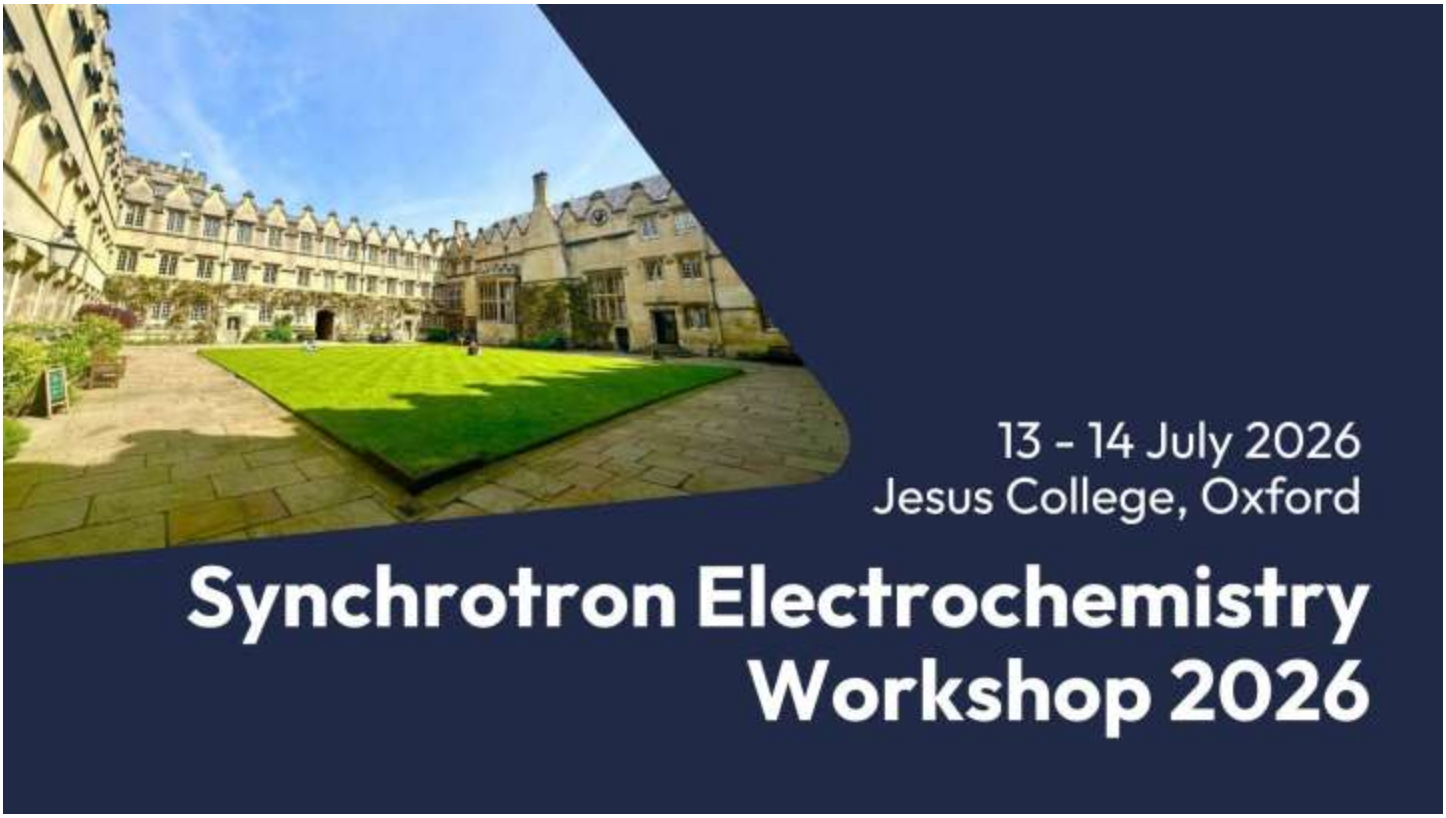

Abstracts

We are grateful to our workshop sponsors:

ALVATEK
BioLogic
THE FARADAY INSTITUTION
HENRY ROYCE INSTITUTE
HIDEN ANALYTICAL
IOP Institute of Physics
RSC INTEREST GROUP SOLID SURFACES
Silson
diamond

Programme

Monday 13th July	
12:00	Registration & Lunch
13:30	**Invited talk** Erwin Reisner, University of Cambridge: *Photoelectrodes for the solar valorisation of CO_2*
14:00	Andrey Shavorskiy: *Time-Resolved Dip-and-Pull APXPS for Studying Kinetics of Electrochemical Reactions*
14:15	Roxy Lee: *Operando Investigations into Nickel-based Electrocatalysts for Green Hydrogen Production in Alkaline Anion Exchange Membrane Environment*
14:30	Edward Brightman: *Operando XAS of polyoxometalate redox mediators for decoupled electrolysis and biomass-to-hydrogen systems*
14:45	Taniya Purkait: *Synchrotron Radiation Enabled FTIR Microspectroscopy for Operando Study of Ca^{2+} Storage Mechanism in Poly(tetra-sulfonamide) based Coordination Polymers*
15:00	**Invited talk** Philip Ash, University of Leicester: *Electrifying crystallography: combining techniques for mechanistic insight*
15:30	Coffee Break
16:00	**Invited talk** Uta Hejral, Chalmers University of Technology: *High energy surface x-ray diffraction from OER model electrocatalysts at work*
16:30	Siyu Jin: *Operando Synchrotron XRD Investigation of Hydrogen Embrittlement in Micro-Forged Al-Zn-Mg-Cu Alloys*
16:45	Liesbet Deconinck: *Advancing hydrogen embrittlement studies using synchrotron in-situ electrochemical GI-XRD with simultaneous mechanical straining*
17:00	Stephen Lyth: *Advanced Characterisation of M-N-C Electrocatalysts*
17:15	Longxiang Liu: *Potential-driven irreversible restructuring of Cu-N-C electrocatalysts*
17:30	**Sponsored presentation** Alvatek
17:50	Group photo
18:30	Dinner
19:30 - 21:00	**Poster Session and drinks**

Tuesday 14th July	
09:00	**Invited talk** Reshma Rao, Imperial College London: *Electrocatalysis for the production of green hydrogen and beyond: Materials and Mechanisms*
09:30	Nipon Deka: *On the microstructure of the electrode-electrolyte interface during oxygen evolution reaction (OER) using operando XAS*
09:45	Raffia Nimal: *Metal-Quinizarin Coordination Complexes as OER Electrocatalysts: Structural Insights from Synchrotron Characterisation*
10:00	James Counter: *Potential Controlled In-Situ Soft X-Ray Spectroscopy for Insights into the Electrocatalysis of Iridium Oxide to the OER*
10:15	Yifeng Wang: *Active Phase of Nickel Electrocatalysts driving Alkaline Hydrogen Evolution*
10:30	Coffee Break
11:00	**Invited talk** Iris Nandhakumar, University of Southampton: *Three-Dimensional Soft-Templated Nanoarchitectures for Enhanced Catalytic Activity*
11:30	Mengnan Wang: *Resolving Ionomer–Catalyst Interactions in Fuel Cell Electrodes via Operando X-ray Absorption Spectroscopy*
11:45	Stefanie Punke: *The electrochemical oxidation of Pt nanocatalysts – an operando X-ray scattering study*
12:00	Chen Han*: Investigating Oxide-Derived Catalysts for Electrochemical CO_2 Conversion to Fuels*
12:15	Soumyajit Maitra: *Investigating the Role of Crystal Phase Driven Surface and Bulk Reconstruction in Triggering Lattice Oxygen-Mediated Oxygen Evolution in Perovskite Oxides*
12:30	Lunch
13:30	**Invited talk** Robert Weatherup, University of Oxford: *Accessing Electrochemical Interfaces in Batteries and Electrolysers with Operando X-ray Spectroscopies*
14:00	Robert Temperton: *Perspectives from MAX IV Laboratory about the future of battery research at synchrotrons*
14:15	James Le Houx: *The Topographic Origin of Chemomechanical Failure in High-Nickel Cathodes Revealed by Operando Dark-Field X-ray Microscopy*
14:30	Farheen Sayed: *Li-ion batteries (LIBs) and Na-ion batteries (NIBs)*
14:45	Li Zhao: *In-Situ and Operando Cell Environments for Reliable Synchrotron Studies of Batteries*
15:00	Discussion and closing remarks
15:30 – 17:00	***The doctor is in!*** Have a coffee and discuss ideas for beamtime and experiment feasibility with the Diamond beamline scientists

Posters

1. **Isabel Antony**, University College London / Diamond Light Source
   Three-Dimensional Soft-Templated Nanoarchitectures for Enhanced Catalytic Activity
2. **Abdul Basith**, University of Reading
   Li+ based methylurea integrated Water in Salt Electrolytes (WiSE) for sustainable energy storage
3. **Liam Bird**, University of Oxford
   High energy surface x-ray diffraction from OER model electrocatalysts at work
4. **Laurence Brazel**, University of Cambridge
   Identifying competing rate effects in Li-O2 batteries with XRD-CT
5. **Francesco Carlà**, Diamond Light Source
   In-situ and operando characterization of electrochemical systems at the I07 beamline
6. **Tengyu Chen**, Barcelona Institute of Science and Technology
   Complementary In-situ X-ray Absorption Spectroscopy Reveals Oxidation Damping in Proximal Ru-O-Cr Motifs for Durable Acidic Oxygen Evolution
7. **Nigel Dyer**, University of Warwick
   Evidence of Room Temperature Bose Einstein Condensates in Water using EXAFS
8. **Kaiqing Fan**, University of Southampton
   Aerosol-assisted chemical vapour deposition of mixed phase tin phosphide thin films, and their sodium storage behaviours
9. **Dario Ferreira Sanchez**, Paul Scherrer Institute
   microXAS at SLS 2.0: Advancing Multimodal Synchrotron Chemical Imaging for Electrochemical Systems
10. **Pilar Ferrer-Escorihuela**, Diamond Light Source
    In Situ Soft X-ray Spectroscopy of Electrochemical Interfaces: From Reaction Cell and Sample Design to Mechanistic Insight
11. **Abolfazl Ghaderian**, University College London
    Ex Situ XANES/EXAFS Investigation of Mo-Based Electrocatalysts for Electrochemical Nitrogen Reduction
12. **Miguel A. Gomez-Gonzalez**, Diamond Light Source
    Unlocking electrochemical mechanisms at the nanoscale: Multi-modal insights from the I14 beamline
13. **Yvonne Grunder**, University of Liverpool
    X-ray Characterisation of Energy Materials at XMaS
14. **Xiuxiu Han**, University of Birmingham
    Electrocatalytic Strategies for Coupling Biomass Conversion with Clean Hydrogen Generation
15. **Thom Harris-Lee**, University of Cambridge
    Electrochemical Etching of GaN: A Call for X-ray Based Mechanistic Insight
16. **Nathaniel Hill**, University of Liverpool / UKRI STFC
    Unravelling the role of additives in the structure of non-aqueous media at the electrode surface under potential control
17. **Shahid Ullah Khan**, Delft University of Technology
    Decoupling Proton Transport and Water Crossover: A Palladium Membrane Reactor for Sustainable Electrocarboxylation

18. **Catherine Kohler**, University of Oxford
19. **Sirui Li**, University of Oxford
    Operando XAS investigation of Size-Selected Cobalt Alloy Nanoparticles for alkaline OER
20. **Menghao Liu**, KTH Royal Institute of Technology
    Revealing passive film evolution on FeMnCrNi medium entropy alloy under anodic polarization by in situ ambient pressure X-ray photoelectron spectroscopy
21. **Karolina Majewska**, Warsaw University of Technology
    Transitional forms in MCFC as key factors in CO_2 processing
22. **Kamana K. Mishra**, Indian Institute of Technology
    Understanding the Role of $NiCoO_x$ Mass Loading in Enhancing Oxygen Evolution Kinetics for AEM Water Electrolysis
23. **Tingting Mo**, Imperial College London
    Deconvoluting the mechanism of ethylene glycol electro-oxidation towards glycolic acid on gold catalysts using Operando-XAS
24. **Ciarán O'Brien**, University of Liverpool / STFC
    Computational Modelling of Electrode-Electrolyte Interfaces, for Applications in Catalysis and Spectroscopy Support
25. **Josh Pickston**, University of Salford
    Electrochemical Nitrogen Reduction Reaction using a Dual Phase Cell
26. **Li Shao**, Diamond Light Source
    Templated electrodeposition of nanoscale materials
27. **Kayleigh Skidmore**, University of Warwick
    Operando X-ray Studies of Proton Exchange Membrane Electrocatalysts for Hydrogen Production
28. **Elisa Solvay**, Imperial College London
    Metal Nitrogen-doped Carbon Catalysts for the Sustainable Electroreduction of Carbon Dioxide into Value-added chemicals
29. **Nita Srinivasan**, University of Surrey
    Deconvoluting the mechanism of ethylene glycol electro-oxidation towards glycolic acid on gold catalysts using Operando-XAS
30. **Andrew Walters**, Diamond Light Source
    Nanoscale X-ray imaging of functional materials in operando: the Diamond-II Coherent Soft X-ray Imaging and Diffraction (CSXID) beamline

POSTER

Three-Dimensional Soft-Templated Nanoarchitectures for Enhanced Catalytic Activity

Antony, I. (UCL/Diamond Light Source)

To meet the automotive industry's demand for longer driving ranges and low-cost battery packs, there is a strong focus on high-capacity cathode materials. $LiNiO_2$ (LNO) is a promising candidate due to its high nickel content and gravimetric capacity exceeding 200 mAh g^{-1}. However, its practical applicability is limited by poor cycling performance, thermal instability, and structural degradation during cycling. The LNO material studied here has previously shown to contain twinned domains within nominally single-crystal particles, consisting of agglomerations of 2-3 crystallites per particle. In this work, we employ spectro-Ptychography at the Ni K-edge on beamline I13-1 at Diamond Light Source to investigate nanoscale Ni charge-state heterogeneity associated with oxygen loss and structural degradation. Particles were studied after first charge and after electrochemical ageing. The measurements reveal spatial indicators of preferential lithiation of single crystallites within a pseudo single crystal particle. These observations suggest a correlation between local Ni charge-state variations and degradation pathways in twinned LNO particles. The result demonstrates the capability of spectro-Ptychography to resolve chemically and structurally heterogeneous degradation processes in high-Ni cathode material.

INVITED TALK

Electrifying crystallography: combining techniques for mechanistic insight

Ash, P. (University of Leicester)

Redox enzymes capable of carrying out multi-electron reactions serve as blueprints for the rational design of bio-inspired catalysts for future green technologies. During catalysis, enzymes transition through multiple 'active' intermediate states. Movement of both electrons and substrate to/from the active site is precisely controlled to achieve the equivalent of high Faradaic efficiencies, with minimal electrons wasted in detrimental side reactions. High turnover frequencies typically require mechanisms for highly-choreographed movement of two quantum particles, protons and electrons. According to Marcus theory, structural rigidity is key to a low reorganisation energy barrier for rapid, outer-sphere electron transfer. However, this is at odds with the requirement for a degree of conformational flexibility to enable rapid proton transfer. Here we exploit a combination of infrared microspectroscopy and structural studies, both under electrochemical control, to resolve biological proton coupled electron transfer with atomic detail.

