report provides guidance on determinations of lateral resolution, analysis area, and sample area viewed by the analyzer in AES and XPS(**8**), and ISO 18516:2006 describes three methods for determination of lateral resolution in AES and XPS. Baer and Engelhard have used well-defined 'dots' of a material on a substrate to determine the area of a specimen contributing to the measured signal of a 'small-area' XPS measurement (**9**). This area could be as much as ten times the area estimated simply from the lateral resolution of the instrument. The amount of intensity in 'fringe' or 'tail' regions could also be highly dependent on lens operation and the adequacy of specimen alignment. Scheithauer described an alternative technique in which Pt apertures of varying diameters were utilized to determine the fraction of 'long-tail' X-ray contributions outside each aperture on the measured Pt photoelectron signal compared to that on a Pt foil (**10**). In test measurements on a commercial XPS instrument with a focused X-ray beam and a nominal lateral resolution of 10 µm (as determined from the distance between the positions for 20% and 80 % of maximum signal when scans were made across an edge), it was found that aperture diameters of about 100 µm and 450 µm were required to reduce the photoelectron signals to 10 % and 1 %, respectively, of the maximum value (**10**). Knowledge of the effective analysis area is important when making tradeoffs between lateral resolution and detectability. In scanning Auger microscopy, the area of analysis is determined more by the radial extent of backscattered electrons than by the radius of the primary beam (**11**, **12**, **13**).

6. Apparatus for the Electron-Gun Method

6.1 *Test Specimen,* preferably a conductor, is required and is mounted in the Auger electron or X-ray photoelectron spectrometer in the usual position for surface analysis. It is recommended that the test specimen be a metallic foil with lateral dimensions larger than the dimensions of the field of view of the electron energy analyzer. The test specimen should be polycrystalline and have grain dimensions much less than the expected spatial resolution of the analyzer or the width of the incident beam on the specimen in order to avoid artifacts due to channeling or diffraction effects. The specimen surface should be smooth and be free of scratches and similar defects that are observable with the unaided eye (see 8.6). It is desirable that the surface of the test specimen be cleaned by ion sputtering or other means to remove surface impurities such as oxides and adsorbed hydrocarbons; the degree of surface cleanliness can be checked with AES or XPS measurements.

6.2 *Electron Gun*—An electron gun must be available on the spectrometer to provide a beam of electrons incident on the test specimen surface with energy typically between 100 eV and 2000 eV (the normal range of detected energies in AES and XPS). The gun must be equipped with a deflection system so

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[5] The boldface numbers in parentheses refer to the list of references at the end of this practice.

that the electron beam can be deflected to different regions of the specimen surface. The width of the electron beam (FWHM) at the test specimen should be less than the spatial resolution desired in the following measurements.

6.3 *Electronic Equipment,* is required to scan the electron beam on the surface of the test specimen and to record and display the selected signals.

6.3.1 *Equipped Spectrometer*—Some commercial spectrometers, particularly those designed for scanning Auger microscopy, have electronic instrumentation, which can be used to scan the electron beam across the test specimen surface, either on a selected line or on a raster pattern with selected dimensions. The selected analyzer signals may be recorded in a computer system or be displayed directly on an oscilloscope or X-Y recorder.

6.3.2 *Unequipped Spectrometer*—If the spectrometer is not equipped with instrumentation for scanning the electron beam, this instrumentation will have to be provided. A line scan can be accomplished with a suitable wave-form generator (either triangular or sawtooth) or a programmable power supply. Another dc supply may be required to define the position of the line on the specimen, that is, in the direction orthogonal to the scan. Raster scans can be made with two waveform generators or two programmable power supplies.

7. Procedure for the Electron-Gun Method

7.1 Choose the energy of the electron beam incident on the surface of the test specimen. This choice should be made depending on the nature of the tests to be made. For example, electron energies between 100 eV and 2000 eV may be chosen for Auger electron experiments with specific choices related to the energies of Auger electron peaks of particular interest. In X-ray photoelectron spectroscopy experiments with magnesium characteristic X-rays, electron energies between approximately 254 eV and 1254 eV might be chosen to determine the analyzer performance for the binding-energy range between 0 eV and 1000 eV.

