



Designation: E 1217 – 00

Standard Practice for Determination of the Specimen Area Contributing to the Detected Signal in Auger Electron Spectrometers and Some X-Ray Photoelectron Spectrometers¹

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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 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, and in which an auxiliary electron gun can be mounted to produce an electron beam of variable energy on the specimen.

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 *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:

E 673 Terminology Relating to Surface Analysis²

E 1016 Practice for Describing and Specifying the Properties of Electrostatic Electron Spectrometers²

2.2 ISO Standards:

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

3. Terminology

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

4. Summary of Practice

4.1 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 a 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 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 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 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.

5.2 This practice is intended as a means for determining the observed specimen area for selected conditions of operation of

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² *Annual Book of ASTM Standards*, Vol 03.06.

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

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 Examples of the application of the methods described in this practice have been published (1-6).⁴

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 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 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

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

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

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 7.2.1; 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 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 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 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 (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 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.

7.7.1 Display of Line Scan—A voltage proportional to the detected electron intensity can be applied either to an X-Y