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Standard Guide for Depth Profiling in Auger Electron Spectroscopy[1]

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1. Scope

1.1 This guide covers procedures used for depth profiling in Auger electron spectroscopy.

1.2 Guidelines are given for depth profiling by the following:

	Section
Ion Sputtering	6
Angle Lapping and Cross-Sectioning	7
Mechanical Cratering	8
Mesh Replica Method	9
Nondestructive Depth Profiling	10

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E673 Terminology Relating to Surface Analysis (Withdrawn 2012)[3]

E684 Practice for Approximate Determination of Current Density of Large-Diameter Ion Beams for Sputter Depth Profiling of Solid Surfaces (Withdrawn 2012)[3]

E827 Practice for Identifying Elements by the Peaks in Auger Electron Spectroscopy

E996 Practice for Reporting Data in Auger Electron Spectroscopy and X-ray Photoelectron Spectroscopy

E1078 Guide for Specimen Preparation and Mounting in Surface Analysis

E1577 Guide for Reporting of Ion Beam Parameters Used in Surface Analysis

E1634 Guide for Performing Sputter Crater Depth Measurements

E1636 Practice for Analytically Describing Depth-Profile and Linescan-Profile Data by an Extended Logistic Function

E1829 Guide for Handling Specimens Prior to Surface Analysis

2.2 *ISO Standard:*[4]

ISO/TR 22335: 2007 Surface Chemical Analysis—Depth Profiling—Measurement of Sputtering Rate: Mesh-Replica Method Using a Mechanical Stylus Profilometer

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this guide, refer to Terminology E673.

4. Summary of Guide

4.1 In ion sputtering, the surface layers are removed by ion bombardment in conjunction with Auger analysis.

4.2 In angle lapping, the surface is lapped or polished at a small angle to improve the depth resolution as compared to a cross section.

4.3 In mechanical cratering, a spherical or cylindrical crater is created in the surface using a rotating ball or wheel. The sloping sides of the crater are used to improve the depth resolution as in angle lapping.

4.4 In nondestructive techniques, different methods of varying the electron information depth are involved.

5. Significance and Use

5.1 Auger electron spectroscopy yields information concerning the chemical and physical state of a solid surface in the near surface region. Nondestructive depth profiling is limited to this near surface region. Techniques for measuring the crater depths and film thicknesses are given in (**1**).[5]

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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 International Organization for Standardization (ISO), 1, ch. de la Voie-Creuse, CP 56, CH-1211 Geneva 20, Switzerland, http://www.iso.org.

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

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5.2 Ion sputtering is primarily used for depths of less than the order of 1 µm.

5.3 Angle lapping or mechanical cratering is primarily used for depths greater than the order of 1 µm.

5.4 The choice of depth profiling methods for investigating an interface depends on surface roughness, interface roughness, and film thickness (**2**).

5.5 The depth profile interface widths can be measured using a logistic function which is described in Practice E1636.

6. Ion Sputtering

6.1 The specimen should be handled in accordance with Guides E1078 and E1829. First introduce the specimen into a vacuum chamber equipped with an Auger analyzer and an ion sputtering gun. Align the ion beam using a sputtering target or a Faraday cup, paying careful attention to the relative spot size of the electron beam, ion beam, and Faraday cup and their respective orientations to ensure accurate convergence of the two beams at the specimen surface.

6.1.1 Place the specimen in front of the Auger analyzer and direct the ion gun towards the analysis area. If the ion beam is not normal to the specimen surface then possible shadowing of the analysis area from the ion beam, due to surface roughness, must be considered. The ion beam conditions should be reported in accordance with Guide E1577.

6.2 Choose the elements to be investigated from previous experience or from an initial Auger electron spectrum or an energy-dispersive X-ray spectrum since the latter spectrum can reveal additional elements present at depths greater than those that contribute to the Auger electron spectrum (**3**). Select a specific transition for each element. During the depth profiling, record the peak-to-peak heights for Auger derivative data, or peak heights or peak areas for $N(E)$ data. The data may be gathered during continuous sputtering or between timed sputter segments. Results may vary between the two techniques.