POSTER

Li+ based methylurea integrated Water in Salt Electrolytes (WiSE) for sustainable energy storage

Basith, A. (University of Reading)

The reliance on toxic and flammable electrolytes in current energy storage systems leads to major challenge in developing safe and sustainable energy storage technologies.[1,2] Water in salt electrolytes (WiSE) (highly concentrated aqueous salt solutions) offer a promising solution for these mentioned challenges, expanding the electrochemical stability window (ESW) of water from 1.2 V to 3 V. Addition of co-solvents like methyl urea have been reported to further improve the ESW up to 4.5 V, but molecular level study for this phenomenon is not clearly understood.[3] This study sheds light on the co-solvent based enhancement of the electrochemical stability window, using dual structure-dynamics approach for rational design of WiSE in future devices. This work focuses on a neutron total scattering study of 21m methyl urea incorporated in 21 m LiTFSI in water (2.62 water molecules to 1 LiTFSI and to 1 methyl urea) with 7 hydrogen/deuterium contrasts. Analysis is performed by reducing the experimental scattering to differential cross section, before performing 'experiment informed' molecular dynamics simulations combined with empirical potential structure refinement. With the help of selective neutron sensitivity to H/D isotopes, the study reveals how Li+ coordination with other molecules in the system and disrupted water networks influence the electrochemical stability window in in the system.[4] In a complimentary NMR investigation, we studied the diffusion coefficients of Li^+, F, and water, furthering our understanding of how the ion transport mechanisms are influenced by the addition of the co-solvent.[5] Future in situ studies will use high energy X-ray techniques to probe the electrode-electrolyte interfacial processes further investigating the SEI formation and associated evolutions in the system. Also cycling studies will be carried out using the same technique to study the cell under realistic operating conditions.

References:

[1] Suo, Liumin, Oleg Borodin, Tao Gao. "“Water-in-salt” electrolyte enables high-voltage aqueous lithium-ion chemistries." Science 350, no. 6263 (2015): 938-943.c

[2] Shen, Yuanhao, Bin Liu, Xiaorui Liu, Jie Liu. "Water-in-salt electrolyte for safe and high-energy aqueous battery." Energy Storage Materials 34 (2021): 461-474.

[3] Lin, R., Ke, C., Chen, J., Liu, S., & Wang, J. (2022). Asymmetric donor-acceptor molecule-regulated core-shell-solvation electrolyte for high-voltage aqueous batteries. Joule, 6(2), 399-417.

[4] Groves., Kieran J. Agg, Shurui Miao, Thomas Headen, Tristan, Gregory N. Smith, Susan Perkin, and James E. Hallett. "Lithium solvation and anion-dominated domain structure in water-in-salt electrolytes." EES batteries (2025).

[5] Abdul Basith, Tim Groves, Neave Taylor, Oliver Alderman, James E. Hallett. "Molecular Engineering of WiSE: Methylurea in LiTFSI systems" (in preparation)

POSTER

High energy surface x-ray diffraction from OER model electrocatalysts at work

Bird, L. (University of Oxford)

Lithium-sulfur (Li-S) cells have high gravimetric energy density but are challenged by capacity degradation as the active material undergoes morphological changes during its reversible conversion from sulfur to lithium polysulfides. Changes in the reversible capacity and overpotential as a function of charge/ discharge rate provide insights into the interface between the active material and electronically conducting host matrix, including the effects of pore size distribution and electrode tortuosity. In-situ synchrotron radiation X-ray computed tomography (SR-XCT) characterizes sulfur distribution in Li-S cathodes as a function of state of charge. Here, we compare electrodes cycled using different charge/ discharge rate protocols including cells undergoing long-term cycling with a constant high C rate and cells undergoing cycling with progressively increasing rates. Correlating the effect of changing C rate on discharge/char capacity with sulfur distribution indicates that dissolution is the rate limiting step, rather than deposition. By characterising the cumulative effects of different cycling protocols on sulfur distribution, we investigate whether capacities derived from variable-rate cycling are representative of long-term high-rate testing and therefore aim to inform future testing protocols with widespread applicability.

POSTER

Identifying competing rate effects in Li-O_2 batteries with XRD-CT

Brazel, L. (University of Cambridge)

Li-O_2 batteries are a post-Li-ion battery technology which promises extremely high energy densities (~3500 Wh/kg). Many technological problems must be overcome before they are commercially viable, one of which is limited capacity at high discharge rates. One of the main reasons for this is the slow mass transport of O_2 through the air electrode, resulting in heterogeneity in the formation of Li_2O_2 through the electrode, both in morphology and quantity. X-ray diffraction computed tomography (XRD-CT) was employed at i18 at Diamond Light Source to characterise the distribution of discharge products through the electrode, allowing for variations in the amount of Li_2O_2 and in the morphology (i.e. crystallite size) to be determined. These heterogeneities reveal into the competing rate effects of O_2 starvation and increased local utilisation of the carbon electrode seen in Li-O_2 batteries. LiOH, the product of parasitic side reactions, could also be spatially characterised, identifying heterogeneity in degradation, informing on what factors drive this degradation.

TALK

Operando XAS of polyoxometalate redox mediators for decoupled electrolysis and biomass-to-hydrogen systems

Brightman, E. (University of Strathclyde)

Decoupled electrolysis using soluble redox mediators offers a promising route to reduce the energy requirements of green hydrogen production while enabling the valorisation of low-value biomass feedstocks. Keggin-type heteropolyacids, such as phosphomolybdic acid (PMA), are attractive candidates due to their reversible multi-electron redox chemistry; however, their behaviour in aqueous electrochemical environments remains poorly understood. Here, we present an operando X-ray absorption spectroscopy (XAS) study of PMA during electrochemical reoxidation in a bespoke proton exchange membrane (PEM) flow cell designed for simultaneous synchrotron analysis and electrochemical operation. Reduced PMA solutions prepared both electrochemically and via thermal digestion of lignocellulosic biomass (including real waste streams) were investigated. Time-resolved Mo K-edge XAS reveals subtle shifts in edge features and changes in EXAFS coordination consistent with partial reoxidation and structural contraction of the Keggin anion. Correlation with electrochemical measurements and mass spectrometry demonstrates a clear relationship between spectral evolution, mediator state-of-charge, and hydrogen production. The results suggest that charge is delocalised across the polyoxometalate framework rather than localized at individual Mo centres, providing new insight into redox mechanisms in solution-phase mediator systems. Additionally, differences between electrochemically and biomass-reduced mediators highlight the complexity introduced by real feedstocks, including evidence of concurrent direct electrochemical oxidation processes. Beyond presenting these findings, we will discuss experimental challenges associated with operando XAS of liquid-phase electrochemical systems, including cell design, beam–sample interactions, and data interpretation. This work establishes a framework for probing redox-active species under realistic operating conditions and motivates further application of synchrotron techniques to emerging electrochemical technologies, including next-generation electrolyser materials.

POSTER

In-situ and operando characterization of electrochemical systems at the I07 beamline

Carlà, F. (Diamond Light Source)

The I07 beamline at Diamond Light Source is a facility dedicated to the characterisation of surfaces and buried interfaces using x-ray diffraction and scattering techniques in a variety of different sample environments. Thanks to the high flux provided by its undulator and the ability to operate in the 5–30 keV energy range, the beamline is particularly well suited to in-situ and operando experiments on surfaces and interfaces. The high transmission of hard x-rays through liquid media makes the beamline an ideal platform for investigating electrochemical reactions. The beamline supports a range of techniques, including x-ray reflectivity (XRR), grazing-incidence x-ray diffraction (GIXD), and grazing-incidence small-angle x-ray scattering (GISAXS), which provide structural and morphological information from surfaces and buried interfaces in real time in relevant reaction conditions. Multiple techniques can be combined simultaneously, allowing different length scales to be probed simultaneously and providing unique insights into structure–reactivity relationships. Different electrochemical setups are available to I07 users, allowing electrochemical experiments to be performed under different conditions (electrodeposition, characterisation of adsorbed layers, electrocatalysis, corrosion, batteries, etc.). User-developed custom setups can also be installed. A versatile detector system enables fast data acquisition and reciprocal space mapping over different length scales. Surface dynamics can also be investigated using time-resolved experiments in the millisecond regime. A fluorescence detector is also available for performing XAS experiments in parallel with diffraction and scattering measurements, providing complementary chemical information.

TALK

Complementary In-situ X-ray Absorption Spectroscopy Reveals Oxidation Damping in Proximal Ru-O-Cr Motifs for Durable Acidic Oxygen Evolution

Tengyu Chen*[1], Kaiwen Wang[1], Lu Xia[1], Bruna Ferreira Gomes[2], Jordi Arbiol[3], F. Pelayo Garcia De Arquer[1*]

[1]ICFO – Institut de Ciències Fotòniques, The Barcelona Institute of Science and Technology, 08860 Castelldefels, Barcelona, Spain. [2]Electrochemical Process Engineering, University of Bayreuth, Universitätsstraße 30, Bayreuth, 95447, Germany. [3]Catalan Institute of Nanoscience and Nanotechnology (ICN2), CSIC and BIST, Campus UAB, Bellaterra, 08193 Barcelona, Catalonia, Spain.

Understanding the dynamic evolution of atomically dispersed active sites under operating conditions remains essential for the development of durable electrocatalysts. Although Ru single-atom catalysts (Ru SACs) exhibit high intrinsic activity toward acidic oxygen evolution (OER), they suffer from progressive overoxidation and dissolution at industrially relevant conditions. Here, we construct RuCr heterogeneous diatomic catalysts (RuCr HDCs) featuring proximal Ru-O-Cr motifs (up to 29.5%), and investigate the redox dynamics using complementary in-situ X-ray absorption spectroscopy (XAS). In-situ X-ray absorption near-edge structure (XANES) measurements reveal dynamically anti-correlated valence-state evolution at the atomic scale. The Ru absorption edge of Ru SAC continuously shifts with increasing potential (2.1 eV), whereas Ru in RuCr HDC exhibits a much smaller overall shift (0.83 eV) together with reversible redox behaviour; meanwhile, the Cr K-edge displays synchronized but opposite changes, indicating local redox buffering within the Ru-O-Cr motif. In-situ extended X-ray absorption fine structure (EXAFS) further reveals distinct coordination trajectories associated with this buffered response. Ru SAC undergoes continuous expansion of Ru-O coordination accompanied by the emergence of Ru-O-Ru scattering, while RuCr HDC preserves a stable Ru–O with only moderate and reversible coordination changes. Cr centers exhibit similarly adaptive yet constrained responses. In addition, in-situ Mn K-edge XAS shows comparable evolution in both catalysts, suggesting that the distinct behaviour originates from local atomic interactions rather than support. These results provide direct spectroscopic evidence that proximal Ru-O-Cr motifs undergo constrained structural evolution, thereby suppressing irreversible Ru overoxidation. The oxidation damping substantially improves stability, decreasing Ru dissolution by 52× relative to Ru SACs in acidic OER. In proton exchange membrane water electrolysis, RuCr HDC operates stably for 285 h at 1 A×cm^{-2} with a degradation rate of 0.28 mV×h^{-1}, compared with 1.90 mV×h^{-1} for Ru SAC. This work demonstrates how complementary X-ray absorption spectroscopy can uncover dynamic cooperative interactions in atomically precise catalysts and establishes proximal redox coupling as a strategy for stabilizing highly utilized Ru sites under harsh oxidative condition.

TALK

Potential Controlled In-Situ Soft X-Ray Spectroscopy for Insights into the Electrocatalysis of Iridium Oxide to the OER

Counter, J. (University of Manchester)

The oxygen evolution half-reaction (OER) for acidic water electrolysis is the rate limiting step and therefore a bottleneck scaling up green hydrogen production from water electrolysis, a key pathway to decarbonizing the hydrogen economy. Iridium oxide (IrOx) is the benchmark OER electrocatalyst due to relatively high stability and catalytic activity. To gain insight into the mechanism, we have combined in-situ X-ray photoelectron spectroscopy (XPS) and near-edge X-ray absorption fine structures (NEXAFS) spectroscopy. These probe the oxidation state, chemical environment, and electronic structure of the electrode surface under conditions not observed in ex-situ measurements. We use these techniques to gain mechanistic insight into IrOx OER electrocatalysis. In addition, we investigate the aging of iridium oxides of different crystallinities to provide insight into the degradation pathways.

To compliment steady-state measurements, a novel potential programmed NEXAFS (PP-NEXAFS) technique was developed. This technique facilitated measurements under dynamic conditions (e.g. during cyclic voltammetry) to measure the NEXAFS signal with <20 mV potential resolution. This was used to distinguish the oxyl speciation on the iridium oxide surface, indicating the formation at bridging, then coordinatively unsaturated sites, hence demonstrating the applicability of PP-NEXAFS at determining transient dynamics of OER catalysts during start-up and shut-down.

TALK

Advancing hydrogen embrittlement studies using synchrotron in-situ electrochemical GI-XRD with simultaneous mechanical straining

Deconinck, Liesbet (Norwegian University of Science and Technology)

Hydrogen embrittlement is a widespread challenge in industry. This phenomenon reduces the ductility and shortens the service life of components through the interaction between hydrogen atoms and metal structure. However, there is currently a literature gap about the active mechanism of hydrogen embrittlement due to the lack of in-situ studies. We have developed an in-situ set-up that enables simultaneous synchrotron-based grazing-incidence X-ray diffraction measurements, specimen straining, and the application of an electrochemical environment. This enables the real-time monitoring of the development of hydrogen-induced changes and the effect of stress. The versatility of the set-up was demonstrated across several case studies. First, the impact of hydrogen on titanium at different strain levels revealed a correlation between hydrogen uptake, hydride precipitation, lattice strain evolution, and deformation behaviour. It was found that the introduction of hydrogen induces anisotropic lattice expansion in specific orientations and lattice compression in others. Furthermore, prolonged hydrogen charging causes partial relaxation in certain crystal orientations. Complementary microscopic characterisation confirmed it to be related to dislocation activity. However, upon plastic deformation, the defect density increased, inducing lattice compression. Secondly, the influence of corrosion and hydrogen charging on pipeline steel was evaluated with this technique. This material is more complex as it contains both the ferrite and pearlite phase. The results indicate an orientation-dependent strain-softening or strain-hardening. The former is explained by preferential dislocation of slip and stress relaxation in specific crystallographic planes, whereas the latter is caused by the increasing compressive strain. Complemented with other high-resolution characterisation techniques, this versatile in-situ set-up offers high potential to acquire new mechanistic insights into hydrogen embrittlement and future studies on environmentally assisted degradation.

