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Surface chemical analysis — Guidelines to sample handling, preparation and mounting —

Part 2: Guidelines to preparation and mounting of specimens prior to analysis

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Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

Contents

Page

Foreword v
Introduction vi
1 Scope 1
2 Normative references 1
3 Terms and definitions 1
4 Symbols and abbreviated terms 1
5 General requirements 1
6 Visual inspection of the specimen 2
7 Specimen considerations 2
7.1 History 2
7.2 Information sought 2
7.3 Analysis order with respect to other analytical techniques 2
8 Sources of specimen contamination 3
8.1 Tools, gloves, mounts, and similar materials 3
8.2 Exposure to gases 3
8.3 Exposure to instrumental vacuum 3
8.4 Exposure to electrons, ions, and X-rays 3
8.5 Contamination of the analytical chamber 4
9 Specimen storage and transfer 4
9.1 Storage time 4
9.2 Storage containers 4
9.3 Temperature and humidity 4
9.4 Specimen transfer 5
10 Specimen mounting procedures 5
10.1 General procedures 5
10.2 Powders and particles 5
10.3 Wires, fibres, and filaments 6
10.4 Pedestal mounting 6
10.5 Reduction of thermal damage during analysis 6
11 Methods for reducing specimen charging 6
11.1 General considerations 6
11.2 Conductive mask, grid, wrap, or coating 6
11.3 Flood gun 7
11.4 Electron and ion beams 7
11.4.1 Angle of incidence of the primary beam in AES 7
11.4.2 Energy of the primary beam in AES and SIMS 7
11.4.3 Current density of the primary beam in AES and SIMS 7
11.4.4 Combined electron and ion beams in AES 7
12 Specimen preparation techniques 7
12.1 General considerations 7
12.2 Mechanical separation 7
12.3 Thinning versus removal 8
12.4 Removal of the substrate 8
12.5 Sectioning techniques 8
12.5.1 General information 8
12.5.2 Sectioning methods 8
12.5.3 Angle lapping 8
12.5.4 Ball cratering 8
12.5.5 Radial sectioning 9

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(standards.iteh.ai)
ISO/DIS 20579-2
https://standards.iteh.ai/catalog/standards/sist/b166cfc9-d852-4af4-8053-20330e678ec6/iso-dis-20579-2

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12.5.6 Mechanical polishing 9
12.5.7 Chemical and electrochemical polishing 9
12.5.8 Crater edge profiling 9
12.5.9 Focused ion beam sectioning 9
12.6 Growth of overlayers 9
12.7 Solvents 9
12.8 Chemical etching 10
12.9 Ion sputtering 10
12.9.1 General information 10
12.9.2 Altered layer 10
12.9.3 Preferential sputtering 10
12.9.4 Chemical changes 10
12.9.5 Sputtering with hydrogen 10
12.9.6 Changes of surface and interface topography 10
12.9.7 Sputtering and heating 11
12.9.8 Sputter-enhanced diffusion 11
12.10 Plasma etching 11
12.11 Heating 11
12.12 Ultraviolet radiation 11
13 Fracturing, cleaving, and scribing 12
13.1 Fracture 12
13.1.1 General information 12
13.1.2 Preparation of specimens 12
13.2 Cleaving 12
13.3 Scribing 12
14 Special specimen-handling techniques 13
14.1 Prepumping of gassy specimens 13
14.2 Viscous liquids 13
14.3 Solute residue 13
Bibliography 14

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(standards.iteh.ai)
ISO/DIS 20579-2
https://standards.iteh.ai/catalog/standards/sist/b166cfc9-d852-4af4-8053-20330e678ec6/iso-dis-20579-2

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Foreword

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The committee responsible for this document is ISO/TC 201.

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Introduction

Common Introduction

This International Standard is intended to assist analysts and those seeking surface chemical analysis in the handling, storage, mounting and treatment of specimens. This is a multipart document, with the first two parts being general requirements for sample handling and storage in ISO 20579-1: Part 1, and mounting and treatment of samples in ISO 20579-2: Part 2. The ensuing parts combine any new requirements of sample handling/storage and/or sample mounting/preparation for new materials classes. Part 3 focuses on biomaterials and Part 4 focuses on reporting needs for nano-objects. Each part of this international standard can be used independently of the other parts although, the general procedures described in Parts 1 and 2 are applicable to a wide range of materials and are not reproduced in the materials specific sections.

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 International Standard will also be applicable to many other surface-sensitive analytical techniques such as ion-scattering spectrometry, scanning probe microscopy, low-energy electron diffraction and electron energy-loss spectroscopy, where specimen handling can influence surface-sensitive measurements. AES, XPS and SIMS are sensitive to surface layers that are typically a few nanometers in thickness. Such thin layers may be subject to severe perturbations caused by specimen handling or surface treatments that may 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..