7.2 Choose the type of scan for the electron beam on the test surface, either line scan or raster scan (6.3). If a line scan is selected, choose the position of the line on the specimen.

7.2.1 A line scan is a relatively simple procedure and can be made for two orthogonal directions. This method for determining the active area of the analyzer may suffice for many applications but has the disadvantage that the active area may not be symmetrical about the two scan lines (**1, 2**). The raster scan method allows convenient observation of any instrumental asymmetries.

7.2.2 The following suggestions are made if the instrument is not already equipped with instrumentation to scan the electron beam. The specific suggestions are made to generate a raster scan for an electron gun equipped with deflection plates. Line scans can be made in a similar way. An analogous procedure would be used for an electron gun operated with an electromagnetic deflection system.

7.2.2.1 *Use of Waveform Generators*—In this approach, use two waveform generators to generate triangular waveforms at frequencies typically in the range of 0.5 kHz to 1 kHz. The waveforms are amplified and coupled through a transformer to the deflection plates of the electron gun, one output being designated for horizontal deflection and the other for vertical deflection. A resistive center-tap is connected across each transformer output with the midpoints grounded. The waveforms are also connected to the horizontal and vertical inputs of an oscilloscope. Adjust the frequencies of the oscillators so that there is a uniform intensity distribution on the oscilloscope, that is, absence of any Lissajou's figures. Select the gains of the amplifiers to deflect the electron beam across the test specimen by amounts corresponding at least to the anticipated analyzer field of view; for a desired deflection on the test specimen, the maximum deflection-plate voltage will scale with the selected electron energy. Make a line scan with a single waveform generator and with the scan voltage applied to either the horizontal or the vertical deflection plates. Apply a dc voltage to the other deflection plates to select the position of the line on the specimen.

7.2.2.2 *Use of Programmable Power Supplies*—Program a computer to control the output voltages of two programmable power supplies. Connect the outputs of the power supplies to the deflection plates of the electron gun. Make these connections as in 7.2.2.1; connect center taps across each power supply, also as in 7.2.2.1. At a given vertical position, step the electron beam horizontally across the test specimen surface. The beam then can be stepped vertically prior to the next horizontal sweep, and so on. Make measurements for each horizontal sweep and for equally spaced horizontal lines within the vertical sweep range. The interval between the positions of the electron beam on the specimen surface together with the width of the beam at the surface will determine the spatial resolution in the measurement of the specimen area contributing to each detected signal.

7.2.3 The maximum amount of deflection of the electron beam on the test specimen should be less than that which would cause significant (>5 %) reduction of incident electron-beam current, for example, reduction due to interception of the beam by electrodes of the electron gun.

7.3 The amount of deflection of the electron beam on the test specimen can be determined from electron intensity measurements with test objects, for example, grids or holes, of known dimensions (**1**). The test object is mounted on the test specimen and features of known shape and size are located in the recorded data (see 7.7). Alternatively, a feature can be located in plots of absorbed current (see 7.4) due to, for example, specimen roughness or a specimen mounting clip (**3**). The specimen can then be moved a known amount using a manipulator and a new plot made of absorbed current. The difference in the positions of the selected feature on the two plots corresponds to the displacement of the specimen.

7.4 It is recommended that measurements be made of the current to the specimen (the absorbed current) as the electron beam is scanned across the specimen surface. These measurements can give information about the topography of the specimen surface and are useful for ensuring that any structure in the other intensity measurements (see 7.5) is not associated with specimen topography.

7.5 Select the electron signals to be measured from the analyzer.

7.5.1 *Elastic Peak*—The electron energy analyzer can be adjusted to detect electrons elastically scattered by the specimen surface, that is, at the energy of the incident electron beam. This choice is recommended for initial survey measurements since this signal is the strongest. A possible disadvantage of this choice is that incorrect intensity measurements may be made if, for energy analyzers with sufficiently high energy resolution, the instrument does not remain aligned on the elastic peak as the electron beam is deflected on the specimen (**4**); see also Guide E1016.

7.5.2 *Inelastically Scattered Electrons*—The electron energy analyzer can be adjusted to detect electrons inelastically scattered by the specimen surface. The electron energy being detected may be between zero and the energy of the incident beam.