TALK

On the microstructure of the electrode-electrolyte interface during oxygen evolution reaction (OER) using operando XAS

Deka, N. (PSI Center for Energy and Environmental Sciences, Paul Scherrer Institute)

Electrified solid-liquid interfaces are ubiquitous in technologies ranging from colloidal science to electrochemistry. Hence, an atomic-level characterization of the electrode-electrolyte interface is crucial for total control and optimization of the processes involved. While the electrode side of the interface has been quite extensively studied, the electrolyte side is comparatively poorly understood. Specifically, probing the near-surface electrolyte species during an electrochemical reaction is a major technical challenge in the field of catalysis and surface chemistry. We have developed an interface-sensitive X-ray absorption spectroscopy (XAS) approach which allows us to directly probe the near-surface cations and anions of the electrolyte under applied potential. In this approach, we employ a mesoporous electrocatalyst film coated on a SiN_x X-ray window. These mesoporous films exhibit an extremely high electrode-electrolyte interface area, enabling us to specifically probe the behavior of interfacial ions via their K-edge spectra. Using this approach, we have recently investigated the interaction of Na^+ cations with IrO_x during the oxygen evolution reaction (OER), which is a bottleneck in major electrochemical processes like green hydrogen production and CO_2 reduction. The cations and anions of the electrolyte reportedly influence the OER activity of electrodes during OER. [2,3] We used operando Na K-edge XAS to directly probe the concentration and coordination environment of the near-surface Na+ cations. Simultaneously, operando O K-edge XAS monitored the interfacial water structure and the evolution of catalyst's surface structure. Contrary to expectations, we discovered that the positively charged Na+ ions are drawn to the IrO_x surface by more positive potentials only at alkaline pH. This finding cannot be explained by any of the electrolyte theories [4] put up thus far, emphasizing the necessity of a detailed investigation of interfacial electrolyte structures.

References

[1] Arminio-Ravelo et al. ChemPhysChem 20, (2019). 2. Tymoczko et al. Catal. Today 244, (2015). 3. Ganassin et al. Phys. Chem. Chem. Phys. 17, (2015). 4. Waegele et al. J. Chem. Phys. 151, (2019).

POSTER

Evidence of Room Temperature Bose Einstein Condensates in Water using EXAFS

Dyer, N. (University of Warwick)

As part of an investigation into the synchronous switching of the ECTO-NOX protein between two catalytic modes with a periodicity of 24 minutes, James and Dorothy Morré performed EXAFS measurements of a CuIICl2 solution at the Pacific Northwest Consortium Collaborative Access Team (PNC-CAT) at the Advance Photon Source (APS) of Argonne National Laboratories in the US. [1] They were able to show that the bond length between the copper ions and the water molecules that form the first solvation layer varied in a complex repetitive manner with a 24-minute periodicity. Unusually, the Morrés also performed 90-minute measurement of the variation in the X ray absorbency at the 8keV energy used for the EXAFS measurements. [2] Independently, they also measured the variation in the IR absorbency of water vapour immediately above a water sample at an ortho-water absorbance line over a 90-minute period. A recent unpublished re-examination of both sets of results showed variations consisting of a superposition of 12 and 14 different sinusoidal variations, respectively. Their frequencies are all close to harmonics of an oscillation with a period of just over 60 minutes with a 4σ significance. I argue that in both cases the specific frequencies seen are consistent with three spatially orthogonal spin-1 Bose-Einstein Condensates (BECs) being present in the water. The low frequency sinusoidal variations seen in the measurements are then consistent with AC-Josephson interactions between the condensates. The periodic variation in the lower resolution EXAFS measurements of the bond length variation between the Copper ion and its water of hydration can then be shown to be consistent with similar AC-Josephson interactions between two spin-1 BECs such that the measured sinusoidal variations are harmonics of an oscillation with a periodicity of 24 minutes. The presence of such macroscopic quantum states would also explain the origin of the synchronisation of the ECTO-NOX transitions between catalytic modes across millions of otherwise independent ECTO-NOX complexes. Recent unpublished experimental work has demonstrated that these experimental results require highly coherent X-ray sources provided by beam-line facilities. They show how such experiments can open a window onto a new aspect of the physics of water and aqueous solutions.

References

[1] Morré, D.J., et al., Structural observations of time dependent oscillatory behavior of CuIICl2 solutions measured via extended X-ray absorption fine structure. Journal of Inorganic Biochemistry, 2007. 101(4): p. 715-726.

[2] Morré, D.J., et al., Regular oscillatory behavior of aqueous solutions of CuII salts related to effects on equilibrium dynamics of ortho/para hydrogen spin isomers of water. Journal of Inorganic Biochemistry, 2008. 102(2): p. 260-267.

POSTER

Aerosol-assisted chemical vapour deposition of mixed phase tin phosphide thin films, and their sodium storage behaviours

Fan, K. (University of Southampton)

SnP/Sn and SnP/Sn_4P_3 thin films were deposited on titanium foils by aerosol-assisted chemical vapour deposition (AACVD) using N-phenyl phosphine carboxamide as an air-stable phosphorus precursor for the first time in CVD processes. By varying the solvent between methanol and chloroform, different phase compositions were obtained. The deposited films were directly employed as free-standing electrodes for sodium-ion batteries without the need for binders or conductive additives. Both film systems exhibited promising electrochemical performance, retaining discharge capacities above 500 mA h g^{-1} after 100 cycles at 200 mA g^{-1}. Ex situ X-ray diffraction and X-ray absorption spectroscopy were used to investigate the structural evolution of SnP/Sn during cycling. The results indicate that the crystalline SnP phase becomes largely amorphous after the initial sodiation process and remains predominantly amorphous upon subsequent cycling, supporting a conversion–alloying sodium-storage mechanism. The amorphous Sn–P framework also demonstrated good structural stability during repeated sodiation/desodiation. This work highlights the potential of AACVD combined with air-stable organophosphorus precursors as a scalable approach for preparing metal phosphide thin-film anodes for sodium-ion batteries.

POSTER

microXAS at SLS 2.0: Advancing Multimodal Synchrotron Chemical Imaging for Electrochemical Systems

Ferreira Sanchez, D. (Paul Scherrer Institute)

The next-generation microXAS beamline at the Swiss Light Source (SLS 2.0) will provide a transformative platform for synchrotron-based chemical imaging of electrochemical systems, enabling unprecedented investigations across ex situ, in situ, and operando conditions. Building on a strong legacy of multimodal microprobe and chemical tomography experiments, the upgraded instrument is designed to address the growing need for spatially and temporally resolved insight into complex, heterogeneous electrochemical processes. The new microXAS beamline is conceived as a dedicated hard X-ray nanoprobe for 4D chemical imaging (space–time), combining techniques such as XRF, XAS, XRD, within a single and dynamically configurable beamline. The SLS 2.0 upgrade will deliver more than two orders of magnitude increase in photon flux on sample, and an extended energy range up to ~40 keV, enabling higher penetration power and access to high-Z elements. These advances will allow imaging of intact electrochemical cells under realistic operating environments, including buried interfaces and complex sample environments typical of batteries, fuel cells, and catalysis systems.

A key feature of microXAS 2.0 is its hierarchical, multimodal imaging capability, combining micro- and nano-focused probes with flexible trade-offs between spatial resolution (down to <100 nm), chemical sensitivity, and temporal resolution. This “multi-scale” approach is particularly suited to electrochemical systems, where relevant phenomena span from particle-level heterogeneity to device-scale gradients. The beamline’s design further emphasizes high-speed data acquisition and advanced tomography schemes, enabling dynamic studies of reaction pathways and kinetics under operando conditions. Recent operando chemical imaging experiments at the present microXAS beamline, such as spatially resolved XRD mapping of all-solid-state batteries, have demonstrated the ability to directly visualize heterogeneous lithiation processes, phase transformations, and degradation pathways within single particles and across electrodes. The enhanced performance of microXAS 2.0 will extend these capabilities toward faster, higher-resolution, and more chemically selective measurements, while supporting more complex electrochemical environments. These aforementioned capabilities are expected to open new perspectives for studying electrochemical energy storage and conversion systems, from solid-state batteries to electrocatalysis and corrosion, under realistic working conditions. With first user operation foreseen at the end of 2027, microXAS 2.0 will provide a unique and powerful tool for the electrochemistry community, enabling a step change in the understanding of structure–property–reactivity relationships in complex materials.

POSTER

In Situ Soft X-ray Spectroscopy of Electrochemical Interfaces: From Reaction Cell and Sample Design to Mechanistic Insight

Ferrer-Escorihuela, P. (Diamond Light Source)

Suitable reaction cells are critical for in situ near-ambient pressure (NAP) soft X-ray photoelectron spectroscopy (XPS) and near-edge X-ray absorption fine structure (NEXAFS) studies. [1-3] These techniques enable the investigation of chemical states and electronic structure of catalytically active materials under realistic operating conditions. To facilitate such experiments, we have developed a top-side illuminated electrochemical flow cell specifically designed for in situ synchrotron-based NAP-XPS and NEXAFS studies on the B07 VerSoX beamline at Diamond Light Source. [3] The flexible cell design incorporates a non-metallic (PEEK) body and replaceable membranes consisting of either X-ray transparent silicon nitride (SiNx) or water-permeable polymer materials (e.g. Nafion™), selected according to the experimental requirements. [3, 4] The modular design also allows rapid sample exchange. NEXAFS measurements can be performed in total electron yield (TEY), Auger electron yield (AEY), and fluorescence yield (FY) modes. These in situ studies provide insight into charge transfer at the liquid–solid interface, the activation of reactant molecules, and the evolution of surface reaction intermediates. Such knowledge is essential for advancing our understanding of electrocatalytically active materials and for the rational design of improved catalytic systems.

References

[1] Grinter, D. C., Ferrer, P., Venturini, F., van Spronsen, M. A., Large, A. I., Kumar, S., Jaugstetter, M., Iordachescu, A., Watts, A., Schroeder, S. L. M., Kroner, A., Grillo, F., Francis, S. M., Webb, P. B., Hand, M., Walters, A., Hillman, M., & Held, G. *VerSoX B07-B: a High-Throughput XPS and Ambient Pressure NEXAFS Beamline at Diamond Light Source*. J. Synchrotron Radiat. (2024), 31, 578–589.

[2] Held, G., Venturini, F., Grinter, D. C., Ferrer, P., Arrigo, R., Deacon, L., Quevedo Garzon, W., Roy, K., Large, A., Stephens, C., Watts, A., Larkin, P., Hand, M., Wang, H., Pratt, L., Mudd, J. J., Richardson, T., Patel, S., Hillman, M., & Scott, S. *Ambient-Pressure Endstation of the Versatile Soft X-ray (VerSoX) Beamline at Diamond Light Source*. J. Synchrotron Radiat. (2020), 27, 1153–1166

[3] Kumar, S., Counter, J. J. C., Grinter, D. C., van Spronsen, M. A., Ferrer, P., Large, A. I., Orzech, M. W., Wojcik, P. J., & Held, G. *An Electrochemical Flow Cell for Operando XPS and NEXAFS Investigation of Solid–Liquid Interfaces*. J. Phys. Energy (2024), 6, 036001.

[4] Counter, J. J. C., Kumar, S., Zalitis, C. M., Ferrer, P., Large, A. I., Grinter, D. C., van Spronsen, M. A., & Held, G. *A Catalyst-Coated Mesoporous Carbon–Membrane Electrode Assembly for In Situ Soft X-ray XPS and NEXAFS Studies of Electrocatalytic Interfaces*. ACS Electrochem. (2026), 2(5), 1226-1237

POSTER

Ex Situ XANES/EXAFS Investigation of Mo-Based Electrocatalysts for Electrochemical Nitrogen Reduction

Ghaderian, A. (University College London)

The electrochemical nitrogen reduction reaction (NRR) is a promising strategy for sustainable ammonia production under ambient conditions. In this study, monometallic Mo-based electrocatalysts were synthesized via a hydrothermal method and deposited onto carbon paper electrodes for electrochemical NH_3 production. Electrochemical measurements were carried out using Pt mesh as the counter electrode under different pH conditions to evaluate catalytic activity and selectivity. Among the tested electrolytes, 0.1 M Li_2SO_4 at neutral pH exhibited the best performance, achieving a Faradaic efficiency of 37% at −0.6 V versus Ag/AgCl. Long-term electrolysis demonstrated good catalyst stability, while surface analysis confirmed a homogeneous distribution of Mo species on the electrode surface. The production of NH3 was verified by both 1H NMR spectroscopy and the indophenol blue method. To investigate the structural and electronic evolution of active sites during catalysis, ex situ synchrotron X-ray absorption spectroscopy was performed on the catalysts before and after electrochemical operation. XANES measurements revealed an approximately −1.02 eV shift in the Mo absorption edge after applying potential, indicating increased electron density at the Mo centers under working conditions. EXAFS analysis further showed changes in the local coordination environment of Mo, including variations in bond distances and coordination numbers, suggesting structural reconstruction and charge redistribution around the active sites. These ex situ XAS results indicate that modification of the Mo local electronic structure promotes N_2 adsorption and activation, thereby enhancing NH3 selectivity and catalytic performance. The combined electrochemical and synchrotron analysis provides important insight into the structure–activity relationship of Mo-based NRR catalysts and demonstrates the value of ex situ XANES/EXAFS techniques for understanding active-site evolution during electrochemical ammonia synthesis.

POSTER

Unlocking electrochemical mechanisms at the nanoscale: Multi-modal insights from the I14 beamline

Gomez-Gonzalez, M. (Diamond Light Source Ltd.)

Nanoscale X-ray microscopy provides a uniquely powerful platform for probing electrochemical systems, enabling spatially resolved investigation of material transformations, phase evolution, and speciation changes. Through the complementary use of X-ray fluorescence (XRF), X-ray absorption near-edge spectroscopy (XANES), X-ray diffraction (XRD), and coherent imaging modalities such as differential phase contrast (DPC) and ptychography, a comprehensive picture of electrochemical processes can be assembled across multiple length scales. Hard X-ray nanoprobes play a critical role, bridging the spatial resolution of electron microscopy with the larger fields of view accessible to bulk techniques, while retaining the penetrating power required for in situ and in operando measurements. The hard X-ray nanoprobe beamline I14 at Diamond Light Source delivers a focused beam of 50 nm over a continuously tuneable energy range of 5–23 keV [1]. Its endstation is engineered for experimental versatility, supporting rapid interchange of sample environments and mounts [2], and thereby opening new pathways for electrochemical research under realistic operating conditions. Electrochemistry underpins technologies of critical societal importance, from energy storage in next-generation batteries to electrocatalysis for clean fuel production. Advancing these technologies toward global net-zero targets requires a mechanistic understanding of how redox chemistry and crystallographic transformations at the micro- and nanoscale govern macroscopic performance, a knowledge that remains incomplete. Hard X-ray nanoprobe techniques are particularly well suited to address this gap. Here, we present a series of user science cases conducted at I14, spanning a range of experimental configurations and material systems: (i) tracking the morphological and chemical evolution of cathode particles through their operating voltage window, with particular focus on vanadium distribution within individual particles of the archetypal sodium-ion polyanionic cathode material $Na_3V_2(PO_4)_3$ (NVP); (ii) investigating structural phase transitions of high-Ni content Li-ion battery cathode particles; and (iii) examining the influence of electrolyte exposure on nickel oxidation states and microstructural integrity in nickel-rich cathode materials under in situ thermal conditions [3]. Finally, we describe the current instrumentation and data infrastructure at I14, including a fully automated processing pipeline capable of delivering meaningful reconstructions to users in near real time. This is a key enabler for efficient, high-throughput nanoscale electrochemical studies at a synchrotron facility.