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Part 2 Introduction

This part of the standard is intended to assist the analyst in understanding the implications of choices in sample mounting and handling in preparation for analysis. Proper preparation and mounting of specimens is particularly critical for surface chemical analysis. Improper preparation may result in the alteration of the surface composition and in unreliable analyses. Specimens shall 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. This International Standard describes methods that the surface analyst may need to use in order to minimize the effects of specimen preparation when using any surface-sensitive analytical technique. The International Standard also describes methods to mount specimens in order to ensure that the desired analytical information is not compromised.

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Surface chemical analysis — Guidelines to sample handling, preparation and mounting —

Part 2: Guidelines to preparation and mounting of specimens prior to analysis

1 Scope

This International Standard gives guidance on methods of mounting and surface treatment for a specimen about to undergo surface chemical analysis. It is intended for the analyst as an aid in understanding the specialized sample-handling conditions required for analyses by techniques such as Auger electron spectroscopy, secondary-ion mass spectrometry, and X-ray photoelectron spectroscopy.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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ISO 18115, *Surface Chemical Analysis — Vocabulary*.

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3 Terms and definitions

For the purposes of this International Standard, the terms and definitions given in ISO 18115 apply.

4 Symbols and abbreviated terms

AES Auger electron spectroscopy

SIMS secondary ion mass spectrometry

XPS X-ray photoelectron spectroscopy

5 General requirements

General information on sample handling is available in two books.[1,2] The degree of cleanliness required by surface-sensitive analytical techniques is much higher than for many other forms of analysis. Specimens and mounts must never be in contact with the bare hand. Handling of the surface to be analysed should be eliminated or minimized whenever possible. Fingerprints contain mobile species that may contaminate the surface of interest. Hand creams, skin oils and other skin materials are not suitable for high vacuum.

Although the handling methods for AES, XPS, and SIMS are basically similar, there are some differences. In general, preparation of specimens for AES and SIMS requires more attention because of potential problems with electron or ion beam damage or charging, or both. This International Standard will note when specimen preparation is significantly different among the three techniques.

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6 Visual inspection of the specimen

A visual inspection of the specimen should be made, possibly using an optical microscope. 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.

Specimen features that are visually apparent when the sample is outside the vacuum system may 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 may then be necessary to physically mark the specimen outside the area to be analysed (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 may leave unwanted detritus on the sample that may be deposited in the instrument or that may affect the analysis. Permanent ink markers may contaminate nearby regions by transport of volatile organics or by surface diffusion of solvent residues.

Changes that may occur during analysis may influence the data interpretation. Following 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.

7 Specimen considerations

7.1 History

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The history of a specimen may affect the surface before surface analysis. For example, a specimen that has been exposed to a contaminating environment may become less reactive, and the need for exceptional care, from an analytical point of view, is then reduced. In such cases, extra care may be required to meet health and safety requirements. Special caution shall be taken with specimens containing potential toxins.

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If a specimen is known to be contaminated, precleaning may be required to expose the surface of interest and reduce the risk of vacuum-system contamination. In such cases, the specimen should be cleaned with a solvent of a suitable grade solvent that is not expected to affect the specimen material.

NOTE Even high-purity solvents can leave residues on a surface. More details on cleaning with solvents are given in 12.7. In some cases, the contamination may be of interest, e.g. where a silicone-release agent influences adhesion. In such cases, precleaning should not be attempted.

7.2 Information sought

The information sought can influence the preparation of a specimen. If the information sought comes from the exterior surface of a specimen, greater care and precautions in specimen preparation shall be taken than if the information sought lies beneath an overlayer that is to be sputtered away in the analytical chamber. It may also be possible to expose the specimen region of interest by in-situ fracture, cleaving, or other means.

7.3 Analysis order with respect to other analytical techniques

It is preferable for surface chemical analysis measurements to be made before the specimen is analysed by other techniques because such specimens may become damaged or be exposed to surface contamination. For example, insulating specimens analysed by electron microscopy may have been 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.:

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8 Sources of specimen contamination

8.1 Tools, gloves, mounts, and similar materials

Specimens for surface chemical analysis shall be prepared and mounted with clean tools to ensure that the surface is not altered prior to analysis and that the best possible vacuum conditions are maintained in the analytical chamber. Tools used to handle specimens should be made of materials that will not transfer to the specimen (e.g. nickel tools will contaminate silicon). Tools should be cleaned in high-purity solvents and dried prior to use. Nonmagnetic tools should be used if the specimen is susceptible to magnetic fields. Tools should not unnecessarily touch the specimen surface to be analysed.

Gloves and wiping materials are sometimes used to handle specimens; it is then likely that their use may result in some contamination of the specimen surface. Care should be taken to avoid contamination by talc, silicone compounds, and other materials that are often found on gloves. "Powder-free" gloves have no talc and may be better suited for use in specimen handling. Unnecessary contact of the specimen with a glove or other tools shall be avoided.

Specimen mounts and other materials used to hold specimens shall be cleaned regularly whenever there is a possibility of cross-contamination of specimens. The use of tapes containing silicones and other mobile species should be avoided.

8.2 Exposure to gases

Breathing on the specimen is likely to cause contamination. 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, must be considered a source of possible 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 ionising nozzle on the gas stream may eliminate this problem.

