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Standard Guide for
Specimen Preparation and Mounting in Surface Analysis[1]

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1. Scope

1.1 This guide covers specimen preparation and mounting prior to, during, and following surface analysis and applies to the following surface analysis disciplines:

1.1.1 Auger electron spectroscopy (AES),

1.1.2 X-ray photoelectron spectroscopy (XPS and ESCA), and

1.1.3 Secondary ion mass spectrometry (SIMS).

1.1.4 Although primarily written for AES, XPS, and SIMS, these methods will also apply to many surface sensitive analysis methods, such as ion scattering spectrometry, low energy electron diffraction, and electron energy loss spectroscopy, where specimen handling can influence surface sensitive measurements.

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

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:*[2]

E983 Guide for Minimizing Unwanted Electron Beam Effects in Auger Electron Spectroscopy

E1127 Guide for Depth Profiling in Auger Electron Spectroscopy

E1523 Guide to Charge Control and Charge Referencing Techniques in X-Ray Photoelectron Spectroscopy

E1829 Guide for Handling Specimens Prior to Surface Analysis

2.2 *ISO Standards:*[3]

ISO 18115–1 Surface chemical analysis—Vocabulary—Part 1: General terms and terms used in spectroscopy

ISO 18115–2 Surface chemical analysis—Vocabulary—Part 2: Terms used in scanning-probe microscopy

3. Terminology

3.1 *Definitions*—For definitions of surface analysis terms used in this guide, see ISO 18115-1 and ISO 18115-2.

4. Significance and Use

4.1 Proper preparation and mounting of specimens is particularly critical for surface analysis. Improper preparation of specimens can result in alteration of the surface composition and unreliable data. Specimens should be handled carefully so as to avoid the introduction of spurious contaminants in the preparation and mounting process. The goal must be to preserve the state of the surface so that the analysis remains representative of the original.

4.2 AES, XPS or ESCA, and SIMS are sensitive to surface layers that are typically a few nanometres thick. Such thin layers can be subject to severe perturbations caused by specimen handling (**1**)[4] or surface treatments that may be necessary prior to introduction into the analytical chamber. In addition, specimen mounting techniques have the potential to affect the intended analysis.

4.3 This guide describes methods that the surface analyst may need to minimize the effects of specimen preparation when using any surface-sensitive analytical technique. Also described are methods to mount specimens so as to ensure that the desired information is not compromised.

4.4 Guide E1829 describes the handling of surface sensitive specimens and, as such, complements this guide.

5. General Requirements

5.1 Although the handling techniques for AES, XPS, and SIMS are basically similar, there are some differences. In general, preparation of specimens for AES and SIMS requires

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

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

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more attention because of potential problems with electron or ion beam damage or charging, or both. This guide will note when specimen preparation is significantly different among the three techniques.

5.2 The degree of cleanliness required by surface sensitive analytical techniques is often much greater than for other forms of analysis.

5.3 Specimens and mounts must never be in contact with the bare hand. Handling of the surface to be analyzed should be eliminated or minimized whenever possible. Fingerprints contain mobile species that may contaminate the surface of interest. Hand creams, skin oils, and other skin materials are not suitable for high vacuum.

5.4 *Visual Inspection:*

5.4.1 A visual inspection should be made, possibly using an optical microscope, prior to analysis. At a minimum, a check should be made for residues, particles, fingerprints, adhesives, contaminants, or other foreign matter.

5.4.2 Features that are visually apparent outside the vacuum system may not be observable with the system's usual imaging method or through available viewports. It may be necessary to physically mark the specimen outside the area to be analyzed (for example, with scratches or a permanent ink marker) so that the analysis location can be found once the specimen is inside the vacuum system.

5.4.3 Changes that may occur during analysis may influence the data interpretation. Following analysis, visual examination of the specimen is recommended to look for possible effects of sputtering, electron beam exposure, X-ray exposure, or vacuum.

6. Specimen Influences

6.1 *History*—The history of a specimen may affect the handling of the surface before analysis. For example, a specimen that has been exposed to a contaminating environment may reduce the need for exceptional care if the surface becomes less reactive. Alternatively, the need for care may increase if the surface becomes toxic.

6.1.1 If a specimen is known to be contaminated, precleaning may be warranted in order to expose the surface of interest and reduce the risk of vacuum system contamination. If precleaning is desired, a suitable grade solvent should be used that does not affect the specimen material (electronic grade solvents if appropriate or available are best suited). Note that even high purity solvents may leave residues on a surface. Cleaning may also be accomplished using an appropriately filtered pressurized gas. In some instances, the contamination itself may be of interest, for example, where a silicone release agent influences adhesion. In these cases, no precleaning should be attempted.

