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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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

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

[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.

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 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

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