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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 *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[2]

E 983 Guide for Minimizing Unwanted Electron Beam Effects in Auger Electron Spectroscopy[2]

E 1127 Guide for Depth Profiling in Auger Electron Spectroscopy[2]

E 1829 Guide for Handling Specimens Prior to Surface Analysis[2]

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 specimens can result in alteration of the surface composition and erroneous 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 subject.

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 nanometres (nm) in thickness. Such thin layers can be subject to severe perturbations due to specimen handling **(1)**.[3]

4.3 This guide describes methods to minimize the effects of specimen preparation on the results obtained using surface sensitive analytical techniques. Methods to mount specimens to obtain desired information are also described. For additional information concerning handling of specimens, see Guide E 1829.

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. Analysts new to AES, XPS, and SIMS often need to be educated regarding these more stringent requirements.

5.3 *Contact*—Any handling of the surface area to be analyzed should be eliminated or minimized whenever possible.

5.4 *Visual Inspection*:

5.4.1 One should make a visual inspection, possibly using a light microscope, prior to analysis.

5.4.2 Features that are visually apparent in the laboratory outside the vacuum system may not be observable with the system's usual imaging method or through available viewports. When such a situation occurs, it may be necessary to mark the specimen with scratches while examining it visually so that the correct location for analysis can be found.

[1] This guide is under the jurisdiction of ASTM Committee E-42 on Surface Analysis and is the direct responsibility of Subcommittees E42.03 on Auger Electron Spectroscopy and XPS.

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

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

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

6. Specimen Influences

6.1 *History*—The history of a specimen should be considered in the course of handling and preparation of its surface. For example, if a specimen has previously been exposed to a contaminating environment, the need for exceptional care in handling and preparation might be less than for a specimen that came from a very clean environment.

6.1.1 If a specimen is known to be contaminated, precleaning may be warranted in order to 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. In some instances, the contamination itself may be of interest, for example, where a silicone influences adhesion. In these cases, no precleaning should be attempted.

6.1.2 Special caution should be exercised with specimens containing potential toxins.

6.2 *Information Sought*—The information sought can influence the handling 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, or can be exposed by in-situ fracture, cleaving, or other means.

6.3 *Specimens Previously Examined by Other Analytical Techniques*—Information available from other analytical techniques can influence the selection of surface-sensitive measurements. However, specimens that have been previously analyzed may have contamination on their surfaces. In particular, specimens examined in an electron microscope typically have been coated to reduce charging. This thick coating renders the specimens unsuitable for subsequent surface analysis. The electron beam in an SEM can also induce damage or additional contamination. In general, it is best to perform surface analysis before applying other techniques or to perform surface analysis 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. Use of clean tools ensures 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 contamination of Si), and these tools should be cleaned in high purity solvents prior to use. Tools should also be demagnetized. Tools should never unnecessarily touch the specimen surface.

7.1.2 Although gloves and wiping materials are sometimes used to handle specimens, it is likely that their use will 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. The surface to be analyzed should never be touched by the glove or other tools unless necessary.

7.1.3 Specimens must never be handled by an ungloved hand, even though the skin does not touch the surface of interest. Fingerprints contain mobile species which may contaminate the surface of interest. Skin oils and other skin material are not suitable for high vacuum.

7.1.4 Specimen holders and other materials used to mount specimens should be cleaned before and after each use where there is a possibility of cross-contamination of specimens. Avoid the use of tapes containing silicones and other mobile species.