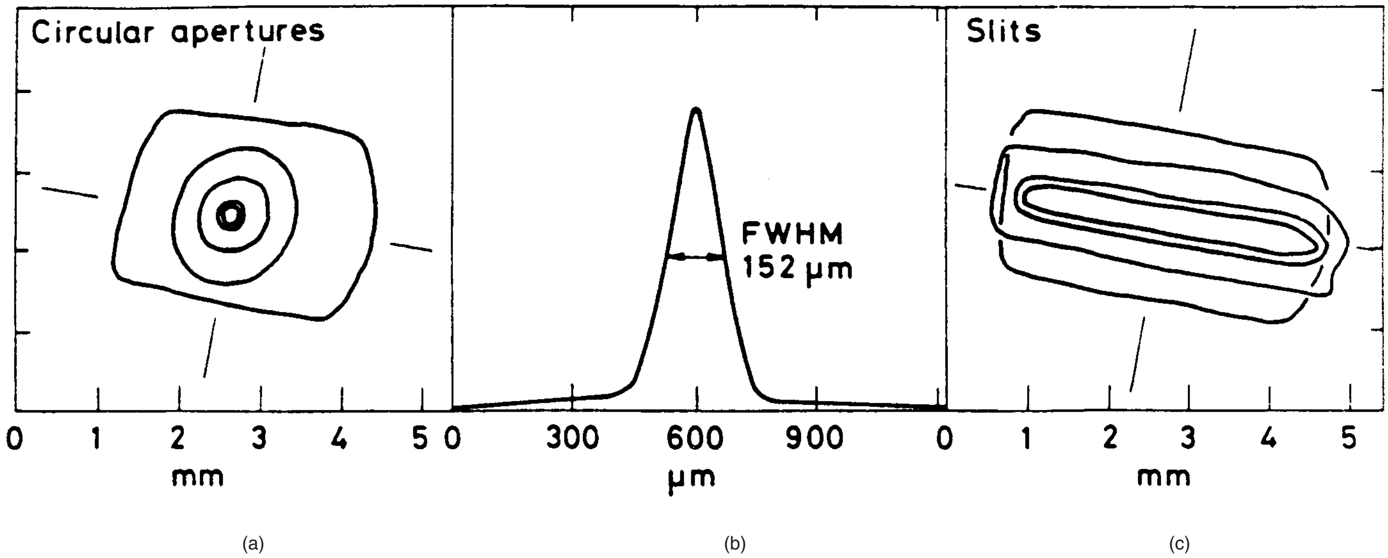
7.5.2.1 This choice is recommended for avoiding the possible artifact described in 7.5.1. It is suggested that the region of the scattered-electron energy distribution about 100 eV below the elastic peak be utilized because the intensity is relatively high. The actual electron energy should be chosen to avoid any structure that may be present in this region due to excitations of inner-shell electrons.

7.5.2.2 A consideration in the choice of signals due to elastically or inelastically scattered electrons is the energy widths (FWHM) of the AES or XPS peaks usually measured by the analyzer. If these widths are less than about 2 eV, it is recommended that the elastic-peak signal be used; if these widths are greater than about 2 eV, it is recommended that the inelastically-scattered-electron signal be used. The reason for these recommendations is that there is a coupling for any analyzer between the detected signal and source position, angle of emission for the source, and electron energy (Guide E1016). As a result, the active specimen area measured with inelastically scattered electrons can be greater than that measured with elastically scattered electrons under otherwise identical conditions. More accurate characterization of the analyzer will be obtained if the energy width of the scattered-electron signal approximates the energy widths of the AES or XPS peaks encountered in practice.

7.5.3 *Auger Electrons*—It may be conveniently possible, particularly with instruments intended for scanning Auger electron microscopy, to adjust the electron energy analyzer to detect Auger electrons emitted from the surface of the test specimen. Even if there is no significant Auger-electron signal from the test specimen at the electron energy of particular interest, instrumental software may be utilized to determine the electron intensity at the selected energy (without subtraction of any background).