6.1.2 Special caution must be taken with specimens containing potential toxins.

6.2 *Information Sought*—The information sought can influence the preparation of a specimen. If the information sought comes from the exterior surface of a specimen, greater care and precautions in specimen preparation must be taken than if the information sought lies beneath an overlayer that must be sputtered away in the analytical chamber. Furthermore, it may also be possible to expose the layer of interest by in-situ fracture, cleaving, or other means.

6.3 *Specimens Previously Examined by Other Analytical Techniques*—It is best if surface analysis measurements are made before the specimen is analyzed by other analytical techniques because such specimens may become damaged or may be exposed to surface contamination. For example, insulating specimens analyzed by electron microscopy may have been coated to reduce charging. This coating will render the specimen unsuitable for subsequent surface analysis. Furthermore, exposure to an electron beam (for example, in a SEM) can induce damage or cause the adsorption and deposition of species from the residual vacuum. If it is not possible to perform surface analysis first, then the analysis should be done on a different, but nominally identical, specimen or area of the specimen.

7. Sources of Specimen Contamination

7.1 *Tools, Gloves, Etc.:*

7.1.1 Preparation and mounting of specimens should only be done with clean tools to ensure that the specimen surface is not altered prior to analysis and that the best possible vacuum conditions are maintained in the analytical chamber. Tools used to handle specimens should be made of materials that will not transfer to the specimen or introduce spurious contaminants (for example, nickel tools contaminate silicon). Tools should be cleaned in high purity solvents and dried prior to use. Nonmagnetic tools should be used if the specimen is susceptible to magnetic fields. Tools should never unnecessarily touch the specimen surface.

7.1.2 Although gloves and wiping materials are sometimes used to prepare specimens, it is likely that their use may result in some contamination. Care should be taken to avoid contamination by talc, silicone compounds, and other materials that are often found on gloves. "Powder-free" gloves have no talc and may be better suited. Unnecessary contact with the glove or other tool shall be avoided.

7.1.3 Specimen mounts and other materials used to hold specimens should be cleaned regularly whenever there is a possibility of cross-contamination of specimens. Avoid the use of tapes containing silicones and other mobile species.

7.2 *Particulate Debris*—Blowing one's breath on the specimen is likely to cause contamination. Compressed gases from aerosol cans or from air lines are often used to blow particles from the surface or to attempt to clean a specimen. They, too, must be considered a source of possible contamination. While particles are removed from specimens by these methods, caution is advised and the methods should be avoided in critical cases. In particular, oil is often a contaminant in compressed air lines. In-line particle filters can reduce oil and particles from these sources. A gas stream can also produce static charge in many specimens, and this could result in attraction of more particulate debris. Use of an ionizing nozzle on the gas stream may eliminate this problem.

7.3 *Vacuum Conditions and Time*—Specimens that were in equilibrium with the ambient environment prior to insertion

into the vacuum chamber may desorb surface species, such as water vapor, plasticizers, and other volatile components. This may cause cross-contamination of adjacent samples and may increase the chamber pressure. It also may cause changes in surface chemistry of the specimens of interest.

7.4 *Effects of the Incident Flux:*

7.4.1 The incident electron flux in AES, ion flux in SIMS, and, to a lesser extent, the photon flux in XPS, may induce changes in the specimen being analyzed (**2**), for example by causing enhanced reactions between the surface of a specimen and the residual gases in the analytical chamber. The incident flux also may locally heat or degrade the specimen, or both, resulting in a change of surface chemistry or a possible rise in chamber pressure and in contamination of the analytical chamber. These effects are discussed in Guide E983.

7.4.2 Residual gases or the incident beam may alter the surface. One can test for undesirable effects by monitoring signals from the specimen as a function of time, for example by setting up the system for a sputter depth profile and then not turning on the ion gun. If changes with time are observed, then the interpretation of the results must account for the observation of an altered surface. This method may also detect desorption of surface species. Care should be taken to account for the possible effects of incident beam fluctuation.

7.4.3 The incident ion beams used during SIMS, AES, and XPS depth profiles not only erode the surface of interest but can also affect surfaces nearby. This can be caused by poor focusing of the primary ion beam and impact of neutrals from the primary beam. These adjacent areas may not be suitable for subsequent analysis by surface analysis methods. In some cases, sputtered material may be deposited onto adjacent areas on the specimen or onto other specimens that may be in the analytical chamber.

7.5 *Analytical Chamber Contamination:*

7.5.1 The analyst should be alert to materials that will lead to contamination of the vacuum chamber as well as other specimens in the chamber. High vapor pressure elements such as mercury, tellurium, cesium, potassium, sodium, arsenic, iodine, zinc, selenium, phosphorus, sulfur, etc. should be analyzed with caution. Many other materials also can exhibit high vapor pressures; these include some polymers, foams, and other porous materials, greases and oils, and liquids.

