Brochure

X-ray photoelectron spectroscopy for surface and interface analysis

A practical guide for research and industry

thermo scientific

Contents

Chapter 1: Introduction to X-ray photoelectron spectroscopy

X-ray photoelectron spectroscopy (XPS) is one of the most powerful and widely used techniques for characterizing the chemistry of surfaces, thin films, and engineered interfaces.

In modern materials science and industrial research, it plays a pivotal role in understanding the chemical identity, composition, and electronic states of atoms located at or near the outermost nanometers of a material's surface. This extreme surface sensitivity—typically probing only the top 10 nanometers—makes XPS essential for industries in which performance is dictated not by the bulk characteristics of a material but by the chemical events occurring at its boundaries. Such applications include advanced semiconductor devices, protective and decorative coatings, battery electrodes, catalysts, corrosion-resistant alloys, biomaterials, polymer engineering, and next-generation energy materials.

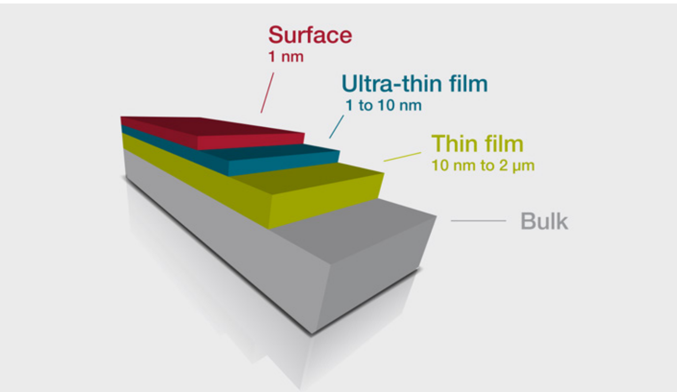


Figure 1: Schematic of a sample showing the typical analysis depths accessible to XPS either directly (up to 10 nm) or through depth profiling.

Surfaces govern behavior in ways that are often invisible to bulk analytical methods. Adhesion failures, corrosion initiation, catalytic activity, interfacial charge transfer, polymer aging, environmental degradation, and coating delamination all originate at or near surfaces. As a result, a technique that can selectively interrogate chemical bonding, oxidation states, and impurity distributions near the surface is indispensable. XPS can fill this role particularly well. Its ability to determine not only what elements are present but also how those elements are chemically bonded sets it apart from other spectroscopic or compositional methods such as energy dispersive X-ray spectroscopy or optical emission techniques.

At the heart of XPS is the photoelectric effect. When a solid is irradiated with X-rays of sufficient energy, electrons can be ejected from atomic orbitals. A spectrometer is used to measure the kinetic energy of these emitted electrons, which makes it possible to determine their binding energy. Because binding energy is highly characteristic of the atomic species and its chemical environment, XPS spectra provide rich chemical-state information. This allows analysts to differentiate, for example, metallic copper from Cu(I) and Cu(II) oxides, or carbon species in polymers such as carbonyls, alcohols, and carboxylates.

Modern XPS instrumentation builds upon decades of technological advancements to provide more stable X-ray sources, higher-energy-resolution analyzers, faster detectors, and increasingly automated workflows. Today's instruments incorporate micro-focused monochromators, hemispherical energy analyzers with multi-channel detection, advanced charge neutralization systems, and sophisticated sample-handling platforms. These innovations ensure that even non-conductive or beam-sensitive materials can be analyzed with high reproducibility. Furthermore, multi-technique integrations such as combining XPS with ultraviolet photoelectron spectroscopy (UPS), ion scattering spectroscopy (ISS), Raman spectroscopy, Auger electron spectroscopy (AES), or reflected electron energy loss spectroscopy (REELS) allows researchers to develop complete chemical and electronic profiles of complex materials.

Industrial adoption of XPS has expanded dramatically in the last 25 years. Semiconductor manufacturers use XPS to measure gate oxide stoichiometry, contamination at metal–dielectric interfaces, and chemical changes induced during plasma processing. Battery researchers rely on XPS to investigate the solid–electrolyte interphase (SEI) layer, track decomposition products responsible for capacity fade, and characterize surfaces of electrode powders. Coatings companies utilize XPS to understand adhesion failures, evaluate the effectiveness of surface treatments, or validate crosslinking in cured polymers. In catalysis research, XPS is often the only technique that can directly quantify oxidation states of catalytically active metals under various pretreatment conditions. Across all these industries, XPS's ability to probe chemical changes with sub-nanometer sensitivity makes it indispensable for diagnosing performance problems and guiding material optimization.

In academia, XPS serves as a foundational technique for surface science. Research groups studying nanomaterials, 2D materials such as graphene or MoS_2, quantum dots, biomolecular interfaces, and thin-film devices rely heavily on XPS because it provides immediate insight into oxidation, doping, defect formation, adsorption, charge transfer, and chemical modifications induced during synthesis or processing. As materials become more complex and functionalization strategies more sophisticated, XPS remains one of the few analytical tools capable of resolving subtle chemical differences at the atomic level.

The power of XPS is magnified when combined with emerging sample-preparation and depth-profiling technologies. Traditional ion sputtering allows analysts to create depth profiles through multilayer structures, revealing buried interfaces, gradients, diffusion layers, or contamination trapped between coatings. However, conventional monatomic ion beams can damage polymers, organics, and certain soft materials, leading to chemical artifacts. To mitigate this, gas-cluster ion sources (GCIS) use clusters of thousands of atoms to gently sputter surfaces with minimal chemical damage. More recently, femtosecond-laser ablation (fs-LA) has revolutionized depth profiling by enabling rapid, artifact-free removal of hundreds of nanometers to micrometers of material at a time, which is particularly invaluable for analyzing thick organic coatings, paints, composite systems, or beam-sensitive perovskite materials.

As XPS evolves, so too does the software and data-processing infrastructure that supports it. Modern platforms incorporate intelligent peak-fitting algorithms, automated component identification, database-driven chemical-state annotation tools, and workflows that reduce operator variability. Tools such as the Thermo Scientific™ Avantage Data System allow both novice and experienced users to analyze data reproducibly, generate publication-quality reports, and manage complex experiments with minimal manual intervention. For industrial labs, automation reduces cost of ownership and ensures consistent results in multi-user environments.

Despite its capabilities, effective use of XPS requires careful experimental design. Analysts must consider surface cleanliness, charging effects, X-ray spot size, analyzer settings, and possible contamination pathways. Moreover, XPS is not a bulk technique, so results must be interpreted with an understanding that subsurface chemistry may differ from surface chemistry. When used appropriately, however, XPS offers unparalleled clarity regarding what is happening at a material's most important interface—the surface.

Chapter 2: Fundamentals of X-ray photoelectron spectroscopy

This guide to XPS will introduce the physical basis of the technique, the instrumentation that is used, and considerations for experimental work and data processing. It also includes case studies on important materials that illustrate how the technique is used.

XPS is fundamentally rooted in the photoelectric effect, a phenomenon first explained by Albert Einstein in 1905. When a material is irradiated with photons of sufficient energy—in most laboratory XPS instruments, these are monochromated Al K-alpha X-rays with an energy of 1,486.6 eV—core-level electrons may be ejected from atoms within the material. These electrons leave the surface with kinetic energies that are directly related to their binding energies, according to the photoelectric equation:

$BE = h\nu - KE - \Phi$

Where $h\nu$ is the X-ray photon energy, KE is the measured kinetic energy, and Φ is the spectrometer work function.

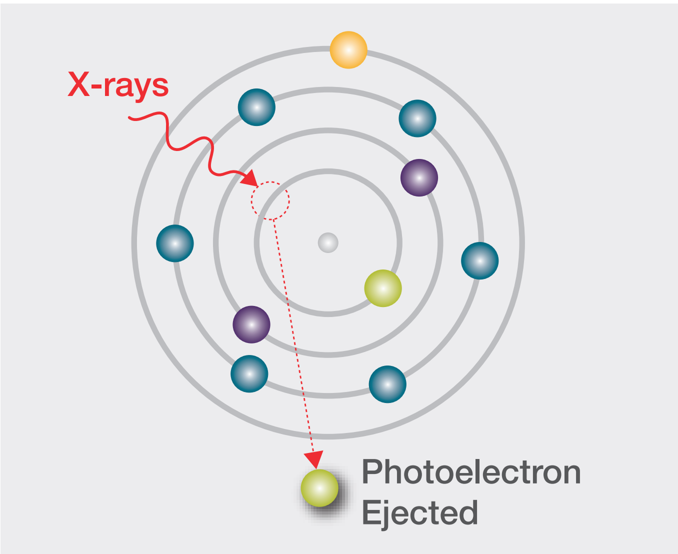


Figure 2: The XPS process, where an atom is irradiated with X-rays and an electron from a core level is ejected.

Because core electron binding energy is influenced by the chemical environment experienced by the atom, XPS provides chemically specific information. The number of electrons emitted is directly related to the number of atoms present, and so the XPS spectra also permit the composition of the surface to be measured. This sensitivity to subtle chemical-state differences, coupled with the ability to quantify them, is the defining strength of XPS and remains unmatched by many other surface-sensitive analytical techniques.

The depth sensitivity of XPS is dictated by the inelastic mean free path (IMFP) of photoemitted electrons. As electrons traverse the solid, they undergo inelastic scattering events that progressively reduce their energy, preventing them from contributing to the detected spectrum. This means that only electrons originating from within approximately the first 10 nanometers of the surface retain sufficient energy to escape the sample and be measured by the spectrometer. As a result, XPS is inherently surface sensitive. This makes it ideally suited for characterizing thin films, passivation layers, interfacial oxides, adsorbed species, organic coatings, and contamination layers. However, it also imposes interpretational constraints. XPS does not provide bulk composition unless the sample is depth profiled. Understanding the IMFP and sampling depth is therefore essential for accurate data interpretation.

The XPS spectrum contains several characteristic features. The most important are the core-level photoelectron peaks that appear at discrete binding energies corresponding to specific atomic orbitals (e.g., C 1s, O 1s, Si 2p, Al 2p). For all peaks arising from orbitals other than s-orbitals, doublets are seen rather than single peaks due to "spin–orbit splitting" determined by the electron's angular momentum and spin quantum numbers. For example, the Mo 3d peak splits into Mo $3d_{5/2}$ and Mo $3d_{3/2}$ components, which have a well-defined intensity ratio and energy separation. These relationships are fundamental and serve as internal consistency checks during spectral interpretation. Chemical shifts—small changes in binding energy caused by differences in oxidation state or bonding—provide the basis for chemical-state analysis. Higher oxidation states typically shift peaks to higher binding energies due to increased effective nuclear charge. For instance, Fe^{2+} and Fe^{3+} oxides exhibit distinct separations from metallic iron in the Fe 2p region, enabling detailed quantification of the oxidation state.

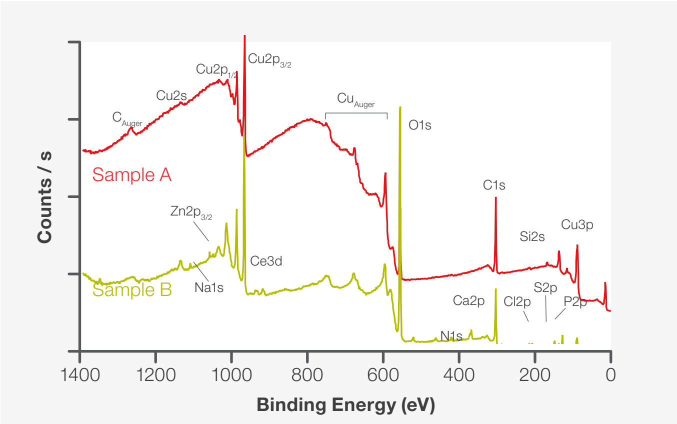


Figure 3: XPS elemental survey spectra from two different copper samples, used to create elemental quantification.

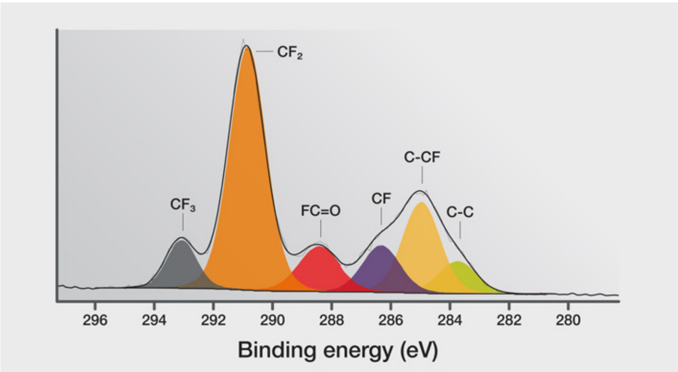


Figure 4: XPS region scan for the C 1s region of a fluoropolymer sample, showing the range of chemistries detected.

Beyond the core photoelectron peaks, spectra contain additional features such as Auger peaks (such as the Cu Auger features seen in Figure 3), satellite features, and plasmon-loss structures. Auger peaks arise from a three-electron process in which the filling of a core-hole created by photoemission by an electron from another orbital results in the ejection of an electron. These peaks are independent of the X-ray energy and can be quite diagnostically useful. For certain elements, such as Zn or Cu, they can be used to assist in the interpretation of XPS spectra. They can also be used to calculate the Auger parameter—another useful diagnostic for identifying chemical states. Plasmon-loss features are satellite peaks caused by collective oscillations of valence electrons. These are particularly prominent in conductive materials such as metals or elemental silicon. Other satellite features maybe seen, arising due to other phenomena within the electronic structure of the investigated atom.

Quantification in XPS is based on the area under a photoelectron peak, corrected for instrumental factors such as the analyzer transmission function and atomic sensitivity factors. Modern XPS software automatically applies these corrections, enabling users to determine atomic percentages with reasonable accuracy, typically within ±10%. However, quantification accuracy depends heavily on peak-fitting skill, the quality of background subtraction, and the analyst's ability to identify and account for overlapping peaks.

High-resolution spectra are essential for this task because they resolve the fine structure associated with complex materials. Advanced peak-fitting strategies involve constraining peak widths, positions, intensity ratios, and shapes based on known physical parameters. This ensures robust fitting and avoids overfitting, which can misrepresent the underlying chemistry.

Charge compensation is another fundamental component of XPS analysis. Non-conductive samples tend to accumulate positive charge as electrons are emitted during X-ray irradiation. This leads to peak shifting and peak broadening, making quantification unreliable or, in the worst cases, impossible. To address this, modern XPS instruments employ electron flood guns that supply low-energy electrons (and sometimes ions) to neutralize surface charge. Achieving stable charge compensation requires careful tuning of electron flux, sample grounding, flood-gun geometry, and X-ray spot size. When properly optimized, charge compensation allows insulating polymers, ceramics, and oxides to be analyzed with high energy resolution and minimal distortion.

The interpretation of XPS results is further strengthened by combining XPS with other spectroscopic and microscopic methods. For example, UPS complements XPS capabilities in the valence band region, enabling determination of work functions and highest occupied molecular orbital (HOMO) energy levels. REELS provides band-gap measurements by analyzing low-energy electron-loss features. ISS offers monolayer-level surface composition, particularly helpful for validating ALD coverage or surface segregation. Raman spectroscopy provides vibrational fingerprints that help distinguish molecular structures and confirm polymer functional groups. Together, these complementary techniques transform XPS from a primarily chemical-state tool into a wide-ranging characterization suite.

Understanding the fundamentals of XPS requires familiarity with the key steps of the measurement process: X-ray illumination, electron emission, energy filtering, and signal detection. Each step is governed by physics and engineering constraints that influence spectral resolution, sensitivity, and accuracy. Advances in monochromator design, electron lens optimization, detector architecture, and UHV engineering continue to push the boundaries of what is possible. As a result, XPS remains not only relevant but essential to modern materials science, helping researchers and engineers decode the subtle chemical and electronic structures that define material behavior.

Chapter 3: Overview of X-ray photoelectron spectroscopy instruments

XPS instruments have evolved dramatically over the past five decades, transitioning from early manually controlled vacuum systems to today's highly automated, multi-technique analytical platforms.

