

Advances in Battery Engineering, Interface Design, and Characterization Mini-Symposium Room 319 - Session BT1+AS+CA+TF-ThM

Interface Engineering in Batteries

Moderators: Prof. Dr. Adriana Creatore, Eindhoven University of Technology, Prof. Alexander Kozen, University of Vermont

8:00am BT1+AS+CA+TF-ThM-1 Plasma and Electrochemical Modification of Battery Cathode Surfaces to Enhance Voltage Stability and Thus Energy Density, Paul Braun, University of Illinois Urbana-Champaign **INVITED**
The energy density of battery cathodes increases dramatically with increases in voltage. However, higher operating voltages, in particular above 4.3V leads to significant capacity degradation. Because the mechanism of capacity degradation is often a surface-initiated reaction, modification of the exposed surface of the cathode material is a promising approach to increase energy density without adding significant mass or volume to the battery. Two approaches we are investigating to increase the voltage stability of LiCoO_2 , a common battery cathode material, include surface modification via a plasma process, and electrochemical deposition of a high voltage stable material. Both methods result in a thin layer of high voltage stable material on the surface of the active cathode material. The plasma modification forms a nanoscale layer of disordered rocksalt on the underlying layered LiCoO_2 while the electrochemical modification grows a layer of lithiated manganese oxide on the underlying LiCoO_2 . In both cases, the cyclable energy density of the LiCoO_2 is increased significantly.

8:30am BT1+AS+CA+TF-ThM-3 Interfacial Engineering of Battery Materials using Atomic Layer Deposition, Neil Dasgupta, University of Michigan, Ann Arbor **INVITED**

Interfaces and interphases play a critical role in battery performance, both in current state-of-the-art Li-ion batteries (LIBs), as well as next generation battery technologies. Therefore, there are great efforts to understand, control, and improve the properties of battery interfaces and interphases. This quickly becomes a multi-dimensional optimization problem, where the required properties of interfaces span from performance (i.e. fast rate capability), stability (i.e. long calendar life) and compatibility with the adjacent chemical and material phases. To address these challenges, there has been increasing interest in the application of 'artificial interphase' layers, which are typically comprised of nanoscale thin-film coatings along electrode-electrolyte interfaces. From a manufacturing perspective, it is critical that these surface coatings be deposited in a conformal, reproducible, scalable, and low-cost manner.

Towards these goals, our research group has developed a range of surface coating layers for battery applications, both for LIBs with liquid electrolytes, as well as next-generation systems such as Li metal anodes and solid-state batteries. In this talk, I will summarize recent progress towards precise and tunable interfacial control in batteries with both liquid and solid electrolytes, using atomic layer deposition (ALD) as an enabling platform. I will describe the development of advanced ALD chemistries, beyond the binary oxide systems that are typically employed. In LIBs, I will describe how surface coatings can simultaneously enable low-temperature and fast charging of graphite anodes without Li plating. In solid-state battery systems, I will describe the stabilization of cathode/solid electrolyte interfaces, enabling high-voltage cycling above 4.5 V. Finally, I will describe our efforts towards scalable manufacturing of ALD-coated battery electrodes, through the development of atmospheric-pressure spatial ALD technologies.

9:00am BT1+AS+CA+TF-ThM-5 Atomic Layer Deposition (ALD) Tuned Interfaces for High-Performance Lithium- and Manganese-Rich Cathodes, Jahnavi Manikantan Sudharma, Argonne National Laboratory; Jeffrey Elam, Argonne National Laboratory, USA