References

[1] Quinn et al. J. Synchrotron Rad. (2021) 28, 1006–1013
[2] Parker et al. J. Synchrotron Rad. (2022) 29 1
[3] Van de Kerkhof et al. J. Microsc. (2025) 299:16–2

POSTER

X-ray Characterisation of Energy Materials at XMaS

Grunder, Y. (University of Liverpool)

XMaS [1] is a diffraction and spectroscopy beamline located at the European Synchrotron Radiation Facility (ESRF) in Grenoble since 1997. It is a designated EPSRC funded National Research Facility with the aim to provide the UK materials science community with access to a state of-the-art X-ray facility and to facilitate training for early career scientists, in advanced scientific methodologies. Although the beamline was originally designed for the exploration of magnetic materials using scattering techniques, it nowadays offers a wide range of x-ray techniques, ranging from diffraction over resonant and small angle scattering to spectroscopic methods (e.g. XAS, EXAFS). XMaS delivers X-ray characterisation methodologies across a range of temporal and spatial length scales and, by the development and use of novel sample environments, enables increasing use of operando studies enabling correlations between structure and functional properties to be determined.

This allows a broad range of scientific areas to be tackled including soft matter, surface electrochemistry, modern magnetic materials, conservation of historical artefacts and medical materials. XMaS has undergone an extensive upgrade through 2019 and 2020 to deliver a state-of-the-art facility that fully exploits the capabilities of the new upgraded ESRF machine. The new facility delivers a much brighter X-ray beam with an extended operational energy range which now extends to 40 keV thereby enabling new activities in materials research, particularly in terms of operando experiments.

The poster will present current capabilities of the beamline and show examples of in-situ/operando characterisation of energy materials and electrochemical systems at the beamline.

TALK

Investigating Oxide-Derived Catalysts for Electrochemical CO_2 Conversion to Fuels

Chen Han (presenter) and Erwin Reisner

Capturing CO_2 emissions and converting them into fuels is an attractive pathway to address greenhouse gas emissions and the energy crisis. Cu-based catalysts have demonstrated outstanding performance in the electrochemical CO_2 reduction reaction (CO_2RR), as Cu is the only known metal capable of producing multicarbon products from CO_2. However, these catalysts often undergo dynamic transformations under reaction conditions, posing challenges for understanding their intrinsic active sites and reaction mechanisms. In this work, we employ precursor engineering to obtain effective oxide-derived catalyst structures, enabling the tuning of selectivity between C_1 and C_2+ products and the selective production of ethylene with high performance. During this process, we use multiple synchrotron-based in situ characterization techniques to provide key insights into material evolution and reaction pathways, including hard X-ray spectroscopy, soft X-ray spectroscopy, infrared spectroscopy, and powder diffraction. These results help establish precursor structure–dynamic active site–performance relationships, benefiting the rational design of catalysts for CO_2RR and other electrochemical synthesis systems, and contributing to sustainable manufacturing.

POSTER

Electrocatalytic Strategies for Coupling Biomass Conversion with Clean Hydrogen Generation

Han, X. (University of Birmingham)

Electrocatalytic upgrading of biomass-derived molecules offers a promising route toward sustainable chemical manufacturing powered by renewable electricity. However, practical implementation is often limited by the persistent activity–stability trade-off in electrocatalysis: highly active sites frequently suffer from structural degradation, unstable intermediate binding, or loss of catalytic selectivity under operating conditions. Addressing this challenge requires catalyst motifs that can simultaneously promote interfacial charge transfer, optimise adsorption energetics, and preserve active-site integrity during reaction. Here, we report an electron-bridge strategy to balance activity and stability in electrocatalysis using layered double hydroxide catalysts as a model platform. In the anodic 5-hydroxymethylfurfural oxidation reaction, the electron-bridge motif enhances substrate adsorption and interfacial activation while stabilising catalytically relevant defect structures, leading to improved reaction kinetics, product selectivity, and operational durability. Density functional theory combined with in situ product analysis reveals that the electron bridges regulate local electronic structure and enable a more favourable reaction pathway. Importantly, the same electron-bridge framework also promotes the hydrogen evolution reaction by accelerating water dissociation and optimising hydrogen adsorption–desorption behaviour. When integrated into a coupled HER‖HMFOR electrolyser, the bifunctional catalyst delivers efficient hydrogen production and biomass upgrading at low cell voltage, high current density, and near-quantitative FDCA selectivity, together with excellent cycling stability. More broadly, this work establishes electron bridges as a general design principle for overcoming the activity–stability trade-off in electrocatalysis and advancing scalable electro-refinery systems.

POSTER

Electrochemical Etching of GaN: A Call for X-ray Based Mechanistic Insight

Harris-Lee, T. (University of Cambridge)

Gallium nitride (GaN) is a wide-bandgap (3.4 eV) III-V semiconductor with excellent thermal stability, chemical resistance, and electronic and optoelectronic properties, being widely used in efficient LED lighting and high-power transistors. More recently, porous GaN has emerged as a promising platform for overcoming key limitations associated with GaN-based devices, such as difficult strain management.[1] The availability of porous GaN architectures also broadens the range of possible applications of GaN, facilitating catalysis and sensing due to the high surface area, and composite formation by pore infiltration. Electrochemical etching (ECE) is a conductivity-selective process that can be applied to n-type GaN to produce porous GaN structures by an anodic electrochemical reaction (Equation 1).

$$2GaN + 6h^{+} \rightarrow 2Ga^{3+} + N_2 \quad (1)$$

Porous GaN research is still in its relative infancy, thus significant knowledge gaps limit the control, reproducibility, and scalability of porous nitride fabrication, preventing its integration into existing devices, which in turn prevents further research on how beneficial porous nitrides could actually be. For example, pore morphology within a porous architecture can be readily tuned empirically by a range of ECE conditions, most prominent are applied bias potential, electrolyte composition, and n-type GaN free carrier density.[2,3] However, the fundamental mechanistic steps of the process are not known and are often disputed. Fundamental questions concerning charge-transfer pathways, interfacial chemistry, transient oxide formation, dissolution mechanisms, and the unusual behaviour of threading dislocations remain unresolved. To date, no direct in situ measurements have been reported. Overcoming this barrier would remove a major bottleneck to the control and tuning of pore morphologies, thereby enabling the effective integration and evaluation of porous GaN within practical GaN-based device architectures, which currently is not possible. Advanced in situ and operando X-ray techniques offer a potential route to address these challenges by directly probing structural and chemical evolution during ECE. Such measurements would provide the first direct experimental insight into the mechanisms governing porous GaN formation and defect-selective electrochemical dissolution, which has the potential to enhance the performance of all nitride-based devices. This poster outlines the current mechanistic uncertainties surrounding GaN ECE and discusses opportunities for operando X-ray studies capable of linking electrochemical behaviour with real-time structural evolution in porous GaN systems.

References

[1] P. H. Griffin and R. A. Oliver, J Phys D Appl Phys, 2020, 53, 383002.
[2] T. R. Harris-Lee et al., ACS Applied Materials and Interfaces, 2025, 17, 64931-64941.
[3] J. Zhang et al., Journal of Applied Physics, 2026, 139, 175702.

INVITED TALK

High energy surface x-ray diffraction from OER model electrocatalysts at work

Hejral, U. (Chalmers University of Technology)

The oxygen evolution reaction (OER) is an essential reaction in several energy-related applications. These include the water splitting reaction for green hydrogen production, which is key in transforming various industrial sectors towards decarbonization. However, OER suffers from sluggish reaction kinetics which necessitates the use of a suitable catalyst. In alkaline electrolysis, Co- and Ni-based electrocatalysts have been found to be promising candidates [1,2] and are favoured due to their low cost and availability. When alloyed with Fe these catalysts show an improved catalyst performance [3,4]. However, to allow a rational design of more efficient electrocatalysts, the correlation between the alloy composition, the catalyst surface structure at the solid. liquid interface and the catalyst performance need to be understood.

In recent years we have shown how synchrotron-based High Energy Surface X-Ray Diffraction (HESXRD, E=70-80 keV) allows a fast probing of reciprocal space and the catalyst surface structure under operando conditions [5,6]. In this presentation I will show how it can be used to study the structural changes on Ni- and Co-based model catalyst as a function of Fe electrodeposition. Moreover, I will show that the technique allows for tracking potential-dependent 2D (oxy-)hydroxide structural changes at the solid/liquid interface [7].

References

[1] M.S. Burke et al. Chemistry of Materials, 27, 7549-7558 (2015).

[2] F. Dionigi et al, Nature Commun., 11, 2522 (2020).

[3] S. Anantharaj et al. Nano Energy, 80, 105514 (2021).

[4] M.S. Burke et al. J. Am. Chem. Soc., 137, 3638-3648 (2015).

[5] J. Gustafson et al. Science, 343, 758-761 (2014).

[6] U. Hejral et al. J. Phys.: Condens. Matter, 33, 073001 (2020).

[7] U. Hejral et al., submitted.

POSTER

Unravelling the role of additives in the structure of non-aqueous media at the electrode surface under potential control [1]

Hill, N. (University of Liverpool/ UKRI STFC)

The bulk performance metrics of electrochemical devices: product selection, selectivity and efficiency; are highly dependent on the micro-environments which exist within the electric double layer (EDL) and direct the reactions at the electrode interface.[2] In electrocatalysis, these microenvironments are perturbed by applying potential, driving reactions, but they can also be perturbed by introducing additives, influencing the interfacial structure, directing different reaction pathways and can improve performance.[3] The current understanding of the fundamental potential dependent interactions between all possible species at the electrode surface is limited. This makes a priori prediction of the EDL challenging and restrains rational design of tuned electrolyte mixtures for improved performance. The interactions of non-aqueous solvents are significantly different to water, which create vastly different microenvironments, leading to different electrochemical performance. Often non-aqueous solvents also have a wider electrochemical window and less Faradaic competition from solvent degradation, while some systems are sensitive to reaction with water which necessitates a non-aqueous solvent. Vibrational Sum Frequency Generation (VSFG) spectroscopy provides a tool for observing interface-specific vibrational signatures, identifying species, and following their relative orientation and net alignment. In this work, VSFG is used to track changes to the structure of the EDL at a thin film gold electrode interface under in-situ potential control. Acetonitrile with TBA PF6 is used as a model non-aqueous system and N-methyl-2-pyrrolidone (NMP) and water are introduced to study the effect of these additives on interfacial microenvironments. When applying small reductive potentials, the sensitivity of VSFG enables the identification of solvent breakdown products at sub monolayer coverage, at far less negative potentials than the onset of large bulk solvent reduction currents. At more negative potentials, these breakdown products become a significant part of the microenvironment. At low concentrations of H_2O (300 ppm), the acetonitrile VSFG features are weak and become much stronger as more H_2O is introduced. The acetonitrile structures are disrupted by the new hydrogen bonds created by the water, leading to greater net alignment at the interface. When a small amount of NMP is added to dry acetonitrile, NMP accumulates at the interface disrupting the acetonitrile structure.

References

[1] N. J. D. Hill, C. O'Brien, P. M. Donaldson, H. Jang, B. Siritanaratkul, G. Teobaldi, A.J. Cowan, A. M. Gardner, Faraday Discuss., 2026, Advance Article
[2] W. Schmickler Chem. Rev., 1996, 96, 3177 —3200
[3] A. M. Gardner, G. Neri, B. Siritanaratkul, H. Jang, K. H. Saeed, P. M. Donaldson, A.J. Cowan, J. Am. Chem. Soc., 2024, 146 , 7130 —7134

TALK

Operando Synchrotron XRD Investigation of Hydrogen Embrittlement in Micro-Forged Al-Zn-Mg-Cu Alloys

Jin, S. (KTH Royal Institute of Technology)

High-strength Al-Zn-Mg-Cu alloys are widely used in aerospace applications, where they are exposed to complex service environments involving mechanical loading, hydrogen, electrolytes, and pH variations. Micro-forging (MF), as an emerging surface strengthening technique, introduces residual stress and work hardening via mechanical hammering, thereby improving fatigue performance. However, it also involves complexity in the underlying mechanisms governing corrosion and hydrogen embrittlement. To establish a direct link between surface integrity (residual stress, work hardening, IMPs, oxide layers. etc.), and electrochemical behavior, this work combines multiple synchrotron-based techniques to investigate untreated (UT) and micro-forged (MF) samples under operando conditions. Operando high-energy X-ray diffraction (HEXRD) measurement at DESY P21 was performed under tensile loading and hydrogen-charging conditions to resolve strain distribution, dislocation density evolution, and H-affected near-surface region varying in-depth from the surface to the bulk. The results show that MF samples exhibit a reduced hydrogen penetration depth, indicating that the high density of dislocations introduced by micro-forging effectively traps hydrogen and limits its inward diffusion.

POSTER

Decoupling Proton Transport and Water Crossover: A Palladium Membrane Reactor for Sustainable Electrocarboxylation

Khan, S. (Delft University of Technology)

Adipic acid is a key industrial chemical used in nylon-6,6 production, yet its conventional production from fossil-based feedstocks results in significant CO_2 emissions. Electrocarboxylation, particularly the direct electrochemical dicarboxylation of 1,3-butadiene with CO_2 to produce 3-hexenedioic acid, a precursor of adipic acid, offers a sustainable alternative by utilizing waste CO_2 and renewable electricity under ambient conditions. However, practical implementation is limited by the reliance on sacrificial metal anodes and the lack of membranes compatible with organic electrolytes. In this work, a novel electrochemical reactor design integrating a proton-selective palladium membrane with a non-sacrificial anodic oxidation reaction is investigated. The palladium membrane enables controlled proton transport from an aqueous anolyte to an organic catholyte while preventing water crossover, thereby enabling stable operation in organic media. This approach eliminates sacrificial anodes and establishes a sustainable platform for electrocarboxylation and broader electrosynthesis applications.