Modern XPS systems are engineered to provide exceptional resolution, high sensitivity, stable long-term performance, and seamless integration with complementary analytical tools. Understanding the architecture and functional design of an XPS instrument is essential for evaluating data quality, optimizing experimental workflows, and selecting the appropriate system for specific research or industrial applications.

This chapter provides a structured examination of the principal components of an XPS instrument: vacuum systems, X-ray sources, sample handling mechanisms, electron optics, energy analyzers, detectors, charge compensation systems, ion and laser ablation sources, and multi-technique modules. Together, these elements define the analytical capabilities, speed, reliability, and ease of use of the complete system.

Ultra-high-vacuum systems

A defining characteristic of XPS instruments is the use of an ultra-high-vacuum (UHV) environment within the analysis chamber. Because XPS relies on measuring the kinetic energy of photoelectrons emitted from the top few nanometers of a surface, it is essential to minimize interactions between emitted electrons and gas molecules in the chamber. Electron scattering by residual gas would broaden peaks, reduce intensity, and compromise quantitative accuracy. Typical operating pressures range from 10^{-7} to 10^{-9} mbar. Achieving such conditions requires a range of pumping, from low-vacuum pumps such as rotary vane or dry scroll roughing pumps to turbomolecular pumps and, in some systems, getter pumps such as titanium sublimation pumps.

Figure 5: XPS UHV systems are typically manufactured from mu-metal or other magnetically shielded metal alloys. Parts are sealed onto the chamber using metal or polymer gaskets to ensure good vacuum.

The vacuum system is often divided into two or more chambers. In addition to the analysis chamber, a load-lock allows samples to be introduced without exposing the main chamber to atmosphere, dramatically reducing pump-down times and contamination risk. In advanced instruments, the load-lock may also be equipped with sample preparation facilities, inert transfer ports, or glovebox docking interfaces used for air-sensitive samples.

X-ray sources

The heart of the XPS instrument is the X-ray source used to generate photons that eject core electrons. Most laboratory XPS systems employ monochromated Al Kα radiation (1,486.6 eV), produced by bombarding an aluminum anode with electrons. The source incorporates a quartz monochromator crystal that energy filters and re-focuses the X-rays, reducing spectral linewidth from about 1 eV in non-monochromated sources to about 0.2 to 0.3 eV. This improvement provides dramatically better spectral resolution. Additional X-ray sources can be added. For example, the Thermo Scientific™ ESCALAB QXi XPS Microprobe can also have a monochromated Ag Lα source (energy: 2,984.3 eV) or a range of non-monochromated sources including Mg or Ti X-rays. Higher energy sources allow access to deeper core levels, and changing the X-ray energy can move overlapping Auger peaks away.

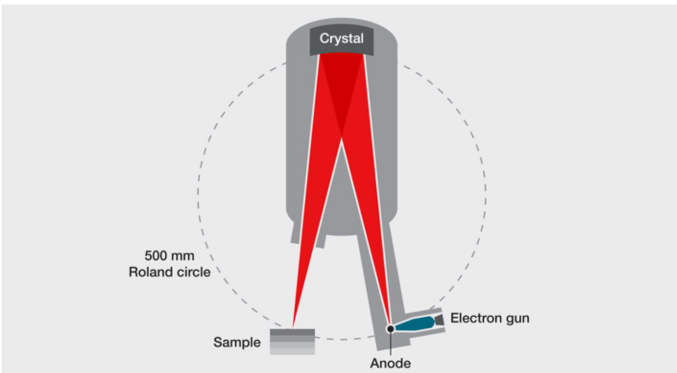


Figure 6: Schematic of a monochromated X-ray source showing the electron source, aluminum anode, and quartz crystal monochromator.

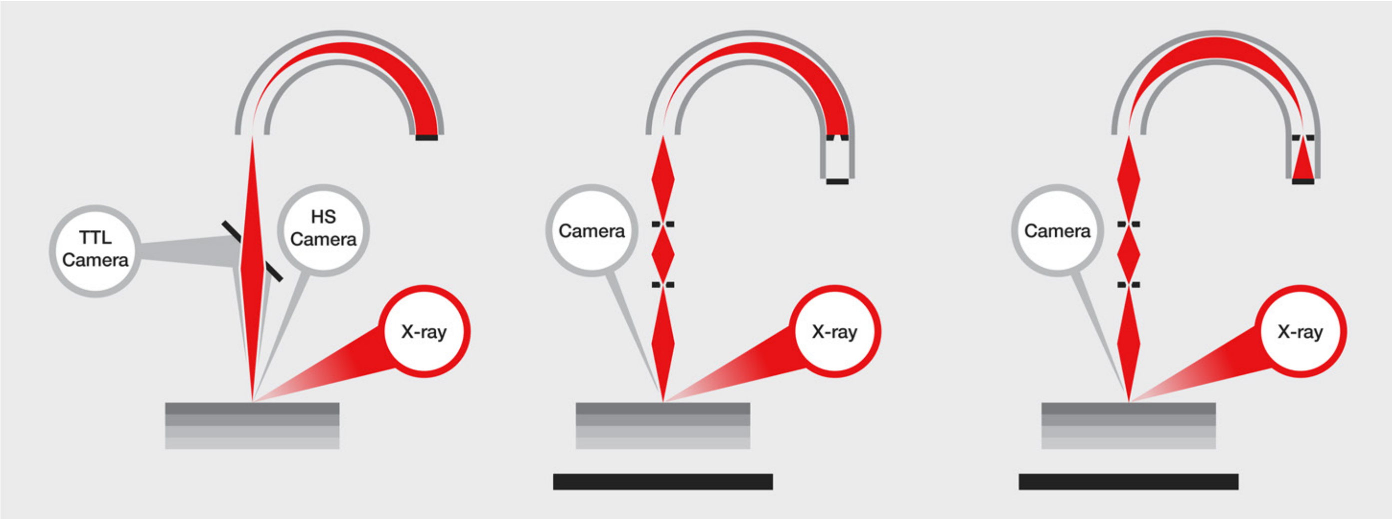


Figure 7: General arrangement of the lenses, analyzer, and detectors for the Thermo Scientific™ K-Alpha and Nexsa™ G2 Systems (left image) and the ESCALAB QXi Microprobe (center and right images).

Electron optics and hemispherical analyzer

Once electrons are emitted from the sample surface, electron optics guide them into the hemispherical analyzer. The electron lens stack is designed to maximize transmission while preserving energy resolution. It typically includes a series of electrostatic and magnetic lenses that focus, accelerate, or decelerate electrons into the entrance slit of the hemispherical analyzer.

The analyzer itself consists of two concentric hemispherical plates across which a voltage is applied. Only electrons with the kinetic energy currently being measured can follow the correct radius of curvature to reach the detector. By scanning the analyzer voltage, the instrument records an energy spectrum containing photoelectron peaks for all elements present in the sample. Analyzer resolution depends on entrance slit widths, pass energy, lens voltages, and electronic stability. Modern analyzers maintain remarkably tight tolerances and are designed for long-term, energy-scale stability to ensure reproducibility.

Detector technology

Modern XPS systems employ multi-channel plate (MCP) detectors or channeltron arrays to detect electrons. These act as amplifiers and electron counters to measure the signal and allow a spectrum to be created. MCP-based detectors enable snapshot spectra, recording entire energy windows in a single exposure rather than stepping sequentially, which can be used for experiments where many spectra need to be collected, such as imaging or depth profiling, and spectral resolution is not critical.

Charge compensation systems

Many samples analyzed by XPS are insulators or partially insulating systems—polymers, ceramics, oxides, organic films, and biomaterials. When photoelectrons are emitted from insulating surfaces, the surface becomes positively charged, which results in shifting peak energies and broadening spectral features. To neutralize this charge buildup, XPS instruments utilize charge compensation systems that maintain a stable electrical environment in the analysis area.

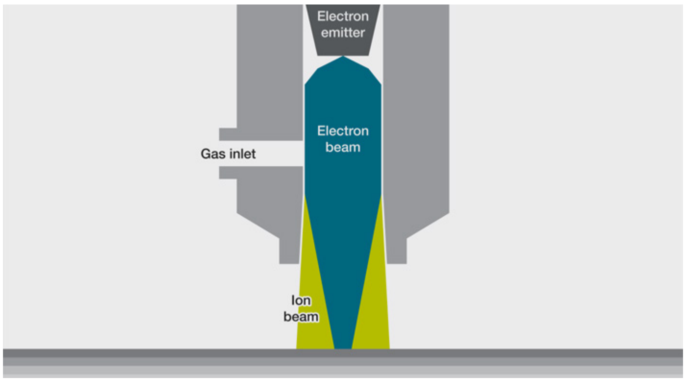


Figure 8: Schematic of a dual-beam flood gun for charge neutralization.

State-of-the-art systems employ dual-beam flood guns that self-regulate charge balance, using both electrons and positively charged ions, usually argon. The electron beam neutralizes positive surface charge, while the ion beam prevents excess negative charge accumulation. This allows stable, reproducible spectra even for highly insulating materials. Successful charge compensation is critical for accurate chemical-state quantification and reliable interpretation of binding-energy shifts.

Ion sources for depth profiling

Monatomic ion sputtering using Ar^+ ions has long been the standard method for XPS depth profiling. It is effective for inorganic materials such as metals, oxides, and nitrides. However, due to high sputter energies, monatomic ions often induce preferential sputtering, reduction, or chemical damage, particularly in polymers and hybrid organic–inorganic materials.

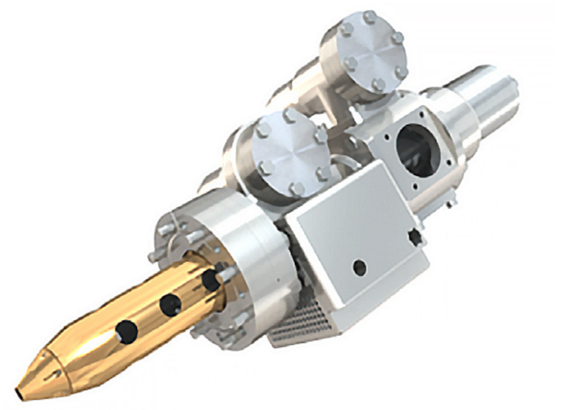


Figure 9: The Thermo Scientific MAGCIS Ion Source can operate in both monatomic and gas cluster ion modes.

Gas-cluster ion sputtering (GCIS) sources overcome many of these limitations. Clusters containing hundreds to thousands of argon atoms impact the surface as a collective mass, sputtering gently with far lower energy per atom. This preserves chemical functionality in organic films and soft materials. GCIS sources have become essential for analyzing OLEDs, polymer surfaces, biomaterials, and thin-film stacks used in flexible electronics.

Femtosecond laser ablation for deep profiling

The newest addition to XPS depth-profiling technology is femtosecond laser ablation (fs-LA). Unlike ion sputtering, fs-LA removes material via ultrafast electronic excitation, eliminating many of the chemical modification pathways that distort depth profiles. It can remove micrometers of material rapidly, enabling analysis of thick coatings, multi-layer paint systems, composite materials, and beam-sensitive perovskites.

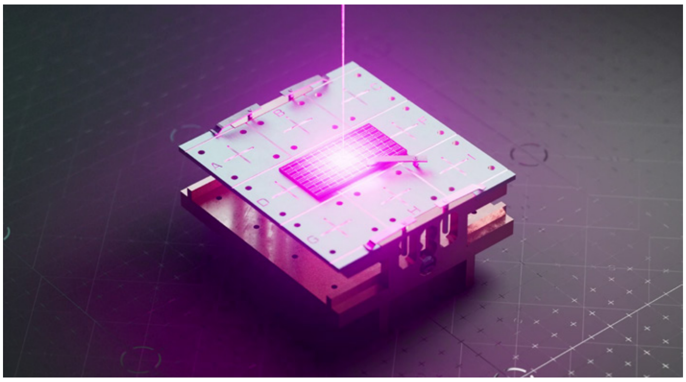


Figure 10: Representation of femtosecond laser ablation.

Fs-LA complements GCIS and monatomic sputtering, offering analysts a full spectrum of material-removal strategies depending on the chemical sensitivity, hardness, thickness, and structural complexity of the sample.

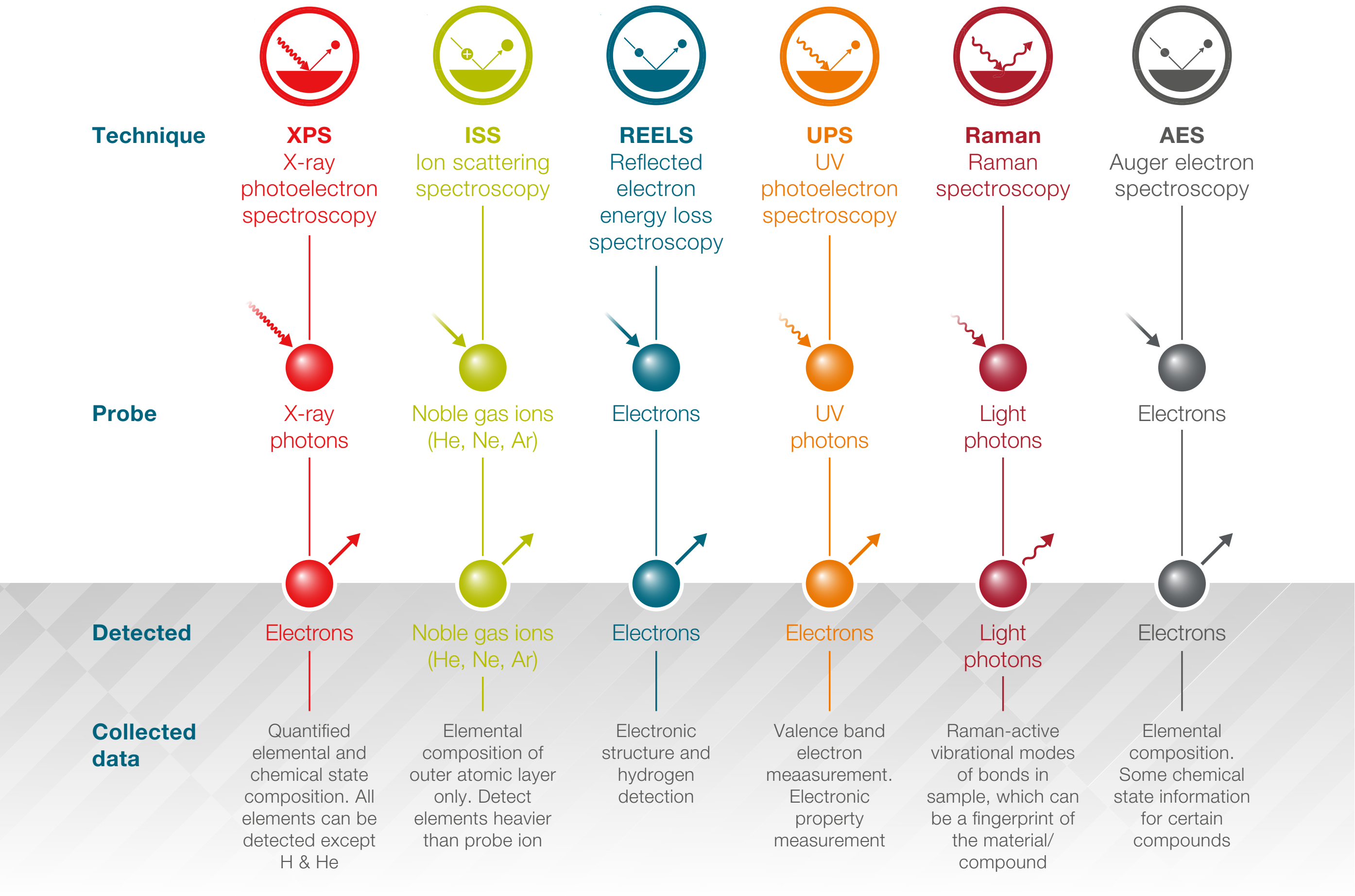


Figure 11: Infographic describing the additional techniques commonly added to an XPS instrument.