7.5.2 Even if an unperturbed specimen meets the vacuum requirements of the analytical chamber, the probing beam required for analysis may degrade the specimen and result in serious contamination, as discussed in 7.4.1.

7.5.3 Contamination by surface diffusion can be a problem, especially with silicone compounds (**3**) and hydrocarbons. It is possible to have excellent vacuum conditions in the analytical chamber and still find contamination by surface diffusion.

7.5.4 In SIMS, atoms sputtered onto the secondary ion extraction lens or other nearby surfaces can be resputtered back onto the surface of the specimen. This effect can be reduced by not having the secondary ion extraction lens or other surfaces close to the specimen. The use of multiple immersion lens strips or cleaning of the lens can help reduce this effect.

7.5.5 The order of use of probing beams can be important, especially when dealing with organic material or other fragile materials (such as those discussed in Section 12).

8. Specimen Storage and Transfer

8.1 *Storage:*

8.1.1 *Time*—The longer a specimen is in storage, the more care must be taken to ensure that the surface to be analyzed has not been contaminated. Even in clean laboratory environments, surfaces can quickly become contaminated to the depth analyzed by AES, XPS, SIMS, and other surface sensitive analytical techniques.

8.1.2 *Containers:*

8.1.2.1 Containers suitable for storage should not transfer contaminants to the specimen by means of particles, liquids, gases, or surface diffusion. Keep in mind unsuitable containers may contain volatile species, such as plasticizers, that may be emitted, contaminating the surface. Preferably, the surface to be analyzed should not contact the container or any other object. Glass jars with an inside diameter slightly larger than the width of a specimen can hold a specimen without contact with the surface. When contact with the surface is unavoidable, wrapping in clean, pre-analyzed aluminum foil may be satisfactory. Containers with a beveled bottom may also be appropriate for storing flat specimens (face down toward the bevel so that only the edges of the sample touch the container).

8.1.2.2 Containers such as glove boxes, vacuum chambers, and desiccators may be excellent choices for storage of specimens. A vacuum desiccator may be preferable to a standard unit and should be maintained free of grease and mechanical pump oil. Cross-contamination between specimens may also occur if multiple specimens are stored together.

8.1.3 *Temperature and Humidity*—Possible temperature and humidity effects should be considered when storing or shipping specimens. Most detrimental effects result from elevated temperatures. Additionally, low specimen temperatures and high to moderate humidity can lead to moisture condensation on the surface.

8.2 *Transfer:*

8.2.1 *Chambers*—Chambers that allow transfer of specimens from a controlled environment to an analytical chamber have been reported (**4-6**). Controlled environments could be other vacuum chambers, glove boxes (dry boxes), glove bags, reaction chambers, etc. Controlled environments can be attached directly to an analytical chamber with the transfer made through a permanent valve. Glove bags can be temporarily attached to an analytical chamber with transfer of a specimen done by removal and then replacement of a flange on the analytical chamber.

8.2.2 *Coatings*—Coatings can sometimes be applied to specimens allowing transfer in atmosphere. The coating is then removed by heating or vacuum pumping in either the analytical chamber or its introduction chamber. This concept has been successfully applied to the transfer of GaAs (**7**). Surfaces to be analyzed by SIMS or AES can be covered with a uniform layer, such as polysilicon for silicon-based technology (**8**). In this case, the coating is removed during analysis, however the influence of atomic mixing on the data must be considered.

9. General Mounting Procedures

9.1 In general, the specimen will be analyzed as received. Surface contamination or atmospheric adsorbates are not usually removed from such specimens because of the importance of analyzing an unaltered surface. In such cases, the specimen should be mounted directly to the specimen mount and held down with a clip or screw. Care should be taken to ensure that the clip or screw does not contact the surface of interest and that it will not interfere with the analysis probes. If specimen charging is a concern, the clip or screw can help to provide a conductive path to ground.

9.2 For some specimens, it is easier to mount the sample by pressing it into a soft metal foil or by placing it on the sticky surface of adhesive tape. The foil or tape is then attached to the specimen holder. Double-sided tape has the advantage of not requiring a clip or screw to hold it onto the specimen mount. Care should be taken to ensure that the surface to be analyzed does not come into contact with the foil or tape. All tape should be pretested for vacuum compatibility and potential contamination.

9.3 *Powders and Particles:*

9.3.1 *Substrates*—Powders and particles are often easier to analyze if they are placed on a conducting substrate. Indium foil is often used because it is soft at room temperature and powders or particles will imbed partly into the foil. (A problem with indium foil is that it redeposits, if sputtering is attempted.) Aluminum, copper, and other metal foils can be used, though only a small percentage of the powder particles may adhere to them. For XPS, powders can be placed on the sticky side of adhesive tape (see 9.2). Metallized tape is usually best and can meet the vacuum requirements of most XPS systems. If any adhesive tape is used, it should be pretested for vacuum compatibility and potential contamination. For some materials pressing a powder into a pliable substrate such as clean room paper tissue could be considered as an alternative, but the substrate must be pretested for vacuum compatability and for potential contamination.

