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Surface chemical analysis — Sample handling, preparation and mounting —

Part 2: Documenting and reporting the preparation and mounting of specimens for analysis

Analyse chimique des surfaces — Manipulation, préparation et montage des échantillons —

Partie 2: Documentation et notification des données de préparation et de montage des échantillons pour analyse

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO document should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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This document was prepared by Technical Committee TC 201, *Surface Chemical Analysis*, Subcommittee SC 2, *General Procedures*.

This first edition of ISO 20579-2 cancels and replaces ISO 18116:2005, which has been technically revised.

A list of all parts in the ISO 20579 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

0.1 General introduction to the ISO 20579 series

Because sample preparation and handling can have a significant impact on the physical and chemical properties of a sample surface, reliable surface analysis depends upon knowing the analysis objective and knowledge of the sample history including aspects of how the sample has been prepared, stored, processed, and handled prior to and during analysis. The ISO 20579 series specifies information that is required to be collected and included as part of the sample history (sample provenance information). The ISO 20579 series describes information that anyone seeking surface analysis is required to provide to an analyst[2] and additional information that an analyst is required to include in the sample provenance record regarding sample handling, storage, and processing.[3] ISO 20579-1 and ISO 20579-2 describe the information to be recorded regarding sample selection, handling, and storage. ISO 20579-1 describes information that is necessary for the sample provenance record and an analyst regarding sample selection and preparation when requesting surface analysis. ISO 20579-2 indicates information about sample handling, preparation, mounting and processing to be recorded and reported by the analyst. ISO 20579-3 and ISO 20579-4 focus on specific reporting requirements associated with biomaterials[5] and nanomaterials,[4] respectively. Each part of the ISO 20579 series can be used independently of the other parts, although the general reporting requirements described in ISO 20579-1 and ISO 20579-2 are applicable to a wide range of materials and are not reproduced in ISO 20579-3 and ISO 20579-4.

Although primarily prepared for the surface-analysis techniques of Auger-electron spectroscopy (AES), X-ray photoelectron spectroscopy (XPS) and secondary-ion mass spectrometry (SIMS), the methods described in this document are also applicable to many other surface-sensitive analytical techniques such as ion-scattering spectrometry (ISS and including low- and medium-energy scattering LEIS, MEIS), scanning probe microscopy (SPM), low-energy electron diffraction (LEED) and electron energy-loss spectroscopy (EELS), where specimen handling can influence surface-sensitive measurements. AES, XPS, and SIMS are sensitive to surface layers that are typically a few nanometers thick. Such thin layers can be subject to severe perturbations caused by specimen handling or surface treatments that can be necessary prior to introduction into the analytical chamber. Proper handling and preparation of specimens is particularly critical for dependable analysis. Improper handling of specimens can result in alteration of the surface composition and unreliable data.[6][7]

0.2 Introduction to ISO 20579-2

This document is intended for the analyst and describes information that is required to be recorded and reported regarding the sample handling, storage, mounting and other aspects of preparing a sample for surface analysis. This information becomes part of sample provenance record to help validate the reliability and usefulness of data obtained from surface-analysis methods.[8]

Although the categories of necessary reporting are similar for all specimens, the details of the required sample handling can vary depending on the nature of the sample and analysis objectives. When the outer surface of a specimen is to be analysed the specimen needs to be handled carefully so that the introduction of spurious contaminants is avoided or minimized. The goal is to preserve the state of the surface during preparation and mounting so that the analysis remains representative of the original specimen. In other cases, sample processing is required to enable access to the surface or interface to be analysed and some aspects of the sample handling might be less stringent. In all cases, the nature of sample handling and preparation for the desired analyses need to be recorded and reported.

Normative annexes to this document describe methods that the surface analyst can use to minimize the effects of specimen preparation when using any surface-sensitive analytical technique. Annexes also describe methods to mount specimens to ensure that the desired analytical information is not compromised. Annex A describes approaches, issues, and good practices regarding sample handling in preparation for analysis. Annex B provides information about sources of contamination, sample handling and storage requirements for differing analysis objectives.

Surface chemical analysis — Sample handling, preparation and mounting —

Part 2: Documenting and reporting the preparation and mounting of specimens for analysis

1 Scope

This document specifies information to be reported by an analyst in a datasheet, certificate of analysis, report or other publication regarding the handling, preparation, processing and mounting of specimens for surface analysis. Appropriate sample handling with adequate documentation is needed to ensure and assess reliability and reproducibility of analyses. Such information is in addition to other details associated with specimen synthesis, processing history and characterization, and should become part of the data record (sometimes identified as provenance information) regarding the source of the material and changes that have taken place since it was originated.

This document also includes normative annexes that summarize important processes and common approaches relevant to sample preparation and mounting for surface analysis. The descriptions of procedures for which records and reporting are required follow the steps that an analyst would follow from receiving the samples, to cleaning or processing outside of the analysis chamber, sample mounting and then treatments in the analysis chamber. The descriptions of the processes and their implications are intended as an aid for the analyst in understanding the reporting requirements for the specialized sample-handling conditions and approaches required for analyses by techniques such as Auger electron spectroscopy (AES), secondary-ion mass spectrometry (SIMS), and X-ray photoelectron spectroscopy (XPS). The methods described are also applicable for other analytical techniques, such as total reflection X-ray fluorescence spectroscopy (TXRF), low energy electron diffraction (LEED), some types of scanning probe microscopy (SPM) including atomic force microscopy (AFM) and scanning tunnelling microscopy (STM), ultra-violet photoelectron spectroscopy (UPS) and medium- and low-energy ion scattering (MEIS and LEIS [also called ion surface scattering, ISS]) that are sensitive to surface composition.

This document does not specify the nature of instrumentation, instrument conditions (e.g., calibration or vacuum quality), or operating procedures required to ensure that the analytical measurements described have been appropriately conducted.

2 Normative references

The following documents are referred to in the text in such a way that some of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 18115-1, *Surface chemical analysis — Vocabulary — Part 1: General terms and terms used in spectroscopy*

ISO 18115-2, *Surface chemical analysis — Vocabulary — Part 2: Terms used in scanning-probe microscopy*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 18115-1 and ISO 18115-2 apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

4 Symbols and abbreviated terms

AES	Auger electron spectroscopy
AFM	atomic force microscopy
EELS	electric energy-loss spectroscopy
ESCA	electron spectroscopy for chemical analysis (alternate name for XPS)
FIB	focused ion beam
ID	Identification
ISS	ion-scattering spectroscopy
LEED	low-energy electron diffraction
LEIS	low energy ion-scattering
MEIS	medium energy ion-scattering
PTFE	polytetrafluoroethylene
SIMS	secondary ion mass spectrometry
SPM	scanning probe microscopy
STM	scanning tunneling microscopy
TXRF	total reflection X-ray fluorescence spectroscopy
UPS	ultraviolet photoelectron spectroscopy
XPS	X-ray photoelectron spectroscopy

5 Provenance information to be collected or retained

5.1 Information record

Clause 5 deals with a sample information record that includes the relevant sample history, sample handling requirements, and analysis objectives. This information is usually provided by those requesting analysis. If it is not provided with the sample, it will need to be created (see 5.2).

Surface analysis is usually undertaken to collect useful information relevant to a sample for a specific reason at specific stages during the lifetime or history of the material. To assess the reliability and usefulness of the analysis, it is important to retain as many relevant sample history and handling details that are available to maintain the provenance[8][9][10] of the sample and data related to them.

