

Applied Surface Science

Room 209 B W - Session AS-MoA

Quantitative Surface Analysis I

Moderator: Lyndi Strange, Pacific Northwest National Laboratory

1:30pm **AS-MoA-1 Quantified Photoemission Using Ga K α (9.25 keV) Hard X-Rays Applied to Advanced Materials**, *Ben Spencer, Abdulrhman Alsaedi, Liam Dwyer*, The University of Manchester, UK; *Benjamin Reed, David Cant, Alexander Shard*, National Physical Laboratory, U.K.; *Michael Baker, Alex Walton, Nicholas Lockyer, Wendy Flavell*, The University of Manchester, UK

INVITED

X-ray photoemission spectroscopy (XPS) sampling through the sub-surface towards the bulk requires higher X-ray photon energies generating higher Kinetic Energy photoelectrons. We detail how this is enabled in the Scienta Omicron Hard X-ray Photoelectron Spectrometer (HAXPES) laboratory instrument utilising a 9.25 keV X-ray source (Ga K α MetalJet, Excillum) and EW4000 electron energy analyser [1-6]. Laboratory systems offer some important advantages over synchrotron X-ray sources including easier access, highly reliable source intensities, and the development of reliable quantification methodologies [7]. We demonstrate how HAXPES can extract information significantly further into the surface compared to traditional approaches, with sampling depths up to twenty times the inelastic mean free path enabled by analysis of the inelastic background [2,5], and we detail the metrology developed to ensure accurate quantification across the measurable Binding Energy range [2-6]. 9.25 keV HAXPES enables measurement of higher Binding Energy core levels, such as 1s core levels of first row transition metals (up to Cu 1s at 8.98 keV), and previously unmeasured Auger transitions. We highlight measurements of higher Binding Energy core levels for quantification and chemical state identification applied to energy materials, where deeper core levels offer enhanced relative sensitivities compared to traditional XPS [8], and including the analysis of satellite peak structures as compared to theoretical calculations. As such we demonstrate the capabilities of laboratory HAXPES with a view to future developments and applications to advanced materials research.

- [1] A. Regoutz *et al.*, Rev. Sci. Instrum. **89**, 073105 (2018)
- [2] B. F. Spencer *et al.*, Applied Surface Science **541**, 148635 (2021)
- [3] M. Bouttemy *et al.*, Coatings **11** (6), 702 (2021)
- [4] D. J. H. Cant *et al.*, Surface and Interface Analysis **54** (4), 442-454 (2022)
- [5] B. F. Spencer *et al.*, Faraday Discussions **236**, 311-337 (2022)
- [6] D. J. H. Cant *et al.*, J. Elect. Spect. and Rel. Phen. **264**, 147311 (2023)
- [7] K. Artyushkova *et al.*, J. Vac. Sci. Technol. A **42**, 052801 (2024)
- [8] S. Maniyarasu *et al.*, J. Materials Chemistry A **10** (35), 18206-18217 (2021)

2:00pm **AS-MoA-3 Quantification of Bulk MoS₂ and Characterization of Mo Oxides by Cr Ka-excited HAXPES : Issues and Tentative Improvements**, *Olivier Renault, Nicolas Gauthier, Mario Ghostine, Roman Charvier*, CEA-Leti, France; *Bruno Domenichini*, Université Bourgogne-France Comté, France

In this presentation, we will present HAXPES results related to Mo compounds of practical interest in device technology. Spectra of bulk MoS₂ were acquired with a Cr K α source following a careful measurement protocol [1]. The latter includes checking for instrument stability, use of pure-element relative sensitivity factors and an improved determination of the spectrometer transmission function. The results indicate large errors in the determination of the stoichiometry depending on the selected core-lines. Preliminary results obtained on pure metallic, homogeneous Mo surfaces show potential improvements if the measured intensity takes into account inelastic losses which are important at high kinetic energies. A second part will be dedicated to sub-stoichiometric Mo oxide films with a particular attention to beam effects, the impact on the determination of the stoichiometry along with suitable protocols for accessing deeply buried interfaces.