POSTER

Kohler, C. (University of Oxford)

Lithium-ion batteries (LiBs) are a complex, interconnected system where relatively minor alterations to the components can trigger a cascade of reactions. These effects are strongly dependent on the choice of battery materials and operating conditions. Identifying the key degradation mechanisms, the interplay between these, and their resulting impact on cycle life is crucial in developing improved LiBs. The state-of-charge (SOC) of a LiB can induce different degradation mechanisms, such as electrolyte oxidation and transition metal dissolution. Therefore, incorporation of emerging cathode materials requires a detailed understanding of the degradation mechanisms that can be triggered in order to better predict and help to mitigate capacity fade. I will present my recent work on SOC interval ageing of the lithium manganese iron phosphate (LMFP)/Graphite system. SOC interval ageing is a methodology to replicate long-term ageing by cycling over a limited SOC at a high C-rate. This is able to mirror long-term full cycle degradation in a shorter time. LMFP is an emerging low-cost cathode material composed of earth-abundant transition metals, but with improved energy density over LFP due to its higher average discharge voltage. However, the higher operating voltage from the Mn plateau increases the risk electrolyte oxidation and Mn dissolution, raising questions about the extent of degradation during long-term cycling and the impact on the SEI formed at the graphite anode. SOC interval ageing enables the isolation of different potential regions of LMFP, in this research we focus on the Fe plateau, Mn plateau and the transition between plateaus. To determine the degradation mechanisms that dominate in these SOC regions, the changes in the electrode interphases and electrolyte composition are carefully characterised. Depth-resolved X-ray photoelectron spectroscopy and soft X-ray absorption spectroscopy provide a surface-sensitive account of the composition and chemical state of the SEI, revealing the influence of Mn crossover, including the extent to which Mn is incorporated into the anode and whether it is redox active. NMR, inductively coupled plasma mass spectrometry (ICP-MS), and Fluoride-ion selective electrode measurements of extracted electrolyte further reveal electrolyte degradation and crossover products. This complementary characterisation approach demonstrates the interconnection between the interphases, electrolyte and SOC degradation. As LMFP becomes a more prevalent chemistry, it is essential to recognize how its potential influences SEI degradation, especially when under conditions of stress in demanding applications. This approach of isolating small SOC regions should provide a greater understanding of the systems degradation mechanisms.

TALK

The Topographic Origin of Chemomechanical Failure in High-Nickel Cathodes Revealed by Operando Dark-Field X-ray Microscopy

Le Houx, J. (University of Greenwich)

The cycle life of high-energy solid-state batteries is significantly limited by the mechanical degradation of high-nickel cathodes (NMC 811), driven by anisotropic lattice volume changes during delithiation. While macroscopic failure modes such as intergranular cracking are well documented, the nanoscale origins of strain localisation within individual primary particles have remained unresolved due to a fundamental trade-off in conventional characterisation: electron microscopy offers spatial resolution but alters the stress state through destructive thinning, while traditional X-ray diffraction methods preserve the bulk environment but average over millions of particles. To bridge this gap, we report the first application of operando dark-field X-ray microscopy (DFXM) to a battery system, leveraging beamline ID03 at the European Synchrotron Radiation Facility. By placing an objective lens in the diffracted beam path, DFXM allows us to spatially resolve the internal lattice evolution of single NMC 811 primary particles under realistic mechanical boundary conditions during electrochemical operation. During operando cycling, we directly observe the nucleation of a Geometrically Necessary Dislocation (GND) wall originating from a pre-existing surface groove at 3.9 V. Quantitative analysis reveals that this surface feature creates a localised stress concentration of approximately 100 MPa, exceeding the material's critical resolved shear strength (~78 MPa) and triggering plasticity, while the bulk particle remains elastic. The resulting dislocation wall partitions the crystal into two kinematic domains separated by a discrete lattice rotation of 0.049 degrees, acting as a high-impedance barrier to lithium transport. In contrast, a spherical control particle cycled to a higher voltage of 4.4 V accommodated the strain entirely elastically. Supported by finite element analysis, we identify a critical surface roughness threshold of approximately 125 nm, above which the plastic failure volume increases 40-fold. These findings provide direct evidence that chemomechanical failure is deterministically governed by surface topography rather than occurring as a stochastic bulk phenomenon. Ultimately, this work demonstrates that in single-crystal architectures, eliminating internal grain boundaries redirects strain energy toward surface features. From an industrial perspective, we propose a new, actionable quality assurance metric for cathode manufacturing: morphological metrology of particle surface roughness against the 125 nm threshold. Implementing these design rules at the powder stage offers a deterministic pathway to grade cathode materials for high-stress applications, reducing downstream cell failure rates in gigafactory scale-ups.

TALK

Operando Investigations into Nickel-based Electrocatalysts for Green Hydrogen Production in Alkaline Anion Exchange Membrane Environment

Roxy Lee,[1] Mathias Hommelgaard,[1] Prajna Bhatt,[2] Mark Isaacs,[3] Lisa Seidl,[1] Yang Hu[1] and Christodoulos Chatzichristodoulou[1]

[1]Technical University of Denmark (DTU), Department of Energy and Storage Fysikvej, Building 310, 2800 Kgs. Lyngby
[2]Consiglio Nazionale delle Ricerche (CNR) Basovizza, 320, 34149 Trieste TS, Italy
[3]Harwell XPS, the UK National Facility for X-ray Photoelectron Spectroscopy Research Complex at Harwell, Rutherford Appleton Labs, Harwell Campus, Didcot, OX11 0FA, UK

Anion exchange membrane water electrolysis (AEMWE) is an emerging technology to produce green hydrogen, which is a major component of a fossil fuel independent future. This technology uses an AEM as a solid polymer electrolyte and non-precious transition metal catalysts such as nickel-based electrocatalysts.[1] Operando soft X-ray spectroscopic methods (XPS and XAS) using an ion exchange membrane (IEM) cell have proven a valuable tool to probe catalysts in this type of environment.[2-4]

In this work we have used two different operando spectro-electrochemical cells - one designed at BESSY-II (ISISS beamline) [2] and one at Diamond Light Source (B07 beamline)[4] with modifications of the membrane electrode assembly (MEA) and current collector. This enabled us to explore potential dependent surface species in Ni-based catalysts under operating conditions for a variety of MEA designs.

References

[1] Li, Q. et al. Journal of Physical Chemistry C 2023, 127 (17), 7901–7912.
[2] Mom, R. et al. J Am Chem Soc 2019, 141, 6537–6544.
[3] Mom, R. V. et al. ACS Catal 2022, 12 (9), 5174–5184.
[4] Kumar, S. et al. JPhys Energy 2024, 6 (3), 036001

POSTER

Operando XAS investigation of Size-Selected Cobalt Alloy Nanoparticles for alkaline OER

Li, S. (University of Oxford)

Understanding the dynamic structural evolution of transition metal electrocatalysts under operating conditions is essential for improving the efficiency of the oxygen evolution reaction (OER) in alkaline water electrolysis. Cobalt-based alloy nanoparticles are promising candidates for anion exchange membrane (AEM) electrolysers, yet the nature of their catalytic surface during OER remains poorly understood. Here we apply operando X-ray absorption spectroscopy (XAS) to follow the electronic structure and coordination environment of size-selected nanoparticles in situ, using element-specific edges to track each element independently as the reaction proceeds.

Size selection resolves a core–shell electronic structure in the electrolyte: a disordered, high-spin (HS) Co^{3+} surface surrounding a low-spin (LS) Co^{3+} core. Under oxidising potential, Co^{4+} forms regardless of particle size, and this change reverses completely as the potential is swept back down. HERFD-XAS supports this picture, with the pre-edge showing a distinct change in the non-local dipole peak.

Building on these findings for monometallic Co catalysts, we then examine the effect of alloying with synergistic elements such as Fe. Alloying with Fe markedly enhances OER activity. Operando Fe L-edge measurements show no change across the oxidation reaction, suggesting that Co is the active site instead of Fe. STEM and EELS mapping reveal that, after OER, a layered CoFe oxy-hydroxide forms irreversibly. It appears more ordered than the amorphous layer that develops on pure Co nanoparticles, pointing to an Fe-induced structural improvement of the active surface.

Combining size selection with element-specific operando XAS allows the surface and core of cobalt nanoparticles to be distinguished. This suggests that Fe enhances activity not by acting as a redox-active site but by segregating to the surface and templating a more ordered, layered CoFe oxy-hydroxide, linking improved performance to structural rather than purely electronic changes. These results identify surface order as a key design parameter for cobalt-based alloy catalysts in alkaline OER.

In-Situ and Operando Cell Environments for Reliable Synchrotron Studies of Batteries

Li, Z. (Imperial College London)

Synchrotron-based in-situ and operando methods have transformed battery research by allowing structural, chemical, interfacial, and spatial changes to be followed during operation. Reliable measurements, however, require more than advanced beamlines, detectors, or analysis. They require cell environments that preserve realistic battery behavior while making the target reaction accessible to the X-ray probe. This challenge is growing as battery research moves from lithium-ion to beyond lithium-ion chemistries, from liquid electrolytes to solid-state batteries, from intercalation to conversion reactions, and from sealed cells to flow-based architectures. This talk will examine operando cell environments as a bridge between electrochemical and X-ray measurement requirements. Early X-ray-transparent coin, pouch, capillary, and tubular cells enabled access to bulk phase evolution, redox processes, and reaction pathways in working batteries. Later fit-for-purpose and radially accessible designs improved compatibility with diffraction, total scattering, X-ray absorption spectroscopy, and imaging. Recent developments have extended studies toward realistic pouch and full-cell formats, spatial mapping, soft X-ray spectroscopy, grazing incidence scattering, X-ray reflectivity, and thin-electrolyte or flow-based environments for buried interfaces. From the electrochemistry side, the cell must maintain representative electrode loading, electrolyte wetting, current collection, stack pressure, sealing, gas and liquid management, temperature, and cycling protocol. From the synchrotron side, it must provide a suitable X-ray path, stable frame and window materials, detector access, low background, controlled beam exposure, a defined probed volume, and stable alignment. These requirements are inseparable: the cell environment defines both the reaction being studied and the signal being interpreted. The talk will also discuss underreported reasons why measurements fail or become misleading, including pressure loss, electrolyte redistribution, window deformation, gas accumulation, beam-induced chemistry, background artifacts, and mismatch between probed and active regions. A central challenge is that future operando battery cells should move beyond X-ray transparency toward joint design of the electrochemical cell and the X-ray measurement. Early collaboration between electrochemists and beamline scientists is essential, so that the target reaction, battery chemistry, X-ray geometry, dose limit, likely failure modes, and essential records are defined before beamtime. As synchrotron facilities move toward brighter beams, higher coherence, faster detectors, and combined scattering, spectroscopy, and imaging, cell design must become more precise rather than more permissive. This approach aims to make operando synchrotron studies of batteries reliable, reproducible, and interpretable.

POSTER

Revealing passive film evolution on FeMnCrNi medium entropy alloy under anodic polarization by in situ ambient pressure X-ray photoelectron spectroscopy

Liu, M. (KTH Royal Institute of Technology)

The occurrence of pitting corrosion is accompanied by the evolution of the passive film, yet existing studies are constrained by limited in situ characterization capabilities and demanding experimental conditions. Using in situ ambient-pressure X-ray photoelectron spectroscopy (AP-XPS) at the HIPPIE beamline of the MAX IV Laboratory (Lund, Sweden), we elucidate the evolution of the surface passive film on a FeMnCrNi medium-entropy alloy (MEA) during stepwise anodic polarization. Within the passive region, anodic polarization primarily increases the hydroxide layer thickness, raising the Cr^{3+} hydroxide component in the hydroxide layer and the Fe^{3+} oxide component in the oxide layer. When polarized at 700 mV (vs. Ag/AgCl) beyond the pitting corrosion potential, the Ni^{2+} hydroxide content in the hydroxide layer increases markedly, and the Mn^{6+} oxide component emerges. At 1000 mV (vs. Ag/AgCl), the oxide layer disappears. The atomic fraction of Fe metal in the near-surface region becomes substantially lower than that in the matrix, and Mn^{6+} oxide is further converted to Mn^{4+} oxide. This work provides a new insight into the dynamic evolution of the passive film on MEA during anodic polarization.

TALK

Potential-driven irreversible restructuring of Cu-N-C electrocatalysts

Liu, L. (University of Oxford)

Copper-nitrogen-doped carbon (Cu-N-C) catalysts featuring atomically dispersed Cu sites have attracted considerable attention for electrochemical conversion reactions, including the carbon dioxide reduction reaction (CO_2RR) and nitrate reduction reaction (NO_3RR). However, their structural instability under negative potentials, involving dynamic dissolution and redeposition, complicates the identification of active sites and obscures mechanistic understanding. Herein, we employ a well-defined copper porphyrin complex supported on carbon black as a model Cu-N-C electrocatalyst to investigate its dynamic evolution under operating conditions during NO_3RR, with the hydrogen evolution reaction (HER) as a control. A combination of multimodal spectroscopic techniques, including in situ Cu K-edge X-ray absorption spectroscopy (XAS), Raman spectroscopy, and infrared spectroscopy, was used to monitor structural transformations in real time. Notably, quick XAS measurements were conducted under cyclic voltammetry (CV) to probe potential-dependent restructuring with temporal resolution, enabling correlation between structural evolution kinetics and electrochemical behavior beyond conventional potentiostatic methods. Operando Cu K-edge XAS reveals that catalytic activity correlates with the formation of metallic Cu species, indicating that these species serve as the active sites for both NO_3RR and HER. Furthermore, analysis of XAS spectral intensity evolution captures the dissolution-redeposition process, while complementary Raman and infrared measurements confirm the irreversible nature of the restructuring. This work demonstrates a methodological advance in operando spectroscopic analysis and provides critical mechanistic insights into the dynamic behaviour of Cu-N-C catalysts under cathodic reaction conditions.

TALK

Advanced Characterisation of M-N-C Electrocatalysts

Lyth, S. (University of Strathclyde)

Polymer electrolyte fuel cells (PEFCs) will perform the crucial role of energy conversion once society shifts towards the hydrogen economy. At the anode, hydrogen molecules spontaneously react to generate protons and electrons, whilst the cathode oxygen combines with the protons and electrons to spontaneously form water. The difference in Gibbs free energy between these two reactions generates a potential difference, which drives protons through an electrolyte membrane and electrons around the external circuit, and is registered as a voltage. However, both the hydrogen oxidation reaction (HOR) and the oxygen reduction reaction (ORR) take place over a finely dispersed platinum catalyst on a carbon black support. Platinum is a critical raw material (CRM) and as such is susceptible to future shocks, such as bottlenecks in supply or volatile prices. As such, it is imperative to investigate alternative platinum-free electrocatalysts. In particular, the cathode requires around 10 times more platinum than the anode due to the more sluggish nature of the ORR. Over the past decade we have developed platinum-free M-N-C electrocatalysts with unique properties and microstructure. The basis for our catalysts is a nitrogen-doped carbon foam, which is obtained by thermal decomposition of metal alkoxides. The materials are exceptional in this field due to their very large macropores which aid gas diffusion, the very large surface area due to microporosity, and the fact that the nitrogen content can be routinely varied. Using these electrocatalysts we have achieved high activity and excellent durability in both acid and alkaline media. One of the advantages of this system is that it can be applied as a model catalyst to independently study the effect of different parameters on catalytic activity. As such, we have made important insights into the synthesis mechanisms of M-N-C electrocatalysts using synchrotron techniques such as in situ X-ray absorption spectroscopy (XAS).