Samples are often provided to an analyst by someone seeking information about one or more samples. Such samples should arrive with a history and the information described in ISO 20579-1 about the nature of the sample, the analysis objective, and any special requirements (ISO 20579-1:2024, 5.2), and with unique sample identifiers (IDs) and information, including dates, about previous handling, storage, and processing as relevant to the analysis objectives (ISO 20579-1:2024, 5.3).[2] Information about different types of

analysis objectives and the implications for sample handling are provided in ISO 20579-1:2024, Annex A and summarized in Annex B of this document. Detailed information records are especially important for nano-objects as described in ISO 20579-4:2018, Clauses 4 and 5.[4]

Information that an analyst shall record and add to the information record regarding the further preparation and handling of samples for surface analysis are described in Clause 6 of this document. This information, along with data collected becomes part of the information record that provides the history of the physical and chemical processes used on a sample that would allow assessment and replication of the measurements. Appropriate information to be retained and passed along with analysis information will vary depending on the nature and history of the sample and the analysis objectives as described in Clause 6. Dates should be provided whenever possible throughout the provenance record.

5.2 Verification or generation of sample information and analysis objectives

When an analyst receives one or more specimens, a necessary step is to examine the sample documentation, or establish it (with the owner) if not provided, including the nature of the sample(s), clear sample IDs, and appropriate analysis objectives. It is also important to determine if the samples have been handled properly to enable appropriate surface analysis and if relevant, that information about specific analysis areas or regions of analysis interest have been identified and documented.

If this information was not provided, the analyst shall assemble as much information as possible to establish a complete information record and analysis plan that will determine the sample handling and preparation necessary to obtain the desired information from the sample(s).

A visual inspection (documented) of each sample is important to verify information, sample condition and identification of any special features or problems such as fingerprints, adhesive, unexpected particles, or contaminants.

6 Information about sample handling and preparation for analysis to be documented and added to the sample information record

6.1 General

Information about the following topics shall be recorded and reported as part of the sample information record.

6.2 Adherence or exceptions to the general sample handling requirements

To maintain the stringent cleanliness required for meaningful surface analysis the general sample handling protocols listed below and in A.1.1 and B.3.2 shall be followed.[7][11] These generic requirements also appear in ISO 20579-1, 2024, B.2.2 and B.2.3. Any exceptions or deviations shall be documented. Justification for these measures and further details are provided in the Annexes of ISO 20579-1 and this document. Annex A of this document gives some additional details about general considerations for sample handling to minimize contamination and is summarized here.

Avoid touching the sample surface to be analysed with any material, including tools, hands, and containers, as well as adventitious contact from gases, liquids, particulates, or outgassing materials near the surface or present in the environment. If possible, air sensitive samples should be introduced using a glove box or a transfer vessel and documented in the reporting.

Thoroughly document all cleaning processes. Be extremely careful with any cleaning processes to make sure they do not alter any aspect of the sample surface important to the analysis objectives (for additional information see A.3.2). Be very careful to use only clean, pure, non-reactive gases (never blow on the sample by mouth) and delivery systems (including lines, nozzles, etc.) if required to dust off particulates. Note that canned air often contains fluorinated propellants which should be avoided.

If smaller samples must be prepared for analysis, thoroughly document any cutting or sectioning procedures, along with any associated cleaning (see A.3.3 for additional information).

Minimizing contamination also requires using cleaned sample handling tools and fixtures involved in sample mounting. It is also relevant to consider if volatile or otherwise mobile contaminants (e.g., Zn, Na, F) from previous samples in the vacuum system or adjacent during sample handling or storage could introduce contamination be detrimental to the desired analysis.

Example descriptions of exceptions or issues related to general sample handling requirements are given in EXAMPLES 1 to 3.

EXAMPLE 1 Because of the small sample size, touching the surface to be analysed with a clean mounting tool was unavoidable.

EXAMPLE 2 Although the analysis chamber and entry system were processed/cleaned between samples, we note that the most recent samples contained fluorine. Therefore, any fluorine identified on the current sample should be viewed with caution.

EXAMPLE 3 Carbon tape was used to mask the sample for charge control. The tape was within the sputter area, so during sputtering, it was redeposited onto the sample surface, resulting in carbon contamination.

6.3 Description of ex situ sample handling

Based on sample information and analysis objectives, the analyst shall determine and report the steps followed to store or prepare the samples for analysis, including any storage, cutting, sectioning, polishing, cleaning, or other preparation before insertion into the analysis chamber in accordance with A.3 (for ex situ handling) and B.3 and B.4 (for handling and storage).

Example descriptions of sample storage and ex situ treatments are given in EXAMPLES 1 to 6.

EXAMPLE 1 As received samples were placed in a desiccator where they were stored for one week before analysis.

EXAMPLE 2 To facilitate AES analysis of the layered structure the sample was polished by angle lapping.

EXAMPLE 3 To minimize sample charge buildup during AES and XPS analysis, the sample was thinned by focused ion beam milling.

EXAMPLE 4 Section was cut from corroded metal plate using a cleaned hack saw blade. Areas to be analysed were identified in an optical photograph.

EXAMPLE 5 Metal samples were machined so that they could be fractured in the analysis chamber to determine grain boundary composition after fracture.

EXAMPLE 6 MgO sample was heated to 800 C in air to remove organic contamination and moisture, and then was inserted into the intro chamber within 30 seconds, before the surface temperature reached 200 C.

6.4 Method of mounting samples for analysis

The analyst shall report details of the approach to sample mounting, which depend on the sample type, the instrument, analysis objectives, and the need for any special environmental control or in situ processing in accordance with A.4.

Example descriptions of sample mounting are given in EXAMPLES 1 to 5.

EXAMPLE 1 Sample was mounted directly onto a specimen holder using a spring clip to ensure good connectivity to spectrometer ground.

EXAMPLE 2 Potentially insulating sample was mounted for XPS analysis using double sided sticky tape, making sure the sample was isolated from the specimen holder so that surface potential could be controlled by the charge neutralization system.

EXAMPLE 3 A portion of the particles of this sample were pressed into indium foil to enable AES analysis of individual particles.

EXAMPLE 4 A solution of nanoparticles was deposited on a silicon wafer. Multiple deposits were made until the substrate was covered. It will be tested to determine that no signal arises from the substrate during XPS analysis.

EXAMPLE 5 Liquid sample was deposited on a LN_2 cooled substrate.

6.5 In situ sample cleaning or other sample preparation or processing

The analyst reports details of any in situ cleaning, and other sample preparation and processing of the sample prior to analysis, including any methods used to expose the region of analysis, in accordance with A.5.

Example descriptions of in situ cleaning and processing are given in EXAMPLES 1 to 6.

EXAMPLE 1 The high vapor pressure sample was degassed by (pre)pumping in the entry chamber (or an auxiliary vacuum system) before insertion into the main analysis chamber.

EXAMPLE 2 The sample was sputter cleaned to remove the thin surface oxide layer using low energy Ar^+ sputtering (0,5 kV). The sputter time for the sputter conditions would have removed approximately 10 nm of SiO_2.

EXAMPLE 3 Using a 20 kV focused Ga ion beam in the AES system, a cross section of the sample was created to enable analysis of the layered structure.

EXAMPLE 4 The sample was scribed inside the system to expose fresh surface for analysis.

EXAMPLE 5 The machined sample was fractured in the liquid nitrogen cooled impact fracture unit to expose grain boundaries for analysis.

EXAMPLE 6 An argon cluster ion source was used to remove organic contamination from the sample surface before analysis (Ar_{1000} at 4 kV). An ion current density of X was applied for 30 seconds (or specify the equivalent sputter removal of X nm of a reference material such as irganox).

6.6 Post analysis handling and storage

The analyst reports the disposition of samples after analysis, calling attention to any specific handling or storage of samples potentially relevant to later analysis or use.

Example descriptions of post analysis sample handling or storage are given in EXAMPLES 1 to 4.

EXAMPLE 1 Samples were discarded after analysis.

EXAMPLE 2 Samples were returned to dry box storage.

EXAMPLE 3 Samples were archived at location ABC.

EXAMPLE 4 Samples were transported to lab XYZ for further characterization.

