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# Standard Practice for Determination of the Specimen Area Contributing to the Detected Signal in X-Ray Photoelectron Spectroscopy and Auger Electron Spectrometers and Some X-Ray Photoelectron Spectrometers<sup>1</sup>

This standard is issued under the fixed designation E 1217; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

<sup>1</sup> This practice is under the jurisdiction of ASTM Committee E-42 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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## 1. Scope

1.1 This practice describes methods for determining the specimen area contributing to the detected signal in X-ray photoelectron spectroscopy (XPS) and Auger electron spectroscopy (AES) spectrometers and some types of X-ray photoelectron spectrometers when this area is defined by the electron collection lens/aperture system. It is recommended as a useful means of determining the observed specimen area for different conditions of spectrometer operation, verifying adequate specimen alignment, lens and characterizing the imaging properties aperture system of the electron energy analyzer.

1.2 This analyzer. The practice is intended applicable only for to those X-ray photoelectron spectrometers in which the specimen area excited by the incident X-ray or electron beams beam is or can be made larger than the specimen area viewed by the analyzer. It is assumed that, under normal conditions of operation, analyzer, in which the specimen is excited by photoelectrons travel in a beam of X rays or electrons that field-free region from the specimen to the analyzer entrance, and in which an auxiliary electron gun can be considered mounted to produce an electron beam of variable energy on the specimen.

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

1.3 This standard does not purport to address all of the safety problems, 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:

E 673 Terminology Relating to Surface Analysis<sup>2</sup>

E 1016 Practice for Describing and Specifying the Properties of Electrostatic Electron Spectrometers<sup>2</sup>

### 2.2 ISO Standards:

ISO/DIS 18115 Surface Chemical Analysis—Vocabulary (in ballot)<sup>3</sup>

## 3. Terminology

3.1 *Definitions*— See Terminology E 673 for terms used in X-ray photoelectron spectroscopy and Auger electron spectroscopy and X-ray photoelectron spectroscopy.

## 4. Summary of Practice

### 4.1 For both Auger

4.1 An electron and X-ray photoelectron spectrometers, an electron beam with a selected energy is scanned across the surface of a test specimen surface: specimen. The beam may be scanned once (, that is, a line scan), or in a pattern (, that is, rastered).

<sup>2</sup> Annual Book of ASTM Standards, Vol 03.06.

<sup>3</sup> The boldface numbers in parentheses refer to the references at the end of this practice.

<sup>3</sup> Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

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 particular 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 a particular kinetic 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 conditions of operation. This information can be used to determine the analysis area (see Terminology E 673 or ISO 18115).

## 5. Significance and Use

### 5.1 X-ray photoelectron

5.1 Auger electron spectroscopy and Auger electron 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 ~~on two types of instruments: (1) those for instruments in which the width of the exciting X-ray or a focused electron beam is much greater than the dimensions of the specimen area viewed by the analyzer, and (2) those for which the dimensions of the specimen region can be scanned by the incident beam are much over a region with dimensions greater than the dimensions of the specimen area viewed by the analyzer.~~

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 can also depend on the adequacy of alignment of the specimen with respect to the electron energy analyzer on instruments that require proper positioning of the specimen 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 in order to generate correction factors if quantitative surface analyses are to be made.

5.4 ~~The dimensions of the observed region of the specimen are a measure of the spatial resolution of the analyzer for the selected conditions. It is important to know the spatial resolution materials in order to determine the extent of regular use have lateral inhomogeneities in other specimens or if specimens with nonideal geometries (for example, small particles) are being examined.~~

5.5 The 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 design manufacturer's specifications.

5.6 Examples of the application of the methods described in this practice have been published (1-4).<sup>4</sup>

## 6. Apparatus

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 ~~suggested~~ recommended that the test specimen be a metallic foil with lateral dimensions sufficiently large that it exceeds larger than the entire 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 ~~target test specimen surface~~ with energy typically between 100 eV and 2000 eV (the normal range of detected energies in ~~XPS AES and AES~~; XPS). The gun must be equipped with a deflection system so 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 ~~test 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 ~~with~~, which can be used to scan the electron beam is scanned 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~~ power 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.

<sup>4</sup> The boldface numbers in parentheses refer to the list of references at the end of this practice.