In a second topic, we will address the case of sub-stoichiometric Mo oxide films which are photochromic materials and are prone to alterations upon X-ray irradiation, with consequences on the determination of the Mo/S ratio. We will comment on the origins of these alterations and propose solutions towards a reliable analysis, before presenting results regarding deeply buried interfaces.

- [1] A. Boyer, N. Gauthier, O. Renault, Surf. Sci. Spectra **31**, 024018 (2024).

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- [2] R. Charvier, O. Renault, B. Domenichini *et al.*, Appl. Surf. Sci. 2025 (under review).

Acknowledgment : This work, carried out on the Platform for Nanocharacterisation (PFNC), was supported by the "Recherche Technologique de Base" and "France 2030 - ANR-22-PEEL-0014" programs of the French National Research Agency (ANR).

2:15pm **AS-MoA-4 Routine, Integrated Ag-La Haxpes – Acquisition and Quantification**, *Miroslav Michlíček*, Thermo Fisher Scientific, CZ, Czechia; *Keith McCourt, Paul Mack*, Thermo Fisher Scientific, UK

Hard X-ray photoelectron spectroscopy (HAXPES) has significantly evolved since its inception and early use in synchrotron facilities, and is nowadays readily available in lab-based instruments. Especially when used in tandem with standard Al-K α XPS spectroscopy, the method offers deep insight into the electronic structure of materials and can help to resolve some ambiguities inherent to standard XPS. Despite its advantages, HAXPES has faced several challenges, including accurate peak quantification.

This work addresses the steps necessary to overcome these quantification challenges for Ag-La HAXPES data. We first discuss critical experimental considerations for ensuring accurate and reproducible measurements, namely the reproducibility of the monochromator crystal. We then examine the three pillars supporting the peak area quantification: relative sensitivity factors, escape depth correction and instrument transmission function. In particular, we detail our approach to estimating the instrument transmission function. The robust, factory-default estimate was acquired by global fitting of noble metals (Au, Ag, Cu) spectral datasets collected from multiple instruments. Finally, we present the combined quantification accuracy on several pure elements (ie. from different transitions) and binary compounds with an expected stoichiometry. In conclusion, the integrated automated anode switching coupled with data quantification software, represents a significant step forward in making routine Ag-La HAXPES acquisition practical and reliable.

2:30pm **AS-MoA-5 Non-Destructive Depth Profiling by Variable Energy PARXPS (VE-PARXPS)**, *Paul Dietrich, Francesca Mirabella, Martin Breitschaft, Andreas Thißen*, SPECS Surface Nano Analysis GmbH, Germany
Chemical analysis of contemporary materials frequently entails the characterization of surface and bulk compositions. EnviroMETROS LAB and FAB provide a monochromated small spot X-ray source with up to three different photon energies giving different surface and bulk sensitivities due to the variation in kinetic energy of the emitted photoelectrons. Moreover, the angle-resolving, wide-angle electron analyzer AEOLOS 150 AD-CMOS together with the software enables high sensitivity and high resolution analysis with direct non-destructive depth profiling.

Variable Energy X-ray photoelectron spectroscopy (VE-XPS) employs tunable X-ray sources, such as synchrotrons or laboratory-based monochromators, to adjust the photon energy. This adjustment enables depth profiling and enhanced sensitivity to different core-levels. By tuning the photon energy, the kinetic energy of emitted photoelectrons can be controlled, effectively changing the inelastic mean free path (IMFP). This capability allows for analysis of surface, subsurface, and bulk regions.

In parallel angle-resolved XPS (PARXPS) photoelectrons at various emission angles are collected simultaneously using a wide-angle analyzer. This method facilitates the concurrent acquisition of spectra from multiple angles, thereby enhancing the efficacy and precision of probing depth and electronic structure. PARXPS allows to create concentrational depth profiles from data that was taken for different photoelectron emission angles. A parallel spectra collection of individual angle channels is possible with the AEOLOS 150 AD-CMOS analyzer, developed especially for performing PARXPS measurements.