9.3.2 *Pellets*—Many powders can be formed into pellets without the use of sintering aids. Alternatively, compression of the powder into a disk such as is used for preparation of KBr disks for infrared spectroscopy can be used. The resulting surface is then gently abraded with a clean scalpel blade prior to use. Forming pellets can be an excellent approach for XPS but often leads to specimen charging in AES and SIMS. Note that pressure and temperature-induced changes may occur. Alternatively, mixing powder with silver flake then polishing afterwards has been very successful although the outside of the powder grain is sacrificed. With electron beam excitation, even insulating powders can be analyzed this way as the powder grain is surrounded by a conductive medium which is also a good heat sink (**9**).

9.3.3 *Transfer of Particles*—Particles may sometimes be transferred to a suitable substrate by working under a microscope and by using a very sharp needle. Non-soluble particles may sometimes be floated on solvents and picked up on conducting filters. Particles can also be transferred onto adhesive tape or replicating compound as discussed in Guide E1829.

9.4 *Wires, Fibers, and Filaments*—Wire, fibers, and filaments may be of such size that it is not possible for the probing beam to remain on the specimen only, and background artifacts may result. In such instances, it may be possible to mount the specimen such that the background is sufficiently out of focus so that it does not contribute to the signal (for example, the sample might be mounted over a hole). Alternatively, many wires, fibers, or filaments can also be placed side-by-side or bundled to fill the field of view. In some cases, these specimens may be mounted like powders and particles (see 9.3).

9.5 *Pedestal Mounting*—For some analytical systems, especially those with large analysis areas, it is possible to mount a specimen on a pedestal so that only the specimen will be seen by the analyzer. This approach may allow analysis of specimens that are smaller than the analysis area.

9.6 *Methods of Reducing Charging:*

9.6.1 *General Considerations*—Specimen charging can be a serious problem with poorly conducting specimens. For many specimens, charging problems are usually more severe with incident electron or ion beams than with an incident X-ray beam. In XPS, charging is usually more severe for a focused monochromatic X-ray beam than for a large-area beam or non-monochromatic X-rays. If the surface is heterogeneous or the probing radiation is focused, the amount of charging can differ across the detection area. Additional reviews of charge control and charge referencing techniques in XPS can be found in Guide E1523.

9.6.2 *Conductive Mask, Grid, Wrap, or Coating*—A mask, grid, wrap, or coating of a conducting material can be used to cover insulating specimens and make contact to ground as close as possible to the surface that will be analyzed. A grid can also be suspended slightly above a surface (**10**). Wraps of metal foils have been used for the same purpose. In AES, it may be important to cover insulating areas of the specimen that are not in the immediate area of analysis so as to avoid the accumulation of scattered electrons and ions that could build up enough charge to deflect the electron probe beam to or from the specimen and perturb the analysis accordingly. Whenever sputtering is used in conjunction with a mask, grid, or wrap, care should be taken to ensure that material is not sputtered from the covering material onto the surface of the specimen. Removable grids have been reported that allow the grid to be moved during sputtering periods and returned for analysis (**11**). Materials such as colloidal silver, silver epoxy, or colloidal graphite can be used to provide a conducting path from near the point of analysis to ground; however, beware that outgassing of the solvent or of the conductive paint may cause a problem. Coating a specimen with a thin conducting layer and subsequently removing the coating by sputtering may be useful, but information regarding the topmost layer of the specimen will generally be lost. This approach can be useful for sputter depth profiling with the warning that charging may reappear as the layers are removed if the walls of the crater remain electrically insulating. Combinations of coatings and masks or wraps may be used.

9.6.3 *Flood Gun*—Low-energy electrons from a nearby filament can be useful for reducing charging of specimens in XPS. The window material in a conventional X-ray source can

also act as a source of electrons to reduce charging. Relative location of electron and ion optics in SIMS analysis of insulators can influence charging phenomena (**12, 13**). Positive ion SIMS depth profiling requires the use of a focused electron beam with similar or greater current density to the ion beam. Negative ion primary beams may be used. A low energy ion flood gun may also be used to minimize charging in AES.

9.6.4 In XPS, selecting an area of analysis within an area that is uniformly charged will help to minimize surface charging. Note that this approach, however, may select an area with properties that are different from adjacent areas.