Annex A
(normative)

Information on approaches, issues and good practices regarding sample handling and mounting in preparation for analysis

A.1 Introduction and overview

Once an analyst knows the nature of the sample, the sample history, and the analysis objectives, it is necessary to plan the approach to preparing a sample for analysis. This includes an assessment of the samples as received including a visual inspection of the samples, determination of storage and special handling requirements, and consideration of other types of analysis requirements (past or future). Depending on the nature of the samples and sample or instrument requirements the plan can involve storage of the samples prior to analysis, some type of ex situ treatment of the sample (usually cleaning and sectioning/cutting), must involve sample mounting for analysis, and there is sometimes the necessity for some type of in-spectrometer (in situ) processing. Each of these items is discussed in the following sections.

A critical aspect of planning is determining what sample handling or preparation is required to be able to examine the surface or interface of interest. If the outer surface is of importance, extremely careful sample handling is required to avoid destroying the information. Often the surface or interface of interest lies beneath a layer of contaminants or other material. This overlayer might require removal without perturbing the surface or interface of interest, as covered in A.3 for ex situ methods, and in A.5 for in situ methods.

Even when the outer surface is not the subject of interest and a buried interface will be exposed, it remains important to minimize processes or handling that add contamination to the sample, such as fingerprints, oils or most adhesives. Such contamination can spread and contaminate a surface of interest once it is exposed and be difficult to remove.

Additional information about sample handling, preparation and mounting can be found from multiple sources. General information on sample handling is available from Stevie, Garcia et. al.,[11] Czanderna, Powell and Madey,[12] and Geller.[6] Information about specific types of materials is also available including for polymers, Easton et al., [13] for Nanomaterials, Baer[14] and Baer et al.[15] and for GaN, Schaber, et al.[16] Information about contamination effects of storage containers is provided by G. Greczynski, L. Hultman[17] and about contamination spread in UHV conditions (relevant to storage and analysis chambers) by Elio et al. and Liu et al.[18][19]

A.1.1 General handing requirements for surface analysis

The degree of cleanliness required by surface-sensitive analytical techniques is much higher than for many other forms of analysis. Therefore, consideration must be given to sample handling and analysis order (see A.2.3) when other measurements are necessary. Sources of contamination are discussed in Annex B of this document and in ISO 20579-1:2024, Annex A,[2] and in ASTM E-1829.[20]

A simple high-level description of important handling requirements includes 1) specimens and mounts shall never be in contact with the bare hand and 2) handling of the surface to be analysed should be eliminated or minimized whenever possible. Fingerprints contain mobile species that can contaminate the surface of interest. Hand creams, skin oils and other skin materials are not compatible with high vacuum.

Analysis order can impact surface analysis results. In almost all cases, surface analytical measurements should be performed before use of bulk analysis methods (see A.2.3).

A.2 Assessment and planning

A.2.1 Introduction

It is important to make sure that the goals and objectives for the analysis are clearly specified. Such specific goals are necessary for determining how samples need to be handled, stored, and processed. For samples with special handling or storage requirements these goals usually should be specified before the samples arrive to the analyst, since actions need to be preplanned. Upon receipt, appropriate sample storage and handling methods shall be implemented to assure that the objectives of the analysis can be achieved. Samples shall be adequately identified. Are there any special sample handling requirements for storage, preparation, or analysis? Do samples require storage and, if so, how? What other analyses are required (or have been completed)?

Special precautions can be necessary for samples that contain or might contain toxins or other hazardous materials, and safety data sheets (SDSs) should be provided to the analyst for this type of sample.

A.2.2 Visual inspection before and after analysis

Upon receiving samples, it is good practice to conduct a visual inspection, using an optical microscope as appropriate. At a minimum, a check should be made for residues, particles, fingerprints, adhesives, contaminants, or other foreign matter. Record the observations in a laboratory notebook; annotated photographs can be useful.

Specimen features that are visually apparent when the sample is outside the vacuum system might not be observable after the sample is placed inside the surface-analysis instrument (for example through use of any available imaging method or through viewports). It can then be necessary to physically mark the specimen outside the area to be analyzed (e.g., by scribing or by a permanent ink marker) so that the analysis location can be found once the specimen is inside the vacuum system. Ensure that any method of marking the sample does not affect the subsequent measurements. Scribing a brittle material can leave unwanted detritus on the sample that can be deposited in the instrument or that could affect the analysis. Permanent ink markers can contaminate nearby regions by transport of volatile organics or by surface diffusion of solvent residues.

Changes that can occur during analysis can influence the data interpretation. Following any analysis, visual examination of the specimen is recommended to look for possible effects of ion-beam sputtering, electron-beam bombardment, X-ray irradiation, or exposure to the instrumental vacuum. Any changes should be documented.

A.2.3 Analysis Order

It is preferable for surface chemical analysis measurements to be made before the specimen is analysed by other techniques because such specimens can become damaged or be exposed to surface contamination. For example, insulating specimens analysed by electron microscopy are sometimes coated to reduce charging. Furthermore, exposure of the specimen to an electron beam (e.g., in a scanning electron microscope) can induce damage or cause the adsorption of surface species from the residual vacuum. Such coatings or modifications render the specimen unsuitable for subsequent surface chemical analysis. If it is not possible to perform the surface chemical analysis first, such an analysis should be performed on a different, but nominally identical, specimen or area of the specimen.

If multiple types of surface analysis are to be used, the order of analysis can have an impact as well. The preferred order can vary with the analysis question and techniques to be used. ISS is the most surface sensitive but involves the use of ion beams that can alter the surface over time. XPS is often considered to be the least damaging and is often done before methods that have greater risk of damage or are inherently destructive. AES does not remove material, but a focused electron beam can damage the surface. Static SIMS and SIMS require surface sputtering which can alter or remove information that might be obtained using XPS or ISS.

Recommendations for order of analysis are also discussed in ISO 20579-1:2024, Annex B[2] and in Lindfors.[7]

A.2.4 Differences in handling and mounting requirements for AES, XPS, SIMS and other surface analysis methods

Although the handling methods for AES, XPS, and SIMS are basically similar, there are some differences to consider in planning measurements. SIMS, for examples is particularly sensitive to silicone contaminants.[21] Mounting requirements can differ as well, for example, during XPS analysis of powder or nanomaterial, the particles are often packed together, but for AES, MEIS and scanning probe microscopy particles must be in a single layer.[14][4][15] The preparation of specimens for AES and SIMS requires attention because of potential problems with electron or ion beam damage or charging, or both. This document will note when specimen preparation is significantly different among the various techniques.

A.2.5 Sample charging issues

Insulating and mixed phase materials are often subject to surface charging during surface analysis. There are multiple approaches to dealing with sample charging.[22][13][23] When preparing an insulating specimen for analysis important considerations and options can be undertaken in external preparation (ex situ), during mounting or in the analysis chamber (in situ). Possible approaches to minimize charging include thinning the sample to lower sample resistance, in situ or ex situ coating of the specimen with a conducting material (wrapping with a foil or depositing a conducting layer in situ or ex situ), and mounting the sample isolated from ground. The approach an analyst can take varies with the specimen, the techniques applied and capabilities associated with the specific instrument.

A.2.6 Storage and transfer prior to analysis

Frequently, it is not possible to immediately analyse sample upon synthesis or receipt. Therefore, part of measurement planning can include the necessity of storing samples for some time before further processing or analysis. Depending on the nature of the sample and the required analysis, the means of storage can vary greatly. Even in ultrahigh vacuum conditions contamination can spread from sample to sample or from a sample holder to the sample.[18][19] The same general principles apply in all circumstances: avoid contamination of the surface to be analyzed, minimize changes to the sample due to environmental conditions and document all storage and transfer conditions (see B.4).

In some cases, samples can be stored and mounted in laminar flow hoods that minimize surface dust, or in a dry box, glove box or sealed container.