## 7. Procedure

7.1 Choose the energy of the electron beam incident on the specimen surface. ~~Make this 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 ~~250~~ 100 eV and ~~1250~~ 2000 eV may be chosen ~~for Auger electron experiments with specific choices related to determine the energies of Auger electron energy analyzer performance for peaks of particular interest. In X-ray photoelectron spectroscopy experiments with magnesium characteristic X-rays (characteristic X-ray energy 1253.6 eV) 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. Other choices would be made if the X-ray photoelectron spectroscopy experiments were to be made with aluminum characteristic X-rays or for Auger electron spectroscopy experiments. eV.~~

7.2 Choose the type of scan ~~of~~ 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 and~~ **(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 ~~on 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 de~~ 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 ~~to provide horizontal and vertical deflections of the electron beam~~ as in 7.2.1; connect center taps across each power supply, also as in 7.2.1. At a given vertical position, step the electron beam horizontally across the test specimen surface. The beam ~~can then~~ can be stepped vertically prior to the next horizontal sweep, and so on. Make measurements ~~at equally spaced points~~ 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 electrodes)-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 ~~the~~ 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 *The Elastic/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 Practice E 1016.

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 secondary electron energy distribution about 100 eV below the elastic peak be utilized since because the intensity is relatively high and there is generally little structure. 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 (Practice E 1016). 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 signals 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 by 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).

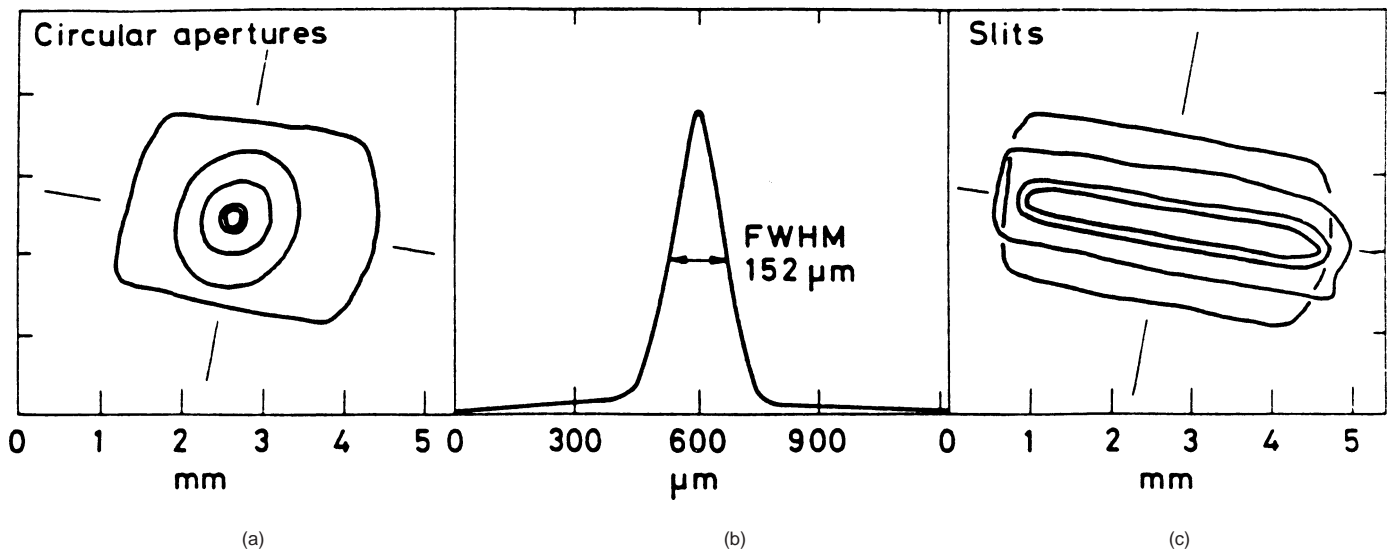
7.6 Select the analyzer operating conditions (for example, analyzer retardation ratio or pass energy if the electrons are retarded before energy analysis, and choice of aperture or slit sizes) and of any variations in the position of the specimen surface with respect to the analyzer. These choices and 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 show examples of the methods.

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) (Fig. 1(b) is an example); y modulation (see Fig. 1(b)).