Investigating the Role of Crystal Phase Driven Surface and Bulk Reconstruction in Triggering Lattice Oxygen-Mediated Oxygen Evolution in Perovskite Oxides

Maitra, S. (University of Oxford)

The oxygen evolution reaction (OER) is the key bottleneck in water electrolysis because of its sluggish four-electron process and high overpotential. Conventional electrocatalysts follow the adsorbate evolution mechanism (AEM), where OH* and OOH* scaling limits the minimum overpotential to ~370 mV. The lattice-oxygen-mediated mechanism (LOM) can bypass this limit by involving lattice oxygen, but the structural origins of LOM activity and the roles of surface and bulk redox remain unclear. Here, we investigate the fundamental role of crystal phase in driving LOM activity in cobalt-based perovskites using phase-pure, dopant-free $SrCoO_3$ (SCO), namely the tetragonal, orthorhombic and hexagonal phases, and compare it with a standard AEM catalyst, $LaCoO_3$ (LCO). While doping can enhance SCO activity according to previous literature, often dopant-induced phase transitions from hexagonal or orthorhombic to tetragonal or cubic phases of SCO are also observed, which appear to be overlooked while attempting to discern the cause of LOM activity. Electrochemical measurements show that tetragonal SCO inherently has much higher OER activity than hexagonal and orthorhombic SCO, demonstrating that crystal phase alone strongly influences LOM. To identify the origin of this phase dependence, we combine electrochemistry with depth-sensitive X-ray spectroscopy techniques and spatially resolved electron microscopy. Soft X-ray absorption spectroscopy in TEY and FY modes reveals higher Co oxidation states, Co-O rehybridization, and molecular oxygen-like features associated with anionic redox. Bulk-sensitive X-ray Raman scattering shows that after OER the O K-edge of hexagonal and orthorhombic SCO remains largely unchanged, whereas tetragonal SCO develops features linked to ligand holes and molecular oxygen, together with increased Co oxidation. HERFD Co K-edge XANES reveals increasing Co-O covalency from LCO to tetragonal SCO, consistent with the OER trend. Operando HERFD shows that tetragonal and orthorhombic SCO undergo bulk reconstruction during OER, while hexagonal SCO is mainly surface-limited. EXAFS, X-ray diffraction, and cross-sectional STEM-EELS/EDX/NBD confirm changes in local coordination, crystal structure, oxidation state, stoichiometry, and phase distribution. Overall, tetragonal SCO has stronger intrinsic Co-O covalency and uniquely promotes bulk redox during OER. It undergoes Sr out-diffusion and transforms into partially amorphized, Co-rich phases with a higher bulk Co oxidation state, whereas hexagonal SCO and LCO remain largely surface-limited. These results establish crystal-phase engineering as an effective strategy to activate lattice oxygen and bulk participation in OER electrocatalysts, enabling OER reactivity beyond the catalyst surface.

POSTER

Transitional forms in MCFC as key factors in CO_2 processing

Majewska, K. (Warsaw University of Technology)

Charge transport in molten carbonate electrochemical systems is conventionally attributed exclusively to carbonate ions. However, under high-temperature conditions and in the presence of water vapor, the formation of protonic species may introduce additional conduction pathways. The present study investigates the occurrence of protonic conduction in molten carbonate-based systems operated in the temperature range of 500–650 °C under controlled gas compositions. Particular attention is given to the role of water vapor in the formation of intermediate species, including bicarbonates, and their potential contribution to charge transport. The influence of operating conditions on transient chemical equilibria and ionic conductivity is systematically analyzed to distinguish between carbonate-dominated and proton-assisted mechanisms. The results indicate the presence of mixed ionic conduction, suggesting that proton-related transport may play a non-negligible role under specific conditions. These findings challenge the classical assumption of purely carbonate-ion conductivity in molten carbonate systems and provide new insight into the physicochemical processes governing their operation. From an application perspective, the identification of protonic contributions to charge transport is relevant for the optimization of molten carbonate technologies for CO_2 separation and capture. Improved understanding of these mechanisms may support the development of more efficient and stable electrochemical systems, with potential future applicability in integrated CO_2 utilization pathways, including synthetic fuel production.

POSTER

Understanding the Role of $NiCoO_x$ Mass Loading in Enhancing Oxygen Evolution Kinetics for AEM Water Electrolysis

Mishra, K. (Department of Physics, Indian Institute of Technology-BHU)

With the rising concern over CO_2 emissions and their severe environmental impact, transitioning from fossil fuels to clean energy from renewable sources has become imperative. Hydrogen production via electrocatalytic and photoelectrocatalytic water splitting offers a sustainable pathway for clean energy conversion and environmental remediation. Among various approaches, Anion Exchange Membrane (AEM) water electrolysis has emerged as a promising and cost-effective technology, particularly due to its compatibility with non-precious metal oxide catalysts. In water electrolysis, the Oxygen Evolution Reaction (OER) at the anode poses challenges due to its sluggish kinetics and high overpotential requirements, limiting large-scale hydrogen production. A valid approach for accelerating the OER process is developing advanced electrocatalysts with enhanced kinetics and reduced overpotentials. This study investigates the influence of varying $NiCoO_x$ (NCO) mass loadings on the anode's catalytic performance and stability in Anion Exchange Membrane (AEM) water electrolyzers. The NCO catalyst was synthesized in a 1:1 ratio using the hydrothermal method. Following successful synthesis, catalyst ink was prepared and coated onto a nickel foam gas diffusion layer (GDL) with loadings of 2 mg/cm², 4 mg/cm², and 6 mg/cm². The performance of NCO as an anode catalyst was tested in an AEM water electrolyzer across a temperature range of 25–70 °C using a 1 M KOH electrolyte. At 70 °C and 1.8 V, current densities of 260, 220, and 260 mA cm^{-2} were recorded for 2, 4, and 6 mg cm^{-2} loadings, respectively. The electrochemical measurements were further conducted to identify the optimal catalyst loading for achieving the best performance. The 2 mg cm^{-2} catalyst loading demonstrated the most favourable OER performance, achieving a low overpotential of 320 mV at 10 mA cm^{-2} and a Tafel slope of 112.35 mV dec^{-1}, supported by its highest Electrochemical active surface area (ECSA) and lowest charge-transfer resistance from EIS analysis. The pre- and post- electrochemical XPS analysis of different mass loadings also supported these results. The durability test of 2 mg cm^{-2} loading over ~70 h showed no significant change in OER activity, confirming the robustness, long-term durability, and excellent stability under harsh operational conditions. Overall, the results underscore that optimizing catalyst mass loading plays a pivotal role in maximizing active surface area and minimizing charge-transfer losses, offering a rational design strategy for high-efficiency, durable, and low-cost AEM water electrolyzers.

POSTER

Deconvoluting the mechanism of ethylene glycol electro-oxidation towards glycolic acid on gold catalysts using Operando-XAS

Mo, T. (Imperial College London)

Hydrogen peroxide may emerge as a by-product within proton exchange membrane fuel cells (PEMFCs) and proton exchange membrane water electrolysers (PEMWEs). Its formation can occur at the electrode catalyst—specifically, at the cathode in PEMFCs and the anode in PEMWEs—or originate from gas crossover, whereby oxygen permeates the membrane to the hydrogen side and, to a lesser extent, hydrogen migrates toward the oxygen side. The presence of peroxide is a concern due to its potential to decompose reductively, leading to the creation of highly reactive hydroxyl and peroxyl radicals. These species can compromise critical device components such as the membrane, catalyst, and gas diffusion layer, thereby diminishing both operational efficiency and service life. In this study, the authors introduce a novel real-time, in-situ approach for peroxide detection that integrates a high mass-transport gas-diffusion electrode with an ultra-microelectrode. This configuration facilitates accurate measurement of peroxide generation under practical operating conditions for fuel cells and electrolyzers, encompassing hydrogen/oxygen reactant environments, and controlled electrochemical potentials. Furthermore, the system demonstrates robust performance even in the presence of hydrogen interference, underscoring its applicability for simulating gas crossover scenarios.

VITED TALK

Three-Dimensional Soft-Templated Nanoarchitectures for Enhanced Catalytic Activity

Nandhakumar, I. [1*] (University of Southampton)

M. Burton [1], J. White [1], S. Akbar [1], A. Squires [4] N. Terrill [2], D. Alba-Venero [3,2]

[1] Department of Chemistry, University of Southampton, Southampton SO17 1BJ, UK.

[2] Diamond Light Source, Diamond House, Harwell Science and Innovation Campus, Didcot, Oxfordshire OX11 0DE, UK

[3]. ISIS Neutron and Muon Source, Rutherford Appleton Laboratory, Didcot OX11 0QX, U.K

[4] Department of Chemistry, University of Bath, South Building, Soldier Down Ln, Claverton Down, Bath, UK

The emergence of synthetic strategies and fabrication technologies that afford control over the architecture of matter at nanometer length scales has provided a new tool kit for engineering functionality in materials. In particular the three-dimensional (3D) nanostructuring of metals and semiconductors has resulted in enhanced optical, magnetic and electronic properties yielding new devices and device concepts. A synthetically attractive route to their preparation is electrochemical templating, which involves electrodeposition at the surface of an electrode around or through a pre-existing soft template. In particular liquid crystal templating using lyotropic phases as soft templates has been shown to be an effective technique for the production of a range of nanostructured materials [1-4] and is applicable to both electrochemical and chemical fabrication methods. Lyotropic liquid crystalline phases can adopt a wide variety of geometries depending on whether the surfactant-water interface curves away or towards the water and are classified as adopting either “normal” or “inverse” topologies. The templated materials are characterised by high interfacial surface areas and nanometric length scales which makes them attractive for many technological applications. Results of recent studies on palladium (Pd) [5] and platinum (Pt) [6-7] demonstrate that this methodology is suitable for producing 3D nanoarchitectures for enhanced catalytic activity superior to commercial Pd black or Pt/C catalysts.

References

[1] M. R.Burton, S.J. Richardson, P.A. Staniec, N.J. Terrill, J.M. Elliott, A.M. Squires, N.M. White and I.S. Nandhakumar, Electrochem. Comm 76, 71-74 (2017)

[2] M. R.Burton, S.J. Richardson, P.A. Staniec, N.J. Terrill, J.M. Elliott, A.M. Squires, N.M. White and I.S. Nandhakumar, Electrochem. Comm 76, 71-74 (2017).

[3] M.R. Burton, C.Lei, P.A. Staniec, N.J. Terrill, A.M. Squires and I.Nandhakumar, Nature Scientific Reports 7, 6405 (2017).

[4] M. Burton, J.M. Elliott, N. Terrill, S. Richardson, I.Nandhakumar, A. Squires, Nanoscale 9, 10227 (2017).

[5] M.Burton, A. Selvam, J. Lawrie-Ashton, N.J. Terrill, A.M. Squires, and I. Nandhakumar, ACS Applied Materials & Interfaces, 10 (43), 37087-37094 (2019).

[6] Joshua S. White, Samina Akbar, A.Squires, D. Alba-Venero, N. J. Terrill and Iris Nandhakumar, J. Colloids & Interfacial Science 704, 139470 (2026).

[7] J.S. White, W. Liu, S.C. Perry, S. Akbar, D. Alba-Venero, NJ Terrill, A. Squires, I.S. Nandhakumar, ACS omega 10 (8), 8082-8088 (2025).

TALK

Metal-Quinizarin Coordination Complexes as OER Electrocatalysts: Structural Insights from Synchrotron Characterisation

Rafia Nimal[1], Anna Warren[2], David Madden[1], Alexandros Terzopoulos[1], Stuart Clarke[1]

[1] Yusuf Hamied Department of Chemistry, University of Cambridge, Cambridge CB2 1EW, UK

[2] Diamond Light Source, Harwell Science and Innovation Campus, Didcot, Oxfordshire OX11 0DE, UK

The oxygen evolution reaction (OER) remains the primary kinetic bottleneck in electrochemical water splitting for green hydrogen production. Noble-metal catalysts (IrO_2, RuO_2) dominate commercial applications but are scarce and costly. Earth-abundant transition metal alternatives have been extensively explored, yet most require high-temperature synthesis and additional conducting binders, adding complexity and cost to electrode fabrication. We report a new class of metal–quinizarin coordination complex (MQ) electrocatalysts synthesised at low temperature (80°C) without binders or conducting additives. The nickel analogue (NiQ) was characterised by single-crystal X-ray diffraction at the Diamond Light Source VMXm microfocus/ nanofocus beamline, revealing discrete NiQ_2 units with a distorted octahedral coordination environment: two bidentate quinizarin ligands occupying equatorial positions and two axial aqua ligands. This crystallographically confirmed structure contrasts with prior reports of polymeric metal–quinizarin frameworks and establishes the molecular basis for catalytic activity. The iron analogue (FeQ) was characterised by XANES/EXAFS at the B18 beamline, confirming Fe(III) oxidation state and a distorted octahedral coordination fully consistent with the NiQ crystal structure, with the active catalyst retaining the organic ligand under OER conditions as confirmed by TOF-SIMS. NiQ@FTO achieves an overpotential of 300 mV at 10 mA cm^{-2} in alkaline media with exceptional stability over 5000 CV cycles. FeQ@FTO exhibits an overpotential of 237 mV and a Tafel slope of 38.8 mV dec^{-1}, among the lowest reported for iron-based coordination complex catalysts. Critically, MQ catalysts has been translated beyond laboratory conditions to an electrochemical flow cell operating at industrial parameters: 7 M KOH electrolyte, 70–80°C, and current densities of 100–1200 mA cm^{-2} on Ni mesh substrates, with accelerated stability testing equivalent to five years of operation. DFT calculations at the TPSSh-D4/def2-TZVPP/CPCM(H_2O) level have been used to investigate three candidate OER mechanisms and identify the active catalytic species. The work is supported by a UK patent (GB2414751.4), EPSRC IAA Follow-on funding, and a MATcelerateZero award targeting commercial deployment. This talk will present the structural basis for activity in the MQ catalyst family, the mechanistic insights from synchrotron and DFT studies.

POSTER

Computational Modelling of Electrode-Electrolyte Interfaces, for Applications in Catalysis and Spectroscopy Support

O'Brien, C. (University of Liverpool / STFC)

Electrochemical interfaces host complex reaction dynamics across the electric double layer (EDL), within which molecules experience highly variable local potentials and field-dependent effects. Mechanistic understanding of electrocatalysis at the electrode/electrolyte interface is critical to optimising the structure and performance of catalysts for energy applications such as CO_2 reduction. My PhD project involves the construction of computational models which build up a sufficiently robust approximation of the EDL to inform the interpretation of interface-specific observables: specifically, those captured using nonlinear vibrational spectroscopy experiments at the Central Laser Facility and the University of Liverpool. Thus far, this work has included field-dependent response studies of organic solvents, cofactors and organometallic precatalysts, DFT-driven ab initio molecular dynamics simulations of simplified interface models, and electronic structure analyses of the cation-mediated CO_2 reduction reaction. At present, the project is focussed on the scale-up of existing dynamical models using Machine-Learned Interatomic Potentials and further dynamics studies incorporating external potentiostatting to simulate charged electrodes; this poster will summarise highlights of my PhD research to-date.

POSTER

Electrochemical Nitrogen Reduction Reaction using a Dual Phase Cell

Pickston, J. (University of Salford)

Ammonia is a key industrial chemical used in the agriculture sector and energy storage sector. The current Haber Bosch process is estimated to release 1-3% of global CO_2 emissions and 1-2 % of global energy supplies, thus a green alternative is highly sought after. The research herein focuses on heterogenous transition metal catalysts that double up as hydrogen transport membranes in order to synthesise ammonia by electrochemically splitting water on one side and transporting H across to react with gas phase nitrogen. This work focuses on better understanding the mechanisms of nitrogen adsorption/desorption, hydrogen permeation and ammonia synthesis on these surfaces under cathodic conditions in order to evaluate the limiting factors of these catalysts and provide insight into how they can be improved for nitrogen activation and hydrogen adsorption.