Multi-technique integration

Cutting-edge XPS platforms integrate complementary analytical tools into the same instrument geometry. These include:

- Ultraviolet photoelectron spectroscopy (UPS), which measures valence band structure and work function
- Ion scattering spectroscopy (ISS), which probes only the top atomic layer
- Reflected electron energy loss spectroscopy (REELS), which determines band gaps and electronic excitations
- Raman spectroscopy, which reveals bonding structure and crystallinity
- Auger electron spectroscopy (AES), which provides nanoscale spectroscopy and mapping

Integration reduces sample handling, improves positional correlation, and allows analysts to correlate chemical, structural, and electronic data from the same region of interest.

Summary

XPS instruments are a synergy of precision engineering, advanced vacuum science, high-stability X-ray generation, electron-optical design, and intelligent detection electronics. Modern systems provide powerful, flexible platforms capable of high-resolution spectroscopy, rapid imaging, depth profiling, and multi-technique correlation. Understanding the instrument's architecture allows users to maximize data quality, optimize workflows, and select the correct analytical configuration for their applications. As XPS technology continues to evolve—including advances in laser ablation, detector architecture, and automation—it will remain a cornerstone of materials characterization for decades to come.

Chapter 4: Sample handling for X-ray photoelectron spectroscopy

Proper sample handling is one of the most critical determinants of data quality in XPS. Unlike bulk analytical techniques that sample large volumes of material, XPS probes only the outermost 10 nanometers of a surface.

This means that any contamination, improper mounting, surface modification, electrostatic charging, or environmental exposure may obscure the true chemistry of the material under investigation. Even subtle mishandling—touching a surface with gloves, improperly storing a sample, or using incompatible adhesives—can introduce carbonaceous residues or atmospheric species that overwhelm or distort the actual chemical information. As a result, mastering best practices in sample preparation, handling, mounting, and storage is essential for achieving reliable and interpretable XPS spectra.

This chapter provides a comprehensive discussion of the principles, strategies, and practical considerations required to prepare a broad range of sample types, including metals, polymers, ceramics, composites, powders, catalysts, biomaterials, hybrid structures, and air-sensitive materials used in batteries, perovskites, and semiconductor devices.

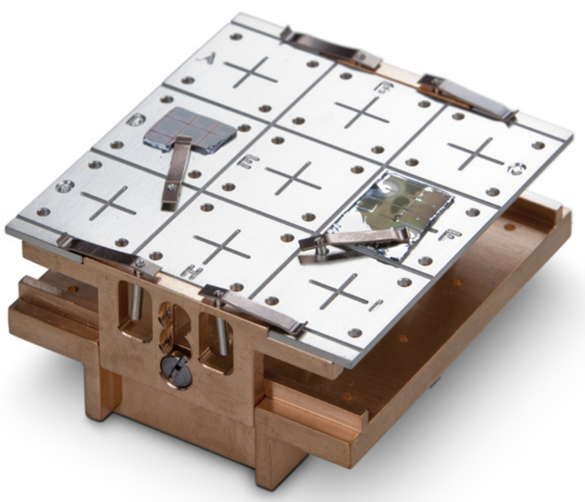


Figure 12: Typical sample mounting using spring clips.

General principles of sample cleanliness

Cleanliness is foundational for XPS sample handling. Because the technique is extremely sensitive to trace contamination, all surfaces that come into contact with the sample—including tweezers, gloves, mounting fixtures, and sample holders—must be thoroughly cleaned. The surfaces to be analyzed should never be touched directly, even when wearing gloves, and fingerprints must be strictly avoided, as even microscopic oils contain carbon and oxygen species that appear in XPS spectra as strong peaks in the C 1s and O 1s regions. When handling multiple samples, analysts should ensure that workspaces are clean, and cleanroom wipes or inert nitrogen streams are available for removing dust. In cases where contamination is a concern, a gentle solvent rinse with isopropanol may be applied, provided the material is chemically compatible with solvent exposure. However, many solvent washes leave thin residues, so it's recommended to test the cleaning protocol on samples that can be disposed.

Mechanical integrity and sample geometry

XPS imposes several mechanical requirements on samples. Samples must physically fit within the instrument's sample holder and must withstand vacuum conditions without outgassing or deforming. For example, polymeric materials with volatile additives or high moisture absorption may outgas significantly in vacuum, delaying pump-down times or contaminating the vacuum chamber. Large or non-flat samples may require sectioning or careful clamping. With composite materials, such as multilayer laminates, analysts should also consider how cutting or fracturing the sample may expose internal layers that were never intended for surface characterization and that may oxidize rapidly.

Mounting approaches

Most samples can be attached to the sample holder using clips or vacuum-compatible conductive adhesive tape. Care may be needed for partially conductive samples to ensure that they are electrically isolated to prevent differential charging from affecting spectra quality. When mounting powders, analysts often press the powder into indented wells on sample holders, adhere it using conductive adhesive tabs, or immobilize it lightly with UHV-stable carbon tape. Great care must be taken to ensure that powder grains do not migrate into the instrument's vacuum system. Powders containing volatile organic components must be pre-dried to remove residual solvents. In some cases, gentle heating under inert gas or vacuum may be applied to remove trapped moisture. For catalytic powders, which often contain air-sensitive surface phases, analysts must minimize oxygen exposure during transfer.

Air-sensitive samples and inert transfer

Many advanced materials cannot tolerate exposure to oxygen, moisture, or carbon dioxide. Examples include halide perovskites, lithiated battery electrodes, sulfide-based electrolytes, metallic lithium, organometallic precursors, freshly deposited metal films, and catalysts in reduced states. To maintain chemical integrity, these materials require inert gas gloveboxes for sample preparation and specialized transfer devices for moving the sample from the glovebox to the XPS analysis chamber without exposure to atmosphere. The ability to preserve reactive surface chemistries opens new avenues for studying degradation, interface formation, and metastable phases that cannot otherwise be observed.

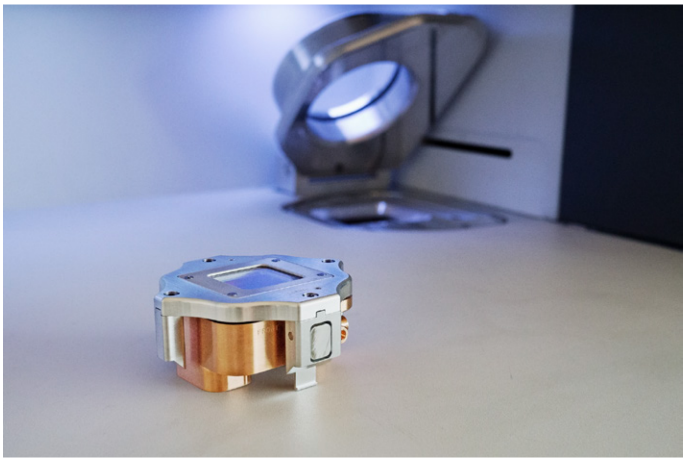


Figure 13: Nexsa G2 System vacuum transfer module for loading air-sensitive samples.

Porous materials such as zeolites, aerogels, battery electrodes, or metal–organic frameworks (MOFs) may also require special handling because their pores trap atmospheric gases and moisture, which may desorb under vacuum and alter chemical states. Such samples should be pre-conditioned under vacuum, in a glovebox, or under inert gas flow depending on their moisture affinity.

Summary

Sample handling is one of the most important aspects of successful XPS analysis. From cleaning and mounting to inert transfer and charge control, proper preparation ensures that the spectra collected truly represent the material of interest. By understanding the unique challenges posed by different classes of materials—metals, polymers, ceramics, powders, catalysts, and air-sensitive compounds—analysts can apply the correct methods to obtain reproducible, high-quality data. Every subsequent step of the XPS workflow relies on proper sample handling, making this an essential competency for all XPS practitioners.

Chapter 5: Analytical methods in X-ray photoelectron spectroscopy

XPS offers a uniquely versatile analytical toolbox. While the technique is perhaps best known for its ability to provide high-resolution chemical-state information, its true power lies in the diversity of analytical methods that can be applied depending on sample type, industry needs, and investigative goals.

Analytical methods in XPS span from simple elemental identification to sophisticated workflows such as angle-resolved XPS (ARXPS), chemical-state mapping, multi-point automation, in-depth quantification, and advanced depth profiling.

This chapter explores the strategies, decision paths, and measurement modalities that transform XPS from a single-spectrum technique into a multidimensional characterization platform capable of addressing the most complex surface-engineering questions.

Survey spectra: The first step in any XPS workflow

The survey scan (also called a wide-scan spectrum) is the foundation of virtually all XPS analyses. It provides a rapid overview of all elements present on the surface with binding energies in the range of the spectrometer. Acquired typically at high pass energies to maximize signal intensity, survey scans reveal elemental composition, identify unexpected contaminants, and inform the selection of high-resolution regions to scan next. Survey spectra also help analysts determine whether a surface is clean, oxidized, contaminated, or contains trace impurities. For example, survey scans can detect adventitious sulfur, halogens, lithium, or organic residues at concentrations as low as 0.1 atomic percent, enabling root-cause analysis of processing defects or environmental degradation.

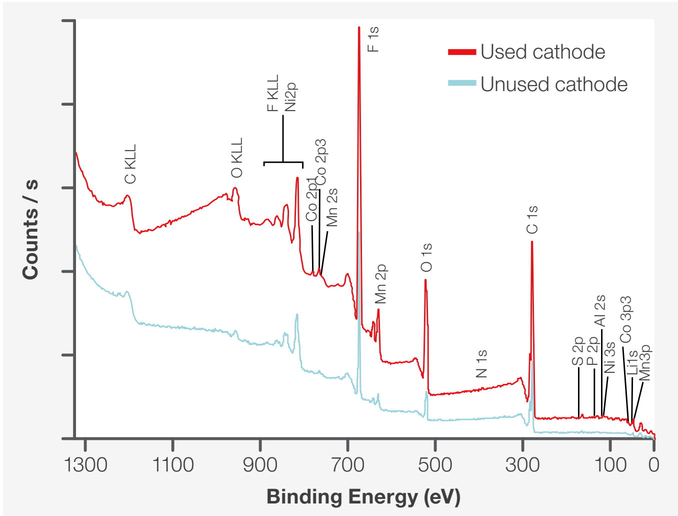


Figure 14: Survey spectra for unused and used Li-ion battery cathodes

High-resolution spectra and chemical-state analysis

High-resolution regional scans are the centerpiece of XPS analytical workflows. These spectra capture fine details in peak shapes, spin-orbit splitting, multiplet structures, shake-up satellites, and chemical shifts. Analysts rely on these features to determine oxidation states (e.g., Fe^{2+} vs Fe^{3+}), coordination environments (e.g., phosphate vs. sulfate), bonding structures (e.g., C–C vs. C–O vs. C=O vs. O–C=O), and subtle differences between chemical species. Chemical shifts as small as 0.1 to 0.2 eV can be resolved in modern spectrometers, provided that appropriate pass energies, count times, and charge compensation settings are selected.

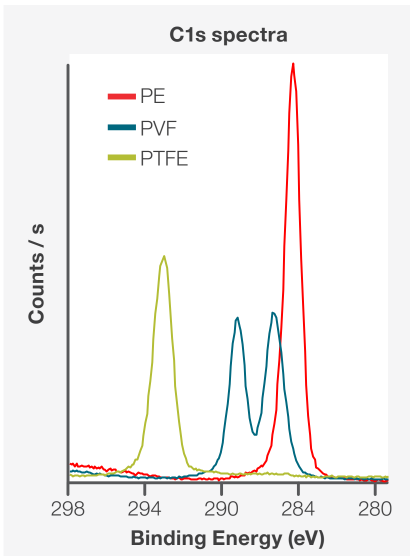


Figure 15: High-resolution C 1s spectra of polyethylene, polyvinyl fluoride, and polytetrafluoroethylene.

XPS quantification

XPS quantification is based on peak areas, corrected using elemental sensitivity factors and analyzer transmission functions. While XPS does not measure absolute concentrations without standards, it provides highly reproducible relative atomic concentrations. Accuracy typically falls within ±10%, though this depends heavily on factors such as sample uniformity, surface roughness, and the presence of adventitious contamination. Quantitative XPS is essential for verifying stoichiometry in oxide films, monitoring elemental cycling in battery electrodes, detecting dopant levels in thin films, and evaluating additive concentrations in polymers or catalysts.

Peak fitting is a key step in chemical-state analysis. Analysts use Voigt or pseudo-Voigt line shapes, constrained spin–orbit ratios, and physically meaningful peak widths to extract chemically relevant information. Overfitting must be avoided—peak fitting should reflect underlying chemistry rather than arbitrary mathematical decomposition. Reference databases, principal component analysis, and intelligent software guidance (e.g., the Knowledge View in Avantage Software) help ensure accurate, consistent peak interpretation across users and laboratories.

Angle-resolved XPS (ARXPS)

Angle-resolved XPS enhances surface sensitivity by varying the orientation of the sample with respect to the analyzer. By doing this, more of the substrate signal is attenuated by the overlayers, as shown in Figure 14. This technique enables non-destructive depth profiling of ultra-thin films (0.5 to 10 nm). Unlike sputtering-based depth profiling, ARXPS preserves chemical states and surface morphology, making it ideal for studies of native oxides, passivation layers, ligand-functionalized nanoparticles, self-assembled monolayers (SAMs), and contamination films.

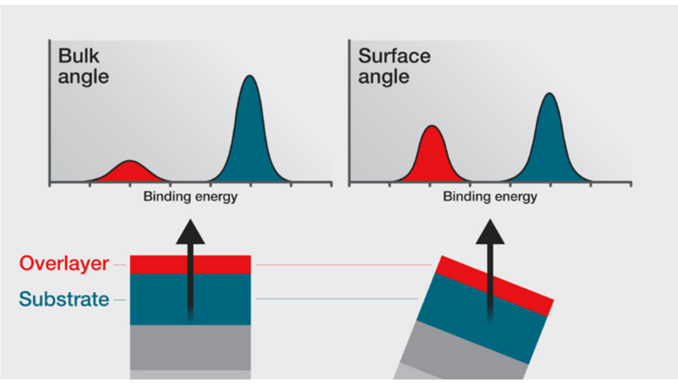


Figure 16: Schematic of angle-resolved XPS.

ARXPS data can be modeled using multilayer thickness algorithms and maximum-entropy reconstruction to determine depth concentration profiles. For semiconductor gate oxides, ARXPS is invaluable for quantifying SiO_2 thickness, detecting interfacial suboxides, and resolving nitrogen profiles in oxynitride dielectrics.

Spatially resolved XPS: Imaging and SnapMap

Traditional XPS is a point-analysis technique, but advances in electron optics and detector architectures have enabled spatially resolved chemical-state imaging. Thermo Scientific SnapMap Technology, for example, collects full XPS spectra at each pixel of a rastered grid. This allows users to visualize chemical heterogeneity across surfaces at micron-scale resolution.

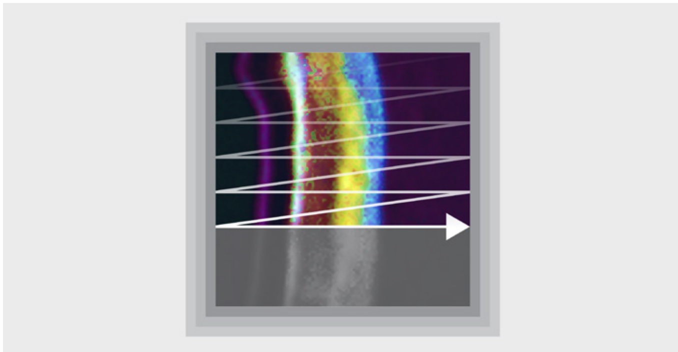


Figure 17: Schematic showing how SnapMap operates for the Nexsa G2 System.

XPS mapping is particularly useful for:

- Identifying failure points in coatings
- Characterizing patterned semiconductors
- Investigating corrosion pitting and oxide formation
- Examining inhomogeneities in battery electrodes
- Mapping contaminants or inclusions

Chemical-state maps extend beyond elemental distribution. They can differentiate oxidation states, polymer functional groups, or phases in mixed-metal oxides. These insights are critical during device development, failure analysis, quality assurance, and materials optimization.

