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## Standard Guide for Specimen Preparation and Mounting in Surface Analysis<sup>1</sup>

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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 *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 Analysis<sup>2</sup>

E 983 Guide for Minimizing Unwanted Electron Beam Effects in Auger Electron Spectroscopy<sup>2</sup>

E 1127 Guide for Depth Profiling in Auger Electron Spectroscopy<sup>2</sup>

E 1829 Guide for Handling Specimens Prior to Surface Analysis<sup>2</sup>

### 3. Terminology

3.1 *Definitions*—For definitions of surface analysis terms used in this guide, see Terminology E 673.

### 4. Significance and Use

4.1 Proper preparation and mounting of specimens is particularly critical for surface analysis. Improper preparation of

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

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 Auger electron spectroscopy (AES), X-ray photoelectron spectroscopy (XPS or ESCA), and secondary ion mass spectrometry (SIMS) are sensitive to surface layers that are typically a few nanometers (nm) thick. Such thin layers can be subject to severe perturbations caused by specimen handling (1)<sup>3</sup> 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 E 1829 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 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.

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

#### 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 (e.g., 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. 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, e.g., 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 thick coating will render the specimen unsuitable for subsequent surface analysis. Furthermore, exposure to an electron beam (e.g. in a SEM) can induce damage or cause the adsorption of surface species from the residual vacuum. If it is not possible to perform the 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, Ni tools contaminate Si). Tools should be cleaned in high purity solvents and dried prior to use. Non-magnetic 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 E 983.

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 the subsequent analysis by surface analysis methods. In some cases, sputtered material may be deposited onto other specimens that may be parked 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 Hg, Te, Cs, K, Na, As, I, Zn, Se, P, S, 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 via 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.

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.

**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.

**9.3.3 Transfer of Particles**—Particles may sometimes be transferred to 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 E 1829.

**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.

**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 (9). 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 (10). 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 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 (11,12) 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.

**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 (13-15).

**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 (11).

**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.