**Royce Industrial Showcase // Applications of Hard X-ray Photoelectron Spectroscopy**

Henry Royce Institute, Scienta Omicron GmbH, National Physical Laboratory, The University of Manchester

**Wednesday 23rd October 2024**

[Core Technology Facility](https://www.conference.manchester.ac.uk/venues/search/details/?property=63), The University of Manchester, Manchester M13 9PL

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| *10:00-10:30* | **Registration** |
| *10:30-10:45* | **Prof. David Knowles, Chief Executive Officer, Henry Royce Institute**  *Welcome and Introduction to Royce* |
| *10:45-11:30* | **Dr. Alexander G. Shard, National Physical Laboratory**  *Practical lab-based HAXPES* |
| *11:30-12:15* | **Prof. Atushi Ogura, Meiji University, Meiji Renewable Energy Institute**  *Tailoring buried interfaces and the performance of electronic devices* |
| *12:15-13:00* | **Lunch**  *Manufacturer and Royce showcase* |
| *13:00-13:30* | **Dr. Andy Naylor, Department of Chemistry, Uppsala University**  *Energy-tuned XPS used to study battery interfaces* |
| *13:30-14:00* | **Dr. Paul Roussel, AWE Nuclear Security Technologies**  *HAXPES applications to nuclear forensics* |
| *14:00-14:15* | **Dr. Ben Spencer, The University of Manchester**  *Case studies from the Royce HAXPES Lab* |
| *14:15-14:30* | **Dr. Benjamen Reed, National Physical Laboratory**  *Beyond the surface — Obtaining surface-free depth distribution information from combined XPS and HAXPES spectra* |
| *14:30-14:45* | **Dr. Megan O. Hill, MAX IV Laboratory**  *Depth-Resolved X-Ray Photoelectron Spectroscopy Evidence of Intrinsic Polar States in HfO2-Based Ferroelectrics* |
| *14:45-15:00* | **Refreshments** |
| *15:00-16:30* | **Panel discussion**  **Chaired by Prof. Wendy Flavell, The University of Manchester**  *Panellists:*  **Dr. David Cant**, National Physical Laboratory  **Dr. Olivier Renault**, Université Grenoble Alpes and the French Alternative Energies and Atomic Energy Commission (CEA)  **Dr. Paul Roussel**, AWE Nuclear Security Technologies  **Dr. Anna Regoutz**, Department of Chemistry, University of Oxford  **Dr. Tien-Lin Lee**, Principle Beamline Scientist, Diamond Light Source |
| *16:30 – 17:30* | **Lab tours (optional)** |

**Abstracts**

**Practical lab-based HAXPES**

Dr. Alexander G. Shard, National Physical Laboratory

Photoelectron spectroscopy with X-ray sources higher than the ‘normal’ 1500 eV has, for a long time, been recognised as useful. Access to deep electronic core levels provides greater clarity in chemical analysis and the increased range of information depths permits better insight into the structure of the material being analysed. The advent of lab-based HAXPES instruments with acquisition times which are reasonable for practical analysis enables HAXPES to follow XPS in the transition from high-end research technique to routine analytical tool. This talk will cover some of the exciting developments in lab-based HAXPES, the analytical opportunities and a roadmap to standard and reproducible practical HAXPES.

**Tailoring buried interfaces and the performance of electronic devices**

Prof. Atushi Ogura, Meiji University, Meiji Renewable Energy Institute

Electronic devices such as LSIs and solar cells have improved performance by replacing chemically unstable surfaces with more stable buried interfaces. In recent years, with increased performance, they have become multilayered and more and more complex. In multilayered structures, the interfaces between the different materials determine the device performance and is therefore of particular interest. Compared with TEM, XRD, XPS and other methods, HAXPES is a powerful non-destructive tool which brings information on the chemical state of buried interfaces down to approx. 50 nm, without sputtering.