VE-XPS allows for the adjustment of probing and information depth, while PARXPS provides angular-resolved data, enabling non-destructive depth profiling with enhanced accuracy. By varying the energy and emission angle, more detailed information can be extracted, which is crucial for materials such as layer stacks and semiconductors. The combination of tunable energy with angular resolution enables the differentiation between surface, interface, and bulk states, thereby facilitating the acquisition of comprehensive structural information. The capacity to adjust depth sensitivity minimizes the occurrence of misinterpretation arising from charging effects or contributions from deeper layers, ensuring the reliability and precision of the measurement process. The combination of VE-XPS and PARXPS, allows to obtain a much richer dataset, enabling a more precise and comprehensive understanding of material surfaces and interfaces

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2:45pm **AS-MoA-6 Efficient, Non-Destructive Characterization of Buried Layer Chemistry - Introducing the Next-Generation High-Intensity Deep HAXPES Instrument**, *Marcus Lundwall¹, Tamara Sloboda, Dick Allansson*, Scienta Omicron, Sweden; *Daniel Beaton*, Scienta Omicron; *Susanna Eriksson*, Scienta Omicron, Sweden

Buried interfaces in electronic devices—such as batteries, solar cells, and transistors—play a critical role in device performance. Reliable instrumentation for characterizing these interfaces is therefore essential for material development. While X-ray photoelectron spectroscopy (XPS) is a powerful technique for probing chemical states at surfaces, it cannot directly access buried interfaces without sputtering or etching, which can introduce artifacts. Hard X-ray photoelectron spectroscopy (HAXPES) offers a solution by providing increased information depth, enabling non-destructive characterization of layered structures. As a result, HAXPES has become an invaluable tool in materials research, particularly for applications involving semiconductor thin films, polymer materials, metal surfaces, and coatings.

This presentation will provide an overview of HAXPES applications, with a focus on non-destructive studies of buried interfaces in electronic devices, operando studies and oxidation processes. Additionally, we will introduce recent advancements in lab-based HAXPES systems. The latest generation features a Ga K α 9.25 keV MetalJet microfocus HAXPES X-ray source, alongside improvements in electron detection efficiency. Compared to its predecessor, the new system offers a several-fold increase in acquisition speed while maintaining a proven probing depth of up to 50 nm. To fully leverage these enhancements, the sample handling and navigation system has been upgraded with a larger mounting area, enabling automated acquisition across a higher number of samples and measurement points. These innovations make high-end, high-energy Deep HAXPES a viable high-throughput technique beyond synchrotron facilities.

3:00pm **AS-MoA-7 Quantification of the Carbon Hybridization State via C 1s XPS: Possibilities and Limitations**, *Filippo Mangolini*, The University of Texas at Austin; *Michael Walter*, Fraunhofer Institute for Mechanics of Materials IWM, Germany; *J. Brandon McClimon*, *Robert W. Carpick*, University of Pennsylvania; *Michael Moseler*, Fraunhofer Institute for Mechanics of Materials IWM, Germany

The determination of the carbon hybridization state in carbon-based materials is of pivotal importance owing to the strong dependence of the physical, chemical, and mechanical properties of these materials on the carbon bonding configuration. Despite the relevance of published studies, the quantification of the hybridization state of carbon atoms by X-ray photoelectron spectroscopy (XPS) is still a surface-analytical problem owing to the challenges associated with the unambiguous identification of the characteristic binding energy values for sp²- and sp³-bonded carbon. Here, we performed density functional theory (DFT) calculations to determine the binding energy values of C 1s core electrons on the absolute energy scale for model structures of a class of carbon-based materials, namely amorphous carbon (a-C). In the case of hydrogen-free a-C, the DFT calculations indicate that the average C 1s binding energy for sp³-bonded carbon atoms is approximately 1 eV higher than the average binding energy of sp²-hybridized carbon atoms. Notably, the introduction of hydrogen in the a-C network reduces the distance between the characteristic signals of sp³- and sp²-bonded carbon due to the increased ability to screen the core hole by neighboring hydrogen atoms as compared to carbon atoms. These results, which are in qualitative agreement with experimental XPS spectra acquired on a-C materials containing different hydrogen content, highlight that the characterization of the carbon hybridization state on the basis of fitting C 1s XPS spectra with two synthetic components, one assigned to sp²-bonded carbon and one assigned to sp³-bonded carbon, is not physically accurate in the case of a-C specimens containing hydrogen. This work can assist surface scientists in the use of XPS for the accurate characterization of carbon-based material.