9.6.5 *Incident Electron and Ion Beams:*

9.6.5.1 *Angle of Incidence*—The secondary electron emission coefficient and the incident beam current density are functions of the angle of incidence of the primary electron beam. Grazing angles of incidence increase the secondary electron emission coefficient and are, therefore, generally better for reduction of charging during AES analysis of flat specimens (**14-16**).

9.6.5.2 *Energy*—The secondary electron emission coefficient is also a function of the energy of the incident electron beam. Generally, incident energies where the secondary electron emission coefficient is greater than unity are better for reducing specimen charging. This usually means that the incident beam energy will have to be lowered, perhaps even as low as 1 keV, to eliminate charging and obtain useful Auger yields. For some layered specimens, it might be possible to achieve reduced specimen charging by increasing the energy of the incident electron beam such that penetration is made to a conducting layer beneath the layer being analyzed. This will result in charge neutralization through the insulating layer to the conducting layer if the conducting layer is suitably grounded. In SIMS, the energy of the incident ion affects specimen charging (**12**).

9.6.5.3 *Current Density*—Specimen charging may be reduced by decreasing the current density of the incident electron or ion beam. Reduction of the beam density can be achieved by reducing the total current, defocusing the beam, rastering the beam over a part of the specimen surface, or by changing the angle of incidence.

9.6.5.4 *Concurrent Electron and Ion Beams*—If a specimen is homogeneous with depth, charging in AES analysis sometimes can be reduced by sputtering the specimen during analysis. The incoming positive charge of the ion beam will partially neutralize the incoming negative charge of the electron beam. Ion-beam induced changes (see 10.9) must be considered. Using coincident low energy ion and low energy electron flood sources may also be used for charge compensation in XPS.

9.7 *Methods of Reducing Thermal Damage*—To reduce thermal damage, specimens can be mounted on a cold probe or stage with liquid nitrogen or other cold liquids or gases flowing through it. Some specimens such as powders could benefit from being compacted to pellets, thereby increasing heat dissipation. Good thermal contact between the specimen and the mounting system should be considered. Wrapping a specimen in a metal foil may be of value in some cases. Reducing the energy input during analysis would also be beneficial as discussed in 9.6.5.2 and 9.6.5.3, but this may result in longer data acquisition times.

10. Techniques for Specimen Preparation

10.1 *General Considerations:*

10.1.1 Often the surface or interface of interest lies beneath a layer of contaminants or other constituents. The problem is then to remove the overlayer without perturbing the surface or interface of interest.

10.1.2 For electronic devices, additional information regarding preparation of specimens can be found in (**17**).

10.2 *Mechanical Separation*—Sometimes it is possible to mechanically separate layers and expose the surface of interest. Except for possible reactions with the atmosphere, a surface exposed in this way is generally excellent for analysis. Delaminating layers and the inside surfaces of blister-like structures are often investigated in this way. Sputter depth profiling is generally not a good method to use on blister-like structures. At the point when the outer skin is penetrated by the ion beam, the data may become dominated by artifacts. Mechanical separation should be carried out just prior to transfer of the sample to the analytical instrument, or in-situ if possible.

10.3 *Thinning Versus Removal*—Complete removal of an overlayer may not be possible or desirable. It may be sufficient to thin the overlayer and continue using sputter depth profiling as discussed in 10.9.

10.4 *Removing the Substrate*—In some specimens, it may be easier to approach the interface of interest by removing the substrate rather than the overlayer, for example, when the composition of the substrate is not of interest, and the composition of the overlayer material is unknown. Chemical etches may be used more effectively and perhaps selectively when the composition of the material to be etched is known. In SIMS, if the overlayers are characterized by nonuniform sputtering, substrate removal may provide improved depth resolution (**18**). As discussed in 10.3, complete removal of the substrate may not be necessary.

10.5 *Sectioning Techniques:*

10.5.1 *General*—Sectioning (cutting) is most often applied to metals, but it can often be applied to other materials equally well. When using sectioning techniques, it is important to section such that minimum alteration occurs to the region of the specimen that will be analyzed. After sectioning, it is usually necessary to clean the specimen by sputtering in the analytical chamber prior to analysis.

10.5.2 *Methods of Sectioning*—Sectioning can be accomplished with an abrasive wheel, sawing, or shearing. The extent of damage is generally increased as cutting speed is increased. Semiconductor samples can also be sectioned by cleaving and polishing or with a focused ion beam (**19**). Chemical changes can be extensive if local heating occurs. Coarse grinding is usually done with abrasive belts or disks. Fine grinding is usually done with silicon carbide, emery, aluminum oxide, or diamond abrasives. Lubricating oils from cutting tools and grinding materials can contaminate the surface and should be avoided. If possible, cutting should be done dry, without lubricants.