7.2 *Particulate Debris*—Compressed gases from cans or from air lines used to blow particles and oil from the surface of a specimen must be considered a source of possible contamination. In particular, oil is often a contaminant in compressed air lines. In-line particle filters can reduce particles from these sources. Blowing one's breath on the specimen is also likely to cause contamination. Certainly, particles are removed from specimens in both methods but caution is advised in critical cases. A gas stream can 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*:

7.3.1 Because AES, XPS, and SIMS are sensitive to even the first atomic layer of contamination, the vacuum conditions in the analytical chamber can have an important influence on the data obtained. Specimens that are sputtered, fractured, cleaved, or scribed in the analytical chamber have surfaces that generally are very chemically active. In such cases, pay special attention to vacuum conditions and exposure time. Assuming a worst-case situation, that every gas atom/molecule striking the surface sticks, then about one atomic layer can absorb in 1 s at a chamber pressure of 1×10^{-4} Pa. The exact time required for absorption of one atomic layer will depend on several factors, including chamber pressure, gas species, chemical reactivity of the surface, and surface temperature. Reactive gas species, such as oxygen, water vapor, carbon dioxide, carbon monoxide, hydrogen, and methane tend to have high sticking coefficients. Their partial pressures are, therefore, of importance. Nearby hot filaments can increase the sticking coefficient of less reactive species, even on chemically inactive surfaces by molecular fragmentation or reaction with the filament. Less volatile species also can be deposited on a specimen from warm surfaces, such as the x-ray anode housing.

7.3.2 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 incident photon flux in XPS, can

cause changes in the specimen being analyzed **(2)**. Such a flux may cause enhanced reactions between the surface of a specimen and the residual gases in the analytical chamber. The incident flux also may locally heat the analysis area 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 One can test for the effects of incident electron or photon beams by monitoring signals from the specimen as a function of time. This could be done by setting up the system for a sputter depth profile and then not turning on the ion gun. If changes occur with time, then the incident beam or residual gases may be altering the surface. This technique also may detect desorption of surface species. Care should be taken to account for the possible effects of incident beam fluctuation or sample damage by the incident beam.

7.4.3 The incident ion beams used during SIMS, AES, and XPS depth profiles not only erode the area being analyzed 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 other specimens.

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. If there are questions regarding possible contamination, tests should be done before the specimen is admitted to the analytical chamber, during insertion for the case of rapid insertion probes, or using low intensity beams for initial analyses.

7.5.3 Contamination by surface diffusion can be a problem, especially with silicone compounds **(3)**. It is possible to have excellent vacuum conditions in the analytical chamber and still have 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 between analyses can help reduce this effect.

7.5.5 The order of incidence of probing beams can be important, especially when dealing with organic material or other materials as discussed in 10.9.4.

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 will not be 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 that volatile species, such as plasticizers, may be emitted from such containers, further 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*—Possible temperature effects should be considered when storing or shipping specimens. Most detrimental effects result from elevated temperatures. Additionally, low specimen temperatures and 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. Other vacuum chambers, glove boxes (dry boxes), and reaction chambers 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.

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 holder 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 tape. The foil or tape is then attached to the specimen holder. Care should be taken to ensure that the surface to be analyzed does not come into contact with the foil or tape. The 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 has been 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 adhesive tape. The metallized kind is usually best and can meet the vacuum requirements of most XPS systems. The adhesive tape used 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. 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, compression of the powder into a disk such as is used for preparation of KBr disks for infra-red spectroscopy can be used. The resulting surface is then gently abraded with a clean scalpel blade prior to use.

9.3.3 *Transfer of Particles*—Particles may sometimes be transferred to suitable substrate by using a very sharp needle and by working under a microscope. Particles that are not soluble 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.

9.3.4 *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.

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, information regarding preparation of specimens can be found in **(9).**

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 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. This could be the case 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 **(10).** As discussed in 10.3, complete removal of the substrate may not be necessary.

10.5 *Sectioning Techniques*:

10.5.1 *General*—Sectioning 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*—Cutting 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 **(11)**. 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, or aluminum oxide abrasives. Lubricating oils from cutting tools and grinding materials may contaminate the surface and should be removed. If possible, sectioning (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 or polished 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. Polishing may alter the chemistry of the surface.

10.5.5 *Mounting Materials*—Compression and thermosetting materials are normally used for mounting specimens for