3:15pm **AS-MoA-8 Correlative XPS and EBSD with Cluster Ion Etching for Enhanced Surface Preparation**, *Simon Bacon*, *Helen Oppong-Mensah*, *Robin Simpson*, *Paul Mack*, *Tim Nunnery*, Thermo Fisher Scientific, UK

Electron Backscatter Diffraction (EBSD) is a critical technique in materials science, providing detailed crystallographic information that is essential for understanding the microstructure and properties of materials. EBSD is widely used to analyse grain size, orientation, and phase identification, which are fundamental for optimising material properties and performance in various applications. The high spatial resolution and sensitivity of EBSD

make it an invaluable tool for characterising such features in metals, ceramics, semiconductors, and other advanced materials.

However, the preparation of samples for EBSD presents several challenges. Surface contamination and damage can significantly affect the quality and accuracy of EBSD data. Traditional mechanical polishing methods often introduce surface artifacts that obscure the true microstructure of the sample. Ensuring the precise alignment and correlation of data between different analytical techniques also poses difficulties and can hinder any characterisation efforts.

To address these challenges, we demonstrate a solution that integrates a correlative XPS-SEM workflow (CISA) with cluster ion beam etch sample preparation. XPS offers detailed surface chemical analysis in the form of elemental composition and chemical state information. Cluster etching provides a controlled method for cleaning and defining surfaces, which helps to minimise contamination and damage. The CISA workflow ensures accurate alignment and correlation of data between XPS and EBSD analyses. By utilising specialised sample holders and advanced software alignment routines, we can achieve precise co-location of analytical data, which improves reliability and reproducibility of the results.

In summary, the combination of XPS and EBSD is a powerful one, offering comprehensive chemical and microstructural characterisation of materials. The use of both cluster ion beam etching as an integrated sample preparation method and the CISA workflow for precise feature location and alignment, ensures high quality, reliable cross-technique data to facilitate advancements in materials science and engineering.

3:30pm **AS-MoA-9 Enhanced Depth Profiling of Polymer Multi-Layer Samples Using Combined Femtosecond Laser Ablation and Cluster Ion Beams in XPS**, *Robin Simpson²*, Thermo Fisher Scientific, UK; *Charlie Chandler*, *Mark Baker*, University of Surrey, UK; *Tim Nunnery*, Thermo Fisher Scientific, UK

X-ray Photoelectron Spectroscopy (XPS) is a critical technique for surface and interface analysis, providing elemental and chemical state information. Depth profiling of polymer multi-layer samples, however, presents significant challenges due to the potential for sample damage and chemical modification during the sputtering process. To address these challenges, we propose a novel approach that integrates femtosecond laser ablation with cluster ion beam sputtering for XPS depth profiling.

Femtosecond laser ablation offers precise material removal with minimal thermal damage, making it ideal for initial bulk material removal. Cluster ion beams, known for their gentle sputtering capabilities, are then employed for fine-scale depth profiling, reducing the risk of chemical alteration and maintaining the integrity of the polymer layers.

In this study, we systematically investigate the combined use of femtosecond laser ablation and cluster ion beams on polymer multi-layer samples. We calibrate the laser and ion beam parameters to optimize ablation rates, minimize surface roughness, and ensure accurate depth resolution. Our results demonstrate significant improvements in depth profiling accuracy and chemical specificity, allowing for detailed characterization of each polymer layer.

The hybrid approach enhances the depth profiling capability of XPS, providing a powerful tool for analyzing complex polymer structures. The integration of femtosecond laser ablation and cluster ion beams enables high-resolution depth profiling, offering new insights into the composition and chemical states of multi-layer polymer samples.

Our findings highlight the potential of this combined technique to advance the field of polymer analysis and other applications requiring precise depth profiling. Future work will focus on further optimization of the parameters and extending the technique to a wider range of materials.

