

Surface analytical considerations for environmental exposure assessment for nanotechnology applications.

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Development of nano-enabled products requires risk assessment. REACH requirements are changing for nanoforms in 2020 and will include evidence-based chemical assessment. Environmental exposure throughout the life cycle of a product will form part of risk assessment providing the relevant chemical hazard is known but what may not be known is how much, where and in what form material may enter the environment. The environment can be considered as a series of reactors, such as waste infrastructure, air, soil, water, sediment and biota, each of which can lead to chemical and physical transformations. Much work has been done modelling these complex issues but validation of models is required. Standards, instrumentation and validated procedures which accurately measure material from the release of engineered nanomaterials are needed. Clearly the fate of nanomaterial in the environment will depend on surface chemistry. This talk will discuss how effective surface analytical methods are for studying nanomaterials in environmental media.

Multi-technique analysis for nano and opto-electronic applications

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The increasingly complex structures and large variety of materials used in modern nano and opto-electronic devices drives the need to develop new approaches for their characterization. To answer the given analytical question it is necessary to combine several techniques to get reliable information and to combine different types of information on the same sample. In many cases, there is also a need to give fast feedback to remain competitive for the development of new technology and the new processes and materials that are involved. This presentation will address developments in TOF-SIMS analysis for applications from semiconductor technology to display technology and the importance of using several techniques such as scanning probe microscopy [1], TEM, X-ray tomography[2], TEM-EDX [3], XPS and plasma profiling time of flight mass spectrometry [4]. The importance of sample preparation to enable multi-technique studies is also critical and several examples will be given involving focused ion beam milling, wedge crater preparation and transfer between instruments under a protected environment (vacuum or inert gas).

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Exploitation of surface characterisation techniques for the improvement of solid/gas interfaces of thin-film solid-oxide electrodes

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Improving energy efficiency, reducing emissions and increasing the share of renewables are among the primary targets of many industrially developed nations. To achieve these goals, solid-state energy devices, including solid oxide fuel and electrolyser cells (SOFC/SOECs), have gathered significant attention. In recent years, advances in materials design have opened up unprecedented opportunities for development. My research focuses on the understanding, controlling and optimising the mechanism of oxygen reduction reactions in the amorphous and crystalline complex transition metal oxides, in particular of thin films grown by Pulsed Laser Deposition (PLD) technique. In my talk, I will present my work on model $\text{La}_{1-x}\text{Sr}_x\text{CoO}_{3-\delta}$ (LSC, $x = 0.2, 0.4$) thin-film electrodes. The control in the temperature and in the background pressure during the PLD deposition governs the thickness, microstructure and the defect structure of the films. The differences in the microstructure and the surface morphology of the films lead to significant differences in the elemental distribution, cation compositions and the oxidation states at the surface and subsurfaces, and hence on the electrochemical behaviour. I investigate the chemical compositions at the solid/gas interface and the depth profiles by the low energy ion scattering spectroscopy (LEIS), and the oxidation states by the X-ray photoelectron spectroscopy (XPS). I also study the ion-exchange kinetics in the films by isotope exchange depth profiling (IEDP) method coupled with time-of-flight secondary ion mass spectroscopy (ToF-SIMS).

A cryogenic 3D OrbiSIMS method for imaging volatile lipids

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3D OrbiSIMS has opened up many new possibilities in biological imaging mass spectrometry. This ultra-high vacuum instrument allows data acquisition with simultaneous high spatial resolution (~2 µm) and high mass resolving power (240,000 @ 200 m/z) [1].

Many compounds, however, are volatile under high vacuum and so refractory to this analytical technique. Here, we develop 3D cryo-OrbiSIMS, a low-temperature method compatible with the imaging of molecules that are volatile under standard ultra-high vacuum conditions. As a proof-of-principle, 3D cryo-OrbiSIMS is applied to the analysis of volatile hydrocarbons. These lipids form a protective barrier on the surface of many plants and animals and have important medical and agricultural applications [2, 3]. Using 3D cryo-OrbiSIMS in spectroscopy and imaging modes, high-resolution surface analysis and depth profiles of hydrocarbons are characterised from the cuticle/skin of insects, plants and humans. In the insect model organism *Drosophila*, hydrocarbons have previously been detected with 100 µm resolution using MALDI with a modified matrix [4]. We now show that 3D cryo-OrbiSIMS can be used to localize and depth profile *Drosophila* hydrocarbons with a spatial resolution of 5µm. In addition, 3D cryo-OrbiSIMS can be combined with the powerful genetic tools available in *Drosophila*, to show that the surface levels of hydrocarbons are regulated by a P450 lipid enzyme. Together, these findings demonstrate the broad utility of 3D cryo-OrbiSIMS for imaging volatile lipids and illustrate how it can be applied to genetic studies of lipid metabolism.