Some types of samples originate from highly specialized environments and laboratory environmental exposure would alter the surface chemistry. Such samples can arrive in environmentally sealed containers and should be transferred into the analysis system without atmospheric exposure. Glove boxes connected to spectrometers or anaerobic transfer containers are often used for such samples.

Specimen transfer from an external environment to a spectrometer might take a few forms depending on the sample type. Anaerobic transfer is briefly discussed in A.2.6.4. Issues of transfer of gassy, viscous, or liquid samples are discussed in relation to sample mounting in A.4.6.

A.2.6.1 Storage time

If a specimen is stored before analysis, care should be taken to ensure that the surface to be analyzed has not been contaminated during storage. Even in clean laboratory environments, surfaces can quickly become contaminated to the depth analyzed by AES, XPS, SIMS, and other surface-sensitive analytical techniques. For example, Liu et al.[19] discuss the evolution of surface contamination in ultra-high vacuum.

A.2.6.2 Storage containers

Containers selected for specimen storage should not transfer contaminants to the specimen via particles, liquids, gases, or surface diffusion. Containers that contain volatile species such as plasticizers (which can be emitted and then contaminate the sample surface) are unsuitable. The specimen surface to be analyzed should preferably not contact the container or any other object. Glass jars should be chosen with an appropriate diameter and height relative to the size of a specimen to constrain/hold the specimen without

glass in contact with the surface to be analyzed. Further details about storage containers are given by G. Greczynski and L. Hultman[17] and in B.4 and Table B.1.

When contact with the surface is unavoidable, wrapping in clean, pre-analysed aluminium foil can be satisfactory. Containers such as glove boxes, vacuum chambers, and desiccators are sometimes excellent choices for storage of specimens. A vacuum desiccator can often be preferable to a normal desiccator and shall be maintained free of grease and mechanical-pump oil.

NOTE Cross-contamination between specimens can occur if multiple specimens are stored in the same container.

A.2.6.3 Temperature and humidity

Possible temperature and humidity effects should be considered when storing or transferring 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.

A.2.6.4 Specimen transfer

Specially designed chambers that allow transfer of specimens from a controlled environment to a surface analysis chamber have been reported and commercial transfer vessels are available for some systems.[24][25][11] The controlled environment could be another vacuum chamber, a glove box (dry box), a glove bag, a reaction chamber, or a deposition chamber. This controlled environment chamber can be attached directly to the analytical chamber with the transfer made through a permanent valve. Glove bags can be temporarily attached to an analytical chamber with the specimen transferred by removal and then replacement of a flange on the analytical chamber. Increasingly XPS and other surface analysis systems have entry systems connected to glove boxes that allow samples to be handled and mounted in anaerobic environments just prior to entry into a spectrometer.

Coatings can sometimes be applied to specimens, thereby allowing transfer in the atmosphere. The coating is then removed by heating or by vacuum pumping in either the analytical chamber or its introduction chamber. This concept has been successfully applied to the transfer of GaAs.[26] Surfaces to be analyzed by AES or SIMS can be covered with a uniform layer, such as polysilicon for silicon-based technology.[27] In this case, the coating is removed by sputtering during analysis; however, the influence of atomic mixing on the analytical results needs to be considered.

The transfer of gassy, viscous or liquid samples into a spectrometer is discussed in A.4.6.

A.3 Ex situ sample handling (e.g. cleaning, cutting/sectioning, polishing, or exposing the region of interest before insertion into the analysis chamber)

A.3.1 Introduction and overview

All samples are handled in some way in preparation for analysis and the nature of handling often impacts the ability to obtain the desired information. As noted above, the guiding rule is do not touch or alter the surface to be analysed to the extent possible or appropriate based on the analysis objective. Often it is possible to simply insert a specimen into the analyser for analysis. However, there can be several reasons for which some type of processing of the sample is needed before it can be mounted for useful analysis. These include:

a) removing a contamination layer from the surface,

b) the need to alter the size or shape of a sample to allow the region of interest to fit in the spectrometer or become available for analysis,

c) exposing the region or interface of interest for the analysis,

d) optimizing the sample for analysis – such as thinning or coating an insulating sample for analysis to minimize charging.

In many situations, ex situ work is required to prepare the sample for mounting and sometimes to enable in situ processing to get to the region of interest. Therefore, in many cases, ex situ processing is necessarily a prelude to additional in situ processing.

A.3.2 Sample cleaning techniques

High-purity solvents can be used to remove soluble contaminants or overlayers that are not of interest. Ethanol (pure, not denatured), isopropanol, and acetone are the most used solvents, and are often used in conjunction with ultrasonic agitation. A residue from the solvent might, however, remain on the specimen, and acetone is hygroscopic and can lead to water adsorption from the atmosphere. Cleaning with acetone can reduce electron emission from lanthanum hexaboride cathodes if used in the AES instrument. Some sources recommend not using acetone for cleaning.[11] Hexane is sometimes used to remove lubricants. In general, a final rinse with isopropyl alcohol or methanol, followed by blowing dry with clean compressed gas can help minimize residue.[11] Wiping a specimen with a tissue or other material that has been soaked with solvent can result in transfer of contaminants from the tissue to the specimen or from one area of the specimen to another.

Compressed gases from aerosol cans or from air lines are often used to try to blow particles from the surface of a specimen or to attempt to clean a specimen. They, too, are necessarily considered a possible source of 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 can eliminate this problem.

A frozen carbon dioxide gas stream (carbon dioxide snow) is also effective for cleaning and can be used to remove some organic or silicone overlayers from specimen surfaces.[28] The cleaning action in this case is based on both solvent action and momentum transfer. However, care must be taken to ensure that the carbon dioxide gas stream does not lead to contamination as discussed in the paragraph above.

Specific cleaning issues related to nanoparticles (also relevant to other types of particles) are discussed in ISO 20579-4[4] and by Baer et. al.[15]

Note that to minimize sample contamination, tools using to handle samples and mounting hardware also must be cleaned, often using methods similar to cleaning specimens.

A.3.3 Sectioning/cutting techniques - altering the size or shape of a sample

A.3.3.1 Overview/introduction

Sectioning, or cutting techniques are most often applied to metals, but can be applied to some other materials. There can be multiple reasons for altering the size or shape of a sample and the processing requirements will again vary with the analysis objective. If the analysis objective relates to an already exposed surface, great care is required. However, if a grain boundary to be exposed by impact fracture is the objective, the primary objective can be to shape the sample to fit into a fracture unit in the spectrometer and the handling requirements are reduced. See A.5.4.2 regarding the design and application of specimens to be fractured in situ.

When using sectioning techniques, it is important to section with the minimum alteration to the region of the specimen that will be analysed. After sectioning, it can be necessary to clean the specimen externally as described above or internally by ion sputtering to remove a contamination or damage layer prior to analysis.

Compression and thermosetting materials are normally used for mounting specimens to be sectioned. These mounting-block materials are often high vapor pressure materials and detrimental to the vacuum environment of the analytical chamber. Consequently, specimens are normally removed from the mounting blocks prior to analysis.

A.3.3.2 Sectioning/cutting methods

Sectioning can be performed using an abrasive wheel or by sawing (perhaps with a cleaned hacksaw blade) or shearing. The extent of damage generally increases as cutting speed increases. Semiconductor samples

can also be sectioned by cleaving and polishing or by cutting using a focused ion beam (FIB).[29] Chemical changes can be extensive if local heating occurs during sectioning. Coarse grinding is usually performed with abrasive belts or disks. Fine grinding is usually done with silicone carbide, emery, aluminium oxide or diamond abrasives. Grinding materials and lubricating oils for cutting tools can contaminate the surface and should be avoided. If possible, cutting should be performed dry (i.e., without lubricants). Clean water can be used as a coolant if water would not impact the analysis.

