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A Comparative Review of Surface Characterization Techniques in Nanotechnology: Strengths, Limitations, and the Central Role of XPS

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Abstract

In nanotechnology the character of the surface will play a critical role where material performance is determined by interfacial phenomena. This review assesses the performance of emerging trends in common surface analysis techniques, related to X-ray Photoelectron Spectroscopy (XPS) as this is the most widely used method in both science and engineering for a wide variety of nanomaterials because of its chemical specificity, quantitative accuracy, and relative ease of application. Other conventional approaches are assessed with respect to their merits and weaknesses. For the near-surface layer of a material, XPS provides unequaled insights into its elemental composition and chemical states. However, its use is limited by the need for a high vacuum, the need to neutralize surface charging, and spatial resolution restricted by the size of the smallest area being analysed. Comparative analysis shows the need to integrate XPS with complementary techniques for a complete characterisation. Current developments including cryogenic XPS, machine learning– assisted spectral analysis, and portable platforms suggest the increasing

availability of in-situ and high-throughput studies. This review presents XPS as a major tool in the development of surface characterisation in nanotechnology.

Keywords: X-ray photoelectron spectroscopy (XPS); Surface characterization; Nanotechnology; Secondary ion mass spectrometry (SIMS); Auger electron spectroscopy (AES); Atomic force microscopy (AFM); Cryogenic XPS.

الملخص

في تقنية النانو، تلعب طبيعة السطح دورًا حاسمًا حيث يُحدد أداء المادة من خلال ظواهر السطح البيني. تُقيّم هذه المراجعة أداء الاتجاهات الناشئة في تقنيات تحليل الأسطح الشائعة، والمتعلقة بمطيافية الأشعة السينية الضوئية الإلكترونية (XPS)، حيث تُعدّ هذه الطريقة الأكثر استخدامًا في العلوم والهندسة على حد سواء لمجموعة واسعة من المواد النانوية نظرًا لخصوصيتها الكيميائية ودقتها الكمية وسهولة تطبيقها النسبية. وتُقيّم الطرق التقليدية الأخرى من حيث مزاياها وعيوبها. بالنسبة للطبقة القريبة من سطح المادة، تُوفر مطيافية الأشعة السينية الضوئية الإلكترونية (XPS) رؤىً لا مثيل لها حول تركيبها العنصري وحالاتها الكيميائية. ومع ذلك، فإن استخدامها محدود بسبب الحاجة إلى فراغ عالٍ، والحاجة إلى تحييد الشحنات السطحية، ودقة مكانية محدودة بحجم أصغر منطقة يتم تحليلها. يُظهر التحليل المقارن الحاجة إلى دمج مطيافية الأشعة السينية الضوئية الإلكترونية مع التقنيات التكميلية للحصول على توصيف كامل. تشير التطورات الحالية، بما في ذلك مطيافية الأشعة السينية الضوئية الإلكترونية بالتبريد العميق، والتحليل الطيفي بمساعدة التعلم الآلي، والمنصات المحمولة، إلى تزايد توافر الدراسات الميدانية وعالية الإنتاجية. يقدم هذا الاستعراض XPS كأداة رئيسية في تطوير توصيف السطح في تكنولوجيا النانو.

الكلمات المفتاحية: مطيافية الفوتون الإلكتروني بالأشعة السينية (XPS)؛ توصيف السطح؛ تقنية النانو؛ مطيافية الكتلة الأيونية الثانوية (SIMS)؛ مطيافية الإلكترون أوجيه (AES)؛ مجهر القوة الذرية (AFM)؛ XPS المبرد.

1. Introduction

Nanotechnology refers to the manipulation of materials at atomic and molecular scales, where the surface to volume ratio is such that the properties of the surface exert an enhanced influence on such material properties as conductivity and reactivity. The large surface-area-to-volume ratio characteristic of nanostructures requires precise analysis of the surface to understand and control its electronic, mechanical, and catalytic properties. A reliable and accurate surface description will enable optimisation of material performance, precise monitoring of degradation processes, and modification of interfaces for optimising specific applications.

