Light Source Enabled Science
Room 319 - Session LS-TuM

Light Source Enabled Energy Science

Moderators: Andi Barbour, Brookhaven National Laboratory, **Philip Ryan**, Argonne National Laboratory, USA

8:00am **LS-TuM-1 Surface Sensitive and In-Situ Characterization of Perovskite Materials for Energy Conversion***, Juanita Hidalgo,* New York University **INVITED**

Developing new energy conversion technologies is a critical scientific challenge to address global energy demand and global warming. Perovskite-type materials are particularly important because they exhibit electronic and or ionic conductivity, enabling a wide range of energy-related applications, including photovoltaics, fuel cells, and electrolysis cells. Lead halide perovskites (APbX3) have emerged as promising absorber layers for low-cost solar cells, while perovskite oxides (ABO3) are widely used as electrodes and catalysts in solid-state electrochemical systems. However, the perovskite crystal structure can be unstable and undergo phase transformations under external stressors. To investigate the surface structural and chemical properties of these materials, my work employs advanced synchrotron-based in-situ characterization techniques, including grazing-incidence wide-angle and small-angle x-ray scattering (GIWAXS/GISAXS) and near-ambient-pressure X-ray photoelectron spectroscopy (NAP-XPS). First, my talk will address the instability of lead halide perovskites under exposure to humidity and air, and the complex relationship between structure, properties, and performance in solar cell applications. Using in-situ GIWAXS, we investigated the undesired phase transformations that occur under humid air and humid nitrogen environments, revealing a synergistic effect between water and oxygen that accelerates degradation. Second, I will discuss the high-temperature reduction of perovskite oxides, which leads to the formation of embedded metal nickel nanoparticles through a process known as exsolution. These nickel nanoparticles can act as catalysts for electrochemical reactions relevant to energy conversion and storage. Using simultaneous in-situ XPS and GISAXS, we tracked nanoparticle formation through the reduction of nickel oxide to the metallic state and the corresponding structural evolution observed by GISAXS. Therefore, understanding the mechanisms governing perovskite phase transformations and establishing correlations between structure, chemistry, and properties is essential for tuning and optimizing these materials to achieve more robust, stable, and efficient energy technologies for real-world applications.

8:30am **LS-TuM-3 Resonant Photoemission Spectroscopy for Resolving Active Oxidation States during Surface Chemical Reactions***, Hao Chen,* Stanford Synchrotron Radiation Lightsource, SLAC National Accelerator Laboratory*; Miquel Salmeron,* Lawrence Berkeley Lab

Identifying active oxidation states of transition-metal oxide catalysts under reaction conditions remains challenging because chemically distinct states often strongly overlap in conventional core-level X-ray photoelectron spectroscopy (XPS). Here, we combine ambient-pressure XPS (APXPS) with resonant photoemission spectroscopy (ResPES) to follow the chemical evolution of ultrathin CoO_x/Au(111) during CO oxidation.[1] While APXPS monitors catalyst composition, adsorbates, and reaction products, ResPES exploits oxidation-state-specific resonant enhancement of valence-band photoemission to distinguish and quantify Co^{2+} and Co^{3+} species.

The catalytic behavior depends strongly on the chemical state of cobalt and the reaction environment. Under oxygen-lean conditions, partially oxidized CoO_x<1 films containing Co^0 and Co^{2+} are reactive toward CO oxidation at moderate temperatures of 100–150 °C, where CO adsorbs on metallic Co sites and reacts with neighboring lattice oxygen. In contrast, stoichiometric CoO containing Co^{2+} readily forms carbonate species in CO, which poison the surface and suppress CO oxidation below 300 °C; at higher temperature, carbonate decomposition is accompanied by oxide reduction, restructuring, and dewetting. Under oxygen-rich conditions, CoO_x>1 phases containing Co^{2+} and Co^{3+} exhibit high activity over a broader temperature range. ResPES directly reveals the dynamic behavior of Co^{3+}: CO reduces Co^{3+} to Co^{2+} at ~100 °C, while O_2 regenerates the Co^{3+} state. Under simultaneous CO/O_2 conditions, the highly active oxide contains a Co^{3+}/Co^{2+} ratio of approximately 1.4, identifying Co^{3+} as a key active site for CO oxidation.