The growing deployment of renewable energy and the rapid expansion of artificial intelligence and machine learning infrastructures place increasingly dynamic demands on modern electrical grids, requiring the need for high-performance, renewable energy-storage technologies. While nickel (Ni)-rich layered oxides remain the state-of-the-art materials for lithium-ion battery cathodes, their reliance on costly and supply-constrained Ni and cobalt (Co), motivates the development of alternative materials. Lithium- and manganese (Mn)-rich (LMR) cathodes offer a compelling solution due to their high energy density and reversible capacities enabled by anionic redox. However, oxygen loss associated with this redox process induces

severe surface and interfacial degradation, including electrolyte decomposition, transition-metal migration, and Mn dissolution, which hinder long-term cycling stability of these cathodes. Suppressing Mn dissolution at the surface is still a key challenge. Advancing surface engineering, structural tuning, and electrolyte optimization is therefore critical to unlocking the full potential of LMR cathodes for next-generation energy-storage systems. Atomic layer deposition (ALD) being a self-limiting thin film growth method characterized by the sequential exposure of chemical species, offers a promising strategy to stabilize the interfaces by depositing tailored ultrathin, uniform and conformal coating layers on the surface and thereby mitigating the surface degradation of the cathode. Surface coatings applied through atomic layer deposition (ALD) offer a highly controlled route to stabilize these interfaces; however, the fundamental mechanisms governing how different ALD chemistries interact with LMR surfaces remain poorly understood. In this presentation, I will discuss our recent study combining in situ measurements of ALD chemistries, ex situ thin film characterization, and electrochemical testing of prototype batteries. By systematically investigating ALD coating chemistries including lithium phosphate (Li_3PO_4) and lithium borate (LiBO_2), we elucidated how ALD surface reactions govern coating composition, structure, and protective functionality and how this functionality affects cycling performance. These insights will inform rational interface-engineering strategies, enabling the practical deployment of high-energy, cobalt-lean Mn-rich cathodes for next-generation lithium-ion batteries.

9:15am BT1+AS+CA+TF-ThM-6 Atomic Layer Deposition for Lithium Metal Anode Stability, Niels Hoogedoorn, Meike Pieters, Cristian van Helvoirt, Mariadriana Creatore, Eindhoven University of Technology, Netherlands

High-performance lithium-ion batteries (LIBs) are hampered by phenomena such as dendrite growth, electrolyte decomposition and uncontrolled interface formation between electrode and electrolyte. This is especially the case of next-generation battery materials, such as Si-anodes, Li-metal anodes and solid-state electrolytes. To address these challenges, atomic layer deposition (ALD) offers a promising pathway by engineering ultrathin conformal interfacial layers with atomic-scale precision.

In this contribution we present our recent work on ALD processes on lithium metal anodes (LMAs). The studies include: Al_2O_3 , LiPO , LiPON and LiF , all developed in a FlexAL ALD-reactor (Oxford instruments). The supercycle approach for the process development of LiPO is highlighted: a novel Li-precursor, Lider™, is combined with an oxygen plasma (O_2^*) and trimethyl phosphate (TMPO). The films are free of carbon impurities, despite that, during the subcycle of Lider + O_2^* , Li_2CO_3 is detected by XPS in combination with a vacuum transfer unit. We find that a chemical vapour transformation [1] occurs, where the subsequent exposure of Li_2CO_3 to TMPO, leads to the abstraction of carbonate from the film, towards C-free LiPO [2].

Additionally, we discuss the deposition of these ALD layers on LMAs and their effect on the critical current density (CCD) of the LMAs, tested in a Li | argyrodite | Li symmetric cell. A home-built vacuum transfer module is used to transport the LMAs between a glovebox and the ALD reactor, where the ALD layers are processed at 120 °C. The CCD is the maximum electrical current per unit area that the anode can withstand before reaching failure, typically caused by lithium dendrite induced shorting. Depending on the material, and layer thickness, the ALD layers improve the CCD by more than 40%. For example, in the case of 10 Al_2O_3 cycles, the maximum sustainable current density increases from 2.4 mA/cm^2 to 3.4 mA/cm^2 , thereby demonstrating enhanced stability against dendrite shorting. These results illustrate how engineering ALD interface layers can improve the CCD of LMAs and mitigate challenges in LMA stability. Moreover, these studies propose CCD measurements as screening approach of ALD chemistry and processing conditions towards their implementation and testing in full LIB cells.