6.2.1 One source of their difference is due to the presence of ion-induced electrons during continuous sputter depth profiling, especially at low-electron kinetic energies, that can become comparable in intensity to the electrons induced by the probing incident electron beam. Unless one or the other of the excitation beams is modulated and detected synchronously these two types of emitted electrons are difficult to distinguish. These ion-induced electrons usually form a featureless background that rises steeply as their kinetic energy decreases, but sometimes ion-induced Auger peaks might be present whose lineshape may be different from those produced by the electron beam (**4**). As a result, care must be taken during continuous sputtering to ensure reliable results. Another source of difference is due to the buildup of adsorbed species during the data acquisition time in the discontinuous sputter depth profile mode (**5**). If portions of the ion-eroded surface expose very reactive phases, then Auger peaks due to adsorbed species, for example, oxygen or carbon, or both, will appear in the spectra and mask the actual depth distribution.

6.2.2 It is advisable when analyzing an unknown specimen to periodically examine survey scans to detect any new elements that were not present in the initial survey scan and to determine if any of the Auger peaks have been displaced outside of their analysis windows (**6**).

6.3 Crater-edge profiling of the sputter-formed crater by using Auger line scans is a technique similar to the analysis of the mechanically formed craters in Section 8 (**7**). Forming the crater by sputtering may introduce the additional complications of ion-induced damage and asymmetric crater dimensions.

6.4 If specimen rotation is used to reduce ion-induced roughness, then the rotational speed, rotation axis runout relative to ion beam sputtered area or wobble and data acquisition rate should be reported (**8, 9**).

6.5 Identify the elements in the survey scans using Practice E827.

6.6 The Auger data and the sputtering conditions should be reported as described in Practice E996.

6.7 There is extensive information available in the literature on the effects of ion bombardment on solid surfaces (**10-15**).

6.8 Special care must be exercised whenever specimen temperature changes are present because effects due to surface diffusion, surface segregation or diffusion limited bulk processes such as point defect migration can occur and dramatically alter the specimen composition, even over depths larger than the ion beam penetration depth which is typically a few nanometers (**16, 17**). The concept of preferential sputtering in multielement, single-phase specimens has altered significantly so that chemical effects such as surface segregation are considered to be at least as important as physical effects such as mass differences in the evolution of the near surface composition during sputter depth profiling (**18-21**). Since the probing depths in Auger electron spectroscopy are usually smaller than the ion-penetration depth these effects are very important in any interpretation of Auger signal intensity in terms of composition during ion-beam profiling. Computer modelling of these and other ion-induced phenomena has been extensively studied and has provided new insights into this field (**22, 23**).

6.8.1 It should be determined for each specimen if compositional changes or other sputter effects are likely to occur. It may be possible to minimize these effects in some instances by adjusting the sputtering parameters.

6.9 Ion guns used in Auger analysis are normally self-contained units capable of producing a focused beam of ions. The specimen is not used as an anode for the gun. Many ion guns are able to raster the ion beam. A rastered ion beam will produce a more uniform ion current distribution on the specimen surface in the region of analysis.

6.10 If the ion gun is differentially pumped, the vacuum pumps may be left on during sputtering, removing most of the sputtered gases. If not, then the chamber must be back filled with gas and provisions for removing the sputtered active gases must be considered. Titanium sublimation is effective in removing these gases.

6.11 Noble gas ions are normally used in sputtering and the most commonly used gas is argon. Xenon is occasionally used

with high beam energies when rapid sputtering is needed. Active gases such as oxygen and metal ions are used in special circumstances.

6.11.1 Ion energies commonly used for depth profiling using noble gases are in the range from 1 to 5 keV where lower ion energies are usually preferred for improved depth resolution. Higher ion energies usually can be obtained with higher ion currents and less preferential sputtering.