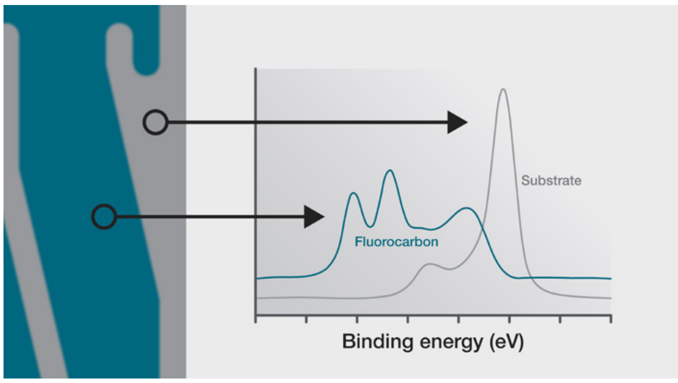


Figure 18: Spectra are behind each pixel in an image, so that the distribution of chemistries can be seen on a sample surface.

Line scans and region-of-interest profiling

Line scans collect spectra at sequential positions across a feature of interest, such as a grain boundary, crack, interphase, or coating edge. For example, in multi-layer coatings, line scans reveal where interdiffusion occurs. In semiconductor wafers, they help evaluate metal–dielectric interface abruptness. In biomaterials, they highlight gradients in surface functionalization. Line scans bridge the gap between point spectroscopy and full imaging, offering fast, targeted insights.

Multi-point and automated workflows

High-throughput XPS instruments support automated multi-point workflows that allow dozens of positions to be analyzed sequentially. These workflows are indispensable in quality-control environments that require consistent analysis across large batches of samples. Automated workflows include:

- Stage automation
- Predefined recipes
- Charge-control optimization
- Auto-focus routines
- Automated report generation

Such automation dramatically reduces operator-to-operator variability and ensures reproducible datasets across shifts, facilities, and product cycles.

Integration with depth profiling

While depth profiling is discussed in detail in Chapter 6, it plays a critical role in many analytical workflows. Analysts often combine chemical-state XPS with monatomic or cluster ion sputtering or femtosecond laser ablation. Depth profiling allows the XPS analysis depth to be extended into the bulk of the material, revealing how the composition changes away from the surface.

Example workflows by industry

Different industries rely on specific XPS analytical methods:

- Semiconductors: Focus on ultra-thin oxide quantification, contamination detection, dielectric stack profiling, metal oxidation states, and ARXPS.
- Batteries: Analyze SEI layers, electrolyte decomposition, transition-metal dissolution, and electrode-surface evolution.
- Polymers and coatings: Chemical-state mapping, surface-additive quantification, plasma-treatment verification, and adhesion-failure analysis.
- Catalysis: High-resolution chemical-state analysis of metal and oxide species and dopant phases, sputter cleaning, and *in situ* analysis.
- 2D materials: Spatial mapping of monolayer chemistry, defect density evaluation, oxidation detection, and multimodal correlation.

Each of these workflows employs a mix of survey scans, high-resolution scans, ARXPS, mapping, line scans, and depth profiling.

Summary

Analytical methods in XPS form a comprehensive suite of tools for understanding surface and interface chemistry. By combining surveys, high-resolution spectra, ARXPS, mapping, automated workflows, and depth-profiling options, modern XPS provides multi-dimensional chemical insight into even the most complex materials systems. Mastery of these methods enables researchers, engineers, and analysts to transform raw spectral data into meaningful scientific and industrial decisions.

Chapter 6: Depth profiling in X-ray photoelectron spectroscopy

Depth profiling is one of the most powerful extensions of XPS, enabling researchers and engineers to examine how chemical composition and electronic structure vary beneath the immediate surface.

While traditional XPS probes only the top few nanometers of a material, depth profiling reveals buried interfaces, diffusion phenomena, layered architectures, chemical gradients, coating adhesion behavior, and degradation pathways. As modern technologies increasingly rely on engineered multilayer systems—such as semiconductor devices, advanced battery electrodes, anti-corrosion coatings, tribological films, optical stacks, thin-film photovoltaics, and polymer laminates—the ability to accurately characterize subsurface chemistry becomes essential.

This chapter provides a comprehensive examination of the methods associated with depth profiling in XPS, with a focus on the strengths, limitations, and best practices of monatomic ion sputtering, gas-cluster ion sputtering, and femtosecond-laser ablation.

Fundamentals of depth profiling

The underlying principle of XPS depth profiling involves alternating cycles of XPS measurement and material removal. After the surface is measured a thin layer is removed—typically between 0.2 nm and several nanometers, depending on the sputtering or ablation technique—a new XPS spectrum is acquired to assess the composition and chemical states at that depth. By repeating this process iteratively, analysts generate a depth-resolved chemical profile.

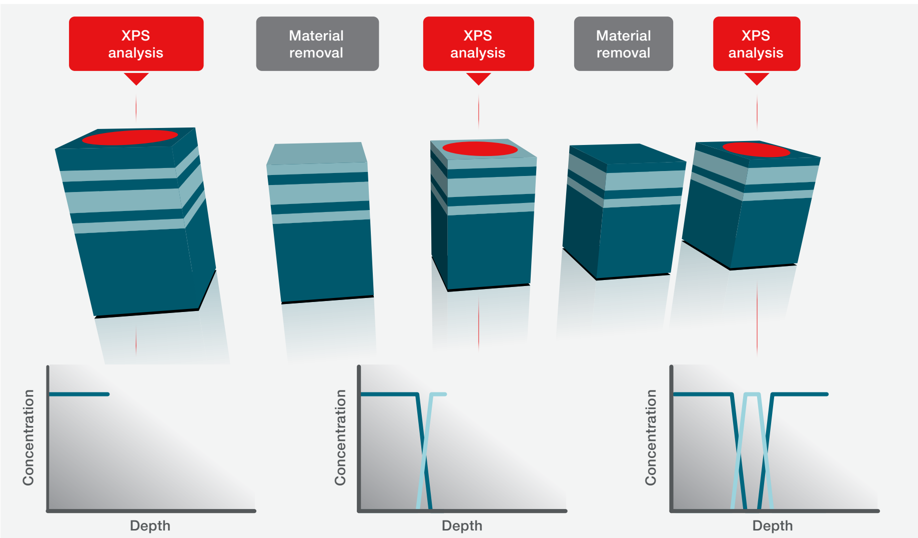


Figure 19: The depth profiling workflow

Monatomic ion sputtering

Monatomic argon ion sputtering has been the standard depth-profiling method for decades. High-energy Ar^+ ions (typically 500 eV to 4 keV) strike the surface, transferring momentum through ballistic collisions that eject atoms from the surface layer. This technique is highly effective for removing metals, oxides, nitrides, and inorganic materials. An example profile for a low-emissivity glass coating is shown in Figure 18.

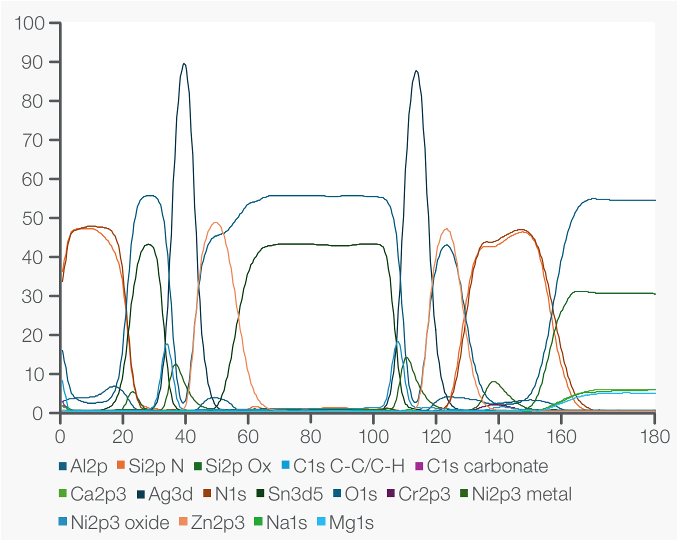


Figure 20: Depth profile of a low-emissivity glass, obtained with monoatomic ion beam profiling.

However, monatomic sputtering can have issues. Preferential sputtering occurs when lighter elements—such as oxygen, nitrogen, sulfur, or halogens—are removed more rapidly than heavier matrix atoms. This distorts stoichiometry, produces false chemical ratios, and may obscure the true composition of buried layers. Ion bombardment can also reduce metal oxides, alter valence states, break chemical bonds in polymers, and produce suboxides or metallic species not originally present. Beam-induced roughening is another artifact: high sputter energies can increase surface topography, lowering depth resolution and broadening buried interfaces. These limitations make monatomic sputtering unsuitable for many organic, polymeric, sensitive, or hybrid organic–inorganic materials.

Gas-cluster ion sputtering

To address the limitations of monatomic sputtering, gas-cluster ion sources (GCIS) were developed. In GCIS, clusters of up to thousands of argon atoms are ionized as a single unit (e.g., Ar_{1000}^{+}). Although the total kinetic energy may be several keV, the energy per atom is extremely low (1 to 20 eV). This dramatically reduces chemical damage, making cluster sputtering suitable for polymers, biomaterials, organic thin films, photoresists, OLED layers, and hybrid materials.

Unlike monatomic sputtering, GCIS does not significantly alter chemical states, preserving functional groups such as C–O, C=O, carbonates, urethane linkages, and aromatic structures. This enables high-confidence depth profiling of polymer laminates, plasma-treated surfaces, organic coatings, and multilayer electronic structures. However, GCIS sputter rates are slower for hard inorganic materials, and crater-edge effects must be monitored. In many workflows, analysts combine monatomic sputtering for inorganics with GCIS for organics, allowing depth profiling across hybrid stacks.

Femtosecond laser ablation

The most recent advancement in XPS depth profiling is femtosecond laser ablation (fs-LA). Unlike sputtering, fs-LA removes material using ultrafast electronic excitation rather than thermal or ballistic processes. Because the laser pulse is extremely short (typically about 160 fs), energy is deposited faster than phonon interactions can occur. This prevents thermal diffusion, melting, or chemical bond rearrangement preventing chemical modification of the remaining material, but also permitting faster removal to quickly get to deep interfaces. As a result, fs-LA offers unprecedented advantages for thick films, beam-sensitive materials, and polymeric or hybrid systems.

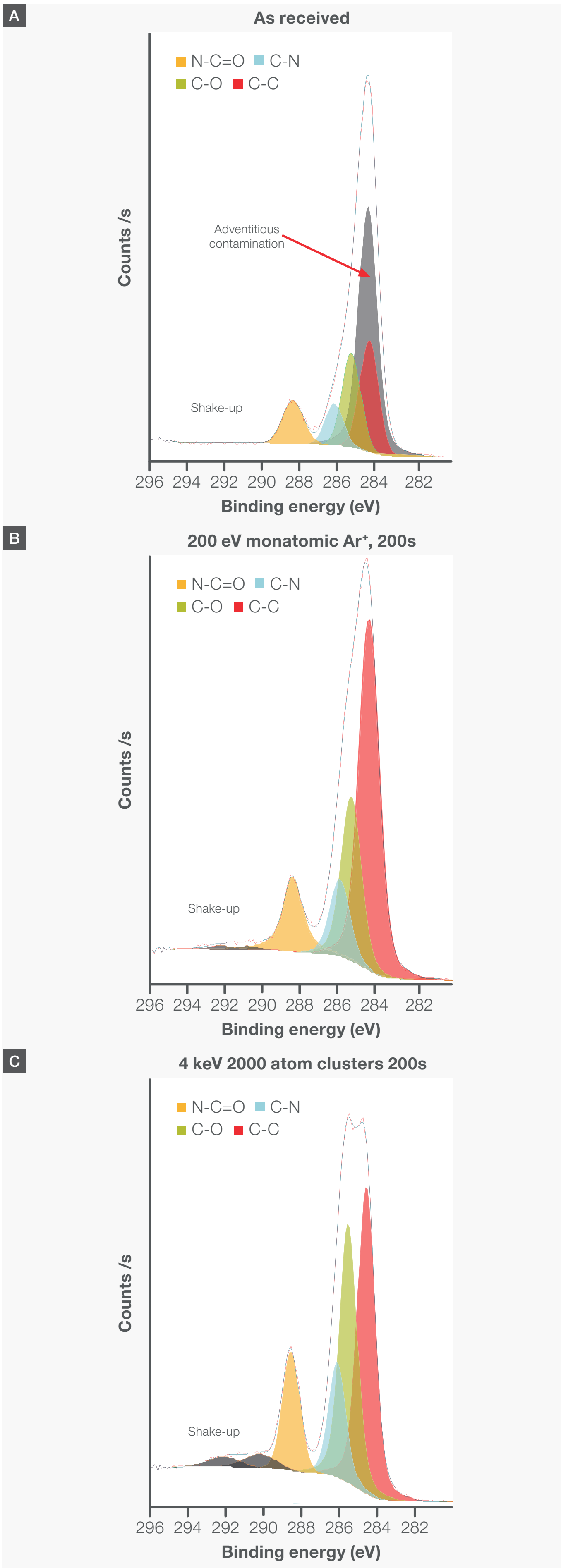


Figure 21: A polyimide sample cleaned using 200 eV monatomic ions (B) and 4 keV 2,000 atom cluster ions (C).

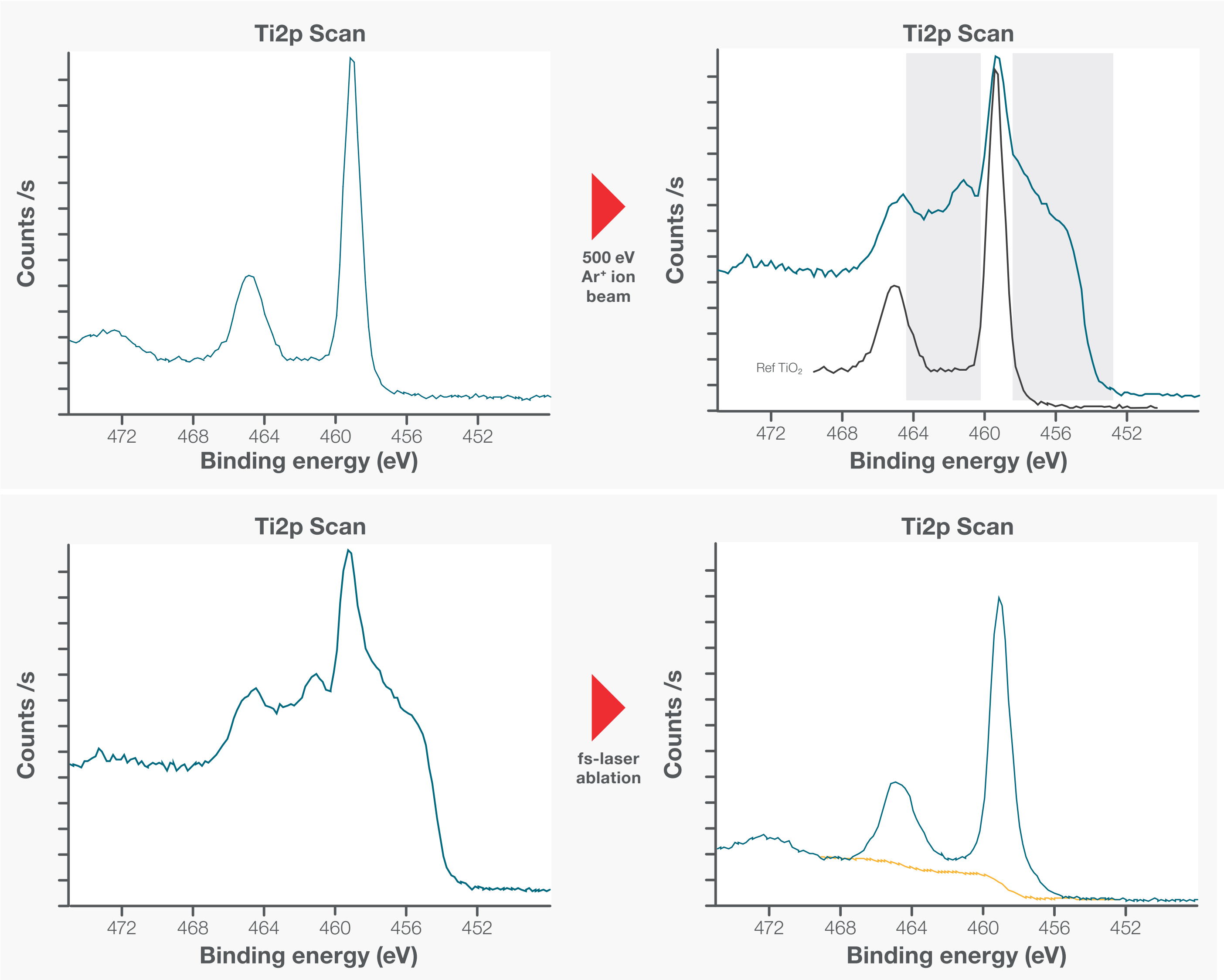


Figure 22: Scans of a TiO_2 sample with damage from monatomic ions (highlighted in grey) and with the damage removed using fs-LA.

