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Standard Guide for Handling Specimens Prior to Surface Analysis[1]

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1. Scope

1.1 This guide covers specimen handling and preparation prior to 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 or ESCA), and

1.1.3 Secondary ion mass spectrometry, SIMS.

1.1.4 Although primarily written for AES, XPS, and SIMS, these methods may 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 1078 Guide for Specimen Preparation and Mounting in 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 handling and preparation of specimens is particularly critical for analysis. Improper handling 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. The goal must be to preserve the state of the surface so that analysis remains representative of the original subject.

4.2 Auger electron spectroscopy (AES), X-ray photoelectron spectroscopy (XPS), and secondary ion mass spectroscopy (SIMS) are sensitive to surface layers that are typically a few nanometres thick. Such thin layers can be subject to severe perturbations from improper specimen handling **(1)**.[3]

4.3 This guide describes methods to minimize the effects of specimen handling on the results obtained using surface-sensitive analytical techniques. It is intended for the specimen owner or the purchaser of surface analytical services and the surface analyst. Because of the wide range of types of specimens and desired information, only broad guidelines and general examples are presented here. The optimum handling procedures will be dependent on the particular specimen and the needed information. It is recommended that the specimen supplier consult the surface analyst as soon as possible with regard to specimen history, the specific problem to be solved or information needed, and the particular specimen preparation or handling procedures required. The surface analyst also is referred to Guide E 1078 that discusses additional procedures for preparing, mounting, and analysis of specimens.

5. General Requirements

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

5.2 Specimens must never be in contact with the bare hand. Handling of the surface to be analyzed should be eliminated or minimized whenever possible.

5.3 Specimens should be transported to the analyst in a container that does not come into direct contact with the surface of interest.

5.4 In most cases, the analysis will be performed on the "as received" specimen. Surface contamination or atmospheric adsorbates are not usually removed because of the importance of analyzing an unaltered surface and as these are often the regions of interest. Care must then be taken in the handling the specimen to ensure that no outside agents come in contact with the surface to be investigated. These agents include: solvents or cleaning solutions, gases (including compressed air) or vapors, metals, tissue or other wrapping materials, tape, cloth, tools, packing materials or the walls of containers. If the specimen

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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.

supplier is uncertain of the requirements for a specific specimen, they should consult the analyst.

5.5 In some cases (for example, for a large specimen), it may be necessary to take a representative sample from the specimen. Selection of a smaller sample from a larger specimen should be done while considering the information being sought because inhomgeneities are often present. It is recommended that this choice be made in consultation with an experienced analyst.

5.6 Numerous methods exist for the mounting of a specimen in preparation for analysis. Refer to Guide E 1078.

5.7 *Hazardous Materials*—Special caution shall be exercised with specimens containing potential toxins or other hazardous materials. Whenever possible chemical hazard data sheets should be supplied with the specimen.

5.8 The severity of the requirement for specimen handling varies dramatically with the condition of the surface and the location of the information being sought. The list in Appendix X1 describes types of specimens by their increasing sensitivity to handling.

6. Specimen Influences

6.1 The analyst should be advised of the specimen history, special storage or transport requirements, exposure to possible contaminants, and the information being sought.

6.2 *History*—The history of a specimen can influence the handling of its surface. For example, a specimen that has been previously 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.

6.3 *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 renders the specimens unsuitable for subsequent surface analysis. Exposure to an electron beam (for example, in a SEM) also can induce damage or deposit additional contamination. If it is not possible to perform the surface analysis work first, then the analysis should be done on a different, but nominally identical, specimen or area of the specimen.

6.4 *Information Sought*—Surface chemical analysis can be performed on a wide range of specimens and can be used to obtain very different types of information about surfaces or interfaces. The degree of care that must to be taken depends upon the type of analysis that is required and the nature of the problem. The information being sought usually falls into four general categories: (A) information on the outermost surface; (B) information as a function of depth (depth profile) or at a buried interface; and (C) information that will require subsequent specimen preparation by the analyst.

6.4.1 Type A specimens include those to be investigated for surface contamination, surface stains, and adhesion failures. This category requires the most care in preparation and packaging. Ideally, nothing should be allowed to contact the surface of interest. In practice, it may be necessary to wrap the samples to avoid damage in transit. (See Appendix X3.)