We will demonstrate how HAXPES Lab helps investigation on chemical properties of devices by showing Bias-applied, angle resolved, and deep probing measurements on memory devices, solar cells and trench structures.

**Energy-tuned XPS used to study battery interfaces**

Dr. Andy Naylor, Department of Chemistry, Uppsala University

Many ageing mechanisms in batteries occur at the electrode-electrolyte interfaces through e.g. phase transformation, surface layer formation, cationic/anionic redox reactions and metal dissolution. Photoelectron spectroscopy allows for a fundamental understanding of such processes, while tuning the incident X-ray energy to non-destructively depth profile gives a surface vs. bulk picture. Hard and soft X-ray photoelectron spectroscopy (HAXPES, SOXPES) are suited to determining composition, oxidation states, and bonding environments down to ~50 nm depth in battery materials. Correlation with electrochemical performance allows for greater understanding of battery ageing and informs strategies to stabilise the interfaces. I will present some of our latest results employing energy-tuned XPS, and steps towards operando XPS experiments.

**HAXPES Applications to Nuclear Forensics**

Dr. Paul Roussel, AWE Nuclear Security Technologies

The use of Hard X-ray Photoelectron Spectroscopy (HAXPES) as an experimental tool for the characterisation of nuclear forensic signatures is explored in this presentation. The limitations of conducting conventional X-ray Photoelectron Spectroscopy on actinide materials will be highlighted. Characterisation of ubiquitous nuclear fuel material uranium dioxide, UO2, using HAXPES data will be presented. Specifically, we address which peaks provide the most information, quantification, and various methods to extract useful information from MNN X-ray excited Auger spectra.

**Beyond the surface — Obtaining surface-free depth distribution information from combined XPS and HAXPES spectra**

Dr. Benjamen Reed, National Physical Laboratory

Gas cluster ion beam technology has enabled damage-free sputtering of organic materials, but low sputtering rates for inorganic materials gives impractical experiment durations. Increasing the energy per atom in clusters will typically improve sputtering rates but also generates a thin altered, or damaged, layer either through chemical changes or preferential removal of elements. To aid in the analysis of these sputter sensitive materials, we present a data processing algorithm which attempts to isolate the spectroscopic information of the undamaged subsurface material from the damaged surface material using the different intensity depth distributions that arise from Al Ka and Ag La X-ray generated spectra. This algorithm generates a *surface-free depth distribution*, and therefore reduces (or completely removes) the intensity contribution of the sputter damaged layer. This method also works for very thin overlayers on homogenous materials, such as native oxides or adventitious carbon contamination.

**Depth-Resolved X-Ray Photoelectron Spectroscopy Evidence of Intrinsic Polar States in HfO2-Based Ferroelectrics**

Dr. Megan O. Hill, MAX IV Laboratory

The discovery of ferroelectricity in industrially popular HfO2-based films has reignited the push for CMOS compatible ferroelectric devices. However, polar states in HfO2 onset only at ultralow thicknesses (<10 nm), in contrast to the degradation of ferroelectricity at low thicknesses in traditional ferroelectrics. This has sparked debated around the nature of ferroelectricity in HfO2-based films — does HfO2 present an intrinsic ferroelectric polarization or is apparent ferroelectric polarization response actually the result of the redistribution of oxygen vacancies under applied fields? This work aims to further elucidate the nature of polarization in HfO2-based ferroelectrics by investigating the electrochemical state at various film depths and polarization states via photoelectron spectroscopy. This work combines experimentation at the I09 beamline at Diamond Light Source and the lab-based HAXPES at the University of Manchester. We compare the ferroelectric behaviour of two HfO2-based ferroelectric device stacks with the observed electrochemical effects of switching by piezoresponse force microscopy. This study demonstrates the complex interplay between field-induced electrochemical changes and polarization switching, and finally provides evidence for an intrinsic polar state in HfO2-based ferroelectrics.