A.3.4 Exposing the surface or buried interface of interest (ex situ)

A.3.4.1 Introduction

In circumstances where the outer surface, even after sample cleaning, is not the area of interest, additional ex situ preparation can be required to either expose or prepare the sample so that the region of importance can be analysed. A wide variety of methods have been applied, some of which are described or referenced below. Note that ex situ efforts can be a prelude to mounting and further in situ actions.

A.3.4.2 Mechanical separation

It is sometimes possible to mechanically separate layers and expose the surface of interest. This is sometimes done ex situ before entry into the analysis chamber or in situ, as described in A.5.4. Except for possible reactions with the atmosphere, a surface exposed in this way is generally suitable for analysis. Delaminated layers and the inside surfaces of blister-like structures are often investigated in this manner. Sputter depth profiling is generally not a good method to use on blister-like structures because, when the outer skin is penetrated by the ion beam, the data can become dominated by artefacts. Mechanical separation should be carried out just prior to transfer of the sample to the analytical chamber or in-situ, if possible.

Occasionally the “adhesive tape” method can be used to pull a loosely adhered layer from a substrate, exposing an interface for analysis.[30] Scraping is sometimes used to remove overlayers (or to expose fresh substrate) as long as the surface of interest is not damaged or contaminated in the process.

A.3.4.3 Chemical etching

Chemical etches can be used to remove or thin an overlayer. In some cases, an etch will be selective and etch down to, but not through, an interface. Specific etches can be found for many types of overlayers.[31] Possible chemical or morphological effects on the substrate should be considered when using this procedure. Complete removal of an overlayer might not be possible or desirable. It can be sufficient to thin the overlayer and then sputter away the remaining material inside the vacuum chamber. (Also see A.3.4.6 about substrate removal.)

A.3.4.4 Exposing layers using cross-sectioning techniques

Individual layers and layered interfaces can sometimes be analyzed by exposing the layers in a cross section of the sample. When the cross sectioning is done at an angle the apparent width of the layers is expanded and they can be analyzed by surface techniques with high spatial resolution such as AES.

A.3.4.4.1 Angle lapping and ball cratering and radial sectioning

Angle lapping (also called taper sectioning) is a technique used to expose and expand the analysis area available from a thin layer at some depth into a specimen.[32] In AES, the diameter of the primary electron beam should be small relative to the expanded dimensions of the layer to be analysed. Considerations related to layer thickness and technique resolution noted above for sectioning techniques are also applicable here. Spalling at weak interfaces can occur during these operations.

Ball cratering is similar to taper sectioning[33] and is applicable when the radius of curvature of the spherical surface is large relative to the thickness of the films being analyzed. Radial sectioning is similar to ball cratering with a cylinder being used to create a crater instead of a spherical ball.

A.3.4.4.2 Focused ion beam (FIB) cross sections

A focused ion beam (FIB) can be used obtain cross section samples for layer analysis. Such cross sectioning can be done in a special FIB system or possibly an SEM with FIB capability. Some surface analysis systems contain FIB capability enabling such sample preparation to be done in situ (See A.5.6). When the FIB cut is at an angle, analysis of the expanded layer can be used to obtain layer information as described above.

A.3.4.5 Mechanical, chemical and electrochemical polishing

Polishing to remove damage is often the most crucial step in the sequence of preparing a lapped specimen. The abrasives used can include aluminum oxide, chromium oxide, cerium oxide, silicon dioxide, silicon carbide, or diamond.[34] The choice of suspension medium (normally oil or water) and polishing cloth should be carefully considered for possible contamination of the specimen, embedding of abrasion material into the specimen, and complete removal of abrasion material from the specimen.

Chemical or electrochemical polishing is sometimes applied after the final mechanical polishing of a specimen.[34] 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 in an appropriate solution. The solution and temperature selected will depend upon the specimen. These polishing methods usually remove surface damage introduced by mechanical polishing. However, any type of polishing can alter the chemistry of the surface.

A.3.4.6 Substrate removal and sample thinning

In some specimens, it can be easier to approach the interface of interest by removing the substrate rather than the overlayer. This is sometimes call “backside removal”. There are a few circumstances for which this approach can be useful. One example occurs when the substrate composition is not of interest or the material composition of the overlayer is unknown. In SIMS, substrate removal can provide improved depth resolution if non-uniform sputtering of the overlayers occurs.[35]

Substrate removal can be accomplished chemically due to selective etching, mechanically by polishing and occasionally by cutting and then polishing.

Substrate thinning is also used to thin high resistivity samples to lower the effective resistance of samples that can be subject to charging during AES or other analyses.

A.3.5 Sample coatings

Coating can be used to both minimize sample charging and to protect a sample surface. Gold coatings (applied ex situ or in situ) have been used to minimize charge buildup for both XPS and AES. There are significant limitations to this method, but it can be useful. When electrical insulators such as ceramic materials are fractured in situ, problems with electrical charging sometimes develop during the surface analysis. To reduce this problem, it can be helpful to coat the outer surface of the insulator with a conducting material, such as gold, prior to fracture.

In some cases, pristine surfaces can be maintained during sample preparation and handling by coating the surface with a substance that can be easily removed prior to analysis. In some cases, the coating is removed with gentle heating and/or vacuum pumping of the sample, either in the sample introduction chamber or in the analysis chamber. This has been done, for example, with GaAs samples.[26] In other cases, the coating has been removed by sputtering,[27] but sputter-induced effects on the analytical results must be considered. Yet another technique is to cap the sample surface with a protective layer that is thin enough so that the surface analytical probe ‘sees through’ the layer to the sample surface below, so that the layer does not have to be removed. An example is the use of Al capping layers on transition-metal nitride thin films.[36] These techniques are not as widely used, since they must carefully be matched to the sample and analysis goals, and thus are often unique to specific samples and conditions.

A.4 Mounting techniques

A.4.1 Introduction

A great deal of creativity has been applied for the mounting of some samples for surface analysis. Useful references for sample mounting include papers by Stevie et al.,[11] Geller,[6] and Lindfors.[7] Only general ideas and high-level descriptions are described below.

A.4.2 General procedures

Many samples will be analysed as received. Surface contamination or atmospheric adsorbates are not usually removed because of the importance of analysing an unaltered specimen surface. In such cases, mount the specimen directly to the specimen holder with a clip or screw. This procedure is particularly important for AES if specimen charging is a concern; the clip can help to provide a conductive path to ground. Care should be taken to ensure that the clip or screw does not contact the area of interest and that it will not interfere with the incident beam or the particles to be detected during the analysis.

For some specimens, it is easier to mount the sample by pressing it into a soft metal foil (e.g., indium) or by placing it on the sticky surface of adhesive tape. The foil or tape is then attached to the specimen mount. Double-sided tape has the advantage of not requiring a clip or screw to hold it onto the mount. Care should be taken to ensure that the surface to be analysed does not contact the foil or tape. All tape should be pretested for vacuum compatibility and potential contamination. These methods are often satisfactory for XPS and some AES and static SIMS studies but are not often used for dynamic SIMS where the particle fluxes are larger.

A.4.3 Powders and particles

Powders and particles are often easier to analyse if they can be placed on a conducting substrate. Indium foil is often used because it is soft at room temperature, and powders or particles can be partly imbedded into the foil. Aluminium, copper or other metal foils can also be used for this purpose, although only a small percentage of the powder or particles might adhere to them. For XPS, powders can be placed on the sticky side of adhesive tape. Metallized tape is usually best; it can meet the vacuum requirements of most XPS systems. If any tape is to be used, it should be pretested for vacuum compatibility and potential contamination of the sample. Particles can sometimes be transferred to a suitable substrate by working under a microscope and by using a sharp needle. Non-soluble particles can sometimes be floated on solvents and picked up on conducting filters. Particles can also be transferred onto adhesive tape or replicating compound.