The versatility of ResPES is further demonstrated for ultrathin FeO_x/Pt(111), where the same approach quantitatively resolves Fe^{2+} and Fe^{3+} contributions in mixed-valence iron oxides.[2]

These results demonstrate the capability of ResPES to resolve dynamic, chemically active oxidation states that are difficult to distinguish by conventional XPS, providing a powerful approach for studying transition-metal oxide surfaces under reaction conditions.

[1] Chen, H.; et al.; Salmeron, M. Nat. Commun. 2023, 14, 6889.

[2] Chen, H.; et al.; Salmeron, M. J. Phys. Chem. Lett. 2025, 16, 10975–10981.

8:45am **LS-TuM-4 Operando Soft X-Ray Spectroscopy of Electrochemical Interfaces: From Reaction Cell and Sample Design to Mechanistic Insight***, Santosh Kumar,* Diamond Light Source Ltd., UK*; James J. C. Counter,* University of Manchester, UK*; David C. Grinter, Matthijs A. van Spronsen, Pilar Ferrer,* Diamond Light Source, UK*; Christopher M. Zalitis, Tugce Eralp Erden,* Johnson Matthey Technology Centre, UK*; Roger A. Bennett,* University of Reading, UK*; **Georg Held**,* Diamond Light Source, UK

Synchrotron-based *operando* soft X-ray photoelectron spectroscopy (XPS) and near-edge X-ray absorption fine structure (NEXAFS) enable element-specific probing of electrochemical interfaces under realistic conditions but require advanced reaction cell and sample designs to bridge aqueous electrochemistry and vacuum-based detection. We present a modular spectro-electrochemical flow cell and window-free working electrode assembly (WEA) compatible with near-ambient-pressure XPS and NEXAFS. The platform combines interchangeable SiN_x or polymer membranes (such as Nafion™) with controlled hydration, enabling stable in-situ measurements under electrochemical operation. Using CuO_x- and IrO_x-based electrodes, we demonstrate tracking of redox processes, surface hydration, and oxygen evolution chemistry. *Operando* XPS and NEXAFS reveal potential-dependent metal oxidation and the formation of reactive oxygen species, providing direct insight into reaction mechanisms at electrochemical interfaces. This approach establishes a versatile methodology for soft X-ray studies of electrocatalysts.

References

1. *Catalyst-Coated Mesoporous Carbon–Membrane Electrode Assembly for In-situ Soft X-ray XPS and NEXAFS Studies of Electrocatalytic Interfaces*, **ACS Electrochemistry** (2026) DOI: 10.1021/acselectrochem.5c00554.

2. *An Electrochemical Flow Cell for Operando XPS and NEXAFS Investigation of Solid–Liquid Interfaces*, **J. Phys. Energy** 6,036001 (2024). DOI: 10.1002/cctc.202400937

9:00am **LS-TuM-5 Substrate-Mediated Doping Reshaping Interfacial Energetics in Organic Electronics***, Xiane Li, Slavomir Nemsak,* Advanced Light Source, Lawrence Berkeley National Laboratory

Energy level alignment (ELA) at electrode/organic-semiconductor interfaces governs charge generation, transport, and injection in organic electronic and energy-conversion devices. However, ELA is typically characterized under ultra-high vacuum, whereas practical devices operate in ambient environments where water is ubiquitous. The influence of water on interfacial energetics therefore remains largely unresolved. Here we probe the evolution of ELA in the presence of water using operando ambient-pressure X-ray photoelectron spectroscopy combined with Kelvin probe measurements. We find that water molecules strongly reshape the interfacial electronic structure by screening the spontaneous charge transfer that forms interface dipoles under vacuum. This screening suppresses dipole formation at interfaces, lowers or increases the work function of the system, and promotes electron or hole accumulation in the organic semiconductor, resulting in pronounced substrate-mediated n-doping/p-doping. Our findings reveal a previously unrecognized environmental influence on ELA, establishing water as a key regulator of substrate-mediated interfacial energetics in organic electronic systems.

