NOTICE: This standard has either been superseded and replaced by a new version or withdrawn.
Contact ASTM International (www.astm.org) for the latest information

Designation: E 1829 – 02

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, 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

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

Current edition approved April 10, 2002. Published April 2002. Originally published as E 1829 – 96. Last previous edition E 1829 – 97.

[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.

iTeh Standards (https://standards.iteh.ai) Document Preview ASTM E1829-02 https://standards.iteh.ai/catalog/standards/sist/9be40e55-2...-4de9-aac9-45fa72c0af44/astm-e1829-02

Copyright © ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States.

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

iTeh Standards
(https://standards.iteh.ai)
Document Preview
ASTM E1829-02