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BRITISH STANDARD

BS ISO
18116:2005

Surface chemical analysis — Guidelines for preparation and mounting of specimens for analysis

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British Standards

National foreword

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INTERNATIONAL STANDARD

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Surface chemical analysis — Guidelines for preparation and mounting of specimens for analysis

Analyse chimique des surfaces — Lignes directrices pour la préparation et le montage des échantillons destinés à l'analyse

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Foreword

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ISO 18116 was prepared by Technical Committee ISO/TC 201, *Surface chemical analysis*, Subcommittee SC 2, *General procedures*.

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Introduction

This International Standard is intended to assist analysts in the handling, mounting and treatment of specimens submitted for surface chemical analysis. 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, 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 nanometres 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 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 have 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. 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. This 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 for preparation and mounting of specimens for 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 specimen-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 referenced documents are indispensable for the application 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, *Surface chemical analysis — Vocabulary*

3 Terms and definitions

For the purposes of this document, 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 specimen 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 specimen is outside the vacuum system may not be observable after the specimen 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 specimen does not affect the subsequent measurements. Scribing a brittle material may leave unwanted detritus on the specimen 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

The history of a specimen may affect the handling of 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.

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 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 Specimens previously examined by 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 ionizing nozzle on the gas stream may eliminate this problem.

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, plasticizers or other volatile components. Desorption of such species may cause cross-contamination of adjacent specimens 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 gun, 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 primary ion beam and

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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 sulfur 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).

9 Specimen storage and transfer

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 plasticizers (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 on 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 specimen 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. 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 specimen. 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 disc, such as the potassium bromide disc used 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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10.3 Wires, fibres and filaments

Wire, fibres and filaments may 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 may 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, fibres and filaments can also be placed side-by-side or bundled to fill the field of view of the analytical instrument. In some cases, these specimens may be mounted in the same way as powders and particles, as described in 10.2.

10.4 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. This approach may allow analysis of specimens that are smaller than the analysis area.

10.5 Reduction of thermal damage during analysis

To reduce thermal damage of specimens during surface analysis, specimens may be mounted on a cooled probe or on a mounting stage cooled with flowing liquids or gases at a desired temperature. Good thermal contact between the specimen and the mounting system is important. Some specimens, such as powders, benefit from being compacted to pellets, thereby increasing heat dissipation. Wrapping a specimen in a metal foil may be of value in some cases. As discussed in 11.4.2 and 11.4.3, reducing the energy dissipated in the specimen can also be beneficial, but this may result in longer data-acquisition times.

11 Methods for reducing specimen charging

11.1 General considerations

Specimen charging can be a serious problem with poorly conducting specimens. Charging is usually more severe for incident electron or ion beams than for an incident X-ray beam. In XPS, charging is usually more severe for a focused, monochromatic X-ray beam than for a large-area beam or for non-monochromatic X-rays. If the surface is heterogeneous or the probing radiation is focused, the amount of charging can differ across the analysis area.

11.2 Conductive mask, grid, wrap or coating

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 may also be suspended slightly above a surface[12]. Wraps of metal foils are used for the same purpose. In AES, it may 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[13] such that 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, beware that outgassing of the solvent may cause a problem. Coating a specimen with a thin conducting layer and subsequently removing the coating by sputtering may 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 may reappear if the walls of the crater remain electrically insulating. Combinations of coatings and masks or wraps may be useful.

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11.3 Flood gun

In XPS, low-energy electrons from a nearby filament can be useful for reducing charging of non-conducting specimens. The window material in a conventional X-ray source can also act as a source of electrons to reduce charging. In SIMS, the relative location of electron and ion optics can influence charging phenomena while analysing insulators.[14],[15] Positive-ion SIMS depth profiling requires the use of a focused electron beam with a current density similar to or greater than that of the ion beam. Negative-ion primary beams may be used.