6.4.2 Type B specimens include those that require the investigation of thick and thin films, single layers, multilayers, metal contact layers on semiconductors, coatings, dopant profiles, and the chemical and physical properties at an interface between two dissimilar materials. For this category the packaging requirements are not as stringent although care should still be taken to not contaminate the specimen. In this class, the information sought comes from a layer below the outermost surface and superficial surface contamination is not an issue. With semiconductor samples, care should be taken to avoid particulate contamination of the surface as this can degrade the quality of the depth profiles.

6.4.3 Type C specimens include those that require preparation by the analyst and includes specimens for in-situ fracture, metallurgical lapping or polishing, and specimens that are part of a larger assembly. Generally, these specimens must be shaped (for example, for fracture), chemically or mechanically altered (as happens with lapping) or disassembled. Few special precautions are needed for samples that are to be fractured, or undergo further sample preparation by the analyst. For specimen in a larger assembly or subassembly, it may be preferable to leave the specimen in place and let the analyst remove it for analysis. Nonetheless, care should still be taken to not contaminate the specimen.

6.5 Clearly identify all specimens with a unique name or identifier. If it is possible to permanently attach this identifier to the specimen (without disturbing the area of interest), do so. Clearly indicate the area of analysis by marking up a drawing or a photograph. If necessary, a scribe or permanent ink marker can be used on an area adjacent to the areas of interest. If there is any doubt as to which side of the specimen is to be analyzed, clearly mark the back of the specimen.

6.6 *Precautions*—Do not touch the surface of interest, either by hand or with a tool. Do not “protect” the surface of interest by covering it with tape, contaminated foil or porous wrapping material. Do not use a diamond scribe to mark semiconductors. Fragile specimens should not be mounted onto double-sided tape.

7. Sources of Specimen Contamination

7.1 An unprotected hand must never handle specimens, even when the skin will not touch the surface of interest. Fingerprints and hand creams contain mobile species that may migrate and contaminate the surface of interest.

7.2 Handling of specimens only should be done with clean tools to ensure that the specimen surface is not altered prior to analysis. Tools should be made of materials that do not transfer to the specimen or introduce spurious contaminants onto surfaces (for example, Ni tools contaminate Si). Tools should be cleaned regularly 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.

7.3 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 tool unless necessary.

7.4 Blowing on the specimen using a compressed gas source (for example, to remove particulates) is likely to cause contamination even when using a noble gas (such as argon or helium) because of the likelihood that the nozzle or the delivery line may contain oils, organics or particulates. Blowing with the mouth is not recommended.

7.5 To minimize the potential for contamination of the area to be analyzed during handling, the preparer should select one of the methods in the list in Appendix X2.

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 become contaminated quickly 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 that volatile species (for example, 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, preanalyzed aluminum foil may be satisfactory. For semiconductor samples, standard wafer carriers are generally adequate.

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 also may 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 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 **(2,3,4)**. Controlled environments could be other vacuum chambers, glove boxes (dry boxes), glove bags, reaction chambers, and so forth, which can be attached directly to an analytical chamber with the transfer made through a permanent valve. Glove bags can be attached temporarily 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 then is removed by heating or vacuum pumping in either the analytical chamber or its introduction chamber. This concept has been applied successfully to the transfer of GaAs **(5)**. Surfaces to be analyzed by SIMS or AES can be covered with a uniform layer, such as polysilicon for silicon-based technology **(6)**. In this case, the coating is removed during analysis.

8.2.3 *Material Transfer*—Material transfer can be of value when the specimen is too large to be inserted into an analytical chamber or is needed for other purposes. The film or particles to be analyzed must transfer from the specimen to the replicating compound or tape. The replicating compound should be conductive for analysis by AES or SIMS **(7)**. Care must be taken to ensure that the material of interest is transferred to the compound or tape and that the compound or tape does not contain elements or compounds suspected to be of concern.

8.3 To minimize the potential for contamination of the area to be analyzed during handling, the preparer should select one of the containers in the list in Appendix X3.

9. Keywords

9.1 Auger electron spectroscopy; secondary ion mass spectrometry; specimen handling; surface analysis; X-ray photoelectron spectroscopy

APPENDIXES

(Nonmandatory Information)

X1. LIST OF SPECIMEN TYPES IN ORDER OF DECREASING SENSITIVITY TO HANDLING

X1.1 Reactive specimens where the reactive surface is to be analyzed.