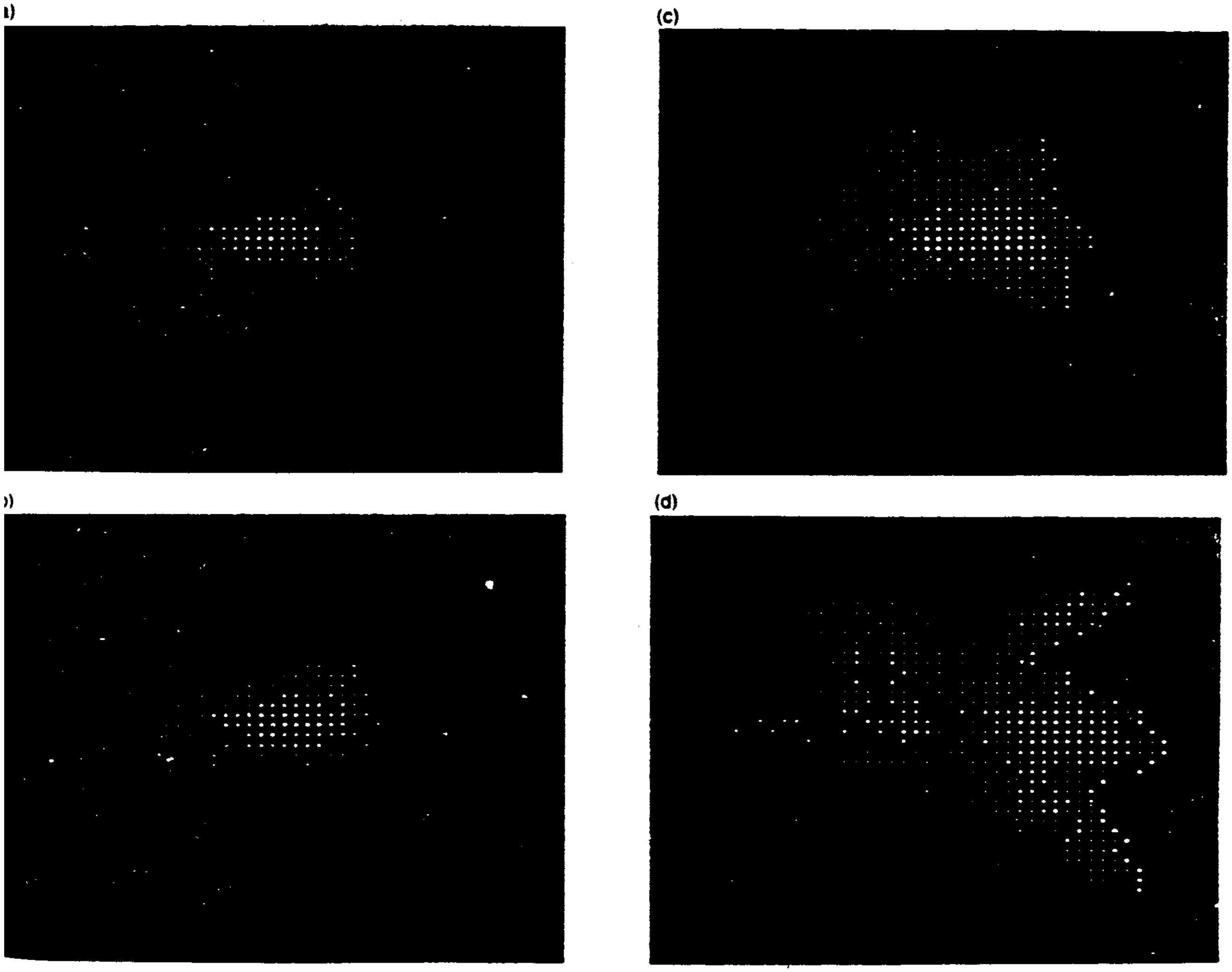
7.6 Select the analyzer operating conditions, for example, analyzer retardation ratio or pass energy if the electrons are retarded before energy analysis, and choice of aperture or slit sizes, and any variations in the position of the specimen surface with respect to the analyzer. These choices and that of the electron energy (see 7.1) are based on the particular measurement conditions for practical measurements for which characterization of the instrument is desired.

7.7 The selected electron signals (see 7.4 and 7.5) can be displayed by several different methods. On scanning Auger electron microscopes, software often will be available for the manipulation and display of the acquired data. On other instruments, the user may be able to export data from the instrumental computer for subsequent analysis and display using software on another computer. If these options are not available, the following suggestions may be useful for data display. Examples of different types of data displays are given in Figs. 1-3.