Fs-LA enables removal of tens to hundreds of nanometers of material per pulse, making it ideal for depth profiling micrometer-thick coatings such as automotive paints, polymer laminates, anti-wear films, perovskite photovoltaic stacks, and composite structures. The method preserves chemical states even for halide perovskites—materials that are notoriously fragile under ion bombardment. Laser-crater geometry, pulse energy optimization, and stage motion allow analysts to generate smooth crater floors and high-resolution profiles. When combined with rapid XPS “snapshot” acquisition, fs-LA enables high-throughput depth profiling that is orders of magnitude faster than sputtering. This makes it possible to perform several profiles on a sample to confirm the conformity of a structure.

Depth-resolution considerations

Depth resolution defines the instrument's ability to distinguish between two adjacent chemical layers. It depends on several factors:

- The sputtering or ablation mechanism
- Surface roughening
- Material hardness
- Crater uniformity
- Analyzer acceptance angle

Monatomic sputtering tends to produce sharper interfaces in hard materials but can damage soft layers. GCIS preserves chemical states but may produce broader interfaces in hard substrates. Fs-LA provides excellent depth resolution for polymeric coatings but requires careful optimization for brittle or crystalline materials to avoid microfractures. Choosing the right technique is often a trade-off between chemical fidelity and structural resolution.

Calibration and reference materials

Accurate depth profiling requires calibration using reference materials with known thicknesses or sputter rates. Silicon dioxide on silicon wafers (SiO_2/Si) or tantalum pentoxide on tantalum (Ta^{2O5}/Ta) are commonly used as reference materials for monatomic sputtering as it is relatively easy to produce well-defined layers of a known thickness. For GCIS, polymer films of known thickness—such as PMMA or polystyrene—provide useful benchmarks. Fs-LA depth calibration can be performed using similar approaches, or by using stylus profilometry or white-light interferometry to measure crater depths directly. For thinner layers or at interfaces, it is possible to use the XPS measurements directly to calculate removal rates.

Best practices

Successful depth profiling requires thoughtful planning:

- Choose the sputtering method based on material sensitivity
- Use appropriate reference materials to calibrate sputter rates
- Monitor surface roughening with SEM or AFM
- Validate chemical states using complementary techniques
- Use GCIS for soft or hybrid materials and fs-LA for thick or sensitive materials
- Ensure consistent data acquisition parameters across depth cycles

Adhering to best practices produces reliable profiles and ensures chemical fidelity across complex stacks.

Summary

Depth profiling extends XPS into the third dimension, allowing users to visualize how chemistry evolves beneath the surface. Monatomic ion sputtering, gas-cluster sputtering, and femtosecond laser ablation each offer unique benefits depending on material type and analytical goals. Understanding their strengths and limitations enables analysts to tailor depth-profiling workflows for accurate, meaningful, and application-driven results.

Chapter 7: Complementary techniques in X-ray photoelectron spectroscopy

Modern XPS is an extraordinarily powerful technique on its own, providing detailed chemical-state information, elemental composition, and quantitative analysis of the top few nanometers of a surface.

However, as materials, devices, and engineered systems continue to grow in complexity, no single analytical method can fully characterize all aspects of a surface or interface. To address the increasing analytical demands of contemporary research and industrial workflows, XPS instruments are now frequently equipped with complementary techniques that expand their capabilities far beyond traditional core-level spectroscopy. These additional techniques—ultraviolet photoelectron spectroscopy (UPS), reflected electron energy loss spectroscopy (REELS), ion scattering spectroscopy (ISS), Raman spectroscopy, and Auger electron spectroscopy (AES)—provide critical insight into valence structure, surface termination, band gap, bonding, crystallinity, and nanoscale heterogeneity. When combined with XPS, they create a comprehensive multi-modal toolbox for evaluating surface and interfacial chemistry.

Ultraviolet photoelectron spectroscopy

UPS focuses on valence electrons rather than core electrons. Using ultraviolet photons—typically He I (21.2 eV) or He II (40.8 eV)—UPS provides high-resolution information about surface electronic structure, including:

- Valence band maximum (VBM)
- Highest occupied molecular orbital (HOMO)
- Density of states near the Fermi level
- Work function and ionization potential
- Molecular orbital features in organic semiconductors

Because UPS examines the outermost electronic states, it is indispensable for research in multiple industries:

- Organic electronics (OLEDs, OPVs, OFETs)
- 2D semiconductors and heterostructures
- Charge transport and injection layers
- Catalytic surfaces
- Corrosion films and oxide states

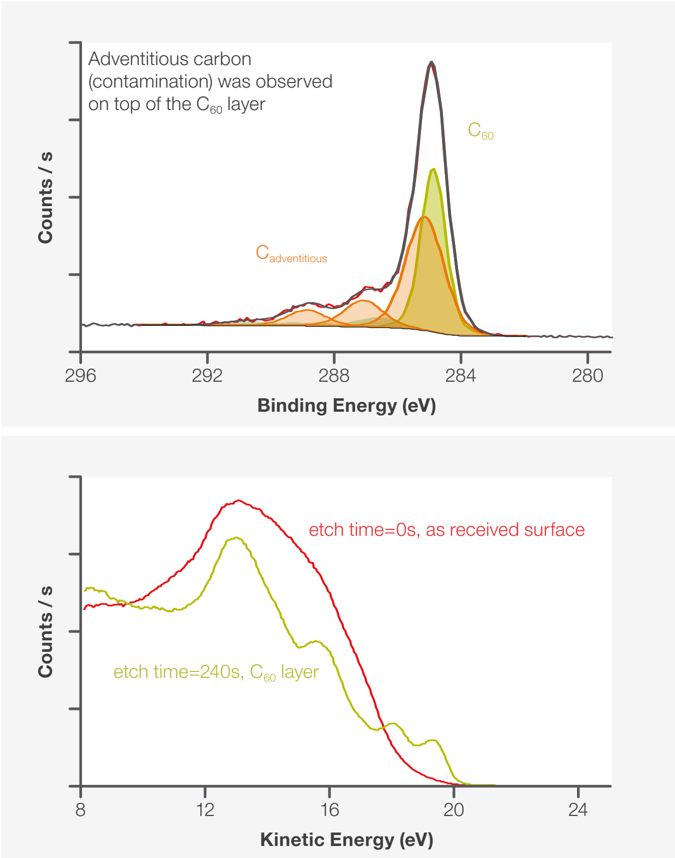


Figure 23: Core-level C 1s and UPS valence band spectra for adventitious carbon and C 60.

UPS data, when combined with XPS core-level information, enables construction of complete energy-level diagrams, providing essential insight into charge alignment, band offsets, and interfacial electronic behavior.

Reflected electron energy loss spectroscopy

REELS measures the energy lost by electrons scattered from the surface, revealing electronic excitations within a material. REELS is particularly valuable because it provides:

- Band-gap measurements
- Information about unoccupied electronic states
- Plasmon loss features
- Hydrogen quantification in polymers

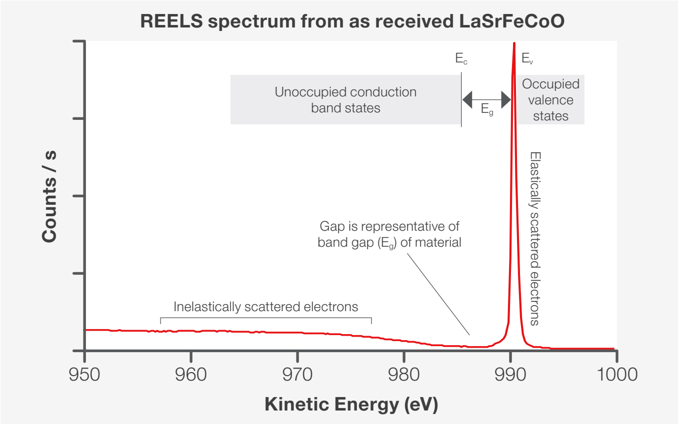


Figure 24: 1 keV REELS spectrum from the double perovskite LaSrFeCoO, used for measuring the sample band gap.

Measuring band gaps is especially valuable for semiconductors, insulators, ultra-thin dielectrics, high-k oxides, and novel materials used in photovoltaics and optoelectronic devices. REELS also complements XPS by verifying oxidation states through characteristic loss signatures. For polymer researchers, REELS provides a rare method for detecting hydrogen, which cannot be observed using conventional XPS.

Ion scattering spectroscopy

ISS is the most surface specific of the complementary techniques. Low-energy He^+ ions scatter off the topmost atomic layer, and the energy of the backscattered ions reveals the elemental identity of surface atoms. Unlike XPS, which probes several nanometers deep, ISS senses only the first atomic layer (0.3 to 0.5 nm). This makes ISS ideal for:

- Evaluating surface coverage in early-stage depositions
- Verifying plasma or chemical treatments
- Measuring segregation in alloys or thin films

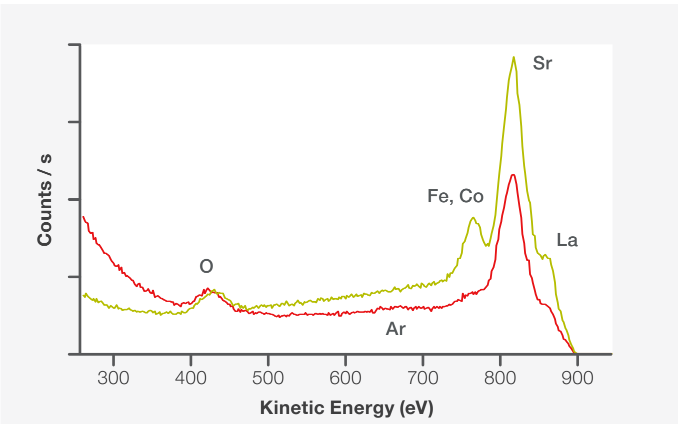
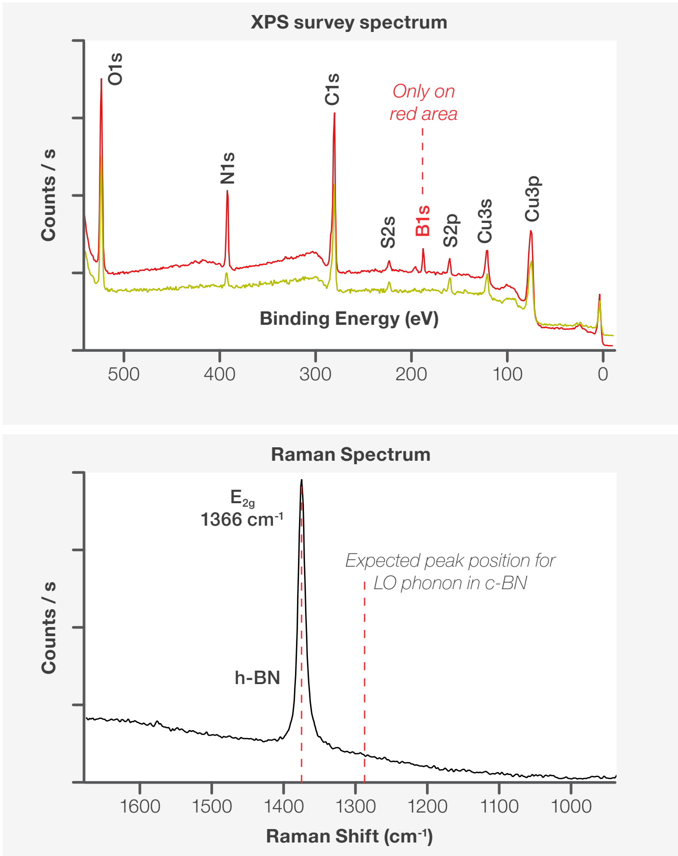


Figure 25: ISS spectra of LaSrFeCoO double perovskite as received (red) and after ion sputtering (green).

Raman spectroscopy

Raman spectroscopy provides vibrational information about molecular bonds, crystal symmetry, phonon modes, and structural disorder. When Raman is integrated directly into the XPS analysis chamber—as in the Nexsa G2 System—it allows for correlated optical and chemical analysis of the same point on a sample. Raman complements XPS by providing:

- Information on polymer functional groups
- Crystallinity and molecular order in carbon materials
- Layer thickness of 2D materials (e.g., graphene, MoS_2)
- Phase identification in oxides and ceramics
- Stress, strain, and defect density



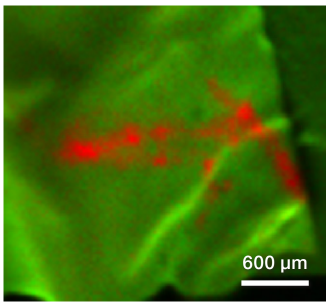


Figure 26: Using XPS SnapMap to locate optically invisible features with XPS and Raman analysis at the same location. The N 1s XPS image shows areas with boron nitride (red) and areas with only the nitrogen precursor (green).

For example, in MoS_2, Raman peak separation between E_2g and A_1g modes indicates layer number, while XPS provides oxidation state and sulfur stoichiometry. This dual analysis provides insights unattainable from either technique alone.

Auger electron spectroscopy

AES probes near-surface composition using an electron beam rather than X-rays. The Auger process yields transition energies unique to each element, enabling analysis sensitive to chemical state. AES provides:

- Nanoscale chemical mapping (<100 nm)
- High-resolution imaging of inclusions, precipitates, or micro-phases
- Complementary insight into metallic bonding environments

AES is particularly valuable in metallurgy, microelectronics, and failure analysis when nanoscale chemical variations must be resolved. When paired with XPS, AES allows for comprehensive depth and lateral resolution in complex systems.

Combining UPS, REELS, ISS, Raman, and AES with XPS

The true advantage of complementary techniques is their integration into unified workflows. For example:

- XPS identifies oxidation states; UPS confirms electronic alignment
- XPS quantifies stoichiometry; REELS verifies electronic band gap
- XPS detects subsurface chemistry; ISS confirms surface termination
- XPS reveals chemical states; Raman provides crystallinity correlations
- XPS maps micron-scale features; AES fills in nanoscale chemical detail

Together, these tools create a multi-dimensional analysis that supports next-generation research in nanotechnology, energy storage, optoelectronics, catalysis, biomaterials, and advanced manufacturing.

Practical considerations for multi-technique integration

Each complementary technique introduces specific requirements. For example:

- UPS requires high vacuum purity and stable He discharge lamps
- REELS requires optimized electron energy and scattering angles
- ISS requires extremely clean sample surfaces to avoid signal masking
- Raman requires optical access, careful focusing, and laser-power tuning
- AES requires conductive samples or charge compensation strategies

Analysts must choose the technique sequence carefully. Generally, XPS is performed first to ensure that the sample is as expected and validate if any cleaning is required prior to continuing the analysis. The additional techniques can then follow to supplement the XPS data.