[1] M. Y. Young et al., *J. Phys. Chem. C* 2019, 123, 9,23783.

[2] M. J. Pieters et al., *J. Phys. Chem. C* 2026, 130, 15, 5458.

9:30am BT1+AS+CA+TF-ThM-7 Buffer Layer Oxidative Polymerization to Stabilize Li Metal Anodes for Lithium Ion Batteries, Amit K. Datta, Blaine D. Kelly, Dilan M. Gamachchi, Indeewari M. Karunarathne, Campbell A. Sweet, Andrew C. Meng, Matthias J. Young, University of Missouri

Li metal anodes promise an order of magnitude higher energy density than conventional graphite anodes in Li ion batteries, however, the practical use of Li metal as an anode remains limited by several factors such as unstable interfacial reactions, continuous electrolyte decomposition, and dendritic Li growth, leading to rapid cell failure. Oxidative Molecular Layer deposition (oMLD) offers the ability to deliver thin film polymer coatings with

electronic, ionic and chemical properties of interest for improving stability of Li metal anodes, but oMLD employs harsh oxidants that are not compatible with Li metal substrates. In this work, we demonstrate an approach we term buffer layer oxidative polymerization (BLOP), to enable the delivery of oMLD coatings onto Li metal. We investigate three BLOP derived polymer coatings on Li metal: polyhydroquinone (pHQ), poly(2,5-dimercapto-1,3,4-thiadiazole) (pDMCT), and poly(p-phenylenediamine) (pPDA). These three coatings were selected as representative classes of coatings based on their electronic and ionic conductivity differences: pHQ exhibits both Li ion conductivity and electronic semiconductivity, pDMCT shows only Li ion conductivity and pPDA shows anionic conductivity and electronic semiconductivity. Galvanostatic charge-discharge measurements in Li-Li symmetric cells show differences in the stability enhancement among the different coatings, with a clear benefit for BLOP vs. oMLD and MLD. Electron microscopy on these coatings helps understand the crosslinking depth achieved from BLOP coatings generated by different organic monomers. Overall, this work demonstrates the oxidative synthesis of multiple polymeric thin films directly on Li metal without degrading the substrate and identifies that coatings with Li ion conductivity and electronic semiconductivity show improved Li metal stability over the alternatives.

9:45am **BT1+AS+CA+TF-ThM-8 Modulation of Anode-Free Sodium Battery Interfaces through Atomic Layer Deposition of NaF**, *Jonathan Thurston, Aaron Melemed, Neil Dasgupta*, University of Michigan

Sodium-based batteries are a promising alternative to lithium-ion technologies due to the higher earth abundance and lower cost of sodium. Furthermore, when Na metal electrodes are used in an “anode-free” configuration, the theoretical energy density is competitive with that of lithium-ion systems. However, the effects of the current-collector surface chemistry on the subsequent Na plating/stripping and solid electrolyte interphase (SEI) composition remains limited.

In this study, we synthesize an “artificial SEI” of NaF using atomic layer deposition onto the surface of Cu current collectors (CCs). Thermal ALD of NaF is successfully demonstrated using sodium *tert*-butoxide and ammonium fluoride precursors. We investigate the impact of the NaF surface layers on the subsequent morphology of deposited sodium using electron microscopy. A multi-modal suite of near-edge X-ray absorption spectroscopy (NEXAFS), X-ray photoelectron spectroscopy (XPS), and infrared spectroscopy (IR) are used to characterize the composition and structure of the resulting SEI as a function of depth and battery cycling. Our results show that the NaF layer encourages more homogenous plating with controlled nucleation sites, while bare Cu CCs have more sporadic nucleation sites for Na metal and a more dendritic mossy plating morphology. Additionally, electrochemical impedance spectroscopy (EIS) and chemical state identification indicate that a more stable SEI is formed in the first few cycles for NaF-coated Cu CCs, while an evolving SEI is observed for uncoated Cu CCs. Our work demonstrates the importance of surface layers in anode-free battery current collectors, while investigating the dynamic nature of the SEI composition and structure throughout cycling.

