Designation: E 1829 – 97

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

This standard is issued under the fixed designation E 1829; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

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, methods also will 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. 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. 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, which results in erroneous data.

4.2 Auger electron spectroscopy, X-ray photoelectron spectroscopy, and secondary ion mass spectroscopy are sensitive to surface layers that are typically a few nanometres in thickness. 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 and preparation 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 specific 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 preparer or owner consult the surface analyst as soon as possible with regard to specimen history, specific problem to be solved or information needed, and 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. Scientists, engineers, and managers new to AES, XPS, and SIMS often need to be educated regarding these more stringent requirements.

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

5.3 *Hazardous Materials*—Special caution should be exercised with specimens containing potential toxins or other hazardous materials.

6. Specimen Influences

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

6.2 *Information Sought*—The information sought can influence the handling of a specimen. If the information sought lies

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

Current edition approved Sept. 10, 1997. Published June 1998. Originally published as E 1829 – 96. Last previous edition E 1829 – 96.

[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.

beneath an overlayer that must be sputtered away in the analytical chamber, or can be exposed by in situ fracture, cleaving, or other means, then more handling may be allowed than if the information sought comes from the exterior surface of a specimen.

6.3 *Specimens Previously Examined by Other Analytical Techniques*—Information available from other analytical techniques can influence the selection of surface-sensitive measurements and handling of a specimen. Specimens that have been analyzed previously may be contaminated 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, although surface composition information can be obtained by sputtering through the overlayer. The electron beam in an SEM also can induce damage or deposit 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 Specimens must never be handled by hand, even though the skin does not touch the surface of interest. Fingerprint materials contain mobile species that may migrate and contaminate the surface of interest. Skin oils and other skin material are not suitable for high vacuum.

7.2 Handling of specimens only should 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 exist in the analytical chamber. Tools should be made of materials that do not transfer to the specimen or introduce spurious contaminants onto surfaces (for example, Ni contamination of Si), and these tools should be cleaned in high-purity solvents and dried prior to use. Tools also should be demagnetized. 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. Clean-room quality 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 is likely to cause contamination.

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 become contaminated quickly to the depth analyzed by AES, XPS, SIMS, and other surfacesensitive 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 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, 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 have been 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*—Possible temperature 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.

9. Keywords

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

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