Summary

Complementary techniques transform XPS into a holistic surface analysis platform capable of probing chemical, structural, electronic, and vibrational information. UPS, REELS, ISS, Raman, and AES each contribute unique insights, allowing analysts to construct complete surface and interface profiles. Modern integrated systems enable rapid switching between techniques, ensuring data consistency and alignment. Mastery of complementary techniques allows researchers and engineers to decode complex materials with unprecedented depth and confidence.

Chapter 8: Applications of X-ray photoelectron spectroscopy

XPS has become an indispensable analytical tool across nearly every technologically significant industry. This chapter explores the wide range of applications in which XPS plays a central role, highlighting both established uses and emerging areas driven by modern research priorities.

Semiconductors and microelectronics

The semiconductor industry is one of the largest and most demanding users of XPS. As transistor gate lengths have shrunk into the single-digit nanometer regime, control of interfacial chemistry has become critical for device reliability. XPS plays a key role in studying:

- High-k dielectrics such as HfO_2, Al_2O_3, and ZrO_2
- SiO_2/Si interface chemistry and suboxides
- Nitrogen incorporation in oxynitride films
- Metal gate work-function tuning
- Surface contamination on wafers
- Photoresist residue analysis
- Passivation layer formation
- Diffusion barrier and adhesion layer performance

XPS is unmatched in its ability to quantify oxidation states, film stoichiometry, and interfacial chemical gradients. Angle-resolved XPS provides non-destructive thickness analysis for ultra-thin films, while depth profiling reveals buried contamination or chlorine/fluorine residues from plasma processing. Advanced complementary techniques such as ISS and REELS are often paired with XPS to verify top-layer coverage and band-gap evolution in dielectric stacks.

Battery materials and energy storage systems

Battery research relies heavily on XPS because the electrochemical stability of electrode–electrolyte interfaces governs battery lifespan, safety, and performance. XPS is used to evaluate:

- Solid-electrolyte interphase (SEI) composition
- Transition-metal dissolution
- Surface reconstruction of Ni-rich layered oxides
- Salt decomposition pathways (e.g., $LiPF_6 \rightarrow$ LiF, P–O, P–F species)
- Polysulfide species in Li–S batteries
- Surface films on solid-state electrolytes

The SEI, which forms spontaneously during the first few charge cycles, is typically only a few nanometers thick and contains inorganic (LiF, Li_2CO_3, Li_2O, Li_3PO_4) and organic ($ROCO_2Li$, alkoxides, polycarbonates) species. XPS is one of the few techniques with sufficient sensitivity to quantify these species. Depth profiling (GCIS or fs-LA) reveals how SEI layers evolve with cycling, temperature, electrolyte additives, and formation protocols. XPS also aids the development of solid-state batteries by characterizing interfacial reactions between sulfide electrolytes, oxide electrolytes, and electrode materials.

Polymer science, surface treatments, and coatings

Polymers and coatings represent another major application area for XPS due to the strong influence of surface chemistry on adhesion, wettability, printability, biocompatibility, and long-term durability. XPS is routinely used to study:

- Plasma treatments (e.g., oxygen, nitrogen, ammonia plasmas)
- UV-ozone treatments
- Surface functionalization and grafting
- Additive migration and blooming
- Oxidative aging and environmental degradation
- Multi-layer polymer laminates
- Paint and coating failures

Surface modifications often involve subtle changes such as increased oxygen functionality (C–O, C=O, O–C=O), nitrogen incorporation, or formation of hydroxyl groups—all readily detectable by XPS. Chemical-state mapping helps correlate surface energies and wetting behavior with specific functional groups. GCIS and fs-LA expand the range of polymer systems that can be depth profiled without damage, enabling analysis of multilayer packaging films, automotive coatings, and biomedical polymer coatings.

Catalysis and surface reactivity

Catalysts operate at the surface, making XPS a natural tool for their characterization. Transition metal catalysts—such as Co(Ni)MoS for hydrotreating, Pt/Pd alloys for automotive emissions control, and Cu-based catalysts for CO_2 reduction—exhibit performance that is highly dependent on oxidation states, ligand environments, surface defects, and adsorbates.

XPS is used to analyze:

- Oxidation states (e.g., Mo^{4+} vs. Mo^{6+})
- Metal–support interactions
- Surface sulfur, oxygen, and nitrogen species
- Catalyst deactivation signatures (coking, oxidation, poisoning)
- Active phases before and after reduction or sulfidation

For example, hydrotreated catalysts are extremely sensitive to air exposure, rapidly oxidizing from the active CoMoS phase to less active oxides. XPS is the only tool capable of unambiguously distinguishing these phases. Combined with REELS and UPS, XPS can also provide insights into surface electronic structure crucial for catalytic activity modeling.

Tribology, lubrication, and wear mechanisms

Wear-resistant surfaces and friction modifiers rely heavily on surface chemistry. Tribofilms formed during sliding contact depend on lubricant additives, load, temperature, and material pairing. XPS is central to evaluating these films, which are often only a few nanometers thick. It is used to:

- Identify anti-wear films (ZDDP-derived phosphates, calcium carbonates)
- Monitor growth and breakdown of friction layers
- Study additive–surface interactions
- Compare high- and low-performing lubricant formulations
- Evaluate corrosion–wear coupling

Depth profiling (GCIS or fs-LA) reveals layered tribofilm structures and their chemical gradients. XPS helps lubricant companies optimize formulations, troubleshoot failures, and validate additive performance under real-world operating conditions.

Corrosion science and protective coatings

Corrosion initiation and mitigation are governed by interfacial chemistry. XPS provides essential insight into:

- Metal oxidation states during early corrosion
- Protective film breakdown
- Composition of inhibitor films
- Conversion-coating chemistry
- Oxide and hydroxide ratios
- Chloride-induced damage

For aluminum alloys, XPS can differentiate between Al_2O_3, $Al(OH)_3$, and Al-substituted oxides. For steels, Fe^{2+} and Fe^{3+} oxide ratios reveal corrosion pathways. Chromate-free corrosion inhibitors and conversion coatings can be evaluated by studying cation and anion incorporation and transformation during exposure.

Optical and photonic materials

Thin-film optics require precise control over multilayer stacks consisting of oxides, nitrides, fluorides, and metals. XPS helps characterize:

- Reflective metal layers
- Anti-reflective coatings
- UV-protective films
- Optical adhesives and epoxies
- Dielectric mirrors and interfering stacks
- Chemical uniformity is essential to achieving consistent optical performance. Depth profiling reveals diffusion, intermixing, and oxygen migration in multilayer structures.

2D materials and nanomaterials

The rise of 2D materials such as graphene, MoS_2, WS_2, h-BN, and MXenes has created strong demand for multi-technique XPS workflows. XPS can measure layer thickness, identify doping, and identify chemical changes. Raman integration helps determine monolayer vs. multilayer domains, while SEM correlation reveals growth morphology. XPS also enables analysis of functionalized graphene, doped 2D semiconductors, and surface termination in MXenes (e.g., –O, –F, –OH groups).

Aerospace and automotive materials

XPS supports the development of advanced coatings, adhesives, composites, and corrosion-resistant alloys. It characterizes:

- Thermal barrier coatings
- Ceramic matrix composites
- Carbon fiber surface treatments
- Adhesion promoters in bonded structures
- Protective inorganic/organic hybrid coatings

In automotive coatings, fs-LA depth profiling enables rapid multilayer characterization (primer, base, clearcoat), supporting color-match studies, finish evaluation, and paint failure diagnostics.

Biomaterials and medical devices

Biocompatibility depends heavily on surface chemistry. XPS helps evaluate:

- Protein adsorption
- Polymer surface functionalization
- Sterilization effects (e-beam, gamma, plasma)
- Passivation layers on implants
- Drug-delivery coatings
- Hydrogel surface chemistry

Many biomaterials are soft or chemically sensitive, making GCIS essential for maintaining chemical fidelity during depth profiling.

Environmental science and surface contaminants

Environmental and industrial contamination studies also rely on XPS to identify:

- Atmospheric oxidation
- Particulate matter on surfaces
- Residues on manufacturing components
- Inorganic scaling and mineral deposits
- Thin organic layers or biofilms

XPS is often used in forensic surface analysis, where identifying trace contaminants is crucial for liability, warranty, or regulatory investigations.

Summary

Across all industries—from semiconductors and batteries to biomaterials and tribology—XPS plays a central role in understanding surface chemistry. The versatility of XPS, combined with depth profiling and complementary techniques, allows researchers to precisely and confidently solve complex problems. As materials systems become more sophisticated, the importance of surface characterization will only continue to grow.

Chapter 9: Case studies

XPS is used across a wide range of application areas. In this chapter, four examples will be discussed to show how the technique is applied to real-world problems.

Oxidation of Co(Ni)MoS catalysts

Cobalt–molybdenum sulfide (CoMoS) catalysts play an essential role in hydrodesulfurization (HDS), a refinery process that removes sulfur from petroleum feedstocks. These catalysts operate through redox cycles involving Co, Mo, and S species on the surface. Their activity depends critically on maintaining specific sulfided phases during operation. However, catalysts are often exposed to air during handling, transport, or storage. Air exposure can partially oxidize sulfide phases, reducing catalytic performance. XPS is uniquely suited for evaluating such oxidation processes because it can directly quantify chemical-state changes for Co, Mo, and S within the top few nanometers—the region most responsible for catalysis.

HDS catalysts are highly sensitive to air oxidation, but the degree of oxidation depends on exposure duration, humidity, temperature, and the presence of stabilizing ligands. In their ideal sulfided state, Co is present primarily as Co^{2+} within a CoMoS-type structure, while Mo is present largely as Mo^{4+} sulfide. Sulfide anions (S^{2-}) dominate the sulfur region. Upon oxidation, Co and Mo can form oxysulfides or full oxides, and sulfur can convert partially to sulfate-like species. Detecting small changes requires high-resolution XPS and precise peak deconvolution. Additionally, catalysts often contain alumina supports whose oxygen signals overlap with Mo and Co oxide signals, requiring careful spectral interpretation.

Two catalyst samples were analyzed: one freshly removed from hexane storage under inert conditions and another exposed to air for three minutes. XPS survey scans confirmed the presence of Al, O, S, Co, Mo, and Ni. High-resolution Co 2p, Mo 3d, and S 2p spectra were then collected to quantify specific species. Peak fitting incorporated multiplet splitting parameters for Co, as well as established reference positions for Mo^{4+}, Mo^{6+}, and S^{2-}.

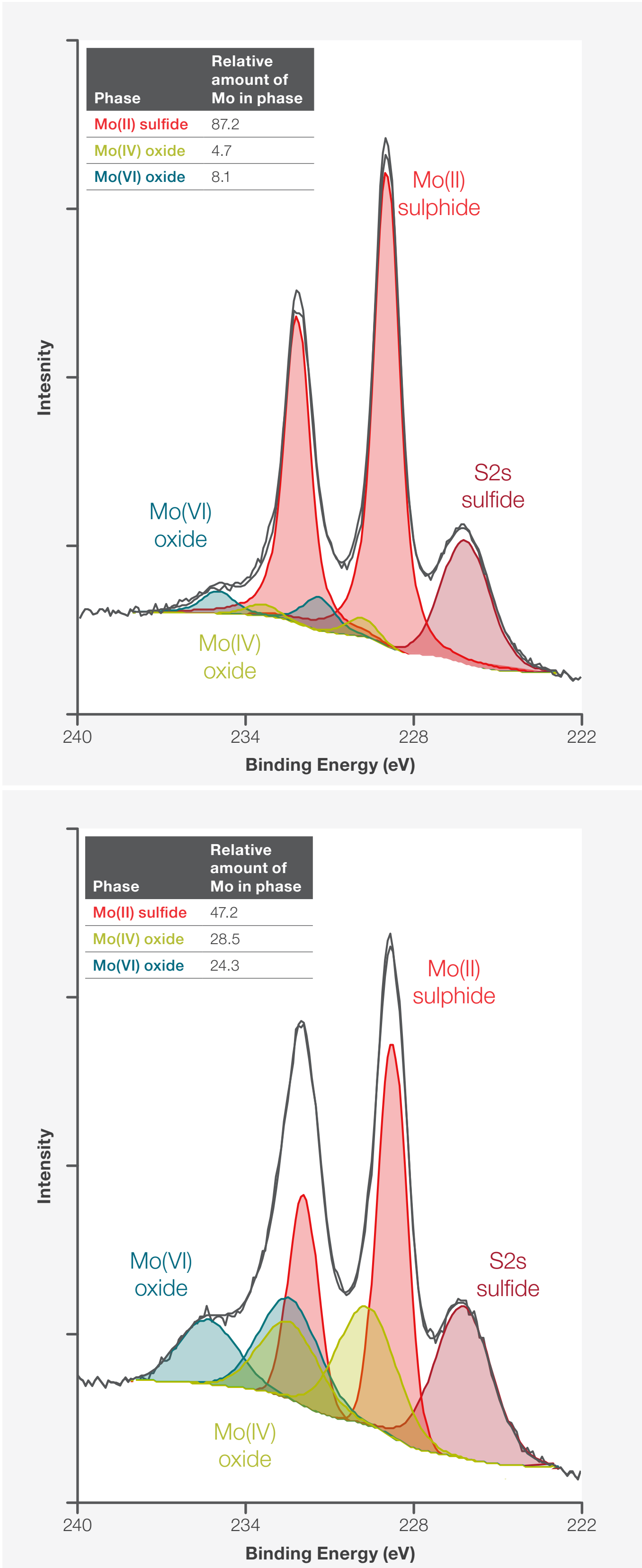


Figure 27: Comparison of the fresh and aged catalysts. Spectra for the $Co2p_{3/2}$ and Mo3d regions are shown

The fresh catalyst exhibited primarily sulfided phases: Co was distributed mainly between Co_9S_8 and CoMoS, while Mo was predominantly Mo^{4+} with minor Mo^{6+} contributions. Sulfur appeared mainly as S^{2-} with trace oxysulfide. After only three minutes of atmospheric exposure, XPS revealed clear oxidation. Co^{2+} oxide signatures increased noticeably, indicating formation of CoO on the surface. The intensity of CoMoS declined, suggesting partial disruption of the active phase. Molybdenum exhibited a significant rise in Mo^{6+} oxide, consistent with rapid aerosol-driven oxidation. Sulfur spectra showed increased contributions from oxidized S species, evidencing transformation toward sulfate-like chemistry.

The sensitivity of CoMoS catalysts to air exposure has major implications for refinery operations and catalyst handling protocols. Even brief exposure leads to measurable oxidation, which can reduce catalytic activity by disrupting the active edge sites where HDS reactions occur. Refineries may need to revise handling procedures, improve inert-atmosphere storage, or incorporate pre-reduction treatments. Catalyst manufacturers benefit from XPS datasets that reveal how formulation changes—such as promoter type, support chemistry, or sulfiding method—affect air stability.

XPS provides rapid, direct, and chemically specific insights into oxidation behavior in CoMoS catalysts. By quantifying shifts in Co, Mo, and S oxidation states, analysts can diagnose catalyst degradation mechanisms, refine handling procedures, and optimize catalyst design for maximum durability and HDS efficiency.

Read the application note

Fs-LA profiling of perovskite solar cells

Halide perovskites represent one of the most promising classes of photovoltaic materials due to their high efficiency, tunable band gaps, low processing temperatures, and solution-phase fabrication.

However, they are notoriously sensitive to ion-beam damage. Even low-energy monatomic sputtering causes catastrophic chemical changes, including instantaneous formation of metallic lead (Pb^0), loss of nitrogen and halides, destruction of organic A-site cations, and broad peak distortions. These artifacts make conventional XPS depth profiling almost impossible for perovskite devices.

To avoid destructive artifacts, fs-LA was used to ablate successive layers of a $FA_{0.95}MA_{0.05}Pb(I_{0.95}Br_{0.05})_3$ perovskite film deposited on a transparent conductive oxide substrate. The laser pulse energy was tuned to gently remove each layer while maintaining phase integrity. Charge neutralization was used during each XPS acquisition step to prevent charging effects common in hybrid organic–inorganic materials.