6.11.2 Ion beam current density can be measured by a Faraday cup or by following Practice E684.

6.11.3 The sputter rate is needed to calibrate the depth scale (**24, 25**, Guide E1634) when depth profiling using ion sputtering. Several reference standards are available for this purpose. One reference material consists of 30 and 100-nm thick tantalum pentoxide films (**26**).[6] Another reference material is an alternating nickel and chromium thin film structure; each layer is nominally 50-nm thick.[7]

7. Angle Lapping and Cross-Sectioning

7.1 In cross-sectioning, polish the specimen perpendicular to the interface, while in angle lapping, polish the specimen at an angle to increase the depth resolution as shown in Fig. 1 (**27**). Polishing usually includes the use of silicon carbide papers, diamond paste, and alumina. Use progressively finer polishing particles to obtain the desired surface finish. Possible limitations of the techniques include smearing of material across the interface, surface roughness, and the electron probe diameter limiting the spatial resolution.

7.2 In angle lapping mount the specimen on a flat gage block and measure the angle with a collimator. The accuracy depends on the flatness of the specimen. In practice an angle of 0.1° can be accurately measured.

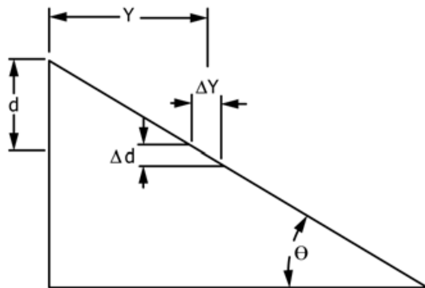
7.3 The depth, d, is given by the following equation:

$$d = Y \tan\theta \quad (1)$$

where (in Fig. 1) θ is the lapped angle and Y is the distance from the edge.

[6] Available from the National Physical Laboratory (NPL), Hampton Road, Teddington, Middlesex, TW11 0LW, UK, http://www.npl.co.uk. Listed as Certified Reference Material NPL No. S7B83, BCR No. 261.

[7] Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 1070, Gaithersburg, MD 20899-1070, http://www.nist.gov. Listed as NIST Standard Reference Material 2135.



NOTE 1—In practice, the angle θ is much smaller than shown, being of the order of 1°.

FIG. 1 Cross Section of Angle-Lapped Specimen

7.4 The depth resolution, Δd, is given by the following equation:

$$\Delta d = \Delta Y \tan\theta \quad (2)$$

where ΔY includes the electron beam diameter and uncertainties in position that may be due to errors in specimen or electron beam positioning.

7.5 Auger analysis can include line scans and point analysis along the lapped surface. Perform the analysis by either moving the specimen using micrometer adjustments or by electronically moving the electron beam.

7.6 Ion sputtering (Section 6) is often used in conjunction with angle lapping to remove contaminants and to investigate interfaces beneath the lapped surfaces.

7.7 Consideration should be given if specimen mounting methods, for example, plastic embedding media, are used which may employ high vapor pressure materials. Out-gassing of the media as well as trapped gases between the media and the specimen may require complete removal of the mounting materials prior to analysis.

8. Mechanical Cratering

8.1 *Ball Cratering:*

8.1.1 First mount the specimen in a device where a rotating steel ball can be placed against its surface. Commercial apparatus is available that uses a rotating shaft with a notch that holds the ball and spins it. The rotational speed and the force against the specimen can be adjusted (**28**).

8.1.2 Coat the ball with an abrasive material to improve the cratering rate. In practice diamond paste is used with a particle size of 0.1 to 1 μm. The larger particle sizes will give the most rapid cratering rates and the finer particle sizes will give the smoothest crater wall surface. The coarser pastes can be used first to form the crater and the fine pastes can be used to smooth the crater wall. As with cross-sectioning and angle lapping, consideration should be given to the possibility of smearing material across the cratered surface.