To address these demands a range of spectroscopic and microscopic techniques has been developed. XPS, can be called Electron Spectroscopy for Chemical Analysis (ESCA), is distinguished by being able to provide accurate quantitative information for the top few nanometers of a material surface concerning elemental composition, oxidation and chemical bonding. Auger Electron Spectroscopy (AES) and Secondary Ion Mass Spectrometry (SIMS) enable enhanced the scope of chemical and depth profiling, while Atomic Force Microscopy (AFM) will provide 3-D surface topography with nanometer resolution. Whereas, Scanning Electron Microscope (SEM) and Transmission Electron Microscope (TEM) can be used to improve the characterisation of form and structure by enhancing spatial resolution. In combination these instruments can produce additional understanding of the nanostructures of complex surfaces.

However, notwithstanding extensive applications, currently most XPS in use is limited by three factors (i) the requirements for an ultra-high vacuum environment, (ii) sensitivity to surface contamination and surface charging, and (iii) relatively

modest lateral resolution. For a complete characterisation of the surface and to overcome these limitations, XPS needs to be coupled with one or more complementary approaches. Recent progress, which includes cryogenic methods for probing hydrated systems (Cryo-XPS) and AI–driven spectral predictive modelling are enabling XPS to be usefully applied to formerly inaccessible areas.

This review presents a review of the application of XPS, as the major technique for surface characterisation, demonstrating its strengths, limitations, and describing emerging trends that will extend its usefulness. The purpose is to describe XPS within the context of analytical nanotechnology, identify where alternative or complementary methods are necessary, and highlight more useful future directions for nanoscale surface analysis.

2. Summary of Surface Characterization Techniques

Techniques of surface analysis are classified as spectroscopic and microscopic. Spectroscopic techniques provide information about chemical composition and bonding, whereas microscopy supplies information about topography and morphology. Cui et al.[1] portray XPS as a method capable for analysing surface properties within approx. 10 nm of the surface, providing essential of surface chemistry through valence band and core level analysis. Andryushchenko et al. [2] report that AES provides elemental composition with a lateral spatial resolution of only a few nanometers and a surface sensitivity suited to conductive materials. Sanada [3] depict SIMS as achieving extremely high elemental detection sensitivity through surface sputtering and secondary ion analysis, although quantification of the results presented a challenge.

Garcia and Tejedor [4] report on the mapping of nanomechanical properties using AFM. They achieved spatial resolution of images of mechanical properties

(indentation nodes) with nanoscale accuracy and high resolution. Senesi and Massaro [5] highlight that AFM can also provide three dimensional representations of surface with sub-nanometer resolution, demonstrating its versatility in imaging plasma-treated films and nanocomposite interfaces. Nene et al. [6] note that SEM provides sample morphology through electron-beam scanning, often complemented with elemental analysis to gain a deeper understanding of sample composition.

Moradifar et al. [7] in regard to electron microscopy have pointed out that scanning transmission electron microscopy (STEM), in particular with in situ and in operando EM, can provide imaging of the crystallographic structure at an atomic scale, making a significant contribution to the perception of structural features in functioning nanomaterials.

The techniques used are selected based on the nature of the sample and the specific surface information sought, enabling the tailoring of the analytical strategies used for nanomaterial characterisation.

3. XPS: Principles and Applications

3.1 Physical Principles and Spectral Interpretation

Cui et al.[1] and Krishna et al. [8] have both emphasized that XPS is primarily a surface-characterisation technique utilizing, as its name suggests, the photoelectric effect. Photon irradiation excites core electrons which can be ejected from the surface and whose kinetic energies can be used to derive their binding energies and hence the chemical state of the surface. The probing depth is limited to approximately 5–10 nm, which is attributed to the short inelastic mean free path of

electrons, and is consistently highlighted as a defining characteristic of XPS surface analysis.

Greczynski et al.[9] when commenting on calibration of the binding energy, emphasised that referring to the carbon C 1 peak is common but can be a source of systematic error if not consistently applied. The authors emphasize the need for standardized, robust, and consistent referencing strategies to improve the accuracy and reproducibility of results across studies. This work provides useful and detailed information on spectral characteristics, including the contribution of peak shape and full width at half maximum (FWHM) to the interpretation of chemical state and determination of oxidation state, further confirming the importance of careful evaluation of these parameters for accurate chemical shift analysis.