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8.3 Exposure to instrumental vacuum

Specimens that were in equilibrium with the ambient environment prior to insertion into the vacuum chamber of the surface-analysis instrument may desorb surface species such as water vapour, plasticisers, or other volatile components. Desorption of such species may cause cross-contamination of adjacent samples and may increase the pressure of the vacuum chamber. Surface desorption may also cause changes in surface chemistry of the specimen of interest. Residual gases in the chamber may adsorb on the specimen surface and react with the specimen.

One can test for undesirable effects by monitoring signals from the specimen as a function of time, e.g. by setting the system for a sputter depth profile and then not turning on the ion gun. If changes in the analytical signals are observed, then the interpretation of the results must account for the observation of an altered surface (e.g. due to desorption of surface species). Care should be taken to account for any fluctuations or drifts of the incident-beam intensity.

8.4 Exposure to electrons, ions, and X-rays

The incident electron flux in AES,[3,4] the ion flux in SIMS[5] and, to a lesser extent, the X-ray flux in XPS[6] may induce changes in the specimen being analysed, for example by causing enhanced reactions between the surface of a specimen and the residual gases in the analytical chamber. The incident flux may also locally heat or degrade the specimen, or both, resulting in a change of surface chemistry and possibly a rise in chamber pressure or contamination of the chamber. One can test for unwanted effects by setting the system for a sputter depth profile and not turning on the ion as described in 8.3.

The incident ion beams used during AES, SIMS and XPS depth profiles not only erode the surface of interest but may also affect surfaces nearby. These effects may be caused by poor focusing of the

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primary ion beam and by impact of neutrals from the primary ion beam. Areas adjacent to a region bombarded by ions may not be suitable for subsequent analysis by surface-analysis methods. In some cases, sputtered material may be deposited on nearby regions of the specimen or onto other specimens that may be parked in the analytical chamber.

8.5 Contamination of the analytical chamber

The analyst should be aware of materials that will lead to contamination of other specimens or of the analytical chamber. High-vapour-pressure elements such as mercury, tellurium, caesium, potassium, sodium, arsenic, iodine, zinc, selenium, phosphorous, and sulphur. should be analysed with caution. Many other materials also can exhibit high vapour pressures (for example, some polymers, foams and other porous materials, greases and oils, and liquids). Even if an unperturbed specimen meets the vacuum requirements of the analytical chamber, the probing beam required for analysis may degrade the specimen and result in serious contamination of the chamber, as described in 8.4.

Contamination of the specimen by surface diffusion can be a problem, especially with silicone compounds[7] and hydrocarbons. It is possible to have excellent vacuum conditions in the analytical chamber and still find contamination by surface diffusion.

In SIMS, atoms sputtered onto the secondary-ion-extraction lens or other nearby surfaces can be resputtered back onto the surface of the specimen. This effect can be reduced by not having the secondary-ion-extraction lens or other surfaces close to the specimen. The use of multiple immersion lens strips or cleaning of the lens can help reduce this effect.

The order of use of probing beams can be important, especially when dealing with organic or other fragile materials (such as those discussed in 14.1).

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9 Specimen storage and transfer

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9.1 Storage time

If a specimen is stored before analysis, care should be taken to ensure that the surface to be analysed has not been contaminated during storage. Even in clean laboratory environments, surfaces can quickly become contaminated to the depth analysed by AES, XPS, SIMS, and other surface-sensitive analytical techniques.

9.2 Storage containers

Containers selected for specimen storage shall not transfer contaminants to the specimen via particles, liquids, gases, or surface diffusion. Containers that contain volatile species such as plasticisers (which may be emitted and then contaminate the surface) are unsuitable. The specimen surface to be analysed should preferably not contact the container or any other object. Glass jars with an inside diameter slightly larger than the width of a specimen may hold a specimen without contact with the surface. When contact with the surface is unavoidable, wrapping in clean, pre-analysed aluminium foil may be satisfactory.

Containers such as glove boxes, vacuum chambers, and desiccators may be excellent choices for storage of specimens. A vacuum desiccator may often be preferable to a normal desiccator, and shall be maintained free of grease and mechanical-pump oil.

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

9.3 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.

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9.4 Specimen transfer

Specially designed chambers that allow transfer of specimens from a controlled environment to a surface-analysis chamber have been reported.[8,9] 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 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.

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.[10] Surfaces to be analysed by AES or SIMS can be covered with a uniform layer, such as polysilicon for silicon-based technology.[11] In this case, the coating is removed by sputtering during analysis; however, the influence of atomic mixing on the analytical results must be considered.

10 Specimen mounting procedures

10.1 General procedures

The specimen will often 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.

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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 come into contact with 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.

10.2 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 may adhere to them. For XPS, powders can be placed on the sticky side of adhesive tape. Metallised 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 may sometimes be transferred to a suitable substrate by working under a microscope and by using a sharp needle. Non-soluble particles can sometime 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. Alternatively, compression of the powder into a disk such as that used for preparation of potassium bromide disks for infra-red spectroscopy can 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 may be modified, however, by pressure or temperature-induced changes during preparation of the pellet.

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