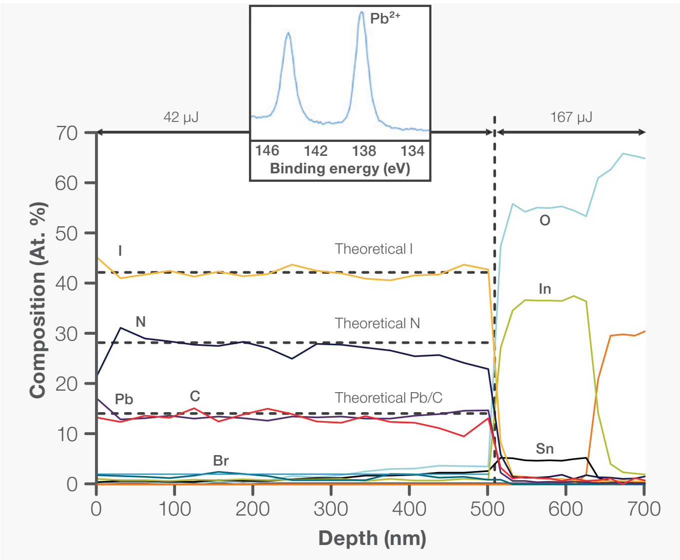


Figure 28: Fs-LA depth profile of the solar cell sample showing relative atomic percentages down to the ITO layer. An example Pb 4f spectrum from a midpoint in the perovskite layer is inset

The fs-LA profile showed stable halide-to-lead ratios (I/Pb and Br/Pb) throughout the absorber layer. Importantly, high-resolution Pb 4f spectra showed no evidence of metallic Pb^0 formation. Nitrogen-containing organic fragments also retained consistent intensities, confirming the preservation of A-site cation chemistry. This stands in stark contrast to Ar^+ sputtering, where Pb^0 formation is visible even after the first sputter cycle, and halide contents drop dramatically.

For perovskite researchers, fs-LA finally provides a method for reliable, chemically meaningful depth profiling. This allows investigation of degradation pathways, interlayer reactions, and processing-induced defects in full stack devices such as encapsulated solar cells and tandem architectures.

Read the application note

Correlative XPS–Raman–SEM analysis of MoS_2 2D materials

Two-dimensional materials such as molybdenum disulfide (MoS_2) have attracted significant interest for use in nanoelectronics, photocatalysis, flexible circuits, and sensing applications. Their properties depend critically on layer thickness, defect concentration, oxidation level, and domain morphology. However, due to the atomic-scale thickness of these materials, no single analytical technique can provide complete insight. For instance, scanning electron microscopy (SEM) reveals morphology but lacks chemical specificity. Raman provides layer-thickness fingerprints but cannot quantify elemental composition. And XPS excels at chemical-state identification but lacks topographical context. The Thermo Scientific CISA (Correlative Imaging and Surface Analysis) Workflow integrates these methods into a seamless platform, allowing analysts to visualize, map, and chemically characterize 2D materials at micrometer scales.

Monolayer MoS_2 grown on SiO_2/Si substrates often forms triangular crystalline domains that vary in lateral size, thickness, and oxidation level. Local defects or partial oxidation can drastically impact electronic properties. Characterizing these nanoscale variations requires correlation across multiple techniques. For example, MoS_2 monolayers exhibit characteristic Raman peak separation between the E_2g and A_1g modes, which increases with layer count. Meanwhile, XPS can distinguish Mo^{4+} in MoS_2 from Mo^{6+} oxides such as MoO_3. SEM reveals domain morphology and continuity, helping analysts correlate crystallographic features with chemical behavior.

The CISA workflow begins with XPS SnapMap, which rapidly collects spectra at each pixel of a defined field of view. For the MoS_2 sample, SnapMap imaging revealed regions with strong Mo 3d and S 2p signals ("bright" domains) and other regions with weak or negligible MoS_2 intensity ("dark" domains). High-resolution XPS spectra from bright domains showed typical MoS_2 signatures (Mo^{4+} with supporting sulfide peaks), while dark regions contained only substrate signals such as Si 2p and O 1s. Some bright domains exhibited additional Mo^{6+} components, indicating localized oxidation.

Raman spectroscopy was then performed on the same positions using the integrated Raman module. Raman spectra taken at bright SnapMap points showed reduced E_2g–A_1g peak spacing, consistent with monolayer or bilayer MoS_2. In contrast, Raman spectra taken from more continuous regions showed wider Raman peak separation, indicating thicker MoS_2 stacking. Raman also revealed defect density through D-band intensity variations.

SEM imaging, carried out next, provided morphological context. Tile maps created using Thermo Scientific Maps Software showed triangular domains of MoS_2 aligned with the areas identified by XPS and Raman. Regions displaying strong MoS_2 XPS signals and monolayer Raman features corresponded to isolated, well-formed triangular flakes. More uniform regions with higher Raman peak spacing corresponded to overlapping or thickened MoS_2 domains.

By combining XPS, Raman, and SEM, analysts obtained a comprehensive understanding of the material. XPS confirmed the presence of both MoS_2 and oxidized Mo species, providing quantitative chemical-state information. Raman analysis linked these chemical variations to layer thickness and structural integrity. SEM established domain geometry and continuity. Together, the data identified regions of pure monolayer MoS_2, thicker multilayer aggregates, and partially oxidized areas. This level of insight would not be achievable with any individual technique alone.

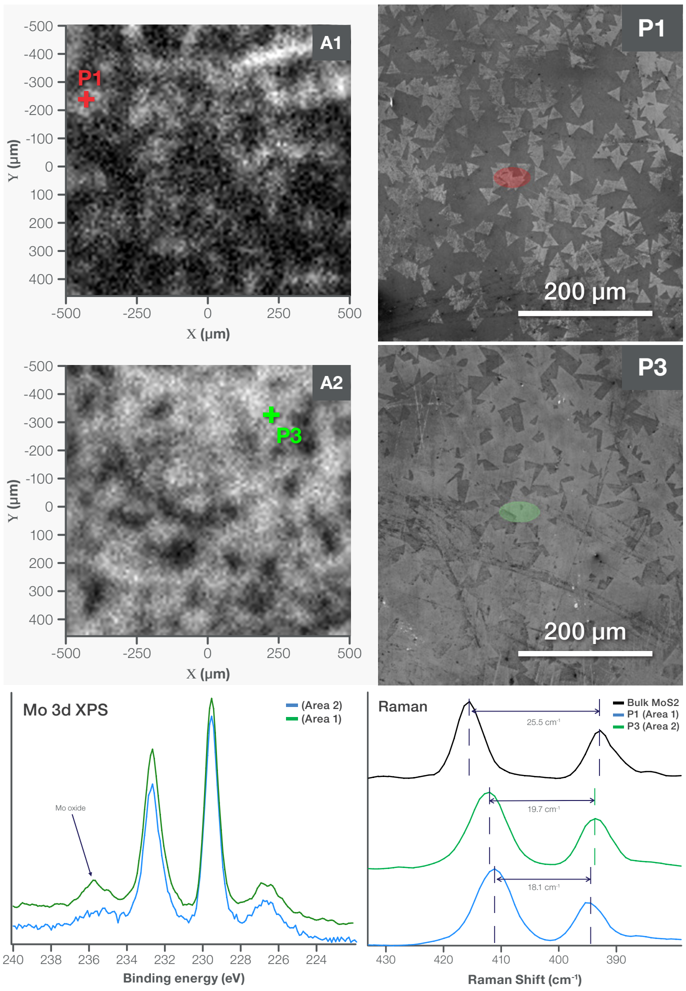


Figure 29: SEM shows a lower density of triangular structures in the area where single-layer MoS2 was identified using the Nexsa G2 System with XPS and Raman.

2D materials researchers benefit significantly from the CISA Workflow. They can directly correlate defects, oxidation hotspots, and thickness variations with device performance. Semiconductor developers can validate growth parameters for wafer-scale 2D material production. Photocatalysis researchers can track how oxidation influences catalytic sites. And device manufacturers gain confidence that materials meet quality and uniformity standards.

This case study demonstrates the value of correlating XPS, Raman, and SEM for full characterization of 2D MoS_2. By leveraging the strengths of each method, the CISA Workflow enables precise mapping of chemical, structural, and morphological features essential for scientific discovery and technology development.

[Read the application note]

Energy-level mapping of OLED polymer PFO

Organic light-emitting diode (OLED) technologies rely on highly engineered organic semiconductors that serve as emissive layers, charge transport layers, and interfacial modifiers. Among these, poly(9,9-dioctylfluorene) (PFO) is a widely studied blue-emitting polymer known for its high brightness, favorable film-forming properties, and compatibility with solution processing. Like other conjugated organic semiconductors, PFO exhibits performance characteristics that depend critically on the alignment of its highest occupied molecular orbital (HOMO) and lowest unoccupied molecular orbital (LUMO) with those of adjacent layers. Accurate measurement of these energy levels is therefore essential for optimizing device efficiency, charge injection, stability, and drive-voltage performance. Traditional electrical characterization methods provide bulk information but cannot reveal the surface electronic structure that governs interfacial charge transfer. A multi-technique surface-analysis workflow combining XPS, UPS, and REELS provides a complete picture of PFO's surface chemistry and electronic energy levels.

Organic semiconductors are often sensitive to ambient exposure and prone to chemical modification, particularly oxidation. In PFO, even slight photooxidation introduces keto-defects that alter emission spectra and reduce device lifetime. Additionally, PFO films typically contain adventitious carbon and surface residues from processing. Analysts must therefore identify and quantify surface impurities, assess chemical states, determine the HOMO onset, measure the ionization potential, and estimate the optical band gap. XPS alone cannot provide all this information. While it is powerful for chemical-state analysis, it cannot characterize unoccupied electronic states or the valence band structure with required sensitivity. UPS provides excellent access to valence electrons but lacks chemical specificity. REELS offers band-gap estimation but requires correlation with other measurements. Only by combining these techniques can a complete electron energy-level diagram be constructed.

The workflow began with XPS survey analysis using a monochromated Al Kα source. The PFO film exhibited a strong carbon signal with minor oxygen contamination (about 0.5 to 1.0 at%), attributed to atmospheric exposure during handling. High-resolution C 1s spectra revealed distinct contributions corresponding to aromatic and aliphatic carbons from the fluorene backbone and side chains. Importantly, the absence of strong C=O peaks indicated minimal surface oxidation, suggesting that the film was well preserved during storage.

Next, UPS measurements using He I excitation (21.2 eV) were performed to determine the HOMO onset and work function. The position of the low-energy cutoff the work function, while the valence band region revealed the onset of the HOMO, enabling determination of absolute HOMO energy. UPS spectra of PFO typically show a clear leading-edge feature corresponding to π-orbital contributions. The ionization potential was calculated by aligning this onset with the low-energy secondary electron cutoff. Unlike metals or inorganic semiconductors, conjugated polymers often require careful background subtraction and signal smoothing to extract precise HOMO energies.

Finally, REELS was used to measure the polymer's band gap. REELS operates by detecting energy losses associated with electron scattering events, including excitations into unoccupied states. PFO exhibits a characteristic π→π* transition and distinct loss edges associated with its electronic band gap. By calculating the onset of inelastic scattering, analysts determined the optical band gap. Combining UPS and REELS allowed direct extraction of the LUMO level from the difference between the band gap and the HOMO energy.

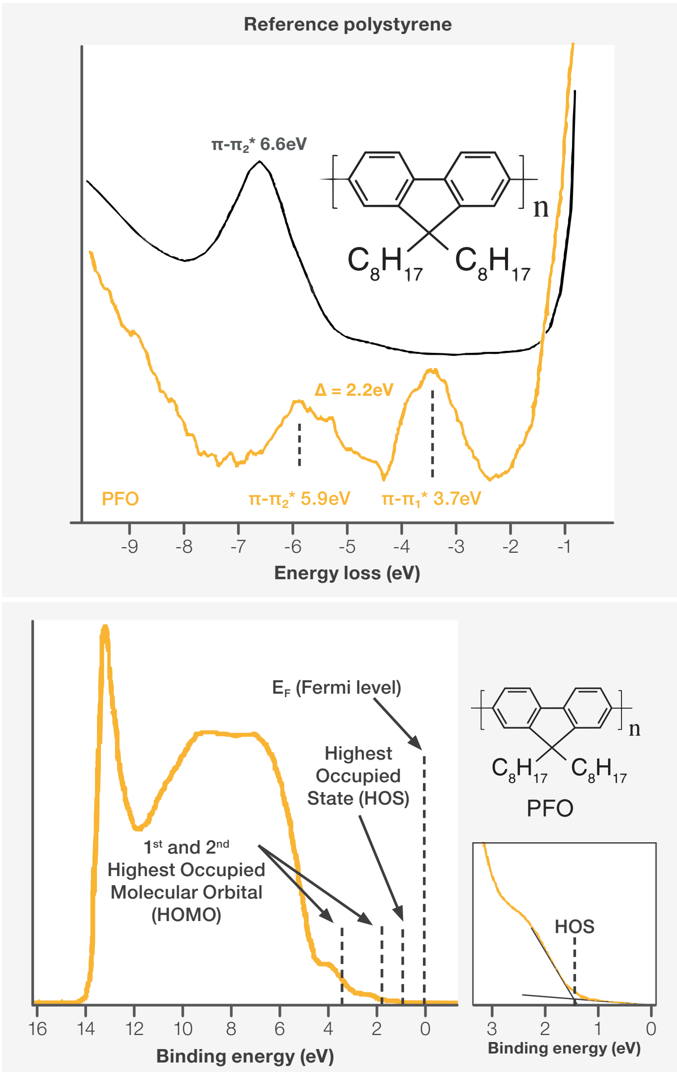


Figure 30: UPS and REELS spectra of the PFO film.

The multi-technique dataset provided rich insight into the PFO film. XPS confirmed a clean, chemically intact polymer with minimal impurities and no evidence of oxidative defect formation. UPS spectra produced a HOMO onset consistent with known values for PFO (about 5.8 eV below vacuum). REELS yielded a band gap of about 3.3 eV, in excellent agreement with optical measurements. Using these values, the LUMO energy level was calculated to be approximately –2.5 eV. The resulting energy-level diagram highlighted the positions of HOMO, LUMO, and the vacuum level, providing a complete electronic picture.

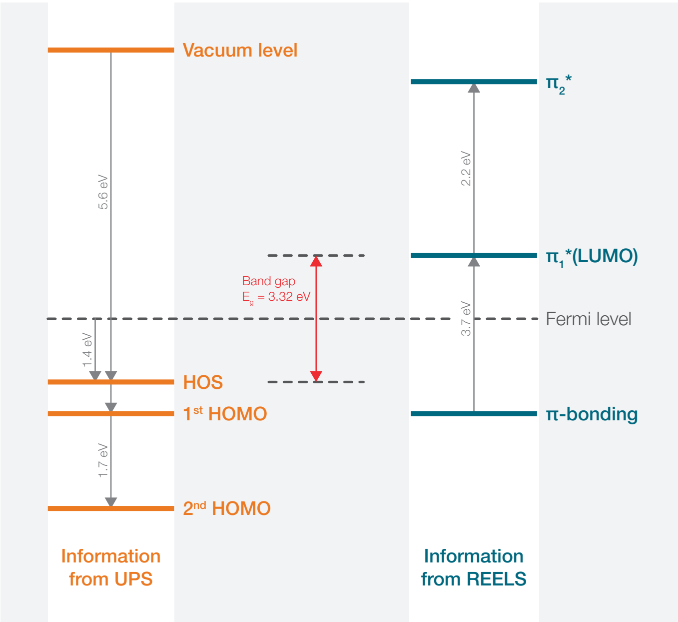


Figure 31: Combining information from REELS and UPS makes it possible to create the energy level diagram of the PFO surface.

This comprehensive surface-electronic characterization is essential for designing OLED stacks with optimal charge balance. Device engineers must match PFO's HOMO with hole injection layers such as PEDOT:PSS or MoO_3 while ensuring the LUMO aligns favorably with electron-transport layers like TPBi or ZnO. Poor alignment leads to charge trapping, recombination inefficiency, device dark-spot formation, and reduced lifetime. The multi-technique workflow also supports evaluation of dopants, blending additives, and interfacial modifications used to tune PFO's emissive properties.