TALK

The electrochemical oxidation of Pt nanocatalysts – an operando X-ray scattering study

Punke, S. (University of Copenhagen)

S. Punke[1], R. K. Pittkowski[1], M. Arenz[2], K. M. Ø. Jensen[1]

[1]Department of Chemistry and Nano-Science Center, University of Copenhagen, 2100 Copenhagen, Denmark,
[2]Department of Chemistry, Biochemistry and Pharmaceutical Sciences, University of Bern, 3012 Bern, Switzerland

Pt nanoparticles are the state-of-the-art catalyst for proton exchange membrane fuel cells (PEMFCs), yet their long-term stability remains limited. [1] A major degradation pathway is metal dissolution, which predominantly occurs during transient oxide formation and reduction. While this oxide formation has been extensively studied on Pt single crystals using techniques such as high-energy surface X-ray diffraction, significantly less is known about the oxide formation on Pt nanoparticles. [2–5] Here, we investigate the oxidation of commercial Pt/C nanoparticles under realistic conditions by combining quasi-simultaneous operando small-angle X-ray scattering (SAXS), X-ray diffraction (XRD), and total scattering with pair distribution function (PDF) analysis in a custom-made operando electrochemical cell. [6] These complementary techniques enable us to follow the morphological and structural evolution across multiple length scales, from nanoparticle morphology to local atomic structure, during electrochemical oxidation. From the features observed in the PDF at 1.3 VRHE, we fingerprint the structural motif of the oxide species forming on the Pt nanoparticle surface and track the growth of this oxide layer by correlating particle size evolution with changes in crystallinity and local structural features.

References

[1] D. A. Cullen, et al. Nature Energy (2021). 6 (5), 462-474.
[2] T. Fuchs, et al. Nature Catalysis (2020). 3, 754-761.
[3] T. Fuchs, et al. Angewandte Chemie International Edition (2023). 62, e202304293.
[4] C. A. Campos-Roldán, et al. Nature Communications (2025). 16, 936.
[5] S. Cherevko, N. Kulyk, K. J. J. Mayrhofer, Nano Energy (2016). 29, 275-298.
[6] G. K. H. Wiberg, R. K. Pittkowski, S. Punke, O. Aalling-Frederiksen, N. Schlegel, K. Jensen, M. Arenz, Chimia (2024). 78 (10), 682.

TALK

Synchrotron Radiation Enabled FTIR Microspectroscopy for Operando Study of Ca^{2+} Storage Mechanism in Poly(tetra-sulfonamide) based Coordination Polymers

Purkait, T. (Institute of Condensed Matter and Nanosciences (IMCN), Université catholique de Louvain, Belgium).

The development of sustainable, high energy-density battery chemistries is critical for the future of energy storage. Among the most promising alternatives to Li-ion batteries are multivalent systems (Ca, Mg). Advances in positive electrode materials for such emerging systems, remain limited due to the strong interactions between divalent cations and rigid inorganic hosts. A new type of coordination polymer cathode, developed at UCLouvain, based on poly(tetra-sulfonamide) (PTtSA) chemistry, offer superior electrochemical performance in multivalent systems, including enhanced energy density, reversibility, and rate capability. Apparently, their high operating volatage (3.24 V vs. Ca^{2+}/Ca for the Ca-Zn-PTtSA) is attributed to the synergistic effect of the intrinsically high redox potential of the sulfonamide functionality and the inductive polarization effect of the nodal metal (Zn); an essential yet poorly understood mechanism. In-depth mechanistic studies were needed to fully grasp the processes of divalent ion storage and transport in these electrodes. Initial lab-scale ex-situ FTIR measurements revealed identical vibrational features in pristine and cycled electrodes. However, it was crucial to gain deeper insights into the underlying electrochemical mechanism, especially to understand how the cation storage proceeds in real time. Hence, in this work, we employed an operando Synchrotron based Fourier transform infrared (SR-FTIR) spectroscopic technique (MIRAS beamline (BL-01), ALBA Synchrotron) to monitor the change in the vibrational fingerprints of these cathode materials at various state-of-charge. We were able to systematically observe the redox mechanism of the PTtSA ligand, through disappearance and reappearance of characteristic vibration bands during real-time battery operation. Interestingly, the absence of any new spectral features or changes around the characteristic solvent bands strongly indicates no solvent uptake by the incoming cation. This suggests that the organic host accommodates essentially "naked" Ca^{2+} ions, without co-inserted anions or solvated species, which is a rare feature in divalent cation storage. Additionally, a sophisticated technique like, SR-FTIR microspectroscopy allowed the access of a microscope, where wider region of the electrode could be investigated via mapping, providing insights into homogeneity of species distribution and reactivity in the bulk of the electrode. Furthermore, we also explored the far-IR region (~600-100 cm-1) with a Bolometer detector to test the feasibility of identifying lower energy metal-ligand characteristic features (M-O/N) and to observe the dynamic evolution of cation solvation structures during oxidation (Ca^{2+} release) and reduction (Ca^{2+} uptake) in Ca-Zn-PTtSA, albeit with limited success.

INVITED TALK

Electrocatalysis for the production of green hydrogen and beyond: Materials and Mechanisms

Rao, R. (Imperial College London)

The efficiency, lifetime and resilience of emerging electrochemical technologies for green energy conversion and storage, chemical synthesis, and pollution control relies on atomic-level processes occurring at complex catalytic interfaces. Therefore, we need experimental probes that can shed light on the underlying physical and chemical processes occurring at these dynamic electrochemical interfaces, at the nanometer scale, and sub-second time resolution, to enable rational design of catalysts.

In this talk, I will demonstrate how we can combine soft and hard X-ray absorption spectroscopy to obtain mechanistic insight about the kinetics of green hydrogen production via water electrolysis on state-of-the-art iridium-based oxide catalysts. The talk will span interfaces with varying degrees of complexity, from model surfaces to industrially relevant systems. I will elucidate how the interaction with ionomer and operation in an electrolyser setup influences the active sites for water oxidation. Using a combination of time-resolved optical and X-ray absorption spectroscopy, I will show how we can identify and quantify the density of potential-dependent intermediates, the degree of interaction between them, and the intrinsic rate of reaction. Based on such mechanistic insight on catalysts ranging from model systems to industrially relevant systems, I will demonstrate how we can develop design rules for discovering next-generation catalysts.

INVITED TALK

Photoelectrodes for the solar valorisation of CO_2

Reisner, E. (University of Cambridge)

The design and construction of prototype solar devices for the direct valorisation of carbon dioxide will be presented. Standalone photoelectrochemical cells and artificial leaves based on integrated semiconductors with immobilised synthetic and biological catalysts have been created for solar-powered CO_2 reduction to produce syngas, CO, formate or ethanol fuel coupled to O_2 evolution from water oxidation. Photocatalyst sheets assembled from immobilised semiconductor powders decorated with (bio)molecular catalysts can cleanly produce formate or acetate from CO_2. Progress is currently being made in the synthesis of complex chemicals through cascade and asymmetric catalysis and engineering biology in vivo. The latest advances concern the direct conversion of atmospheric CO_2 using semiconductor particles through gas-phase photocatalysis, bringing us a further step closer to the mimicry of photosynthesis. The materials and devices presented are aimed to stimulate discussions on the use of synchrotron methods to characterise state-of-the-art (photo)electrochemical systems in the future.

TALK

Operando Electrochemical-Structural Characterisation of Promising Li-ion Battery Cathode Materials at Diamond Diffraction Beamlines

Sayed, F. (University of Cambridge).

To meet the growing energy demands and support the adoption of renewal energy generation, the secondary (rechargeable) batteries such as Li-ion batteries (LIBs) and Na-ion batteries (NIBs) need to be improved. The positive electrode material (cathode) is still the limiting component in these rechargeable battery technologies. It is crucial to understand the origins of structural degradation and capacity fade related to cathode materials in order to overcome the issues which impede the adoption of many promising cathodes. Ex-situ or postmortem characterisation of phases formed by powder diffraction during the electrochemical cycling is challenging owing to the metastable nature of the majority of phases formed. To gain commercially relevant insights, structural characterisation should be performed under realistic, operando conditions, in a 'full-cell' configuration using commercially viable anodes instead of an alkali metal anode ('half-cell' setup). Furthermore, it is important to evaluate the performance of cathode materials over an extended period of cycling (100s or 1000s of charge-discharge cycles) to develop an understanding of the long-term mechanisms which deteriorate the battery.
Previously, we have successfully shown the value of a long duration experiment (LDE) at I11-1 synchrotron x-ray diffraction beamline in identifying the minute reversible phase transformations and demonstrated the formation of a new fatigued phase on layered Ni-rich cathode particles after prolonged cycling in the full-cell configuration. In our current LDE experiment we are investigating the heat treatment modified Ni-rich cathode material ($LiNi_{0.8}Mn_{0.1}Co_{0.1}O_2$, NMC811) for structural stability over prolonged cycling in comparison with the commercial NMC811 cathode material. In contrast to layered cathode materials, polyanion e.g. $LiFePO_4$ (LFP) and LiMn0.8Fe0.02PO4 (LMFP), are known to perform better under fast cycling conditions. The modified polyanion cathode, i.e. LMFP, also has better electronic conductivity and shows redox reactions up to a higher potential 4.1 V vs Li metal anode. The commercially viable LMFP-graphite cells can achieve a practical capacity of 140 $mAhg^{-1}$, exhibiting the dual plateaus associated with $Fe^{2+/3+}$ and $Mn^{2+/3+}$ redox processes at 3.3 and 4.0 V, respectively. Owing to the presence of two redox TMs and related two distinct (de)lithiation plateaus, it's crucial to perform the rate capability in these two distinct voltage ranges. Furthermore, in conditions relevant to commercial cells, the rate performance in full-cell against graphite anode is crucial to understand the effect of two different (de)lithiation voltage cut-offs over an extended period of cycling. In this presentation, we will discuss the latest operando-electrochemistry - SXRD results on Ni-rich layered and LMFP polyanion cathodes from our long duration experiment at I11-1 beamline.

POSTER

Templated electrodeposition of nanoscale materials

Shao, L. (Diamond Light Source)

Electrodeposition within mesoporous silica hard templates provides a versatile route for fabricating highly ordered nanostructures with dimensions below 10 nm. In this work, in-situ synchrotron surface characterisation techniques were used to investigate the electrodeposition of Bi_2Te_3 and CdTe nanowires inside mesoporous silica thin films with 2D and 3D pore architectures. Experiments were performed using grazing-incidence techniques at beamline I07 at Diamond Light Source. In-situ GISAXS was used to monitor pore filling, mesostructural evolution, and template stability during electrodeposition, while GIWAXS/GIXD measurements followed phase formation and crystallisation of the Bi_2Te_3 and CdTe nanowires. XRR measurements were further used to characterise film thickness, interface roughness, and structural integrity before and after deposition. During electrodeposition, characteristic GISAXS peak splitting was observed at intermediate pore filling, indicating coexistence of empty and semiconductor-filled mesophases within the silica framework. These results demonstrate the sensitivity of grazing-incidence synchrotron techniques for probing dynamic electrochemical growth processes at buried interfaces. This work highlights how combined GISAXS, GIWAXS/GIXD, and XRR measurements can provide real-time insight into confined electrodeposition and nanoscale interface evolution in semiconductor nanostructured systems.

TALK

Time-Resolved Dip-and-Pull APXPS for Studying Kinetics of Electrochemical Reactions

Shavorskiy, A. (MAX IV Laboratory)

Dip-and-pull (also known as “meniscus”) Ambient Pressure XPS has been increasingly popular in studying solid-liquid interfaces of a wide range of electrochemical systems, such as, e.g., batteries, electrocatalysts, or corroding alloys. Its popularity roots in the method’s high surface sensitivity, chemical specificity, non-contact nature of the probe, and intrinsic in situ / operando capabilities. Despite its limitations, which have been carefully examined in several recent studies, the method is ideal for investigating the fundamental properties of the interfacial double layer, of both capacitive and faradic nature.

In this study, we examined the influence of the ion composition of the electrolyte on the capacitive properties of EDL by employing the time-resolved APXPS and chronoamperometry during modulation of the sample bias. A time evolution of the local potential in the form of photoemission peak shifts has been measured with 0.1 s time resolution from 1M KOH with and without 1M KCl at different distances from the bulk electrolyte after changing the applied potential by 1.0 V (+/- 0.5 V vs OCV). Nine such modulations have been averaged to improve the signal-to-noise ratio. As expected, the time constant for photoemission peak shift by 1.0 eV (equal to the potential jump) increases with the increase of the distance from the bulk electrolyte. This trend holds true for KOH and KOH + KCl electrolytes. However, in the case of pure KOH, the time constants are significantly smaller than when the chloride is added, implying a decrease in the capacitance of the EDL. We employ a simple meniscus model based on an RC circuit to explain the observed trends and discuss the implications for electrocatalysis.

POSTER

Operando X-ray Studies of Proton Exchange Membrane Electrocatalysts for Hydrogen Production

Skidmore, K. (University of Warwick)

Richard. I. Walton[1], Katharina Brinkert[1,2], Julie Macpherson[1], Lucy Mcleod[3], Kayleigh Skidmore[1]

[1]University of Warwick, Coventry, United Kingdom CV4 7AL

[2]University of Bremen, Zarm, Germany 28359

[3]Johnson Matthey, Blouts Ct Road, Reading, RG49NH

Green hydrogen is gaining interest and investment from governments and industry due to the growing need to switch from fossil fuels towards renewable energy sources. Hydrogen can be produced using renewable energy with a high gas purity and at high current densities using proton exchange membrane (PEM) electrolysis. In PEM electrolysis, the oxygen evolution reaction (OER) process occurring at the anode is one of the main bottlenecks due to its sluggish electrode kinetics. Currently, the best electrocatalysts for the OER in acidic conditions are based on IrO_x. Given the associated costs with iridium as a precious metal, it is important to improve the mechanistic understanding of the catalytic reaction to target new materials. Operando X-ray studies can be used to shed light on the rate-limiting step and elucidate structural changes of the electrocatalyst during the OER. We report on a modified commercially available electrochemical cell for operando X-ray diffraction (XRD) and X-ray absorption spectroscopy (XAS) studies of the OER using a calcium iridium pyrochlore (Ca_2-$xIr_2O_6 \cdot H_2O$) electrocatalyst spray-coated onto boron-doped diamond and Toray carbon paper [1]. Electrochemical techniques such as chronoamperometry and cyclic voltammetry are utilised in acidic electrolyte (1 M $HClO_4$) to mimic the conditions which will be used in commercial electrolyser devices. Changes to the catalyst surface, degradation rates and crystallinity are monitored in operando by XRD, whereas XAS provides an overall oxidation state change of the iridium pyrochlore throughout the OER. We report on cell modifications accounting for mass transport and gas bubble formation at the working electrode and changes to the overall design that allow an interchangeable use in both, transmission and reflection measurements at a synchrotron source (ESRF, XMaS beamline). This allows us to monitor catalyst activity, stability, and oxidation state changes under realistic operating conditions that are currently unprecedented in the literature.