11.4 Electron and ion beams

11.4.1 Angle of incidence of the primary beam in AES

The secondary-electron emission coefficient and the incident-beam current density are functions of the angle of incidence of the primary-electron beam. Grazing angles of incidence increase the secondary-electron emission coefficient and are, therefore, generally better for reduction of charging during AES analysis of flat specimens.[16],[18]

11.4.2 Energy of the primary beam in AES and SIMS

The secondary-electron emission coefficient is also a function of the energy of the primary-electron beam in AES. It is generally better to select a primary energy where the secondary-electron emission coefficient is greater than unity to reduce specimen charging. For layered specimens, it might be possible to achieve reduced charging by increasing the energy of the incident electron beam so that penetration is made to a conducting layer beneath the analysed layer. Charge neutralization may occur if the conducting layer is suitably grounded. In SIMS, the energy of the incident ion affects specimen charging.[14]

11.4.3 Current density of the primary beam in AES and SIMS

Specimen charging may be reduced by decreasing the current density of the primary electron or ion beam in AES and SIMS. Reduction of the current density can be achieved by reducing the total current, defocusing the beam, rastering the beam over a part of the specimen surface, or changing the angle of incidence.

11.4.4 Combined electron and ion beams in AES

If a specimen is homogeneous with depth, charging in an AES analysis can sometimes be reduced by sputtering the specimen with positive ions during an analysis. The positive charge of these ions will partially neutralize the negative charge generally created by the primary-electron beam. Ion-beam-induced changes of surface composition must be considered, as discussed in 12.9.

12 Specimen preparation techniques

12.1 General considerations

Often the surface or interface of interest lies beneath a layer of contaminants or other material. This overlayer must often be removed without perturbing the surface or interface of interest.

For electronic devices, additional information regarding preparation of specimens is available.[19]

12.2 Mechanical separation

It is sometimes possible to mechanically separate layers and expose the surface of interest, as described in Clause 13. 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,

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when the outer skin is penetrated by the ion beam, the data may become dominated by artefacts. Mechanical separation should be carried out just prior to transfer of the specimen to the analytical chamber or *in situ*, if possible.

12.3 Thinning versus removal

Complete removal of an overlayer may not be possible or desirable. It may be sufficient to thin the overlayer and continue using sputter depth profiling as discussed in 12.9.

12.4 Removal of the substrate

In some specimens, it may be easier to approach the interface by removing the substrate rather than the overlayer. This approach may be useful, for example, when the substrate composition is not of interest or the material composition of the overlayer is unknown. In SIMS, substrate removal may provide improved depth resolution if non-uniform sputtering of the overlayers occurs.[20] As discussed in 12.3, complete removal of the substrate may not be necessary.

NOTE A chemical etch is more effective and more selective when the composition of the material to be etched is known. Additional information on chemical etching is given in 12.8.

12.5 Sectioning techniques

12.5.1 General information

Sectioning (cutting) techniques are most often applied to metals, but can often be applied to other materials equally well. When using sectioning techniques, it is important to section with minimum alteration to the region of the specimen that will be analysed. After sectioning, it is usually necessary to clean the specimen by sputtering in the analytical chamber prior to surface analysis.

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

12.5.2 Sectioning methods

Sectioning can be performed using an abrasive wheel or by sawing or shearing. The extent of damage generally increases as cutting speed increases. Semiconductor specimens may also be sectioned by cleaving and polishing or by a focused ion beam.[21] Chemical changes can be extensive if local heating occurs during sectioning. Coarse grinding is usually performed with abrasive belts or discs. Fine grinding is usually done with silicone carbide, emery, aluminium oxide or diamond abrasives. Grinding materials and lubricating oils for cutting tools may contaminate the surface and should be avoided. If possible, cutting should be performed dry (i.e. without lubricants).

12.5.3 Angle lapping

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.[22] In AES, the diameter of the primary electron beam must be small relative to the expanded dimensions of the layer to be analysed. The considerations outlined in 12.5.1 are also applicable to lapping. Spalling at weak interfaces may occur during these operations.

12.5.4 Ball cratering

Ball cratering is similar to angle lapping[23] and is applicable when the radius of curvature of the spherical surface is large relative to the thickness of the films being analysed.