Advances in Battery Engineering, Interface Design, and Characterization Mini-Symposium Room 319 - Session BT2+AS+CA+TF-ThM

Advanced Battery Electrodes

Moderators: Prof. Alexander Kozen, University of Vermont, Dr. Tanguy Terlier, Rice University, Dr. Andrew Westover, Oak Ridge National Laboratory

11:00am **BT2+AS+CA+TF-ThM-13 Understanding the Electrically-Limited Reaction Kinetics and Dynamics for Conversion Electrode Iron Fluoride**, *Chuan-Fu Lin*, Catholic University of America **INVITED**

To maintain efficient reaction kinetics in standard electrodes, fast and stable ionic and electronic conductive networks are required for long-term reversibility, and this poses the major challenges for conversion-type electrode materials. The phase separation reaction, $CM + Li^+ + e^- \rightarrow M$ (reduced metal) + LiX (Li compounds), induced by the lithiation process deteriorates the structural integrity, which certainly destroys the original ionic and electronic pathways and reconstructs new ones. However, it is still unclear whether the transports of conversion electrode are benefited or hindered from the reconstruction of transport pathways due to phase separation, yet the dynamic evolution of electrical conductivity in these systems remains overlooked.

This work aims to investigate the electrically-limited reaction of conversion electrodes upon phase separation and recombination by studying the structural, electrical, and electrochemical properties of amorphous and crystalline iron (III) fluoride (FeF_3) thin films synthesized via sputtering deposition. Using a binder-free lateral device architecture and probing measurement, we dynamically mapped resistivity evolution during electrochemical cycling. While pristine crystalline films exhibit lower initial resistivity than amorphous films, deep lithiation triggers a dramatic drop in resistivity by over five orders of magnitude in both structural variants. Despite the massive volume fraction of the insulating LiF byproduct (1 : 4.17 for Fe : LiF ratio), the resistivity reduced to $\sim 0.35 \Omega\cdot m$, demonstrating a common conduction pathway formed by a metallic Fe network within the nanocomposite. Correlating this resistivity evolution with X-ray Photoelectron Spectroscopy (XPS) spectra reveals the electrically-controlled reaction dynamics of the thin-film conversion electrodes. Conversely, delithiation results in a resistivity increase of two orders of magnitude, driven by a rate-dependent electrochemical “trapping effect.” Fast charging causes isolated Fe domains electronically stranded within the LiF matrix and driving irreversible capacity loss. These results elucidate the coupling between chemical quantifications, reaction dynamics, and electrical properties, providing understanding of the influence of the electrical conductivity for high-energy-density conversion cathodes.

11:30am **BT2+AS+CA+TF-ThM-15 Correlating Electrical Property Evolution and Reversibility in Sputtered ZnO Thin-Film Conversion Electrodes**, *Sukanya Goswami, Chuan-Fu Lin*, The Catholic University of America

During conversion reaction in Lithium-ion batteries, electrical properties shift as reaction kinetics vary with phase separation, state of charge, and evolving conductivity. Understanding how these dynamic electrical transitions govern reaction control is essential for optimizing conversion electrodes. This study probes the evolution of ZnO conversion electrodes during cycling, examining the electron transport, reversibility and capacity retention. Because the literature on ZnO remains limited, it provides a simple benchmark for testing broader conversion-mechanism concepts and for comparing phase separation and recombination process across conversion materials in LIBs. **Sputter-deposited ZnO thin films** were cycled in a lithium-ion half-cell configuration and their electrical transport properties tracked ex-situ using **two-probe resistivity measurements** after each set of cycles.