10.5.3 *Mechanical Polishing*—Polishing is often the most crucial step in the sequence of preparing a lapped specimen. The abrasives used may be aluminum oxide, chromium oxide, magnesium oxide, cerium oxide, silicon dioxide, silicon carbide, or diamond. Choice of suspension medium (normally oil or water) and polishing cloth must be carefully considered.

10.5.4 *Chemical or Electrochemical Polishing*—Chemical or electrochemical polishing is sometimes applied after the final mechanical polishing. In chemical polishing the specimen is immersed in a polishing solution without external potentials being applied. In electrochemical polishing, a constant current or voltage is applied to the specimen. The solution and temperature selected will depend upon the specimen. These polishing methods usually prevent surface damage introduced by mechanical polishing. However, any type of polishing may alter the chemistry of the surface.

10.5.5 *Mounting Materials*—Compression and thermosetting materials are normally used for mounting specimens for sectioning. These mounting block materials are often of high vapor pressure and detrimental to the vacuum environment of the analytical chamber. Consequently, specimens are normally removed from the mounting blocks prior to analysis.

10.5.6 *Angle Lapping*—Angle lapping (also called taper sectioning) is a technique used to expose and expand the analysis area from a thin layer at some depth into a specimen (**20**). In AES, the diameter of the probing electron beam must be small relative to the expanded dimensions of the layer to be analyzed. The same considerations and techniques outlined in 10.5.1 would also be applicable to lapping. Spalling at weak interfaces may occur during these operations.

10.5.7 *Ball Cratering*—Ball cratering is similar to angle lapping (**21**) and is applicable when the radius of curvature of the spherical surface is large relative to the thickness of the films being analyzed.

10.5.8 *Radial Sectioning*—Radial sectioning is similar to ball cratering with a cylinder being used to create a crater instead of a spherical ball.

10.5.9 *Crater Edge Profiling*—Crater edge profiling is similar to angle lapping. Craters left by fixed or rastered ion beams often have a slightly slanting sidewall. An electron beam can be translated across the crater wall to obtain composition versus depth information (**22**).

10.5.10 *Focused Ion Beam Sectioning*—FIB sectioning with a liquid metal ion source can be used to expose the various layers within a material or expose buried regions of interest, such as a particle. Detailed analysis across the face of the FIB cut correlates to an analysis by depth. The FIB cut should be made with the appropriate step cuts so that the shape of the crater is appropriate for the analytical technique to be used. Note that atoms from the ion beam can be implanted and remain on the crater surface with concentrations approaching several percent. Also note that other residues may be present from in situ chemical assistance commonly used during the FIB process. Shallow etching of the implanted surface by a noble atom ion beam prior to analysis may be necessary to remove these residual materials. Additionally, redeposition of sputtered materials may occur.

10.6 *Growth of Overlayers*—The interface between an overlayer material and the substrate can be analyzed by AES and XPS if the overlayer can be grown slowly or in discrete steps (for example, increments of about one monatomic layer thickness). AES and XPS can thus be used to probe interface properties and possible reactions as the interface is grown. The composition at the interface measured in this way, however, may not always be identical with that for a thicker overlayer film. Gas-metal, metal-polymer, metal-semiconductor, and metal-metal interactions can be studied in this fashion.

10.7 *Solvents:*

10.7.1 High purity solvents can be used to remove soluble contaminants or overlayers if these materials are not of interest. Ethanol, isopropanol, and acetone are the most commonly used solvents and are often used in conjunction with ultrasonic agitation. A residue from the solvent may, however, be left on the specimen; for example, acetone is hydroscopic and can absorb water from the atmosphere. In addition, acetone could temporarily reduce emission from lanthanum hexaboride (LaB_6) cathodes used in analytical equipment.

10.7.2 Wiping a specimen with a tissue or other material that has been soaked with solvent can result in transfer of contaminants from the tissue to the specimen or from one area of the specimen to another.

10.7.3 A frozen carbon dioxide gas stream (carbon dioxide snow) is also effective for cleaning and can be used to remove organic or silicone overlayers from a specimen surface. The cleaning action is based on both solvent action and momentum transfer (**23**). The concerns of section 7.2 should be noted, however.

10.8 *Chemical Etching*—Chemical etches can be used to remove or thin an overlayer. In some cases an etch will be selective and etch down to, but not through, an interface. Specific etches can be found for many types of overlayers (**24**). Possible chemical or morphological effects on the substrate should be considered when using this procedure.

10.9 *Sputtering:*

10.9.1 *General Conditions*—Sputtering (ion etching) is often used to expose subsurface layers or, combined with analysis, to produce sputter depth profiles. One typically uses noble gas ions at 0.25 to 5 keV incident energy for sputtering. The effects of sputtering in surface analysis can be quite complex (**25, 26**), and reviews of sputtering can be found in Guide E1127. Some of the more important aspects are discussed in sections 10.9.2 through 10.9.8.