Many powders can be formed into pellets without the use of sintering aids or compressed into a disk. For example, equipment used for preparation of potassium bromide disks for infra-red spectroscopy could be used. The resulting surface is then gently abraded with a clean scalpel blade prior to use. The use of pellets can be an excellent approach for XPS but often leads to specimen charging in AES and SIMS. Some specimens can be modified, however, by pressure or temperature-induced changes during preparation of the pellets.

Sometime powders have been deposited and pressed onto scribed metal (often copper) substrates where the ridges help capture the powder.

Nanoparticles are a special case of powders (see ISO 20579-4).[4] Sometimes they are dry powders and sometimes suspended in solution. Depending on the analysis objective, some type of cleaning can be required. Mounting processes for such particles for surface analysis can take many forms such as deposition of liquid suspensions on a substrate, use of spin coating methods, or even dipping of samples in solution. Dry powders can be suspended in a liquid and deposited if the liquid does not impact the desired information. We note here that for XPS analysis an opaque layer or mound of particles is satisfactory. For other analyses such as ISS, MEIS and SPM a particle layer of “isolated” particles (no particle on top of another particle) is often required. For a comprehensive description of nanoparticle surface analysis issues see Baer et al.[15]

A.4.4 Wires, fibers, and filaments

Wire, fibers, and filaments can have sufficiently small dimensions that it is not possible for the primary beam to remain only on the specimen during the analysis. As a result, the recorded spectra will contain contributions from the material on which the specimens are mounted. In such instances, it might be possible to mount the specimen so that the unwanted signal is minimized or that the mounting material is out

of focus; for example, the specimen could be mounted over a hole. Alternatively, many wires, fibers, and filaments can be placed side-by-side or bundled to fill the field of view of the analytical instrument. In some cases, these specimens can be mounted in the same way as powders and particles, as described in A.4.3.

A.4.5 Pedestal mounting

For some analytical instruments, especially those with large analysis areas, it is possible to mount the specimen on a pedestal so that only the specimen will be "seen" by the analyser. Effectively other parts of the sample holder are outside the area detected by the analyser. This approach can allow analysis of specimens that are smaller than the analysis area. The method might be used for wires or filaments as described above but also for other types of samples smaller than the analysis beam.

A.4.6 Special cases: gassy specimens, viscous liquids, solute residues, freezing samples

Some specimens will emit gases and cannot be analysed because they degrade the vacuum environment in the analytical chamber. These specimens can be pre-pumped in an entry or an auxiliary vacuum chamber and quickly transferred to the analytical chamber. Perhaps the easiest method for pre-pumping is in the introduction chamber of a fast insertion probe. Removal of the volatile components can change the surface chemistry of the specimen. Cross-contamination between specimens can occur in such cases if multiple samples are in the chamber at the same time. Also see A.5.1.

Viscous liquids can be analysed by XPS by placing a thick layer on a smooth substrate material and wiping away most of the liquid. The remaining specimen layer is often of such a thickness that no signal from the substrate is detected yet the vacuum requirements of the analytical chamber are met.

If solute residues from a solution are to be analysed, the solvent can be placed in a small pan or other substrate (that would not interfere with the analysis) and the liquid is evaporated. The solute residue will remain on the pan and can be transferred to the analytical chamber for analysis.

Some analysts use rapid freezing (cryo-XPS) to analyse biological materials and solid-liquid interfaces.[37] In this work a sample is deposited onto a precooled substrate. Cold substrates are also useful for analysis of any high vapor pressure substance.

A.4.7 Masking for charge control

Specimen charging during analysis can be a serious problem with poorly conducting specimens (also see A.2.5), and several documents, reviews and ISO 19318 are devoted to describing ways to minimize the effects of charging.[23][22][11] Charging can sometimes be mitigated by the way that the specimen is mounted. 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 area that will be analysed. A grid can also be suspended slightly above a surface.[38] Wraps of metal foils are used for the same purpose. In AES, it can be important to cover insulating areas of the specimen that are not in the immediate analysis area to avoid the accumulation of sufficient charge (from scattered electrons and ions) that could deflect the primary electron beam or perturb the analysis. Whenever sputtering is used in conjunction with a mask, grid, or wrap, care should be taken to ensure that the covering material is not sputtered onto the analysis area. Removable grids have been reported[39] where the grid is moved while the ion gun is on and then returned during analysis. Materials such as colloidal silver, silver epoxy or colloidal graphite can be used to provide a conductive path from near the point of analysis to ground; however, be aware that outgassing of the solvent can cause problems. Coating a specimen with a thin conducting layer and subsequently removing the coating by sputtering can be useful, but information regarding the topmost layer of the specimen will generally be lost. This approach can be useful for sputter depth profiling although charging can reappear if the walls of the crater remain electrically insulating. Combinations of coatings and masks or wraps are sometimes useful.

A.5 In situ sample cleaning or other sample preparation or processing to expose the region of interest

A.5.1 Sample heating: cleaning/decontamination, segregation studies, reactive layer formation

In situ heating of samples can be done for several reasons. Heating is not generally used to clean specimens because only a small number of materials can withstand the temperatures required to desorb most contaminants. The technique can be useful for refractory metals and, possibly, ceramics. Since heating can cause many changes to a specimen, such as segregation of minor species to the surface or decomposition of some species,[40][41] this technique should be used with care. Heating is also useful for the outgassing of specimens, the removal of implanted rare-gas ions, and annealing out lattice damage caused by ion bombardment of single crystals. Some research studies are conducted by heating a sample in vacuum. This can be done to create a reactive layer, such as oxide formation, or to examine segregation of contaminants to a sample surface. Specimens can be heated indirectly (by conduction) and directly by resistive, electron-bombardment, quartz-lamp, or laser heating.

A variation of the heating technique is to expose the specimen to a reactive environment such as oxygen or hydrogen, and to heat the specimen to a lower temperature than might normally be necessary for contamination removal. Contaminants can then react with the environment and be transformed to volatile species that can be pumped away. This approach would normally be used in a chamber separate from the analysis chamber. Specialized ultra-high vacuum chambers for controlled exposure of specimens to special environments are available that allow for specimen modifications by thermal or chemical treatments. Such chambers are separated from the analytical chamber by an ultra-high-vacuum valve; a suitable specimen-transfer mechanism is also used to minimize possible contamination of the analytical chamber.

Gaseous sample and volatile overlayers can be “cleaned” or degassed by sitting in vacuum (with or without mild heating) in an analysis chamber or an auxiliary vacuum chamber. This approach can require several days and is generally applicable to organic overlayers on inorganic substrates.

A.5.2 Ion sputtering and cluster cleaning

Sputtering (also called ion etching) is sometimes used to clean the sample surface, expose subsurface layers or, when combined with surface analyses, to produce a sputter depth profile (i.e., composition as a function of depth, which is not covered in this document).

Traditionally, noble gas ions with 1 keV to 5 keV incident energy were commonly used for sputtering of multiple materials. The effects of sputtering in surface analysis can be complex and have been described in several reviews.[42][43][44] The development of cluster ion beam (e.g., Ar gas clusters) have made cluster sputtering a highly useful method for cleaning organic layers from the surface of inorganic substrates.[45][46]

A brief description of some of the major considerations and issues with sputtering are given here.

Whatever method is used it is important to provide some measure of the extent of ion exposure. This can be accomplished by indicating the ion dose or by some other measure/indicator of the equivalent amount of sputtered material.

Mono-atomic ion bombardment will normally mix the top layers of a specimen to a depth that is comparable with the information depth for analyses by AES and XPS.[47] The particular advantage of using Ar cluster ions for sample cleaning is the major difference in sputter rates for organic molecules in comparison to inorganic materials. Consequently, an organic contamination layer can be removed without significant impact on the substrate of interest.