The resistivity data reveal a clear and progressive deterioration in electronic transport over cycling. Starting from values on the order of $10^{-4} \Omega\cdot m$ in the early cycles, resistivity increases by several orders of magnitude, reaching **1-7 $\Omega\cdot m$ by cycle 30**. This monotonic rise reflects a gradual breakdown of the conductive percolation network as the conversion reaction proceeds. Persistent differences in resistivity during early cycling reveal an asymmetry between lithiation and delithiation, likely caused by sluggish reconversion kinetics of the phase-separated products. These electrical changes correlate directly with coulombic efficiency. When the electrode maintains sufficient conductivity, capacity retention is upto **95%**. Conversely, as resistivity rises sharply with progressive cycling, coulombic efficiency becomes irregular and drops. This correlation implicates degradation of electronic transport, driven by delamination caused by the volume expansion during phase transformation, as the primary mechanism governing capacity fades.

To understand the structural and chemical origins of these changes, cycled electrodes were characterized by **XRD, XPS, and SEM**. XRD tracked phase separation and recombination between ZnO, metallic Zn and Li_2O . XPS provided insight into surface chemical states and irreversible interfacial species, while SEM captured morphological degradation including delamination. Together, these results establish a direct link between electronic conductivity, conversion reaction reversibility and coulombic efficiency, offering a framework for mitigating kinetic limitations in ZnO based thin film cathodes.

11:45am **BT2+AS+CA+TF-ThM-16 Molecular Mapping Investigations of Prelithiation Alloy Formation via Thermal Evaporation on Silicon Based Electrodes**, *Gabriel Parker, Amanda Musgrove, Gabriel Veith, Ivan Matyushov, Xiao-Ying Yu*, Oak Ridge National Laboratory, USA

Abstract: Silicon based composites have become increasingly popular as potential anodes for lithium-ion batteries due to their large storage capacity, relative abundance, and low cost. However, these anodes often see reduced initial coulombic efficiency (ICE) due to disruptive volume expansion up to 300% and continuous unstable solid electrolyte interphase (SEI) layer formation. Prelithiation, the process of adding excess reservoir of Li to the electrode to compensate for irreversible SEI formation losses during their sample preparation, has proven to solve the issue of

immediate capacity loss. Thermal evaporation is a prelithiation technique with limited studies on its effectiveness. In this study, time-of-flight secondary ion mass spectrometry (ToF-SIMS) is used to investigate the benefits of prelithiation via thermal evaporation. ToF-SIMS provides chemical mapping and spatial information in 2D and 3D visualizing the deposition of lithium, identifying Li_xSi_y alloy and $\text{Li}_x\text{Si}_y\text{O}_z$ silicate formation, and the distribution of lithium passivation into the electrodes. Passivation under different atmospheric conditions, such as inert Argon (Ar) and Ar/carbon dioxide (CO_2), highlights the impact of the environment on the passivation effectiveness and formation of Li_xSi_y alloy and $\text{Li}_x\text{Si}_y\text{O}_z$ silicate. The ToF-SIMS molecular imaging and depth profiling results indicate that prelithiation via thermal evaporation effectively distributes lithium throughout the depth profile thickness of several hundred nanometers. It induces a greater degree of $\text{Li}_x\text{Si}_y\text{O}_z$ silicate formation over Li_xSi_y alloy. The results provided by the ToF-SIMS are corroborated by electrochemical measurement of the electrodes under inert Ar and Ar/ CO_2 environments indicating that the Ar/ CO_2 electrode retained a higher specific capacity over multiple cycles. Our ToF-SIMS characterization results show the effectiveness of thermal evaporation in producing a more stable electrode and an electrode with an effective lithium reserve that can preserve its capacity.

Keywords: Solid-state batteries, solid-electrolyte interface, ToF-SIMS, prelithiation, molecular imaging.