9:15am **LS-TuM-6 Investigation on Topotactic Phase Transition of $LaCoO_3$ Thin Films with in Situ XRD and Ambient Pressure Hard X-Ray Photoelectron Spectroscopy**, *Hyunsuk Shin, Youngmin Yun,* Gwangju Institute of Science and Technology, Republic of Korea*; Okkyun Seo,* National Institute for Materials Science, Japan*; Seongeun Kim,* Chosun University, Republic of Korea*; Minsik Seo, Dongwoo Kim,* Gwangju Institute of Science and Technology, Republic of Korea*; Hojoon Lim,* Myongji University, Republic of Korea*; Hojun Oh, Subin Jang, Kyungmin Kim, Sae Hyun Kang,* Gwangju Institute of Science and Technology, Republic of Korea*; Adrian Hunt, Iradwikanari Waluyo,* Brookhaven National Laboratory*; Do Young Noh,* Gwangju Institute of Science and Technology, Republic of Korea*; Hyon Chol Kang,* Chosun University, Republic of Korea*; **Bongjin Simon Mun**,* Gwangju Institute of Science and Technology, Republic of Korea

To understand the correlation between the structure phase transition (SPT) and electronic/chemical properties of LaCoO3 (LCO), multimodal techniques, including in situ XRD, I-V measurement, and ambient pressure hard X-ray photoelectron spectroscopy (AP-HAXPES) are employed. Especially, AP-HAXPES not only allows operando analysis under wide pressure and temperature ranges, but also provides the direct bulk sensitive information of the electronic/chemical states. The AP-HAXPES measurement overcomes the difference of the probing depths between conventional XPS and XRD, thereby providing bulk-sensitive chemical/electronic structure information under reaction condition. The observed oxidation states in core level spectra, Co 2p, clearly showed that the oxygen vacancies play an important role in the SPT of LCO thin films. Importantly, the analysis of the valence band (VB) spectra of AP-HAXPES showed the presence of enlarged band gap formation, demonstrating the presence of the complex interplay between the oxygen vacancies and electronic structure. The modified VB structures across the SPT are also compared to the in situXRD and transport measurements.

9:30am **LS-TuM-7 Advances in Hydrogen Technology Enabled by Synchrotron X-ray Methods**, ***Nicholas Strange***, SLAC National Accelerator Laboratory **INVITED**

Hydrogen energy represents a critical frontier in securing the United States' long-term energy resilience and global technological dominance. Achieving cheap, sustainable, and equitable hydrogen production at scale requires rapid advancement of the underlying materials science and process technologies, a challenge that has mobilized significant federal investment and a growing network of national laboratory partnerships. This talk will highlight previous and ongoing research within the materials science division at the Stanford Synchrotron Radiation Lightsource (SSRL), contextualized within the broader scopes of the HyMARC, H2NEW, H2NUC, HydroGEN, and HyBlend energy materials networks (i.e., DOE-led consortia uniting national laboratories around hydrogen production, storage, and transport).

The hydrogen R&D landscape is undergoing a decisive shift from foundational discovery toward application-focused development of materials optimized for specific performance and durability requirements. American leadership in this transition is essential, both to drive down the cost of clean hydrogen production and to establish the domestic industrial base necessary for long-term energy independence. *In situ* and *operando* X-ray scattering measurement designs developed at SSRL will be discussed, with results presented from a selection of collaborative projects aimed at accelerating this national effort.

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