Preferential sputtering occurs when the constituents of a specimen are not removed at uniform rates. Within the altered layer, for example, the species that sputters most rapidly will be depleted relative to the local composition of the material. This phenomenon can be an important consideration in quantitative studies, especially when dealing with metal alloys.[48]

Sputtering and heating of the specimen (either simultaneously or sequentially) can be used to remove bulk impurities from metal foils or crystals when impurities segregate to the surface during heating. With single crystals, heating should be the final step to remove lattice damage.

A.5.3 UV radiation

Exposure of a specimen to ultraviolet radiation in air can remove organic contaminants, including photoresist residues, from the surfaces of specimens.[49] Some specimens, however, can decompose on exposure to ultraviolet radiation.

A.5.4 Fracturing, cleaving, and scribing in situ

A.5.4.1 Fracturing

In-situ fracture has been extensively applied to metal specimens. However, it can be applied equally well to a broad range of materials, and has found considerable use with composite materials, glasses, and ceramics.

Impact fracture is used more frequently than tensile fracture, possibly because such devices are simpler and readily available, and many specimens can be fractured, then analysed without breaking vacuum. In some cases, cooling the specimens to liquid-nitrogen temperatures can facilitate fracture. Devices for tensile fracture have been reported[50] and are commercially available. Such devices are usually limited to single specimens per pump-down of the vacuum chamber. Specimens can be intergranularly fractured at the proper strain rate by tensile devices at liquid nitrogen temperatures.

It is possible to pretest specimens for impact fracture by mounting the specimen in a vice and hitting it with a hammer (or by other means) to simulate the action of the fracture stage. If an intergranular surface is exposed in this fashion, it is likely that an intergranular failure will also occur using the impact-fracture mechanism in the analysis chamber.

A.5.4.2 Facture specimen design and preparation

Impact fracture and tensile fracture devices generally have a preferred geometry for the specimen to be fractured. The specimens are usually notched to control the location of the fracture.

Specimens with non-ideal geometries for impact fracture can still be fractured in the impact device by using additional pieces to allow a non-ideal shape to approximate the ideal shape or by using special mounting in the fracture device. When the geometry of a specimen does not fit the mounting mechanism well, or if the specimen is brittle, it is advisable to wrap the end of the specimen held in the mount with a foil such as aluminium or indium. This procedure should prevent premature and poorly located fractures.

Metal specimens can be charged with hydrogen to increase the probability of intergranular fracture.[51] The time and temperature required for charging depends upon the specimen. Also, some metals can be embrittled by exposure to liquid metals such as gallium or mercury.[52] However, interpretation of the results will be difficult because of the presence of residual liquid metal atoms in the fracture surface or because of the formation of amalgams that affect the specimen composition and chemistry. Hydrogen-charged specimens will usually lose the hydrogen if they are allowed to remain at room temperature for a relatively short time. Such specimens can be shipped in dry ice and stored in liquid nitrogen for many days without serious degradation of the charging. Pre-testing is recommended for hydrogen- or liquid-metal charged samples.

A.5.4.3 Cleaving

Cleaving a single-crystal specimen in an analytical chamber requires a special mechanism.[53][54] Cleaving can lead to the deposition of particles on the specimen surface.

A.5.4.4 Scribing

In-situ scribing to expose bulk material can be performed by scraping the specimen with a hard, sharp point. Caution should be observed to avoid smearing of the constituents of either the sample or the scribe.

The scribe mark should be wide enough to contain the primary beam for the selected surface-analysis technique. A variation of this concept is to use a wire brush within a load-lock chamber. Scribing can lead to the deposition of particles on the specimen surface.

A.5.5 Coatings and growth of overlayers

The interface region between an overlayer and its substrate can be analysed by AES and XPS if the overlayer can be grown in situ slowly or in discrete steps (e.g., in increments of about one monatomic layer in thickness).[11] AES and XPS can then be used to probe interface properties and possible reactions as the interface is grown. Gas-metal, metal-polymer, metal-semiconductor, and metal-metal interfaces can be studied in this manner.

A.5.6 FIB cross sections and crater edge profiling

Sectioning can be performed with a focused ion beam equipped with a liquid-metal ion source to make a crater of suitable dimensions in a specimen.[29] Surface analyses can then be performed at selected points across the crater; such analyses can then give information on composition as a function of depth from the original surface. The specimen should be tilted during crater formation so that the shape of the crater is appropriate for the analytical technique to be used. Note that atoms of the incident ion beam can be implanted and remain on the crater surface with concentrations approaching several percent. Shallow etching of the implanted surface by a noble-atom ion beam prior to analysis might be necessary.

Because the sputtering at the edges of ion beams is often slower than the sputtering at the centre of a sputter crater, ion sputtering can expose multiple layers during a depth profile. Such layers appear as rings around the centre of the sputter crater. In such cases AES can sometime be used to extract layer profiles similar to those created by cross section sputtering, angle lapping, or ball cratering (see A.3.4.4).

Annex B
(normative)

Sources of contamination, sample handling and storage practices

B.1 Introduction

Contamination is a major concern in preparing samples for surface analysis. This short overview of sources of contamination and good sample handling practices is included because of the high importance of handling samples to minimize contamination. The elements of B.3.2 are the basis of the requirements in 6.2 in this document. The material included here is discussed in greater detail in the Annexes of ISO 20579-1[2].

The focus of ISO 20579-1 is on the information that the sample "owner" is expected to provide to an analyst about the sample for which surface analysis is desired. Information in the informative annexes is intended to assist the sample owner in collecting and preparing the sample for analysis in a manner that allows the desired data to be obtained. The annexes of 20579-1 provide useful information for both the sample "owner" and the surface analyst, and some components are summarized here.

The normative annexes of ISO 20579-1 provide the following information.

— ISO 20579-1:2024, Annex A - Overview of issues and methods related to sample handling and a table identifying typical handling requirements that vary with the nature of the sample and desired information. This annex focuses on analysis objectives, with a table providing the overview of handling methods and containers for different objectives and sample types.

— ISO 20579-1:2024, Annex B - Critical information about sample handling and order of analyses to minimize contamination.

— ISO 20579-1:2024, Annex C – Information for sample storage and transport.

B.2 Potential sources of contamination during handling

Because of the sensitivity of surface analysis methods, there are many different actions or processes that can contaminate a sample during selection or handling including:

a) Handling a sample with bare hands, even without touching the surface to be analysed. Fingerprints and hand creams have molecules that can migrate and contaminate a surface of interest.

b) Handling a sample with tools that have not been solvent cleaned will transfer and spread contamination to a surface. Tools with high Ni-content have been found to contaminate silicon. Use of nonmagnetic tools is also important to both avoid magnetizing a sample and having it move in unintended ways during mounting for analysis.

c) Any unnecessary contact with a sample is to be avoided if possible. Powder-free and silicone-free gloves are useful in handling clean tools but are not to be used to contact the sample unless absolutely essential.

d) Blowing on a sample by mouth is totally unacceptable for removal of unwanted particulates. Many sources of compressed gas also contain unwanted contaminants. High purity non-reactive gas sources are sometimes used to remove dust from a sample, but verification that the source, and delivery hardware (such a tubing or nozzles) do not contaminate a clean sample should be conducted.

e) Storing samples alongside materials which outgas (including storage containers) will contaminate the sample.

B.3 Handling procedures to minimize contamination

B.3.1 Introduction

The extent/level of special handling depends on several factors including the condition of the surface, depth from the surface of the information being sought, and the detection level required to get the desired information. Generic sample handling recommendations that are useful for most samples are given below, followed by more specific recommendations.

Special precautions are necessary for samples that possibly contain toxins or other hazardous materials, and safety data sheets should be provided to the analyst for this type of sample.

B.3.2 Description of important generic sample handling requirements

Specimens should never be in contact with the bare hand. Eliminate or minimize contact with the sample surface to be analysed with handling tools or other equipment.