NOTE 1—In (a) and (c), contour maps of constant elastic-peak intensity are shown for 50 % of the maximum intensity for different choices of apertures and slits on the instrument. Fig. 1(a) shows use of circular apertures in the instrument of diameter 10, 5, 3, 1, and 0.5 mm; the contours for the two smallest apertures almost overlap. Fig. 1(b) shows a line scan with a plot of the elastic-peak intensity as a function of electron-beam position on the test specimen with use of the 0.5-mm aperture. Fig. 1(c) shows use of rectangular slits of length 15 mm and widths of 6, 3, 1.5, and 0.75 mm.

FIG. 1 Examples of Contour Maps and Line Scans Obtained from Elastic-Peak Measurements with an XPS Instrument (2)



NOTE 1—Shown are examples of raster scans obtained by z-modulation of an oscilloscope for an AES/XPS instrument operated with an elastically scattered electron beam of 1000 eV and an analyzer pass energy of 25 eV. The largest dots show intensities above 75 % of the maximum, the middle-sized dots those between 50 and 75 %, and the smallest dots those between 25 and 50 %. The horizontal width of each photograph corresponds to an electron-beam deflection of 7.5 mm on the test specimen and the vertical height to a deflection of 10 mm. Fig. 2(a) is test specimen located 1.5 mm from the focal point of the double-pass cylindrical-mirror analyzer in the direction away from the analyzer. Fig. 2(b) as for Fig. 2(a), but with the specimen 0.5 mm from the focal point. Fig. 2(c) is specimen 0.5 mm from the focal point in the direction towards the analyzer. Fig. 2(d) as for Fig. 2(c), but with the specimen 1.5 mm from the focal point.

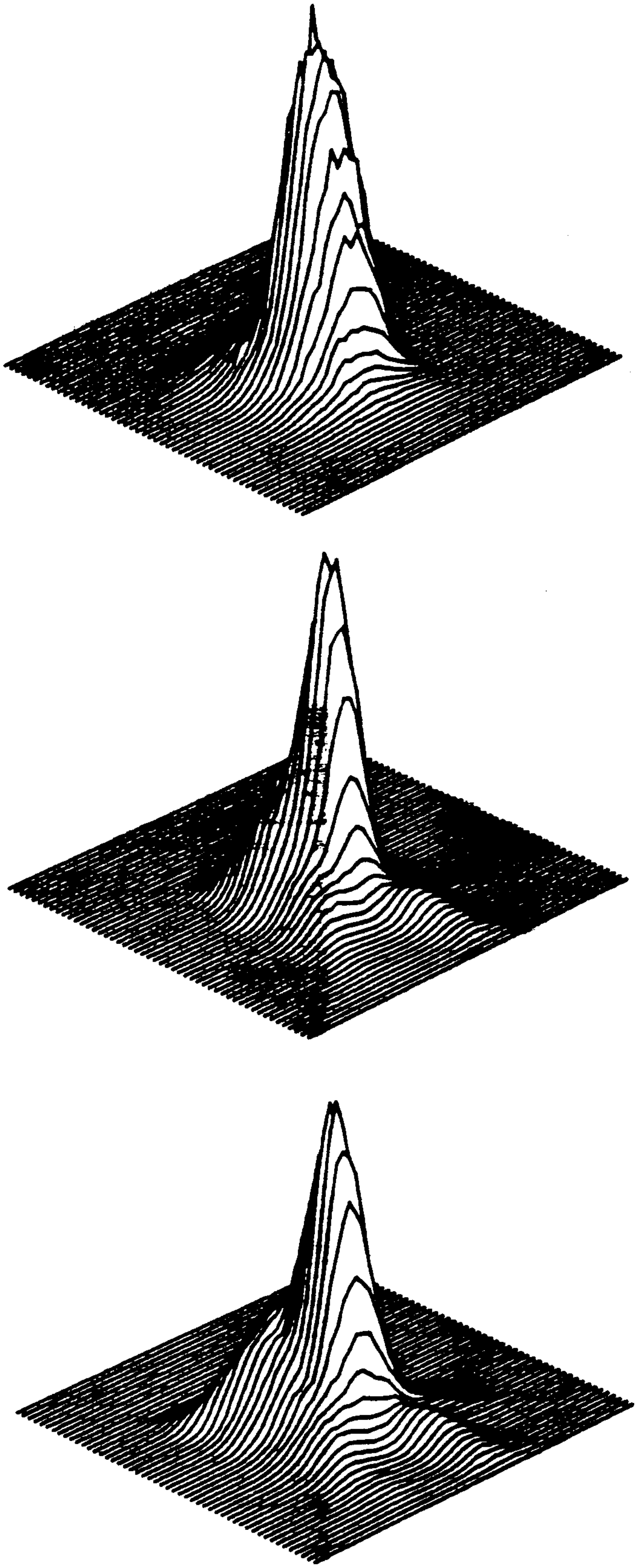
FIG. 2 Examples of Raster Scans (1)