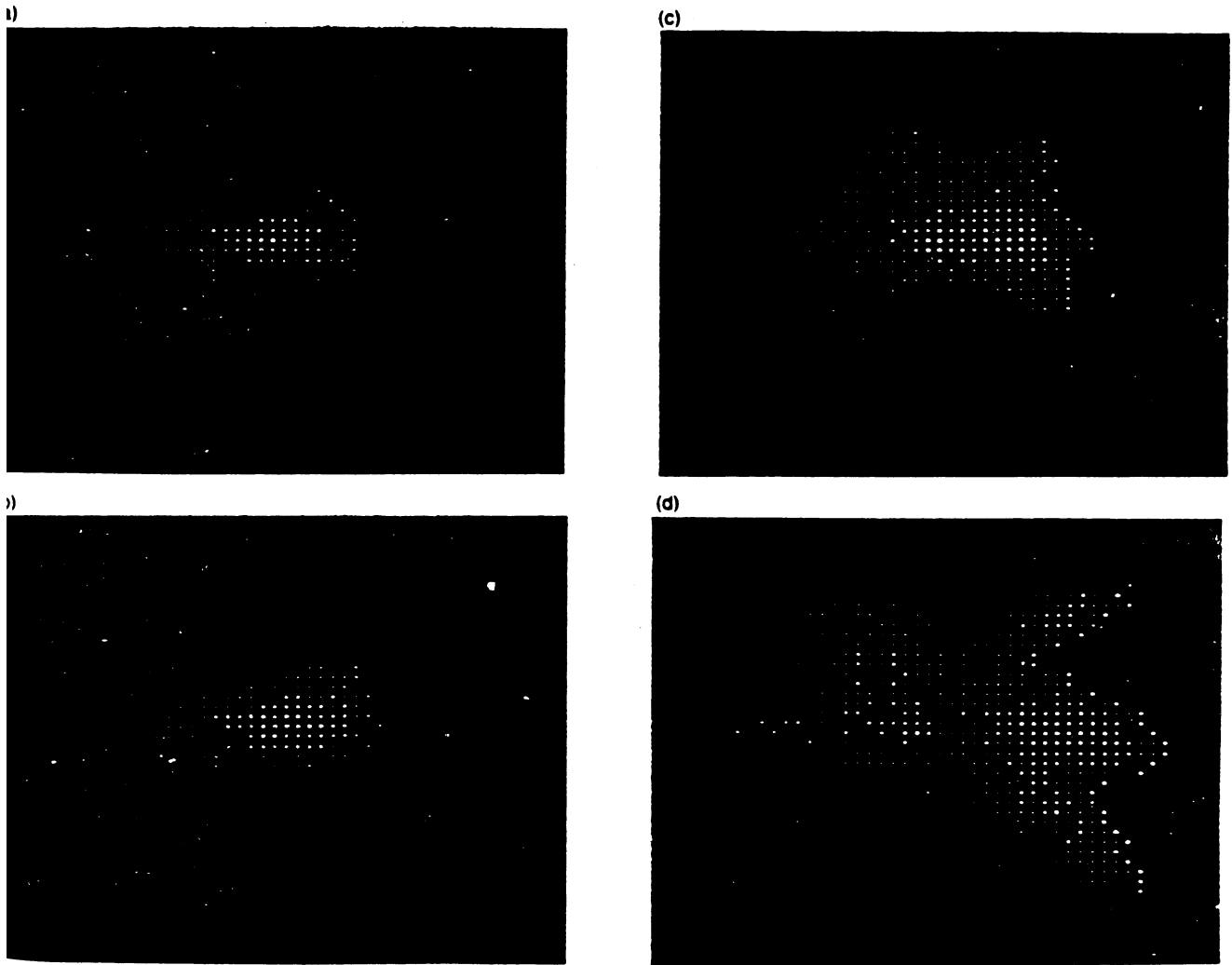
7.7.2 Display of Raster Scan—A choice can be made of several methods for displaying measured intensities.

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 multiplier 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 can also be made in real time (5, 6).



NOTE 1—In (a) and (c), contours of constant elastic-peak intensity are shown for 50 % of the maximum level for different choices of apertures and slits on the instrument. (a) 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. (b) a line scan showing a plot of the elastic-peak intensity as a function of electron-beam position on the specimen with use of the 0.5-mm aperture. (c) use of rectangular slits of length 15 mm and widths of 6, 3, 1.5, and 0.75 mm.

FIG. 1 Examples of Data 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 (1). 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 specimen and the vertical height to a deflection of 10 mm. (a) 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.