12:00pm **BT2+AS+CA+TF-ThM-17 Engineering Conformality of Lithium-Terephthalate Thin Films via Precursor Selection**, *Anish Philip*, *Joakim Jestilä*, *Milad Madadi*, Aalto University, Finland; *Jussi Kinnunen*, Chipmetrics Ltd, Finland; *Antti Karttunen*, *Maarit Karppinen*, Aalto University, Finland

Lithium-terephthalate (Li-TP) is recognized as a highly promising anode material for lithium-ion batteries.¹ The advantages of atomic/molecular layer deposition (ALD/MLD) technique have enabled the realization of Li-TP thin films in a highly controllable manner needed for its use in thin-film microbattery configuration.² However not much attention has been paid to understand the importance of selecting an appropriate lithium precursor for controlling the thin film properties and conformality, even though Li-TP materials are highly desirable from the battery application point of view. Given their potential application in 3D thin-film microbatteries, understanding both conformality and thin-film coverage (penetration depth) are also crucial. In this research, we investigated the impact of different lithium precursors on the Li-TP thin film properties, conformality and penetration depth (PD). The lithium precursors evaluated were: lithium tert-butoxide (LiO^tBu), lithium bis(trimethylsilyl)amide (Li-HMDS) and lithium 2,2,6,6-tetramethyl-3,5-heptanedionate (Li-THD); for the first two precursors the ALD/MLD process was developed/optimized as they had not been previously employed for the Li-TP thin film growth. For conformality measurements, we utilized lateral high-aspect-ratio (LHAR) PillarHall³ structures and a novel imaging ellipsometry technique that enables film thickness determination even at Ångström level. To support the experimental work, a detailed computational survey of the precursors was conducted. Through DFT optimizations combined with highly accurate DLPNO-CCSD(T) energy calculations the most prevalent oligomeric forms under typical ALD conditions were determined; the effective diameter (d_{eff}) and mean free path (MFP, λ) of the precursor's oligomeric form were found to play a major role in determining the conformality characteristics of the resultant Li-TP thin films (Figure 1). Among the three precursors investigated, Li-HMDS was found to result in Li-TP thin films with superior conformality characteristics. The current approach of using theoretical calculations to predict film conformality based on precursor molecular structure can significantly reduce the amount of experimental work required and streamline the screening process for precursors aimed at enhancing conformal coating.

References

- [1] M. Armand et.al., *Nat Mater*, **8**, 120–125, 2009.
- [2] M. Nisula, M. Karppinen, *Nano Lett.*, **16**, 1276–1281, 2016.
- [3] A. Philip et.al., *Small*, **20**, 2402608, 2024.

Advances in Battery Engineering, Interface Design, and Characterization Mini-Symposium Room 319 - Session BT-ThA

Advanced Characterization of Battery Interfaces

Moderators: Dr. Samantha Rosenberg, Kairos Power, Prof. Dr. Xiao-Ying Yu, Oak Ridge National Laboratory, USA

2:15pm BT-ThA-1 Designing Materials for High Performance Energy Storage Applications at Solid-Solid and Solid-Liquid Interfaces, *Ajay Karakoti*, Pacific Northwest National Laboratory **INVITED**

The performance of energy storage materials is fundamentally governed by interfacial phenomena, where ion transport, nucleation, and chemical degradation dictate cycle life and energy density. Probing these buried, dynamic interfaces requires combining advanced characterization, computational modeling, and augmented by data-driven workflows. This talk highlights two complementary efforts that leverage multiscale characterization, in-situ probes, and emerging computational frameworks to elucidate and engineer interfaces in next-generation solid-state and grid-scale lead-acid batteries.