X1.2 Specimens that will be fractured or freshly prepared outside the analysis chamber, including materials prepared in a controlled atmosphere.

X1.3 Specimens that have been exposed to the atmosphere and that are to be analyzed “as received.”

X1.4 Specimens with a contamination layer that is the object of analysis.

X1.5 Specimens with atmospheric adsorbates that may interfere with analysis.

X1.6 Non-uniform thin films that do not adequately cover the layer of interest.

X1.7 Thin films that will be delaminated prior to insertion

into the analysis chamber.

X1.8 Specimens with a contamination layer (or other topmost layer) that is of no interest and that will be removed in the analytical chamber.

X1.9 Specimens that will be fractured or freshly prepared in the analysis chamber or an adjacent chamber and kept *in vacuuo*.

X1.10 Uniform thin films that will be delaminated in the analysis chamber.

X1.11 Uniform thin films that are to be removed by ion etching or scraping in the analysis chamber to expose a layer or interface of interest.

X1.12 Uniform thick films that are to be removed by ion etching or scraping in the analysis chamber to expose a layer or interface of interest.

X1.13 Bulk materials where the information sought is on bulk properties.

X2. LIST OF HANDLING CONCERNS IN ORDER OF DECREASING SEVERITY

X2.1 The selection of the specific method should be made in careful consideration of the type of sample (see 4.7) and the information being sought.

X2.2 Use a clean specialty tool (for example, wafer tweezers or non-magnetic grippers) to hold the specimen. The tool should be washed in spec grade ethanol and dried between uses. A powder-free, silicone-free latex glove should cover the hand.

X2.3 Use clean, dry tweezers or grippers to hold the specimen. The tool should have been washed in ethanol. A powder-free, silicone-free latex glove should cover the hand.

X2.4 The hand, covered by a powder-free, silicone-free, latex glove, can be used to hold the specimen by the edges. Do not make contact with the analytical surface.

X2.5 The hand, covered by a polyethylene glove, can be used to hold the specimen by the edges. Do not make contact with the analytical surface.

X2.6 An ungloved hand can be used to manipulate a clean specialty tool, tweezers or grippers to hold the specimen. The tool should be dipped or rinsed in spec grade ethanol and dried after each use.

X2.7 Use a piece of lint-free paper (for example, a chemical wipe) to grip the specimen by the edges. The paper must not come into contact with the analytical surface.

X3. LIST OF CONTAINER TYPES IN ORDER OF DECREASING SPECIMEN PROTECTION

X3.1 The list is in order from most protective to least protective. The selection of the specific container should be made in careful consideration of the type of sample (see 4.7), previous handling (see 6.5) and the information being sought.

X3.2 Place the specimen in a specialty container (for example, a wafer carrier) that is specifically designed to transport the specimen securely. The internal surfaces of the container should have minimum contact with the surface to be analyzed.

X3.3 Place the specimen in a clean glass jar. A piece of double-sided adhesive tape can be placed in the bottom of the jar to hold the specimen in place and to keep it from coming into contact with the lid of the jar. The jar should be large enough so that the sides of the jar do not make contact with the edge of the specimen. The lid should composed of a chemically inert material and securely fastened onto the jar.

X3.4 In the absence of a suitable lid, aluminum foil can used to cover the opening. The foil should not contact the specimen.

X3.5 In the absence of aluminum foil, plastic wrap can be used to cover the opening. The wrap should not contact the specimen.

X3.6 Place the specimen in a polypropylene container. A piece of double-sided adhesive tape can be placed in the bottom of the container to hold the specimen in place and to keep it from coming into contact with the lid. The container should be large enough so that the sides of the jar do not make contact with the edge of the specimen. The lid should screw on or snap on and be composed of a same material as the container.

X3.7 Place the specimen in a polyethylene container. A piece of double-sided adhesive tape can be placed in the bottom of the container to hold the specimen in place and to keep it from coming into contact with the lid. The container should be large enough so that the sides of the jar do not make contact with the edge of the specimen. The lid should screw on or snap on and be composed of a same material as the container.

X3.8 Wrap the specimen in clean aluminum foil, taking care to minimize contact with the surface to be analyzed. The specimen can then be transported in a mailing container.

X3.9 Wrap the specimen in lint-free paper (for example, a chemical wipe), taking care to minimize contact with the surface to be analyzed. The specimen can then be transported in a mailing container.

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