3.2 Instrumentation: X-Ray Sources and Configurations

Cui et al. [1] have recently described how typical XPS systems commonly use conventional soft x-ray sources Mg Kα (1253.6 eV) and Al Kα (1486.6 eV). These techniques have already proved suitable for a large number of elements (except hydrogen and helium due to their small cross-sections). XPS can now be used commercially for many materials ranging from ceramics through contaminants and glues to wood. However, Cui et al. [1] also highlighted recent developments involving higherenergy X-ray sources, such as Ag and Cr, together with synchrotron-based setups, which can significantly improve sensitivity and enable non-destructive profiling to a depth of several tens of nanometers. Cui et al. [1] also described the utilisation of angle-resolved XPS (ARXPS), where differences in the photoelectron take-off angle allow the non-destructive examination of compositional gradients and layered interfaces within approximately 10 nm of the surface, providing a

complementary approach to sputter-depth profiling. We note that Baer et al. [10] had also previously emphasized the widespread use of Al Kα and Mg Kα as sources in routine measurements.

3.3 Advanced and Functional Add-ons

Cui et al.[1] describe how modern XPS instruments are increasingly equipped with advanced functionalities that significantly expand their analytical abilities. Of these, Near Ambient Pressure XPS (NAP-XPS) enables spectroscopic characterisation with, e.g., the simultaneous measurement of catalytic processes, in gaseous atmospheres comprising any or a mixture of at least CO, CO2, H2, H2O, O2 and NH3 at pressures ranging from sub-atmospheric to several tens of millibars. These operando investigations allow direct observation of reaction intermediaries without ultra-high vacuum conditions, a major development that makes the study of surface reactions in real-world conditions possible.

The integration of Ion Scattering Spectroscopy (ISS) with XPS (also known as low energy ion scattering (LEIS)) provides highly sensitive insights into the surface by providing compositional analysis at the level of the single atomic layer at the surface of the material. Further, the development of inert transfer systems and high-temperature reactor setups to support quasi in situ XPS measurements, are providing essential information for maintaining the chemical integrity of oxygen-sensitive or pre-activated samples[1].

Collectively, these advancements extend XPS applications beyond conventional UHV-prepared surfaces, making it possible to obtain meaningful data under realistic and reactive environments[1].

3.4 Applications in Nanotechnology

Today XPS is a key analytical technique with multiple, applications in nanotechnology. First, for nanoparticles and thin films, XPS determines oxidation states, identifies surface functional groups, and maps elemental distributions in nanostructured materials, including core–shell particles and surface coatings[11]. Second, in catalysis, XPS is widely employed to identify catalytic active sites and oxidation states, and its power is enhanced when measurements are performed under operando conditions using near-ambientpressure (NAP) XPS [1]. Third, for semiconductor and oxide interfaces, XPS is used to quantify interfacial band alignment, measure valence-band offsets, and resolve chemical layering within heterostructures[8]. Fourth, in polymer functionalisation and biomaterials, X-ray photoelectron spectroscopy enables the analysis of surface composition of functional coatings or biointerfaces, but because most polymers are insulating, effective charge compensation is essential to achieve high-resolution spectra [12].

4. Comparative Evaluation

To determine whether a surface characterisation method is suitable for nanoscale studies a systematic assessment is required. Each technique will have its own unique strengths regarding provision of chemical data, resolution, and sensitivity, but no one method is universally optimal. Thus, assessing XPS with AES, AFM, SEM and SIMS should highlight their complementarity for studies in nanoscience. Cui et al.[1] emphasize that the selection of XPS, AES, AFM, SEM or SIMS will depend on the desired balance between sensitivity, lateral resolution and chemical specificity. XPS can consistently provide quantitative detection sensitivity of ~0.1–

1 at.% for chemical-state information while spatial resolution would generally be limited to the micrometer scale [2]. In contrast, SIMS can detect very low concentrations (e.g., ppm or even ppb of dopants or impurities) with a lateral resolution of tens of nanometers [8]. AES can deliver high-resolution elemental mapping of ~10 nm [[10, 11] while AFM and SEM can correlate nanoscale topography and morphology to provide compositional data[4]. This framework places XPS as the benchmark technique for chemical specificity see Table 1 and Figure 1.