References

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Lead halide perovskite photovoltaics: challenges and pitfalls with their surface analysis

Dr Alexandra Ramadan

Lead halide perovskite photovoltaics (PV) have emerged in the past decade as a field with great potential to provide cheap renewable energy. Efficiencies of perovskite PV devices are approaching 26 %, bringing the technology in line with conventional silicon photovoltaics and making them the fastest developing solar technology to date. However, despite the potential of perovskite PVs, their incorporation into commercial products has not yet been realised. In spite of the impressive strides made in device performance and stability, fundamental characterisation of these materials is still severely lacking.

The instability and complex chemistry of these perovskite materials have made understanding the relation between the chemistry and electronic structure of these materials difficult. Attempts to measure the electronic structure and composition of various lead halide perovskite materials using photoemission based techniques have shown a lack of consensus in the compositional and electronic properties of these materials.

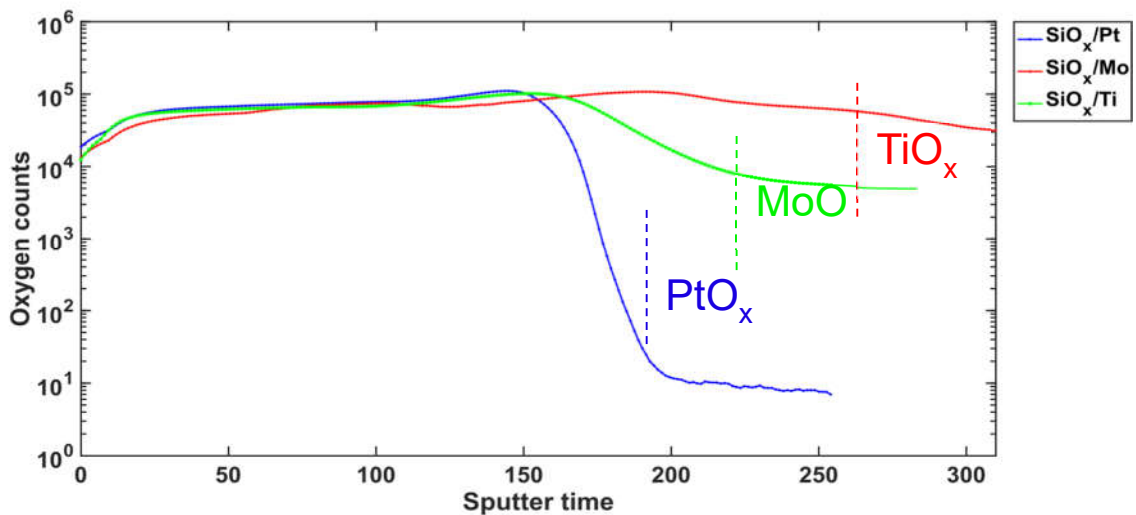
In this talk I will discuss the problems encountered when attempting to characterise the surface of these materials. I will then discuss our work using photoemission, scanned probe and electron microscopies and ion scattering to investigating the influence of sample preparation and chemical composition on the electronic structure of various lead halide perovskite systems.

One thousand trillion reasons why – silicon oxides (SiO_x) memristors

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Neuromorphic systems have the potential to transform the fields of Artificial Intelligence and Machine Learning by emulating the continuous, parallel communication between neurons found in the human brain. A promising route to this is the use of resistance switching devices as synapses due to their continuous states and compatibility with present semiconductor fabrication processes. Unfortunately, the physical mechanism that underpins the switching remains contentious, leading to problems in the stability and cycle lifetime of current devices and limiting their applicability. The device studied here, developed by our group, consists of a 40nm thick amorphous silicon oxide “switching layer” sandwiched between two metal electrodes



As localised chemical changes are at the heart of the switching process, Secondary Ion Mass Spectrometry (SIMS) is used to map the chemical changes that occur in these devices providing new insight into the role of oxygen in switching. Regions of SiO_x were electrically stressed using a Conductive Atomic Force Microscope in place of the top electrode. Three devices were made using different metals as bottom electrodes — metals with increasing affinities for oxygen — in order to probe the importance of the oxygen exchange at and across the $\text{SiO}_x/\text{Metal}$ interface. Subsequent SIMS depth profiling of pristine devices revealed an increase in the width of the metal oxide at the interface as the oxygen affinity of the electrode increased from $\text{Pt} \rightarrow \text{Mo} \rightarrow \text{Ti}$. This plays a crucial role in the behaviour of the devices, providing electrical resistance and thus making it more difficult to change their state. Under electrical biasing it was shown that Mo having a higher oxygen affinity than Pt allowed it to exchange and store oxygen with the switching layer more readily. In this way it acted as an oxygen “reservoir”, improving device performance and revealing the importance of the Metal/ SiO_x interface to resistance switching behaviour.

Through fine tuning this interface and closer matching of the Gibbs energy of oxide formation to that of the switching layer it should be possible to reduce oxygen loss and hence device damage. This will enable the development of much smaller, more reliable devices with orders of magnitude lifetime improvements