4:00pm **AS-MoA-11 Contribution of Imaging X-Ray Photoelectron Spectroscopy (Xps) to Characterize Spatial Chemical Distribution of Zn Nanoparticles in a Protective SiO₂ Matrix for Luminescence Application**, *Vincent Fernandez*, IMN-CNRS, France; *Michel Féron*, LCC-CNRS, France; *Neal Fairley*, CasaXPS, France; *Myrtil Kahn*, LCC-CNRS, France; *Richard Clergereaux*, LPCE-CNRS, France; *Mireille Richard-Plouet*, IMN-CNRS, France

Due to their potential applications, white-light emitting materials have attracted extensive research interest. The covered fields of applications include information display, fluorescent sensors, and solid-state lighting. Among them zinc oxide with its photoluminescence characterized by two main bands: one related to excitons below 400 nm and a broad emission in the visible range could enable to obtain white light emission. In order to

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protect nanocrystalline phosphors such as ZnO nanoparticles (NPs) and keep their emission performance, a strategy is to embed them in transparent matrix. Among the possible oxides fulfilling the specification, silica was identified as a suitable candidate thanks to its chemical stability and non-toxicity.

Nanocomposite (NC) thin films are prepared by a hybrid process [1] combining injection, at low pressure, of colloidal ZnO solution, forming an aerosol, in a Plasma Enhanced Chemical Vapor Deposition (PECVD) reactor. Thus innovative thin films consisting of ZnO nanoparticles and SiO₂ using this hybrid process were deposited by injection of nanoparticles stabilized by organic ligands and dispersed in 20%vol pentane-80%vol HMDSO solution in an Ar plasma. These films were characterized by XPS and by parallel Imaging energy scan XPS [2] on a Kratos Nova optimized instrument [3]. Data were analyzed using CASAXPS 2.3.27 [4]. Zn 2p, O1s, C1s and Si 2p Core levels were measured in parallel Imaging energy scan XPS with an energy resolution of 1.12 ± 0.05 eV measured on Ag 3d5/2. Data analyses show anti-correlation between the atomic concentration of Zn, associated to ZnO nanoparticles, and both the atomic percentages of Si, associated to SiO₂ Fig 1 and the atomic concentration of Carbon fig 2. Moreover the energy resolution is good enough to map separately the amount the O bound to Zn, at 530.8 eV and the O linked to Si, at 532.6 eV.

These results highlight the possibilities offered by XPS imaging, allowing us to shed light on mechanisms involved during drying of the aerosol droplets at the substrate surface.

Acknowledgments

“Measurements were performed using the IMN’s characterisation platform, PLASSMAT, Nantes, France.”

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- [3] V. Fernandez, et al., *Surface and Interface Analysis*, vol. n/a, no. n/a, Mar. 2024, doi: 10.1002/sia.7296.
- [4] N. Fairley et al., *Applied Surface Science Advances*, vol. 5, p. 100112, Sep. 2021, doi: 10.1016/j.apsadv.2021.100112.

4:15pm **AS-MoA-12 Uncertainties in XPS Elemental Quantification**, **Alexander Shard**, Benjamin Reed, David Cant, National Physical Laboratory, UK

X-ray photoelectron spectroscopy (XPS) is routinely employed to measure surface compositions. The standard approach to quantification treats the surface region as if it is homogeneous and applies sensitivity factors to measured peak intensities to calculate the ‘equivalent-homogeneous composition’ of the sample expressed as a mole fraction, which is usually converted to atomic percent. In this talk, we briefly summarise the main contributions to uncertainty in XPS composition measurements and provide mathematical expressions through which the uncertainty in the measured composition can be estimated and reported. Contributions to the measurement uncertainty from peak area measurement, detector linearity, sensitivity factors, spectrometer transmission and analytical practice are outlined and combined. We show that the statistical uncertainty in peak area measurement usually contributes negligibly to the accuracy of the measurement, but is the main factor that determines the precision. The best practices to improve the precision and accuracy of standard ‘equivalent-homogeneous composition’ XPS quantification are outlined.