8.1.3 The geometry of the crater is shown in Fig. 2. The depth of the crater, d, is given by the following equation:

$$d = D^2/8R \quad (3)$$

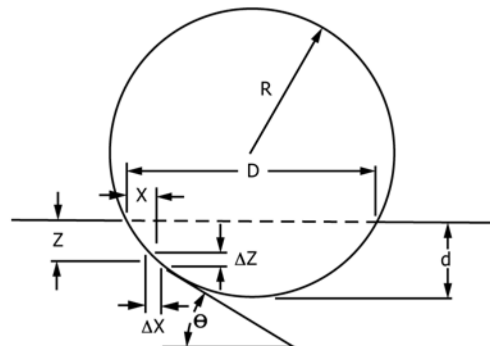


FIG. 2 Cross Section of Specimen After Ball-Cratering Using a Sphere of Radius, *R*, to a depth, *d*

where:

D = the diameter of the crater,
R = the radius of the ball, and
R = >> $D/2$.

8.1.4 The Auger analysis is the same as described in 7.5 and 7.6.

8.1.5 The depth at any point in the analysis, Z, is given by the following equation (**2**):

$$Z = (R^2 - x^2 + Dx - D^2/4)^{1/2} - (R^2 - D^2/4)^{1/2} \quad (4)$$

where x is the lateral distance from the crater edge. The depth may also be given by the approximation as follows:

$$Z = x(D - x)/2R \quad (5)$$

8.1.6 The depth resolution, ΔZ, is given by the following equation:

$$\Delta Z = \Delta x \tan\theta \quad (6)$$

where Δx includes the electron beam diameter and other uncertainties in lateral position and θ is the taper angle. In contrast to angle lapping (Section 7), the taper angle, which is defined as the angle between the surface and the tangent to the crater, varies in value along the crater wall. Its value is given by the following equation:

$$\sin\theta = (0.5D - x)2/R \quad (7)$$

The best resolution is when θ is the smallest at the crater bottom.

8.2 *Radial Sectioning*—A technique similar to ball cratering that uses a cylindrical grinding tool instead of a spherical one (**29**).

9. Mesh Replica Method

9.1 ISO/TR 22335: 2007 describes a method for determining ion-sputtering rates for depth profiling measurements with Auger electron spectroscopy (AES) where the specimen is ion-sputtered over a region with an area between 0.4 mm^2 and 3.0 mm^2. The Technical Report is applicable only to a laterally homogeneous bulk or single-layered material where the ion-sputtering rate is determined from the sputtered depth, as measured by a mechanical stylus profilometer, and sputtering time.

9.1.1 The Technical Report provides a method to convert the ion-sputtering time scale to sputtered depth in a depth profile by assuming a constant sputtering rate. It is not applicable to the case where the sputtered area is less than 0.4 mm^2 or where the sputter-induced surface roughness is significant compared with the sputtered depth to be measured (**30**).

10. Nondestructive Depth Profiling

10.1 Methods for nondestructive depth profiling with Auger electron spectroscopy are based upon varying the effective electron escape depth from the specimen and are limited to characterizing the outermost 2 to 5 nm.

10.2 For certain elements, a depth dependence may be found by examining Auger transitions of different energies (**31**). The lower energy Auger electrons will have a shallower escape depth than the more energetic electrons and therefore, different transitions for the same element will have different sampling depths.

10.3 The sampling depth may also be varied to a limited degree by varying the incident electron beam energy to produce a weak depth dependence in the excitation volume of the specimen (**32**).

10.4 Angle-resolved Auger electron spectroscopy, which involves varying the collected take-off angle of the emitted electrons, has been used for depth profiling (**33**), but the technique is limited due to surface roughness and an often observed angular anisotropy in the Auger signal strength (**34, 35**).

10.5 A general formulation that incorporates electron-solid interactions to characterize the low kinetic energy loss features of an Auger peak can be inverted to produce a nondestructive model depth profile within a depth of almost five times the inelastic mean free path. The technique has been reported (**36**) to be able to distinguish island growth from layer-by-layer growth of adsorbed species.

11. Keywords

11.1 angle lapping; angle-resolved AES; Auger electron spectroscopy; ball cratering; compositional depth profiling; cross sectioning; depth profiling; depth resolution; sputter depth profiling; sputtering; thin films

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