This case study demonstrates the power of combining XPS, UPS, and REELS to fully characterize an OLED polymer's electronic structure. The correlation of surface chemistry with electronic functionality provides actionable insights for OLED material development, device engineering, and performance optimization.

[Read the application note]

Chapter 10: Choosing the right X-ray photoelectron spectroscopy system

Selecting the right XPS system is a strategic decision that shapes the analytical capabilities, productivity, and research trajectory of any laboratory.

XPS instruments differ significantly in spectral resolution, imaging performance, sample-handling workflows, multi-technique integration, and depth-profiling capability. With the introduction of the Thermo Scientific™ Hypulse™ Surface Analysis System, the landscape has expanded beyond traditional sputter-based depth profiling, providing a new avenue for micrometer-scale, damage-free, high-throughput profiling. This chapter provides a detailed, practical guide to the Thermo Scientific K-Alpha, Nexsa G2, ESCALAB QXi, and Hypulse Systems.

Factors to consider when selecting an XPS platform

Every laboratory must consider its specific analytical requirements before choosing a system. These typically revolve around:

- Material types: metals, polymers, ceramics, catalysts, semiconductors, hybrid stacks
- Depth-profiling needs: nanometer vs. micrometer scale, organic vs. inorganic, beam sensitivity
- Multi-technique workflows: need for UPS, Raman, REELS, ISS, AES, SEM
- Sample sensitivity: insulators, air-sensitive materials, easily damaged surfaces
- Imaging requirements: small-feature mapping, chemical-state imaging
- Throughput and automation: QC workflows, multi-user labs, unattended analysis
- Future scalability: ability to expand to multi-technique or deep-profiling capabilities

Understanding these requirements ensures that the selected XPS instrument aligns with long-term needs and minimizes analytical trade-offs.

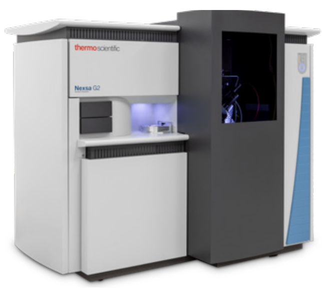
Overview of Thermo Scientific XPS instruments

Thermo Scientific offers four primary XPS instruments:

1. The K-Alpha System is accessible, robust, and suited to routine or multi-user environments
2. The Nexsa G2 System offers multi-technique, correlative analysis for research with diverse surface analysis needs
3. The ESCALAB QXi Microprobe delivers high-resolution imaging and spectroscopy for advanced research environments
4. The Hypulse System is a novel fs-laser XPS platform enabling deep, damage-free, high-throughput profiling

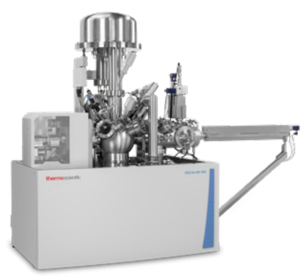
Each platform builds on Thermo Fisher Scientific's proven core technologies—high stability, reproducibility, robust charge compensation, and the Avantage Data System—but serves distinct analytical niches.

K-Alpha System



Nexsa G2 System

Hypulse System



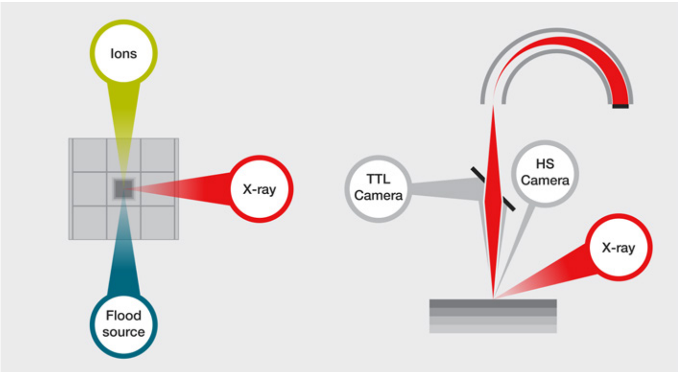
ESCALAB QXi Microprobe

K-Alpha System: Simplicity, robustness, and productivity

The K-Alpha System is designed for environments where reliability, usability, and robust day-to-day performance are essential. Its guided workflows allow even novice users to perform high-quality XPS analysis with minimal training.

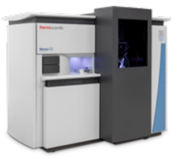
Key features

- Rapid survey and high-resolution scanning
- User-friendly interface and automation
- Multi-camera navigation system
- Self-optimizing charge compensation
- Efficient sample throughput



The K-Alpha System is well suited for routine surface analysis, contamination studies, polymer surface chemistry, oxide states, adhesion failures, and general-purpose XPS.

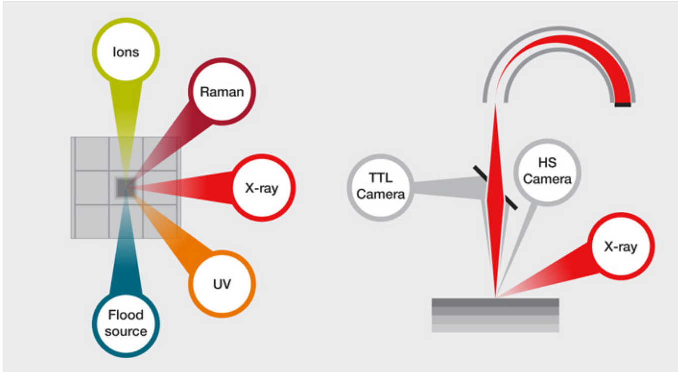
Nexsa G2 System: Multi-technique correlation and analytical flexibility



The Nexsa G2 System is engineered for laboratories that need deep analytical flexibility. It integrates several complementary techniques in a single system.

Key features

- UPS for valence bands, HOMO, work function
- REELS for band-gap determination and hydrogen analysis
- ISS for monolayer-level surface termination
- Raman for structural and vibrational information
- GCIS for damage-free sputtering of polymers



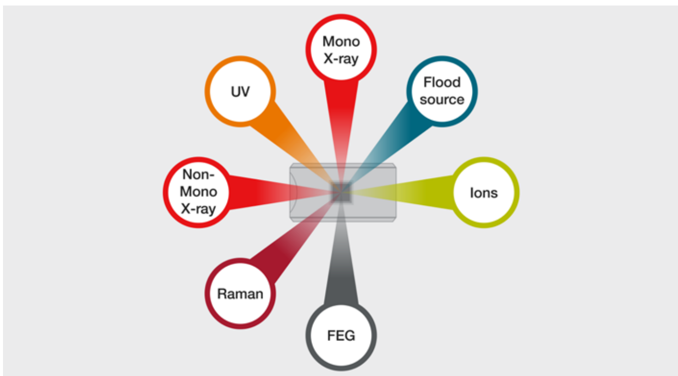
This makes the Nexsa G2 System suited for researchers studying OLEDs, polymers, semiconductors, catalysts, nanomaterials, and hybrid systems where multi-dimensional understanding of surface and interface behavior is essential. It supports high sample throughput, correlative mapping workflows, and automated recipe-driven operation. It is our most versatile system for laboratories that need a broad analytical toolbox.

ESCALAB QXi Microprobe: The high-performance microprobe

The ESCALAB QXi XPS Microprobe is built for advanced research where high spectral resolution, imaging performance, and instrument flexibility are key.

Key features

- High-flux micro-focused dual anode X-ray monochromator
- Quantitative XPS imaging (<3 µm)
- Multi-sample automation
- Dual-mode ion-beam sputtering
- Optional SEM and AES integration



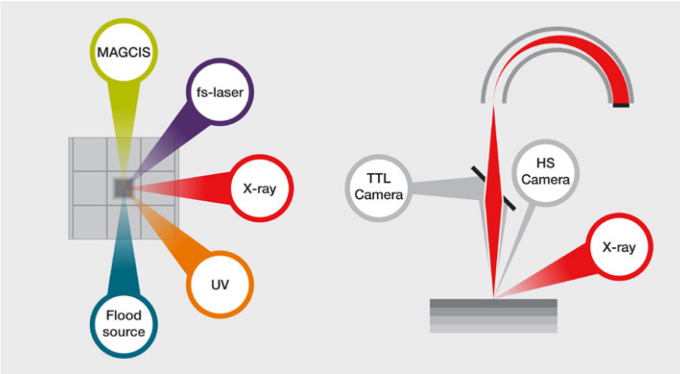
The ESCALAB QXi Microprobe excels in semiconductor research, advanced coatings, nanotechnology, catalysis, and quantitative imaging for laboratories that require maximum system performance and imaging resolution with flexibility for sample preparation.

Hypulse System: The fs-LA depth profiling revolution

The Hypulse Surface Analysis System fundamentally changes what XPS can achieve. It is a novel XPS platform equipped with femtosecond laser ablation (fs-LA).

Key features

- Micrometer-scale depth profiling (up to tens of micrometers)
- Chemical-state preservation even for beam-sensitive materials
- Fast profiling of thick coatings or polymers
- True-to-depth stoichiometry without preferential sputtering
- Rapid cycle times for thick, multi-layer samples



The Hypulse System is particularly useful for:

- Perovskite solar cells for maintaining halide chemistry during deep profiling
- Polymer and paint multilayers for rapid, damage-free profiling through tens of micrometers
- Tribological coatings for analyzing MoS_2:Ti systems without sulfur loss
- Battery electrodes for studying buried SEI and CEI structures
- Catalyst analysis for depth-profiling oxide–sulfide transitions
- Any hybrid organic–inorganic device where ion sputtering distorts chemistry

If deep, accurate, and non-destructive depth profiling is essential, the Hypulse System is a clear choice.

Choosing the right depth-profiling method

Different systems support different types of depth profiling:

- K-Alpha System: Monatomic ion sputtering for inorganics only
- Nexsa G2 System: Monatomic and gas cluster ion sputtering for all material types
- ESCALAB QXi Microprobe: Monatomic and gas cluster ion sputtering for all material types
- Hypulse System: Ultimate depth profiling with fs-LA plus monatomic and gas cluster ion source

For organic materials, GCIS or fs-LA is mandatory to preserve chemistry. For thick films, multilayer paints, perovskites, and hybrid stacks, fs-LA is superior to all sputter-based approaches. For nanometer-resolved profiling of inorganic films, monatomic sputtering remains appropriate.

Automation, workflow efficiency, and user experience

Automation is a defining factor for many labs. All Thermo Scientific XPS systems use Avantage Software, which is designed to scale to meet your requirements and support automated workflows and complex data processing.

K-Alpha System

- Highly automated workflows
- Simple operation for non-expert users
- Minimal training overhead
- Push-button calibration

Nexsa G2 System and ESCALAB QXi Microprobe

- Automated multi-point acquisition
- Advanced stage control
- Sample queues and recipes
- Automated processing

Hypulse System

- Automated fs-LA profiling sequences
- Integrated sputter and laser workflows
- High-throughput analysis of thick films

Summary and recommendations

Selecting the correct XPS platform requires understanding analytical needs, depth-profiling requirements, sample diversity, and long-term research goals. The Thermo Scientific XPS portfolio is structured as follows:

- K-Alpha System: Best for routine XPS and multi-user labs
- Nexsa G2 System: Best for multi-technique, correlative workflows
- ESCALAB QXi Microprobe: Best for high-end imaging and research flexibility
- Hypulse System: Best for deep, damage-free, fs-LA depth profiling across challenging materials

The Hypulse System is our most technologically advanced platform for laboratories working with multi-layer structures, beam-sensitive materials, polymers, paints, perovskites, and complex hybrid architectures. When deep profiling is critical, it delivers outstanding performance. For labs focused on high-resolution imaging, advanced multi-technique studies, or semiconductor research, the Nexsa G2 System and ESCALAB QXi Microprobe remain exceptional choices. And the K-Alpha System continues to deliver reliable, accessible XPS capability for routine analysis.

By aligning system selection with scientific and operational needs, laboratories can ensure the chosen platform provides the capabilities required today and the flexibility needed for tomorrow's materials challenges.

Chapter 11: Summary

XPS has become a cornerstone technique in modern materials science, providing researchers and industry professionals with a uniquely powerful means of probing the chemistry of surfaces, interfaces, and engineered thin films.

Across sectors—semiconductors, energy storage, catalysis, tribology, biomedical materials, optical coatings, protective films, advanced polymers, and 2D materials—XPS plays a critical role in ensuring that surfaces function as designed.

This final chapter summarizes the key themes discussed throughout the book and provides a forward-looking view of where XPS technology is heading as material systems become increasingly complex and application demands grow more stringent.

The enduring strength of XPS as a chemical-state technique

XPS remains unmatched in its ability to resolve the chemical states of elements with nanometer-scale surface sensitivity. Whether identifying oxidation states in semiconductor devices, evaluating SEI chemistry in lithium-ion batteries, monitoring polymer surface treatments, or determining ligand environments in catalysts, XPS consistently provides quantitative and actionable data. Unlike bulk spectroscopy techniques, which average signals over large volumes of material, XPS focuses specifically on the surface—the region where most real-world interactions occur.

Integrating complementary techniques dramatically expands insight

While XPS excels at chemical-state identification, modern research increasingly requires a multi-dimensional understanding of materials. Integrated complementary techniques—such as UPS for valence-band mapping, REELS for band gap measurement and hydrogen detection, ISS for topmost-atomic-layer analysis, Raman spectroscopy for vibrational information, and AES/SEM for nanoscale imaging—transform XPS systems into comprehensive analytical platforms. These correlative workflows allow scientists to evaluate the electronic, structural, and chemical landscape of a material from a single integrated system, reducing experimental time while improving interpretive clarity.

Depth profiling: A new era with fs-LA

XPS depth profiling has entered a new era with the introduction of femtosecond laser ablation, as implemented in the Hypulse System. Traditional ion sputtering—using monatomic Ar^+ or gas-cluster ion beams—remains essential for many inorganic and hybrid films. However, beam-sensitive materials such as polymers, perovskites, organic semiconductors, tribofilms, and multi-layer coatings demand a gentler and more rapid approach. Fs-LA enables micrometer-scale profiling without chemical distortion, opening entirely new application domains that were previously inaccessible. This advancement redefines how XPS can be used in manufacturing, research, and large-volume industrial workflows.

Increasing automation and usability

As demand for XPS grows across industries, modern systems emphasize automation, workflow repeatability, and user-friendly software interfaces. Automated charge control, recipe-based acquisition, intelligent peak identification, batch processing, and multi-sample staging have made XPS accessible to users with a wide range of expertise. Instruments like the K-Alpha System, Nexsa G2 System, ESCALAB QXi Microprobe, and Hypulse System integrate digital intelligence that reduces training burden, accelerates throughput, and ensures that data integrity is preserved even in multi-user environments.

Expanding application frontiers

Innovation in materials science continues to push XPS into new frontiers. Key emerging application areas include:

- Solid-state batteries: understanding sulfide and electrolyte chemistry and buried interphases
- Perovskite photovoltaics: monitoring degradation pathways and stabilizing device lifetime
- 2D materials and heterostructures: correlating defect states with device performance
- Biomaterials: identifying surface modifications for improved cellular responses
- Additive manufacturing: characterizing surface oxidation in advanced printed alloys
- Quantum materials: linking surface electronic structure to quantum-device behavior

Each frontier brings unique analytical challenges, and XPS continues to adapt through improved resolution, expanded energy ranges, and multi-modal integration.

The future of XPS

As technological challenges grow more complex, XPS remains an essential part of the materials characterization landscape. The technique's unique ability to resolve surface chemistry, combined with powerful depth profiling and complementary multimodal capabilities, ensures that XPS will continue to drive innovation across fields.

Instruments such as the K-Alpha System, Nexsa G2 System, ESCALAB QXi Microprobe, and Hypulse System provide laboratories with scalable options to meet current and future analytical demands—from routine quality control to advanced research on emerging materials. The continual evolution of XPS technology ensures that scientists and engineers can meet the challenges of next-generation materials and devices with clarity, precision, and confidence.