References

[1] Exploiting the Flexibility of the Pyrochlore Composition for Acid-Resilient Iridium Oxide Electrocatalyst

POSTER

Metal Nitrogen-doped Carbon Catalysts for the Sustainable Electroreduction of Carbon Dioxide into Value-added chemicals

Elisa Solvay* Magda Titirici, Jesús Barrio (Imperial College London)

Electrochemical CO_2 reduction (CO_2R) offers a promising route to remove anthropogenic CO_2 from the atmosphere while yielding valuable chemicals. Although CO_2R has previously relied on Ag or Au-based catalysts for the production of carbon monoxide (CO), these metals (now considered as critical materials within the European Union [1] have been outshone by Metal Nitrogen-doped Carbon catalysts (MNCs) offering enhanced selectivity towards CO_2RR at reduced over-potentials. [2] This class of material comprise isolated metal atoms coordinated within a conductive nitrogen-doped carbon matrix. Their controlled synthesis remains challenging due to the high temperature pyrolysis step of C-, N- and metal precursors, that result in metal aggregation and formation of oxides and carbides. [3] In this work, employing 2,4,6-Triaminopyrimidine and $MgCl_2.6H_2O$ as organic building block and active site template [4, 5], a decoupled synthesis has be used to synthesise four different MNCs that benefit from an identical C- and N- containing framwork in which Ni, Fe, Co and Cr were respectively introduced at low temperature. [6] Characterisation of the materials showcase remarkable micro- and mesoporosity (> 3300 $m^2\ g^{-1}$) and the absence of metal aggregates. At applied potentials between -0.3 V_{RHE} and -0.75 V_{RHE}, the investigation of these various MNCs revealed near-unity Faradaic efficiency towards CO (FE_{CO}). Results proved particularly encouraging with regard to Co-NC achieving FE_{CO} ~ 75% at -0.5 V_{RHE} as compared to the performance of other Co-contaning MNCs discussed in literature, reporting FE_{CO} circa. 20% at the same applied potential,[7] emphasizing the need for controlled morphological development during MNC-synthesis.

References

[1] Milan G, Constanze V, DG, EC. European Commission, Study on the Critical Raw Materials for the EU 2023 – Final Report. 2023. Report No.: ET-07-23-116-EN-N

[2] Bagger A, Ju W, Varela AS, Strasser P, Rossmeisl J. Single site porphyrine-like structures advantage over metals for selective electrochemical CO_2 reduction. Catal Today. 2017;288:74-8.

[3] Mehmood A, Pampel J, Ali G, Ha HY, Ruiz-Zepeda F, Fellinger TP. Facile Metal Coordination of Active Site Imprinted Nitrogen Doped Carbons for the Conservative Preparation of Non-Noble Metal Oxygen Reduction Electrocatalysts. Advanced Energy Materials. 2017;8(9).

[4] Pampel J, Mehmood A, Antonietti M, Fellinger TP. Ionothermal template transformations for preparation of tubular porous nitrogen doped carbons. Materials Horizons. 2017;4(3):493-501.

[5] Barrio J, Thomas A. Synthesis of Functional Materials Using N-heterocyclic Amines Beyond Melamine. Adv Sci (Weinh). 2025:e10137.

[6] Sarma SC, Barrio J, Bagger A, Pedersen A, Gong M, Luo H, et al. Reaching the Fundamental Limitation in CO_2 Reduction to CO with Single Atom Catalysts. Advanced Functional Materials. 2023;33(41).

[7] Ju W, Bagger A, Hao GP, Varela AS, Sinev I, Bon V, et al. Understanding activity and selectivity of metal-nitrogen-doped carbon catalysts for electrochemical reduction of CO_2. Nat Commun. 2017;8(1):944.

POSTER

Deconvoluting the mechanism of ethylene glycol electro-oxidation towards glycolic acid on gold catalysts using Operando-XAS

Srinivasan, N. (University of Surrey)

The electrochemical conversion of plastic waste into clean fuels and commodity chemicals has gained significant interest due to its economic and environmental benefits. [1] Electrolysis of ethylene glycol (EG), a promising approach for sustainable hydrogen production, can be directly linked to the chemical recycling of polyethylene terephthalate (PET) plastics. [2,3] This process not only reduces plastic waste but also enables the partial oxidation of EG to the high-value product glycolic acid (GCA), a thermodynamically favourable route that enhances green hydrogen production. However, achieving selective oxidation to glycolic acid remains challenging due to the concurrent formation of by-products, particularly formic acid. [4]
To address this challenge, we systematically investigate the oxidation mechanism of EG on state-of-the-art gold catalysts. Electrochemical analysis using cyclic voltammetry (CV) and chronoamperometry (CA) in a RDE and three-electrode set up, coupled with ex-situ high-performance liquid chromatography (HPLC) for product quantification, was performed under various applied potentials in 1 M KOH. A primary oxidation peak for EG at 1.15 V corresponding to oxidation to GCA with a peak current density of approximately 60 mA cm^{-2}. Upon further increasing the potential, the current density decreased due to the formation of inactive gold oxide. A secondary oxidation peak was observed at 1.0 V in the reverse CV scan, which was attributed to the oxidation of adsorbed intermediates following regeneration of active gold catalyst. exhibited limited electrochemical activity, confirming the higher GCA selectivity previously reported for Au catalysts. However, two distinct oxidation peaks were observed at 1.5 V with further oxidised products being detected when the potential was held at this value likely related to the adsorption of GCA on an oxidised Au catalyst surface. These findings suggest that the decreased GCA selectivity at potentials above 1.5 V may result from the simultaneous oxidation of both EG and GCA to further oxidised products on gold oxide.

References

[1] Coates, G.W. and Getzler, Y.D.Y.L. (2020), Nature Reviews Materials. Nature Research, pp. 501–516.
[2] Si, D. et al. (2021), Chem Catalysis, 1(4), pp. 941–955.
[3] Yan, Y. et al. (2023), Journal of the American Chemical Society, 145(11), pp. 6144–6155.
[4] Xin, L. et al. (2012), Applied Catalysis B: Environmental, 125, pp. 85–94.

TALK

Perspectives from MAX IV Laboratory about the future of battery research at synchrotrons.

Temperton, R. (MAX IV Laboratory)

The challenge of developing better batteries is a true multiscale problem. The length scales range from electron transfer at the atomic scale through to the engineering challenges of deploying large-scale assemblies for grid-level storage. The timescales also span a similarly vast range from electron charge transfer (fs) to ion dynamics (μs-s) to the degradation mechanisms (years). And on top of this, the wide variety of different battery chemistries can be overwhelmingly extensive. X-rays obviously can be used to help understand a huge amount of this parameter space. MAX IV is no exception to this, which has thriving battery research programs at numerous beamlines spanning spectroscopy, scattering, diffraction and imaging; using both hard and soft X-rays. However, it is becoming clear that simply providing access to these tools is rarely enough to be truly transformative. We really need to consider how we make these tools available. The normal operating model of a synchrotron is that a single research group applies for time at a single technique/beamline. This generates pockets of expertise, that are often not transferred to the broader community, and the findings are typically restricted to a very small area of the parameter space outlined above. This talk explores how we as a facility can better support the research area to break out of this model of small, localized knowledge with the aim of better enabling transformative breakthroughs in battery technologies using X-rays.
Firstly, we look at the internal perspective, regarding how as a lab we can enable information and knowledge exchange between different beamlines. At MAX IV, we have been trialling a “matrix structure” to allow beamline scientists and engineers to consider a wider perspective. Secondly, we will consider the external perspective about how users interact with the lab. Specifically, we are piloting the “hub access” model, which was pioneered at the ESRF, to really allow a consortium of users to tackle a big research problem in a multidisciplinary way. Taken together, we believe MAX IV (and other synchrotrons) can act as a true enabler of the sorts of collaborations needed to make impact in the complex area that is battery materials research.

TALK

Active Phase of Nickel Electrocatalysts driving Alkaline Hydrogen Evolution

Wang, Y. (Imperial College London)

Nickel-based cathodes are widely used in alkaline water electrolysis, yet the nature and stability of the active surface under operating conditions remain unclear due to the dynamic evolution of metal/oxide/hydroxide species. In particular, the role of metal/oxo–hydroxo interfacial structures in governing hydrogen evolution activity are not well understood. Here, we employ a multimodal, depth-sensitive approach combining operando Ni L-edge X-ray absorption spectroscopy, depth-sensitive X-ray absorption measurements in total electron yield and Auger electron yield modes, X-ray photoelectron spectroscopy, isotopically labelled nano secondary ion mass spectrometry, and online electrochemical mass spectrometry to directly track the evolution of Ni/NiO_xH_y interfaces during the hydrogen evolution reaction. Using well-defined sputtered Ni thin films as a model system, we show that progressive reduction of near-surface oxide/hydroxide species is accompanied by a gradual loss of hydrogen evolution activity. Depth-resolved measurements reveal a predominantly metallic outermost surface under cathodic bias, while NiO_xH_y reforms the surface upon relaxation to open-circuit conditions. Importantly, mild anodic pre-conditioning regenerates subsurface NiO_xH_y species, resulting in a sustained increase in hydrogen evolution activity upon subsequent cathodic polarisation. These results establish the crucial role of metal/oxo–hydroxo interfaces as active phases for hydrogen evolution and provide a framework for engineering robust, earth-abundant HER cathodes capable of operating under dynamic, real-world electrolysis conditions.

TALK

Resolving Ionomer–Catalyst Interactions in Fuel Cell Electrodes via Operando X-ray Absorption Spectroscopy

Wang, M. (University of Exeter)

Building on this mechanistic understanding, we further developed a lignin derived freestanding catalyst layer using a dual templating strategy to create an interconnected hierarchical pore network. This architecture improved bulk mass transport in gas diffusion electrodes by enhancing access to active sites and reducing transport limitations typically associated with conventional catalyst layers. Together, these studies demonstrate how synchrotron electrochemistry can link local catalyst environment, support architecture, and electrode scale transport in ORR catalyst layers. The findings provide design principles for sustainable, nanostructured carbon electrodes and highlight the value of operando X-ray characterisation in revealing structure performance relationships under realistic electrochemical conditions.

References

[1] Wang, M. et al. Resolving optimal ionomer interaction in fuel cell electrodes via operando X ray absorption spectroscopy. Nature Communications, 2024, 15, 9390.

[2] Wang, M. et al. Enhanced ORR performance with biomass derived freestanding catalyst layers: advancing mass transport in gas diffusion electrodes. Journal of Materials Chemistry A, 2024, 12, 24878 to 24885.

POSTER

Nanoscale X-ray imaging of functional materials in operando: the Diamond-II Coherent Soft X-ray Imaging and Diffraction (CSXID) beamline

Walters, A. (Diamond Light Source)

The Coherent Soft X-ray Imaging and Diffraction (CSXID) beamline at Diamond-II will be a flagship facility for nanoscale imaging of both quantum and functional materials using coherent soft X-rays. It will include the Functional Materials Imaging (FMI) endstation, which will be dedicated to the study of functional materials for use in catalysis and batteries. Commercially available operando cells, originally developed for transmission electron microscopy studies, will be used at the FMI endstation to enable soft X-ray imaging experiments in gas and liquid environments at elevated temperatures with a spatial resolution of around 10 nm. In particular a three-electrode electrochemical cell will also be available, enabling the CSXID beamline to not only study innovative battery materials but also opening the door to the study of electrocatalysts. The beamline will primarily employ X-ray ptychography, a coherent diffractive imaging technique, to provide X-ray microscopy with very high spatial resolution. The high brilliance of the Diamond-II synchrotron will provide a coherent flux around an order of magnitude higher than currently available with the existing machine, which will dramatically speed up X-ray ptychography measurements. Moreover, the wide energy range of the CSXID beamline (250 eV to 3500 eV) will enable spectroscopic measurements at multiple X-ray absorption edges to be performed within the same experiment, allowing the physicochemical behaviour of multiple components to be determined in the investigated system. The overall design of the CSXID beamline will be presented, together with the latest conceptual design of the FMI endstation. We will describe the current status of the Diamond-II project and outline the timescales of the CSXID beamline build project.

INVITED TALK

Accessing Electrochemical Interfaces in Batteries and Electrolysers with Operando X-ray Spectroscopies

Weatherup, R. (University of Oxford)

Understanding how electrochemical interfaces evolve under operating conditions is a central challenge in energy storage (batteries) and conversion (electrocatalysis). While X-ray spectroscopies offer powerful element-specific insight, extracting interface-sensitive information under realistic conditions is far from straightforward.[1] In this talk, I will show how combining complementary X-ray techniques and extending them to operando conditions enables us to disentangle bulk and interfacial processes and directly resolve the chemical state of electroactive materials. I will first present our recent work on Ni-rich cathode materials for Li-ion batteries, where we integrate multiple spectroscopic probes to obtain a depth-resolved picture of redox processes spanning ~10 nm to >10 µm. [2,3] By combining soft X-ray absorption spectroscopy (sXAS) in electron and fluorescence yield modes, X-ray Raman spectroscopy, and electron energy loss spectroscopy, we distinguish reversible bulk redox from near-surface degradation. These measurements reveal that bulk redox in $LiNiO_2$ proceeds via Ni–O rehybridization, reducing electron density on oxygen without the formation of molecular O_2. Instead, trapped O_2 is linked to surface degradation at the electrode–electrolyte interface and is accompanied by Ni reduction. To directly probe electrochemical reactions at solid–liquid interfaces, we have developed operando electrochemical cells incorporating thin (<100 nm) silicon nitride membranes, enabling sXAS measurements under realistic conditions.[4] Using modulated X-ray illumination and electron yield detection, we directly track the formation of the solid–electrolyte interphase (SEI) on silicon anodes and reveal how the widely used additive fluoroethylene carbonate modifies interphase chemistry and stability.[5] For electrocatalysis, we employ fluorescence yield measurements in combination with controlled nanoparticle size to achieve effective surface sensitivity. By comparing 3 nm and 9 nm Co nanoparticles during the oxygen evolution reaction, we separate bulk and surface contributions, showing that high-spin Co^{3+} forms at the catalyst surface and oxidises toward Co^{4+} with increasing potential, while the bulk remains dominated by low-spin Co^{3+} due to its distinct coordination environment. Together, these results demonstrate that operando X-ray spectroscopies can now directly resolve the chemical evolution of electrochemical interfaces under reaction conditions, enabling a more accurate and mechanistic understanding of energy materials. I will conclude by highlighting emerging opportunities to extend these approaches and further bridge the gap between model studies and real operating systems.

References

[1] Wu et al. Phys. Chem. Chem. Phys. 2015, 17, 30229
[2] An et al. Energy Environ. Sci. 2024, 17, 8379
[3] Poletayev et al. Nature Commun. 2025, 16, 9379
[4] Weatherup et al. J. Phys. Chem. B 2018, 122, 2, 737–744
[5] Swallow et al. Nature Commun. 2022, 13, 6070