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12.5.5 Radial sectioning

Radial sectioning is similar to ball cratering, with a cylinder being used to create a crater instead of a spherical ball.

12.5.6 Mechanical polishing

Polishing is often the most crucial step in the sequence of preparing a lapped specimen. The abrasives used may include aluminium oxide, chromium oxide, cerium oxide, silicon dioxide, silicon carbide or diamond.[24] The choice of suspension medium (normally oil or water) and polishing cloth must be carefully considered for possible contamination of the specimen, embedding of abrasion material into the specimen, and complete removal of abrasion removal from the specimen.

12.5.7 Chemical and electrochemical polishing

Chemical or electrochemical polishing is sometimes applied after the final mechanical polishing of a specimen.[24] 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 avoid the sort of surface damage introduced by mechanical polishing. However, any type of polishing may alter the chemistry of the surface.

12.5.8 Crater edge profiling

Craters left by fixed or rastered ion beams used for sputter depth profiling often have a slightly inclined sidewall. An electron beam can be translated across this sidewall to obtain composition versus depth information by AES.[25]

12.5.9 Focused ion beam sectioning

Sectioning can be performed by a focused ion beam equipped with a liquid-metal ion source to make a crater of suitable dimensions in a specimen. 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 may be necessary to remove this residual material. Additionally, redeposition of sputtered material may occur.

12.6 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 slowly or in discrete steps (e.g. in increments of about one monatomic layer in thickness). 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.

12.7 Solvents

High-purity solvents can be used to remove soluble contaminants or overlayers that are not of interest. Ethanol, isopropanol and acetone are the most commonly used solvents, and are often used in conjunction with ultrasonic agitation. A residue from the solvent may, however, be left on the specimen. 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. 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. The cleaning action in this case is based on both solvent action and momentum transfer.[26]

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NOTE 1 The utilization of acetone to clean specimens or components of AES instruments will temporarily reduce electron emission from lanthanum hexaboride cathodes if used in the AES instrument. In addition, acetone is hygroscopic and can absorb water from the atmosphere.

NOTE 2 Use of a carbon dioxide gas stream may also lead to unwanted specimen contamination, as described in 8.2.

12.8 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 overlayer.[27] Possible chemical or morphological effects on the substrate should be considered when using this procedure.

12.9 Ion sputtering

12.9.1 General information

Sputtering (also called ion etching) is often used to expose subsurface layers or, when combined with surface analyses, to produce a sputter depth profile (i.e. composition as a function of depth). Noble-gas ions of 1 keV to 5 keV incident energy are commonly used for sputtering. The effects of sputtering in surface analysis can be complex, and have been described in several reviews.[5],[28],[29]

12.9.2 Altered layer

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.[30] The extent of atomic mixing will depend upon the composition of the specimen, the incident-ion species and the energy of the incident ions. Reducing the ion energy, changing the angle of incidence and using an ion of higher mass (e.g. xenon) will generally reduce the depth over which atomic mixing occurs.

12.9.3 Preferential sputtering

The constituents of a specimen may not eroded by sputtering 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 may be an important consideration in quantitative studies, especially when dealing with metal alloys.[31]

12.9.4 Chemical changes

Bombardment of a specimen by energetic ions can cause chemical changes in the specimen depending upon the composition of the specimen. For example, nitrates, phosphates and carbonates can be converted to oxides under bombardment by 1 keV to 3 keV argon ions.[32] If a metal has multiple oxidation states, the oxide with maximum valency is particularly susceptible to reduction. In general, the composition of polymers will be significantly changed by ion bombardment.