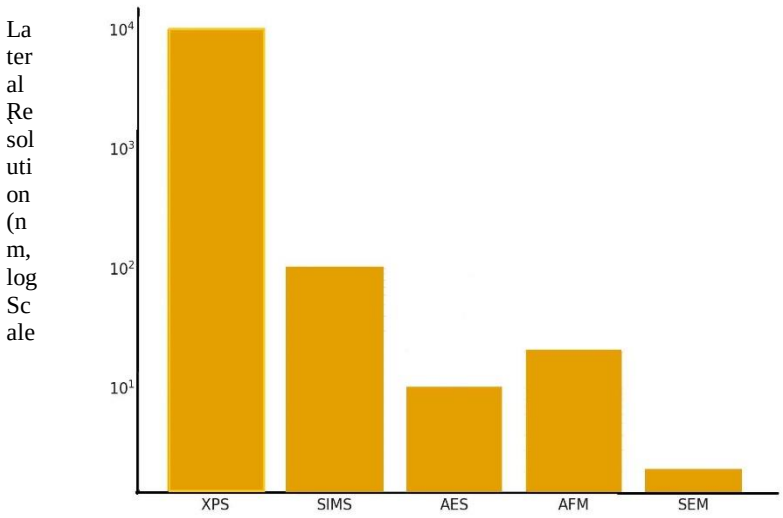

Fig. 1 Comparative Lateral Resolution of Surface Characterisation Technique

Table 1 compares major techniques in terms of sensitivity, resolution, and analytical depth.

Technique	Primary Output	Sampling Depth	Lateral Resolution	Sensitivity / Detection	Notable Strengths
XPS (incl. NAP-XPS)	Elemental + chemical states	~1–10 nm	~10–20 µm (microprobe); down to ~1 µm (imaging)	~0.1–1 at.%	Quantitative, chemical specificity; operando at near-ambient pressure
ToF-SIMS / NanoSIMS	Trace elemental + molecular ions	Top monolayers; depth profiling	~100–200 nm (ToF-SIMS); ~50 nm (NanoSIMS)	ppm–ppb	Ultra-trace sensitivity; isotopic imaging; 3D profiling (GCIB)
AES (SAM)	Elemental (surface)	~1.5–15 nm	~10 nm (field-emission)	~0.1–1 wt.%	High lateral resolution; rapid surface mapping
AFM / AFM-IR	Topography; mechanical; nanoscale IR spectra	Surface contact; AFM-IR ~10–20 nm effective	Sub-nm vertical; few–20 nm lateral	N/A	Non-destructive; nm-scale property mapping
SEM/EDS	Morphology + elemental (X-rays)	~0.5–2 µm interaction volume	~1–2 nm (imaging); mapping limited by volume	≥0.1 wt.%	Fast survey; broad Z range; mapping

As we see from Table 1, XPS provides the most sensitive chemical analysis, SIMS provides the best elemental analysis, while AES can provide nanometerscale imaging of conductive materials. Each method can be used to complement the others for multi-modal analyses.

5. Applications in Industry and Scientific Research

Surface characterization methods, especially XPS, are indispensable in both industry and scientific research due to their ability to determine the chemistry of interphase boundaries with high accuracy. In catalysis, NAP-XPS enables operando monitoring of oxidation states, advancing the design of efficient catalytic systems [1]. With devices for energy storage, XPS can provide insights into the interfaces between electrode/electrolyte as well as redox mechanisms in batteries and fuel cells [3].

In semiconductor technology, interface alignment and thin film chemistry can be explored to improve device reliability[8].

In biomedical and biomaterial applications, XPS supports biocompatibility and functionality studies of implants and, especially, polymeric coatings, enhancing biocompatibility[12].

The following list represents only a small portion of the wide range of XPS applications in both industry and research:

- Catalysis: Using NAP-based XPS to monitor oxidation states in real time, which is essential for understanding active sites.
- Energy Devices: Studying interfaces in batteries and fuel cells using in situ XPS to determine redox behavior.
- Thin Films and Coatings: Studying surface chemistry in microelectronic devices to better control adhesion and passivation.
- Biomedicine: Improved functioning of implants and polymeric materials via XPS, often used with cryo-transfer to avoid contamination.