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

In some cases, it is necessary to take a representative sample from the specimen. Selection of a smaller sample from a larger specimen requires consideration of the information being sought because inhomogeneities are often present. It is recommended that this choice be made in consultation with an experienced analyst. Specific care can be required to avoid contaminating the surface of interest during the cutting procedure. It is often better to request that the analyst, rather than a sample owner, perform any cutting or sectioning of the sample to ensure that it will fit inside the analysis chamber and be suitable for the analysis objectives.

B.3.3 Analysis objectives determine specific sample handling requirements

B.3.3.1 Overview

Surface chemical analysis can be performed on a wide range of specimens and multiple approaches can be used to obtain very different types of information about surfaces or interfaces. The degree of care in handling the sample that is necessary depends upon the type of analysis that is required and the nature of the problem. The information being sought usually falls into three general categories: (Analysis Objective 1) information requiring integrity of the outermost surface; (Analysis Objective 2) information as a function of depth (depth profile) or at a buried interface; and (Analysis Objective 3) information that will require subsequent specimen preparation by the analyst, including bulk analysis or results from some type of sample processing. Subclause B.3.3.5 provides a summary table of the general sample handling and storage requirements for the different analysis objectives.

Independent of the specific analysis objective, minimizing contamination of the surface of any sample to undergo surface analysis is an essential requirement which places important requirements on sample selection, handling, storage, transport, and related documentation. Some specific requirements related to analysis objectives are indicated in B.3.3, general sample handling requirements were described B.3.2, A.1.1 and 6.2. Additional information related to the specific analysis objectives and useful methods can be found in ASTM E 1829,[20] in a paper by Stevie et al.[11], and book chapters by Lindfors[7] and Geller.[6]

Sometimes very special sample requirements are necessary to obtain the desired information. Examples include the analysis of catalysts after activation and analysis of soils or other environmentally relevant materials from non-ambient environments. It is useful for surface analysts to have discussions with those requesting analysis before such samples are submitted, to provide input and guidance on requirements and opportunities.

Vacuum systems that have been recently used for analysis of mobile species such as organics or materials with low vapor pressure can be a source of contamination. If recent system use has the potential to compromise planned analyses, contamination tests or system baking can be necessary to assure reliable measurements.

B.3.3.2 Objective Type 1

Objective 1 specimens include those to be investigated for surface contamination, surface organic coatings, biomaterials (except live organisms such as cells, bacteria, etc.), surface stains, semiconductors, adhesion failures, etc. Two types of samples can fit into this category, those with highly reactive surfaces that can require handling in controlled environments, and those for which the ambient-exposed surface is to be analysed in the as-received condition. This category requires the most care in preparation and packaging. Nothing should be allowed to contact the surface of interest. If certain elements are to be analysed at low levels, ensure that, as far as possible, those elements are not contained in any handling tools, gloves or container materials. Objective Type 1 specimens fall in the first two rows in Table B.1.

Types of specimens that fit analysis Objective Type 1.

a) Reactive specimens where the reactive surface is to be analysed, without special processing by the analyst or in the instrument, although a reactive surface can require handling in a protective or anaerobic environment to minimize additional reaction.

b) Specimens with hydrocarbons, molecular films, or biomaterials on the surface that are the objective of the analysis.

c) Specimens with a contamination layer that is the object of the analysis.

d) Specimens that have been exposed to the atmosphere and are to be analysed as received.

B.3.3.3 Objective Type 2

In this class, the information sought comes from a layer below the outermost surface and identification of superficial surface contamination is not the primary goal of the analysis. Objective 2 specimens include those that require the investigation of thick or thin films, single layers, multilayers, metal contact layers on semiconductors, coatings, dopant profiles, and the chemical and physical properties at an interface. For this category, the packaging requirements are not as stringent, although care is still required not to contaminate the specimen. Surface diffusion, however, can play a role in the interpretation of the results. In this case the objective is not primarily analysis of the outer surface, but material below this surface, often requiring some treatment in the vacuum system by the analyst. Care is necessary to avoid carbonaceous and particulate contamination of the surface as these can migrate and degrade the quality of depth profiles. Objective Type 2 specimens are in the third row of Table B.1.

Types of specimens that fit analysis Objective Type 2.

a) Specimens with atmospheric adsorbates that might interfere with analysis.

b) Specimens with a contamination layer (or other topmost layer) that is of no interest and that will be removed just prior to insertion in the analytical chamber (e.g., treatment by solutions, abrasion, plasma, exposure to radiation, etc.).

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

B.3.3.4 Objective Type 3

Objective 3 specimens include those that require preparation by the analyst or other special handling to get at the desired information. Examples include specimens for in situ fracture, metallurgical lapping or polishing, and specimens that are part of a larger assembly. Generally, these specimens must be shaped (e.g., for fracture), chemically or mechanically altered (as happens with lapping) or disassembled. Fewer special precautions are required for samples that are to be fractured or undergo further sample preparation by the analyst. Nonetheless, care must still be taken not to contaminate the specimen. For specimens in a larger assembly or subassembly, it might be preferable to leave the specimen in place and let the analyst remove it prior to analysis. Objective 3 specimens are in the fourth (last) row of Table B.1.

Examples of specimens that fit analysis Objective Type 3.

a) Thin films that will be delaminated by the analyst prior to insertion into the analysis chamber.

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

c) 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.

d) Samples that will be fractured in situ.

e) Materials where the information on the bulk properties is desired (any surface layers might require removal).

B.3.3.5 Overview of relationships between analysis objectives and sample handling and storage requirements

The minimum handling and specimen container requirements for different categories of information requirements are summarized in Table B.1.

Table B.1 — Minimum handling methods and specimen containers for different analysis objectives

Specimen Objective Type	Specimen category/ Depth of Information	Handling Method	Transport and Storage Container
1	Reactive specimens for which analyses are to be performed as close to original condition as possible.	Often handled in a specialized environment using clean, non-magnetic, uncoated stainless steel or specialty tools only; handled using polyethylene gloves.	Argon or nitrogen glove box or vacuum transfer vessel. Two flat specimens, face-to-face, sealed with PTFE tape.
1	Specimens requiring surface hydrocarbon, molecular, contaminant, or ambient surface layer analysis (e.g., static SIMS and XPS analyses).	Clean, non-magnetic, uncoated stainless-steel tweezers or grippers only, handled using polyethylene gloves.	Clean glass container with glass, PTFE tape, or clean Al foil stopper. High quality polypropylene wafer holder
2	Specimens where the surface of interest is obscured by a surface contamination layer from handling or environmental exposure	Powder-free, polyethylene disposable gloves holding specimen by edge. Powder-free, silicone-free, latex gloves holding specimen by edge.	Any of the above Clean Al foil. Polyethylene box or bag.
3	Specimens with buried interfaces or layers of interest, fracture specimens, bulk analysis.	Clean tools or handheld by edges with gloves. Acid-free, lint-free paper to hold specimen by edge.	Any of the above Acid-free, lint-free, paper.

B.3.3.6 Sample handling priorities

In cases where the analysis will be performed on the “as received” specimen, surface contamination or atmospheric adsorbates are not usually removed because they are the item of interest. Special **c**are shall be taken in the handling of these specimens to ensure that nothing, apart from air or clean inert gas contacts the surface to be investigated. In such cases, it is often appropriate to avoid contacting the specimen surface with solvents or cleaning solutions, gases such as compressed air or solvent vapours, metals, tissue or other wrapping materials, tape, cloth, tools, packing materials, or the walls of containers.

To minimize the potential for contamination of the analysis area during handling, select one of the methods in the list a) to e) below. The list is in approximate order from most stringent to least stringent handling conditions and columns 1 and 2 of Table B.1 show the relationship to the specimen category and analysis