10.9.2 *Mixed Layer*—Ion bombardment will normally mix the top layers of a specimen to a depth that is comparable with the depth of analysis for AES and XPS (**27**). The extent of mixing will depend upon the composition of the specimen, the incident ion species, and the energy of the incident ions. Reducing the incident energies, changing the angle of incidence, and using a higher mass ion beam (for example, xenon) will reduce the depth of the mixed layer.

10.9.3 *Preferential Sputtering*—The constituents of a specimen may not sputter at uniform rates. This means that within the mixed layer the species that sputters most rapidly will be depleted, relative to the bulk composition of the material. This

may be an important consideration in quantitative studies using AES or XPS, especially when dealing with metal alloys (**28**).

10.9.4 *Chemical Changes*—The energetic ion beam used for sputtering can cause chemical changes in the specimen. The composition of the specimen will be dominant in determining if this will occur. For example, nitrates, phosphates, and carbonates can be converted to oxides under bombardment by 1 to 3 keV argon ions (**29**) . In some materials the ion milling process may also cause the formation of carbides resultant from adventitious carbon being mixed into the matrix. If a metal has multiple oxidation states, the maximum-valent oxide particularly is susceptible to reduction. In general, polymeric chemistry will be changed significantly during ion bombardment. In most cases there is a breakdown in the chemical structure to that of a graphitic species under higher kinetic energy primary ion fluence (**30, 31**). This process may be reduced by using cluster ion sources, such as SF_5^+, C_{60}^+, and argon cluster ions (**32, 33, 34, 35**), or with the use of certain low energy atomic beams (<200 eV O_2 or Cs^+) (**36, 37**).

10.9.5 *Sputtering with Hydrogen*—Sputtering with hydrogen might remove contaminants, in some cases with minimum alteration of the surface of interest (**38**).

10.9.6 *Surface and Interface Topography*—Unidirectional ion bombardment often produces changes in surface topography which seriously reduce the chances of properly exposing or determining a subsurface interface. The depth resolution is usually 3 to 15 % of the sputtered depth (**39**). Use of two ion guns incident at different angles can reduce sputter induced topographical features (**40**). Specimen rotation during sputtering may improve depth resolution (**41**). Alignment of the ion gun, analysis area, and rotation center is especially important if rotating a specimen whose chemistry varies across its surface. Lower incident energies can also improve depth resolution (**42**). Both smaller (**43**) and higher (**42**) angles of incidence have been shown to improve depth resolution for certain specimens.

10.9.7 *Sputtering and Heating*—Sputtering and heating (either simultaneously or sequentially) can be used to remove bulk impurities from metal foils or crystals when impurities segregate to the surface during heating. With single crystals, heating should be the final step to remove lattice damage.

10.9.8 *Sputter Enhanced Diffusion*—Sputtering can result in enhanced diffusion away from or toward the surface layer, producing distorted depth profiles. This can be a particular problem in SIMS (**44**) but may also be observed in XPS and AES profiles.

10.10 *Plasma Etching*—Plasma etching, using a reactive ion species such as oxygen, has been used to etch specimens when directional ion beams would produce artifacts in the data.

10.11 *Heating:*

10.11.1 Heating is not often used to clean specimens because only a small number of materials can withstand the temperatures required to drive off most contaminants. The technique should be considered for refractory metals and, possibly, ceramics. Heating can cause many changes in a specimen, so this technique should be used with discretion. Heating is also useful for the outgassing of specimens, the removal of implanted rare gas ions, and annealing out lattice damage caused by ion bombardment of single crystals. Methods of heating include resistive, electron bombardment, quartz lamp, laser, and indirect heating by conduction.

10.11.2 A variation of the heating technique is to combine lower temperatures with a reactive environment, such as oxygen or hydrogen. Contaminants may then be transformed to volatile species that can be pumped away. This approach would normally be used in a chamber separate from the analysis chamber.

10.12 *Vacuum Pumping*—When the overlayers to be removed consist of materials with higher vapor pressures than the surface of interest, then the overlayers may be pumped away in an auxiliary vacuum chamber. As discussed in 10.11.2, vacuum pumping may be used in conjunction with heating. This approach may require several days and is generally applicable to organic overlayers on inorganic substrates.

10.13 *Ultraviolet Radiation*—Exposure of a specimen to ultraviolet radiation in air can remove organic contaminants, including photoresist residues, from the surfaces of specimens (**45**). Note that some specimens may decompose or polymerize under ultraviolet radiation.

11. Fracturing, Cleaving, and Scribing

11.1 *Reaction Chambers*—Specialized ultra high vacuum (UHV) chambers for controlled exposure of specimens to unique environments are available that allow for specimen modifications by chemical or thermal means. Generally, these chambers are separated from the analytical chamber by UHV valves and a suitable specimen transfer mechanism to minimize possible contamination to the analytical chamber.