7.7.1 *Display of Line Scan*—A voltage proportional to the detected electron intensity can be applied either to an X-Y recorder or to an oscilloscope as the electron beam is scanned across the test specimen, that is, y modulation (see Fig. 1(b)).

7.7.2 *Display of Raster Scan*—A choice can be made of several methods for displaying measured intensities.

7.7.2.1 *z-Modulation of Oscilloscope*—A voltage proportional to the detected electron intensity can be used to modulate the intensity of an oscilloscope, that is, z-modulation. The pulse signal from the electron detector can be used in the same way. The oscilloscope beam is deflected in the same way as the electron beam is rastered on the specimen so that the oscilloscope display represents the selected signal intensity distribution as the electron beam is scanned. This display can be photographed, and Fig. 2 shows examples. Displays of this type also can be made in real time (**5, 6**).

7.7.2.2 *Images*—Successive line scans of the raster can be displayed on an oscilloscope or an X-Y recorder as in 7.7.1, preferably with small vertical separations in the displayed intensity distribution for successive lines. Alternatively, if a suitable computer is available, successive line scans can be plotted on an X-Y recorder with both horizontal and vertical separations for successive lines and with suppression of certain segments of the line scans to give the appearance of a “three-dimensional” image as viewed from a designated point. Fig. 3 shows examples of this latter method.



NOTE 1—Elastic-peak images are for an analyzer pass energy of 50 eV and for electron energies of 100 eV (top), 500 eV (center), and 1000 eV (bottom). The horizontal distance scanned by the electron beam on the specimen (corresponding to the bottom left to right line scan in each image) was 13 mm and the vertical distance was 15 mm.

FIG. 3 Examples of Images Obtained with an XPS Instrument (3)

7.7.2.3 *Contour Maps*—A map can be made with lines of constant intensity to represent the measured intensity distribution as the beam is rastered on the surface of the test specimen. Fig. 1(a) and Fig. 1(c) show simple examples.

8. Analysis of Data for the Electron-Gun Method

8.1 Line scans (7.7.1) such as that in Fig. 1(b) can give information on the width of the region along the line that contributes a given fraction of the maximum measured signal. Fig. 1(b) shows a line scan for an XPS instrument with a width of 152 µm corresponding to 50 % of the maximum intensity. This width is a measure of the spatial resolution of the electron energy analyzer for the particular conditions of operation. Other examples of line scans for different conditions of analyzer operation have been published (**6**).

8.1.1 Some experimentation is required to select the region on the test specimen for the line scan. Measurements should be made in two orthogonal directions that pass through the point on the specimen with maximum signal intensity.

8.2 Raster-scan data in the form of photographs (7.7.2) can give information on the specimen area contributing to the detected signal by the electron energy analyzer for the particular operating conditions. For the examples shown in Fig. 2, the intensity information is presented in only three bands, but it is readily possible to determine the active specimen area that contributes a given percentage (>25 %, >50 %, or >75 %) of the maximum elastic-peak signal. The data in Fig. 2 indicated that the minimum image width did not occur at the analyzer focal point found by the technique of adjusting the specimen position on the analyzer axis until a symmetrical elastic peak at a calibrated energy of 2 keV was obtained (**1**).

8.2.1 Raster scans in the form of images (7.7.2.2), as shown in Fig. 3, contain more detailed information than usually can be presented in photographs. It is readily possible to determine the analysis area, namely the area of the specimen from which the entire signal or a specified percentage of that signal is detected (see Terminology E673 or ISO 18115:2001). Furthermore, it is obvious visually that the top image in Fig. 3 is almost symmetrical while the bottom image has noticeable asymmetries (**3**).