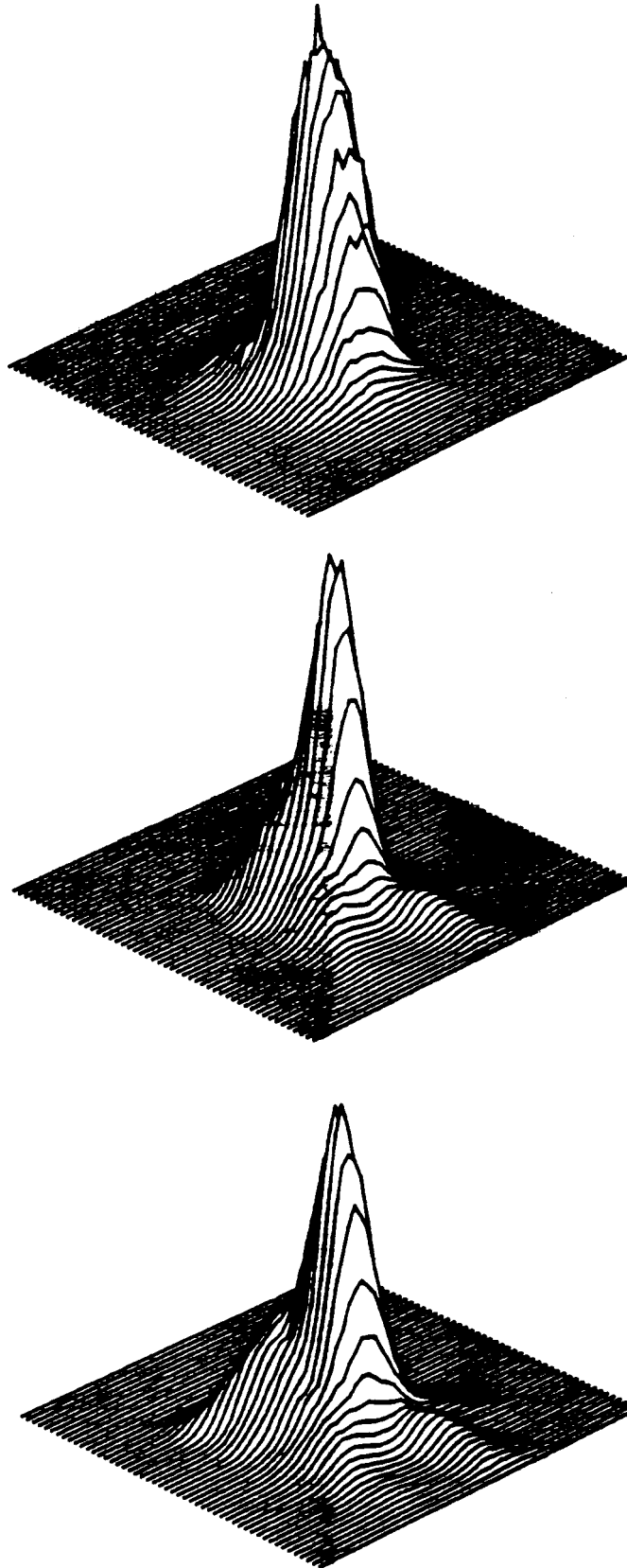
FIG. 2 Examples of Raster Scans (1)

7.7.2.2 *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.

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 specimen surface. Fig. 1(a) and Fig. 1(c) show simple examples.

## 8. Analysis of Data

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  $\mu\text{m}$  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).



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 15 mm.

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

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 can usually be presented in photographs. It is readily possible to determine the active analysis area, namely the area of the specimen that contributes from which a given specified percentage of the signal is detected (see Terminology E 673) or the area of the specimen from which the entire signal or a specified percentage of that signal is detected (ISO 18115). Furthermore, it is visually obvious 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 calculated available from manufacturer’s specifications or from design data for the particular instrument and for particular operating conditions (1, 3) ; see also Practice E 1016. The raster-scan data of the type shown in Figs. 2 and Fig. 3 show directly the imaging properties of the analyzer (1, 3, 4). Other examples have been published (6) .

~~8.6 If quantitative surface analyses are to be made and if changes are found~~

~~8.6 Contrast in the active specimen area as a function of measurement conditions (for example, electron energy or analyzer pass energy), correction factors need to be calculated to relate AES or XPS intensity measurements made at one set of measurement conditions to those made at another set of conditions. Suitable correction factors can be calculated conveniently from raster-scan data in the form of images that have been recorded for conditions corresponding to the AES or XPS measurements of interest. For each image, a calculation is made of the integral intensity (that is, the volume under the surface shown in Fig. 3) after the maximum intensity has been normalized to unity. The correction factor is then the inverse of the ratio of the integral intensities for the two measurement conditions.~~

~~8.7 Structure 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.~~

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