11.2 *Fracture:*

11.2.1 *General Conditions*—In-situ fracture has been extensively applied to metal specimens. However, it can be applied equally well to a broad range of materials and has found considerable use with composite materials, glasses, and ceramics.

11.2.2 *Impact or Tensile Fracture*—Impact fracture is used more than tensile fracture, possibly because such devices are simpler and readily available, and multiple specimens can be analyzed without breaking vacuum. In some cases, cooling the specimens to liquid nitrogen temperatures can facilitate fracture. Devices for tensile fracture have been reported (**46**) and are commercially available. Such devices are usually limited to single specimens per pump-down of the vacuum chamber. Specimens can be intergranularly fractured at proper strain rate at liquid nitrogen temperatures by tensile devices.

11.2.2.1 *Pretest*—It is possible to pretest specimens for impact fracture by mounting the specimen in a vise, and hitting it with a hammer or other methods that simulate the fracture stage. If an intergranular surface is exposed in this fashion, then it is likely that an intergranular failure will occur using the impact fracture mechanism in a UHV chamber. Pretesting is also suggested for hydrogen charged specimens (11.2.3.3).

11.2.3 *Preparation of Specimens:*

11.2.3.1 *Geometry, Location of Fracture*—Impact and tensile fracture devices generally have a preferred geometry for

the specimen to be fractured. The specimens are usually notched in an attempt to control the location of the fracture.

11.2.3.2 *Nonideal Geometries*—Specimens with nonideal geometries for impact fracture can still be fractured in the impact device by using additional pieces to allow the nonideal shape to approximate the ideal shape or by using special mounting in the fracture devices. When the geometry of a specimen does not fit the mounting mechanism well, or if the specimen is brittle, then it is advisable to wrap the end of the specimen held in the mount with a foil, such as aluminum or indium. This should help prevent premature and poorly located fractures.

11.2.3.3 *Hydrogen and Liquid Metal Charging*—Many metal specimens can be charged with hydrogen to increase the probability of intergranular fracture (**47**). The time and temperature required for charging will depend upon the specimen. Also, some metals can be embrittled by liquid metals, such as gallium or mercury (**48**). However, interpretation of the results will be made more difficult by the presence of residual liquid metal atoms in the fracture or by the formation of amalgams that affect the chemistry and composition of the specimen. Hydrogen-charged specimens will usually lose the hydrogen if they are allowed to remain at room temperature for a relatively short time. Such specimens can be shipped in dry ice by means of overnight express and stored in liquid nitrogen for many days without serious degradation of the charging. Also, hydrogen-charged specimens may need to be stressed or slowly strained in order for hydrogen embrittlement and in-situ fractures to occur.

11.2.3.4 *Coatings on Electrical Insulators*—When electrical insulators such as ceramic materials are fractured, problems with electrical charging may develop during analysis. To reduce these problems, it may be helpful to coat the outer surface of the insulator with a conducting material such as gold, prior to fracture.

11.3 *Cleaving*—Cleaving a single crystal specimen in an analytical chamber requires a special mechanism (**49, 50**).

11.4 *Scribing*—In-situ scribing to expose bulk material can be done by scraping the specimen with a hard, sharp point. Caution should be observed to avoid smearing of the constituents. The scribe mark should be wide enough to contain the probing beam. A variation of this concept is to use a wire brush within a load-lock chamber.

NOTE 1—Cleaving (11.3) and scribing (11.4) may introduce particles onto the surface.

12. Special Handling Techniques

12.1 *Prepumping of Gassy Specimens*—Some specimens will emit gases and cannot be analyzed because they degrade the vacuum environment in the analytical chamber. These specimens may be prepumped in an auxiliary vacuum chamber and quickly transferred to the analytical chamber without appreciable pickup of gases during the transfer. Perhaps the easiest method for prepumping is in the introduction chamber of the spectrometer. Removal of the volatile components may change the chemistry of the surface. Cross contamination between specimens may occur if multiple samples are in the chamber at the same time.

12.2 *Viscous Liquids*—Viscous liquids can be analyzed by XPS by placing a thick layer on a smooth substrate material and wiping away most of the liquid. Often the remaining specimen layer is of such thickness that no signal from the substrate is detected, yet the vacuum requirements of the analytical chamber are met.

12.3 *Solute Residue*—If solute residues from a solution are to be analyzed, the solvent can be placed in a small pan and the liquid evaporated. The solute residue will remain on the pan and may be transferred to the analytical chamber for analysis.

13. Keywords

13.1 Auger electron spectroscopy; secondary ion mass spectroscopy; specimen mounting; specimen preparation; specimen treatment; surface analysis; X-ray photoelectron spectroscopy

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