The first part of the talk demonstrates a discovery framework for designing solid-solid interfaces in lithium-ion solid-state batteries by coupling state-of-the-art machine learning potentials with physics-based simulations on cloud high-performance computing. Screening across millions of candidate compositions yielded a tractable, shortlisted set of promising solid electrolyte candidates that were subsequently synthesized and experimentally validated. Postmortem characterization of cycled cells using SEM-EDS, XPS, and PXRD reveals how cathode-electrolyte interfacial stability and structural evolution govern long-term performance, informing design rules for composite electrolytes.

The second part of the talk identifies opportunities for utilizing data-driven frameworks to bridge our understanding of failure mechanisms in lead-acid batteries. The complex chemistry of this system necessitates probing reactions using a multi-modal toolkit spanning in-situ atomic force and optical microscopy, solid-state NMR, XPS, and synchrotron methods that link macroscopic failure modes, such as, irreversible sulfation and electrolyte stratification to atomic-scale interfacial structure. The rich, multimodal datasets emerging from these studies present a unique opportunity to bridge length scales and provide inputs for future machine learning and autonomous workflows aimed at formulating an overarching mechanistic understanding of battery failure and accelerating the discovery of new materials.

2:45pm BT-ThA-3 Advances in Neutron and X-Ray Imaging and Tomography Methods for in Situ Characterization of Lithium-Ion Batteries at NIST, *David Jacobson, Jacob LaManna, Elias Baltic, Daniel Hussey*, National Institute of Standards and Technology (NIST) **INVITED**

Neutron imaging has played an important role in characterizing electrochemical systems in the last 20 years. Neutrons penetrate metallic components of battery systems yet remain sensitive to changes in lithium concentration and/or hydrogenous electrolyte concentration, thanks to the large attenuation cross-section for ^6Li and H. When combined in operando with X-rays, it is possible to obtain a multi-modal picture of the electrochemical system, allowing for phase separation that is not possible with individual probes alone.

At NIST, the neutron imaging program operates two instruments, both with simultaneous neutron/X-ray tomography capabilities: the BT2 NIST Neutron Imaging Facility (NNIF), and the NG6 Cold Neutron Imaging Instrument (CNII).

In this presentation, we will discuss the current status of these facilities, planned future upgrades, the impacts on battery characterization, and examples of the techniques as applied to imaging lithium batteries. During the NIST Center for Neutron Research (NCNR) shutdown, we performed experiments at other facilities that will be discussed here. In addition, we have pursued upgrades to the NIST imaging facilities to improve spatial resolution, field-of-view, data acquisition, and data analysis speeds.

Because neutron sources are lower in intrinsic brightness than synchrotron X-ray sources, they require longer data collection times for 3D data sets. To improve neutron data acquisition speeds NIST has deployed machine-learning methods to reduce image noise allowing for shorter image acquisition times. NIST is also incorporating a high-speed server and network to provide near real-time data analysis to facility users.

Furthermore, we will discuss new cold neutron imaging methods for multi-scale characterization and the status of the NIST neutron microscope project.

3:15pm BT-ThA-5 Solid Electrolyte Interphase Formation between a Li-Metal Anode and an Ionic Liquid Electrolyte Using an Ab Initio Molecular Dynamics Approach, *Hafsa Ashraf, Luis A. Selis*, Texas A&M University; *Jorge M. Seminario*, Texas A&M University

Understanding and controlling lithium-metal interfacial phenomena is critical for developing safe, high-performance next-generation batteries. In this study, ab initio molecular dynamics simulations are used to investigate solid-electrolyte interphase (SEI) evolution and electrochemical interfacial stability for an ionic-liquid electrolyte in contact with a lithium-metal surface. The electrolyte consists of 1-ethyl-3-methylimidazolium bis(trifluoromethanesulfonyl)imide with 1 M LiTFSI. To improve interfacial performance, tetrafluoroborate is partially introduced as a counterion, and ethylene carbonate is included as a functional additive. The simulations show limited interfacial reactivity, suggesting strong chemical stability of the electrolyte formulation. Decomposition pathways indicate that lithium fluoride is the dominant SEI product, highlighting the potential of this IL-based electrolyte system to stabilize lithium-metal interfaces for advanced battery applications.