12.9.5 Sputtering with hydrogen

Sputtering with hydrogen may remove contaminants in some cases with minimum alteration of the surface of interest.[33]

12.9.6 Changes of surface and interface topography

Unidirectional ion bombardment often produces increased surface and interface topography (roughness) and can create topographical features such as pits, pyramids, cones, whiskers and ripples.[5] As a result, sputter depth profiles will be distorted, and it may be more difficult to detect and characterize a subsurface interface. The depth resolution in sputter depth profiling is usually 3 % to 15 % of the sputtered depth.[34] Use of two ion

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beams, incident on the specimen at different angles, can reduce sputter-induced roughness and topographical features.[35] Specimen rotation during ion sputtering may improve depth resolution,[36] especially for polycrystalline metallic layers. Alignment of the ion gun, analysis area and rotation centre are especially important while rotating a specimen of varied surface composition. Lower incident energies can also improve the depth resolution.[37] Both smaller[38] and larger[37] angles of incidence can improve the depth resolution for certain specimens.

12.9.7 Sputtering and heating

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.

12.9.8 Sputter-enhanced diffusion

Ion bombardment of a specimen can result in enhanced diffusion of constituent species away from or towards the surface, thereby producing distorted sputter depth profiles. This phenomenon can be a particular problem in SIMS.[39]

12.10 Plasma etching

Plasma etching, using a reactive ion species such as oxygen, has been found helpful in etching specimens in cases where ion beams would produce artefacts in the data.

12.11 Heating

Heating is not often used to clean specimens because only a small number of materials can withstand the temperatures required to desorb most contaminants. The technique may be useful for refractory metals and, possibly, ceramics. Since heating can cause many changes to a specimen,[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. Specimens can be heated indirectly (by conduction) and directly by resistive, electron-bombardment, quartz-lamp and 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. Contaminants may then 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.

When the overlayers to be removed consist of materials with vapour pressures higher than that of the surface of interest, the overlayers may be pumped away in an auxiliary vacuum chamber. This approach may require several days, and is generally applicable to organic overlayers on inorganic substrates.

12.12 Ultraviolet radiation

Exposure of a specimen to ultraviolet radiation in air can remove organic contaminants, including photoresist residues, from the surfaces of specimens.[42] Some specimens, however, may decompose on exposure to ultraviolet radiation.

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13 Fracturing, cleaving and scribing

13.1 Fracture

13.1.1 General information

In situ fracture has been extensively applied to metal specimens. However, it can be applied equally well to a broad range of other 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 analysed without breaking vacuum. In some cases, cooling the specimens to liquid-nitrogen temperatures can facilitate fracture. Devices for tensile fracture have been reported[43] 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. Pretesting is also suggested for hydrogen-charged specimens.

13.1.2 Preparation of specimens

Impact fracture and tensile fracture devices generally have a preferred geometry for the specimen to be fractured. The specimens are usually notched in an attempt 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 made of e.g. aluminium or indium. This procedure should prevent premature and poorly located fractures.

Metal specimens may be charged with hydrogen to increase the probability of intergranular fracture.[44] The time and temperature required for charging depends upon the specimen. Also, some metals may be embrittled by exposure to liquid metals such as gallium or mercury.[45] 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 specimens.

When electrical insulators such as ceramic materials are fractured, problems with electrical charging may develop during the surface analysis. To reduce this problem, it may be helpful to coat the outer surface of the insulator with a conducting material, such as gold, prior to fracture.

13.2 Cleaving

Cleaving a single-crystal specimen in an analytical chamber requires a special mechanism.[46],[47]

NOTE Cleaving may lead to the deposition of particles on the specimen surface.

13.3 Scribing

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

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NOTE Scribing may lead to the deposition of particles on the specimen surface.

14 Special specimen-handling techniques

14.1 Prepumping of gassy specimens

Some specimens will emit gases and cannot be analysed because they degrade the vacuum environment in the analytical chamber. These specimens may be prepumped in an auxiliary vacuum chamber and quickly transferred to the analytical chamber without appreciable pickup of gases during the transfer. Perhaps the easiest method for prepumping is in the introduction chamber of a fast-insertion probe. Removal of the volatile components may change the surface chemistry of the specimen. Cross-contamination between specimens may occur in such cases if multiple specimens are in the chamber at the same time.

14.2 Viscous liquids

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.

14.3 Solute residue

If solute residues from a solution are to be analysed, the solvent can be placed in a small pan and the liquid evaporated. The solute residue will remain on the pan and may be transferred to the analytical chamber for analysis.

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