6. **Advantages and Limitations**

Krishna[8] et al. describe XPS as unique in its ability to precisely identify chemical states, operando operation under near-ambient pressure, and its capacity to probe compositional gradients via ARXPS. Such abilities allow real-time tracking of the chemical composition and states of a semiconductor surface, both of which are essential for the understanding of catalytic activity. Nevertheless, important constraints remain: restricted lateral resolution, charge compensation required when testing samples of insulating materials, and the risk that sputter-based depth profiling

will modify the surface[3]. Recent advances in gas-cluster ion beams and cryogenic transfer have been shown to reduce these problems and provide a fuller analysis at the nanoscale but at the cost of having to combine XPS with another technique.

We summarise the relative advantages of XPS as: (i) Ability to identify precisely the chemical constituents of a surface and their oxidation states, (ii) It can be used for real-time monitoring under conditions where the surface is undergoing reaction when combined with NAP, and (iii) XPS-ARXPS imaging can enhance compositional gradient analyses and surface mapping.

However, certain limitations remain: (i) Lateral resolution of standard XPS is limited to about ~10 μm, though this can be improved by adding imaging systems, (ii) Samples of insulating materials may need charge compensation to produce accurate spectra, and (iii) Sputtering can produce distorted results for the depth profiles of multicomponent materials such as polymers, though this can be significantly reduced by the use of a gas cluster ion beam.

7. Future Trends

Analysis of current trends in surface characterisation techniques in nanotechnology reveal that XPS is moving away from stand-alone use which focuses on chemical composition of surfaces and interfaces, and towards integration with other techniques making multi-dimensional analysis of a given specimen possible. Such combined applications, where the different physical characteristics can be examined in one location during the same test period, removes the necessity of physically moving the specimen from one set of instrumentation to another. Such developments are being combined with automated platforms on which machine learning is being increasingly integrated into, for example, spectral deconvolution, advancing

chemical-state assessment and reproducibility [8]. Simultaneously, these multi-modal platforms are being re-configured to have a smaller environmental footprint.

Integrating AFM and SEM to combine imaging systems can deliver a more effective correlative approach that has been shown to provide enhanced insights at the nanoscale into surfaces and interfaces [14]. Importantly, miniaturized, portable XPS systems are being developed, these should provide analytical capabilities outside the major laboratories.

Cryogenic XPS, where the sample is plunge freezed in liquid N2 (-196 °C) to preserve its original chemical state, and other inert transfer systems, are expanding the application of XPS to biomaterials and volatile/reactive nanostructures.

Collectively, these developments demonstrate the place of XPS as a core, and versatile method for the future of nanoscience.

To summarise, if present trends continue, we expect the following to feature large in the use of XPS. (i) Multi-mode platforms which combine XPS, AFM, EDX and SEM techniques for enhanced depth analysis, (ii) The use AI (machine learning) to improve spacial precision and classification of spectra, (iii) The increasing use of compact, portable XPS systems for in-field analysis, and (iv) Cryogenic XPS and Inert Transfer to preserve sensitive surfaces of soft matter such as biomaterials, and (v) Analytical systems with reduced environmental impact.

8. Conclusion

XPS remains key for nanoscience, due to its unrivalled near-surface, characterisation, identification of chemical states and elemental composition. However, the scope of XPS is being progressively extended to enable investigations

to be made under more realistic and operando conditions by, for example, using machine learning for improved spectral analysis, cryogenic transfer for materials with relatively high vapour pressure and near-ambient-pressure operation to simulate more realistic conditions

Important challenges remain. Key are (i) sputter induced damage, (ii) lateral resolution and (iii) sample charging. However, it is expected that the use of balancing techniques including AES, AFM, SIMS and TEM will help overcome these and ensure a more complete and accurate portrayal of complex systems at the nanoscale.

Combining XPS with multi-instrument, portable platforms with sustainable analytical practices will retain its role as an indispensable technique and make it an evolving and indispensable method for the next generation of nanotechnology research and subsequent industrial applications.

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