8.3 Raster-scan data can be used to generate contour plots, that is, lines of constant signal intensity as a function of electron beam position on the test specimen (7.7.2.3). Contour plots are useful for presenting the intensity data in compact form and for avoiding uncertainties associated with the "hidden" regions of images, such as those shown in Fig. 3.

8.4 Measurements of the widths of line scans or of raster-scan data can be used to verify adequate alignment of the specimen and to determine changes in the active specimen area as a function of the experimental conditions. Fig. 2 shows examples of raster-scan data as a specimen was moved along the axis of a double-pass cylindrical-mirror analyzer (**1**). Similar measurements can be made as a function of electron-energy-analyzer pass energy or electron emission angle (**1**).

8.5 Measurements of the widths of line scans or of raster-scan data can be compared with any values available from manufacturer's specifications or from design data for the particular instrument and for particular operating conditions (**1, 3**); see also Guide E1016. The raster-scan data of the type shown in Figs. 2 and 3 show directly the imaging properties of the analyzer (**1, 3, 4**). Other examples have been published (**6**).

8.6 Contrast in the absorbed-current images (scanning electron micrographs) is due to variations of the secondary-electron yield caused either by differences of chemical composition or by specimen topography, or due to electron-beam nonuniformities across the scanned area (see 7.2.3). If the test specimen is a metallic foil with no surface impurities detected by AES or XPS (for example, if the test specimen had been adequately cleaned by ion sputtering after insertion into the instrument), contrast in the absorbed-current images will be due almost entirely to the specimen topography. It is desirable that the test specimens be sufficiently smooth so that variations in absorbed current are less than about 5 % as the electron beam is deflected across the surface. Topographical variations would then be expected to have a correspondingly small effect on the intensities of elastically scattered electrons, inelastically scattered electrons, and Auger electrons.

9. Apparatus for the Sharp-Edge Method

9.1 *Test Specimen*—Preferably a wafer (such as Si [001]) that could be easily cleaved to produce a long side with a sharp edge without irregularities. The specimen should be mounted in the analysis chamber in front of the photoelectron spectrometer in the usual position for surface analysis. It is recommended that the surface is covered with a metallic film (deposited prior to cleaving) with lateral dimensions larger than the dimensions of the field of view of the electron energy analyzer. The test surface should be polycrystalline and have grain dimensions much less than the expected spatial resolution of the analyzer or the width of the incident beam on the specimen in order to avoid artifacts due to channeling or diffraction effects. The specimen surface should be smooth and be free of scratches and similar defects that are observable with the unaided eye (see 11.3). It is desirable that the surface of the test specimen be cleaned by ion sputtering or other means to remove surface impurities such as oxides and adsorbed hydrocarbons; the degree of surface cleanliness can be checked with AES or XPS measurements.

9.2 *Sample Manipulator*—The analysis chamber should be equipped with a sample manipulator with a positioning precision much smaller than the length of the analysis area in the scanning direction (that is, about five times smaller than the distance between two consecutive measurements as described in 10.2). It is preferable that the sample surface is parallel to the scanning direction (which is usually chosen as one of the axes of the manipulator), and that the sharp edge is perpendicular to the scanning direction.

10. Procedure for the Sharp-Edge Method

10.1 A representative photoelectron peak of the metallic surface should be chosen such that its area could be determined with high precision through regular peak-fitting procedures.

10.2 Spectra for the chosen peak should be acquired at different positions of the sample in the scanning direction. Enough equidistant sample positions should be considered to clearly identify the plateaus for the maximum (when the sampled area is completely contained by the sample surface) and minimum (when there is no overlap between the analysis volume and the sample surface) peak intensities (at least two positions for each side). In addition, enough sample positions (at least four) should be considered between the plateaus to assess the full shape of the curve.

10.3 It is preferable to make the measurements by moving the sample consecutively in only one direction to optimize the positioning resolution of the manipulator.

10.4 For electron spectrometers with well defined axes of symmetry, it is preferable that the chosen scanning direction is in each of the directions of the axes of symmetry of the electron analyzer. For hemispherical spectrometers, one of the symmetry axes corresponds to the line between the point of entrance of the electrons to the hemisphere and the center of the channeltrons, and the other to the perpendicular direction in the plane of the flat base of the hemisphere.