4:30pm **AS-MoA-13 Revisiting Detector Linearity and Deadtime Correction for Photoelectron Spectrometers**, **Benjamin Reed**, **Alexander Shard**, National Physical Laboratory, U.K.

In x-ray photoelectron spectroscopy (XPS) data, the integrated area under a core level peak is proportional to the atomic fraction of that element in the sample. Therefore, confidence in the intensity scale of a photoelectron spectrometer is vital for quantitative analysis. Sample dependent factors aside, the XPS analyst must calibrate for the relative intensity response (or transmission function) of their analyzer, which is largely a result of the electrostatic lens column and is unique for each instrument. However, even before a spectrum is acquired, there must be confidence that the measured count rate of the analyzer’s detection system is correct. At high count rates, the photoelectron detector may exhibit non-linearity that significantly affects measured count rates, and therefore the peak intensities from which atomic fractions are calculated. At even higher count rates, detector saturation may occur.

So, although it is tempting to conduct XPS measurements with increased count rates (e.g. by increasing x-ray emission and using high-throughput lens modes / collimation settings) with the intent to improve signal-to-noise and reduce experiment times, this approach is stymied by the limits of the detection system itself. Awareness of this limitation of photoelectron detectors may not be universally known throughout the XPS users’ community; in a recent VAMAS interlaboratory study on XPS intensity calibration, a number of datasets on sputter-cleaned gold were submitted that exhibited evidence of detector non-linearity.

With the international standard on detector linearity (ISO 12170) due for systematic review in 2026, it seems appropriate to revisit these concepts for the benefit of the surface analysis community. We present a method for assessing the linearity and deadtime correction of photoelectron detectors based on the *spectrum ratio* method from ISO 12170 with improved data selection criteria for multi-channel detectors. We also discuss the effect of non-linearity and incorrect deadtime correction on XPS quantification.

4:45pm **AS-MoA-14 Beyond the Standard Elemental Analysis of Surfaces - What more can be done with XPS?**, **Donald Baer**, Pacific Northwest National Laboratory; **Merve Taner Camci**, Turkish Energy, Nuclear and Mineral Research Agency, Turkey; **David Cant**, National Physical Laboratory, UK; **Scott Chambers**, Pacific Northwest National Laboratory; **Hagai Cohen**, Weizmann Institute of Science, Israel; **Pinar Aydogan Gokturk**, Koc University, Turkey; **David J. Morgan**, Cardiff University and HarwellXPS, UK; **Andrey Shchukarev**, Umeå University, Sweden; **Peter Sherwood**, University of Washington; **Sven Tougaard**, University of Southern Denmark; **Sefik Suzer**, Bilkent University, Turkey; **John Watts**, University of Surrey, UK

Because of the importance of surfaces and interfaces in many scientific and technological areas, the use of XPS in publications has been growing exponentially. Although XPS is being used to obtain useful information about the surface composition of samples, much more information about materials and their properties can be extracted from XPS data than commonly obtained. This presentation describes some of the areas where alternative analysis methods or experimental design can obtain information about the near surface region of a sample, often information not available in other ways. Experienced XPS analysts are familiar with many of these methods, but they may not be known to new or casual XPS users and sometimes they have not been used because of an inappropriately assumed complexity. Information available includes, optical, electronic, and electrical properties, nanostructure, expanded chemical information and enhanced analysis biological materials and solid/liquid interfaces. Many of these analyses can be conducted on standard laboratory XPS systems, with either no or relatively minor system alterations. Topics discussed include i) Considerations beyond the “traditional” uniform surface layer composition calculation to obtain nanostructure of the near surface region, ii) using the Auger parameter to determine a sample property, iii) use of the D parameter to identify sp² and sp³ carbon information, iv) extracting phase and enhanced chemical information from the XPS valence band, v) using cryocooling to examine the solid/liquid interface and expand range of natural and biological samples that can be analyzed and minimize damage, and vi) Using electrical potential effects on XPS signals to extract chemically resolved electrical measurements including band alignment, electrical property information, double layer formation and charge dynamics.

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