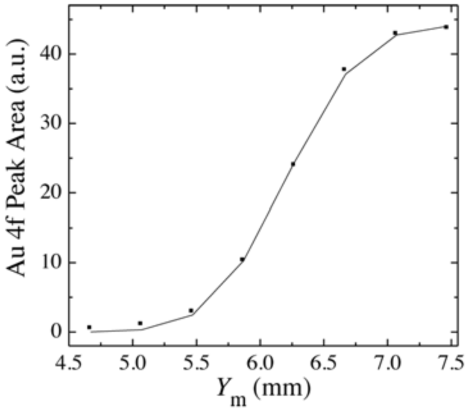
10.5 Measurements in two perpendicular directions should be done by either using a sample with two sharp edges (as defined in 9.1) perpendicular to each other (producing a corner) or by rotating the sample by 90° after the first measurement.

11. Analysis of Data for the Sharp-Edge Method

11.1 Peak-intensity curves (obtained as described in 10.2) such as that in Fig. 4 can give information on the width of the analysis area in the scanning direction.

11.2 It is possible to reproduce the data by assuming a model for the dependence of the electron analyzer efficiency with position. If a Gaussian decay is assumed, the curve should be fitted to an expression of the form:

$$a_1 + a_2 \int_{-\infty}^{x} dx' e^{-(x' - x_{0/\sigma})^2} \tag{1}$$



NOTE 1—Shown is an example of a knife-edge experiment. The FWHM of the analysis area of the electron analyzer assessed from the calculations is 1.05 mm.

FIG. 4 Example of a Curve Obtained with the Sharp-Edge Method (7)

where x is the reading of the micrometer manipulating the sample position, and a_1, a_2, σ, x_0 are fitting parameters. For the purpose of assessing the width of the analysis area, the only significant parameter is σ, which is related to the FWHM of the analysis area in the scanning direction by a factor of $2\sqrt{1n2}$. x_0 corresponds to the micrometer reading when the sample edge is positioned at the center of the analysis area in the direction of the scan, a_1 is the signal intensity when the sample is away from the analysis area (except for noise, it should be equal to zero), and a_2 is related to the peak intensity when the analysis area is completely contained by the sample surface. This formulation has been applied in the analysis of ARXPS data (**14**). If a different dependence of the electron analyzer efficiency with position is assumed, the expression should be modified accordingly.

11.3 Measurements of the spectrometer analysis area made through the sharp-edge method can be compared to other type of measurements and to manufacturing specifications for spectrometers with some degree of symmetry. Complex shapes, as those for cylindrical mirror analyzers described in Refs (**3** and **4**), cannot be simply described in terms of curves as that shown in Fig. 4 or parameters such as σ. Two parameters are sufficient for spectrometers with elliptical shapes of the spatial dependence of the spectrometer efficiency.

11.4 The specific way to make the comparison depends on the definition employed for the spectrometer analysis area:

11.4.1 If it is done in terms of the distance between the positions for, say, 20 % and 80 % of maximum signal when scans are made across an edge (**8**) the comparison can be made directly from the curves of the type shown in Fig. 4.

11.4.2 If the spectrometer analysis area is defined in terms of the length of the region for which the XPS signal is larger than, say, 80 % of the maximum signal, the length should be compared to $2\sigma\sqrt{-1n\ 0.8}$.

11.4.3 If the definition is done in terms of the region contributing to a certain percent of the signal (for example, 80 %), the length of that region should be compared to 2σ InvErf (0.8), where InvErf is the inverse error function.

11.5 Deviation from the expected behavior could be due to several reasons. If the region illuminated by the X-ray is not much larger but only comparable to the analysis area viewed by the analyzer, a more complex analysis should be applied (**7**). Scratches on the sample surface, irregularities on the sample edge, or deviation on the orientation (such that the edge is not perpendicular to the scanning direction), can also affect the behavior.

12. Keywords

12.1 aperture; auger electron spectroscopy (AES); knife-edge experiments; sharp edge; specimen area; spectrometer; surface analysis; X-ray photoelectron spectroscopy (XPS)

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