3:30pm BT-ThA-6 ASSD Student Award Finalist Talk: Strategies for ToF-SIMS of Buried Interfaces in Solid Polymer Electrolyte Batteries, *Reyhane Shavandi, Meghan Burns, Neelam Sunariwal*, University of Illinois at Chicago; *Sanja Tepavcevic*, Argonne National Laboratory; *Luke Hanley*, University of Illinois at Chicago

The solid electrolyte interphase (SEI) formed at lithium metal interfaces plays a critical role in determining the stability and performance of solid state batteries with polymer-based electrolytes. Time-of-flight secondary ion mass spectrometry (ToF-SIMS) combined with ion sputtering was used here to investigate the composition and spatial distribution of the buried SEI formed with a mixed of bis(fluorosulfonyl)imide (LiFSI) and poly(ethylene oxide) (PEO) electrolytes between lithium electrodes, $\text{Li}|\text{PEO-LiFSI-LiNO}_3|\text{Li}$. Gaseous cluster ion beam (GCIB) sputtering revealed that the SEI remained adhered to the Li electrode rather than to the polymer electrolyte, indicating its mechanical robustness and chemical stability. Depth profiles from ToF-SIMS analyses further revealed that addition of small amounts of LiNO_3 to the LiFSI/PEO electrolyte led to the formation of a nitrogen-rich SEI layer adjacent to the Li electrode surface, characterized by a strong CN $^-$ signal identified by high mass accuracy peak assignment (S.R. Shavandi, *et al.*, *J. Vac. Sci. Technol. B* 44, 2026, 012401). The presence of CN $^-$ signal indicated the presence of nitrogen-containing species within the inner SEI and a corresponding reduction of LiF-content within the SEI. Based on these findings, an anode free system using a Cu electrode, which both less expensive and nonflammable was examined. The Cu electrode was coated with a thin Au layer and Al_2O_3 nanoparticles were incorporated into the solid-state electrolyte to form $\text{Li}|\text{PEO-LiFSI-LiNO}_3+\text{Al}_2\text{O}_3|\text{Au/Cu}$. LiNO_3 was added to create a nitrogen-rich interfacial layer with excellent Li-ion transport ability, while Al_2O_3 nanoparticles served to mechanically reinforce the pure polymer electrolyte. Li plating onto bare Cu during electrochemical cycling is hindered by a large nucleation overpotential that leads to non-uniform and dendritic growth, but the Au coating onto Cu is thought to create a lithiophilic interface by forming Au-Li intermetallics that reduce the nucleation barrier and enable uniform Li deposition. Current efforts focus on combining the benefits of the nitrogen-rich interface with this lithiophilic Au-alloying strategy to better understand where Li preferentially plates and how the nitrogen-rich layer forms and evolves on Au/Cu versus metallic Li surfaces. ToF-SIMS directly probed Li nucleation and interfacial chemistry with depth profiling using either O_2 or GCIB sputtering guns to achieve high sensitivity to Li and buried interfacial species. Both two-dimensional depth profiling and three-dimensional chemical imaging were utilized to resolve the spatial distribution of Li relative to the Au interlayer.

3:45pm BT-ThA-7 Correlative Imaging and Surface Analysis of Air-Sensitive Materials, *Lu Ping*, Thermo Fisher Scientific, USA; *Hsiang-Han Tseng, Helen Oppong-Mensah, Tim Nunney*, Thermo Fisher Scientific, UK

Understanding complex materials increasingly requires the correlation of structural, morphological, and chemical information acquired across multiple analytical platforms. Imaging techniques such as scanning electron microscopy provide essential context on microstructure, defects, particles, and regions of interest, while surface-sensitive spectroscopies such as X-ray photoelectron spectroscopy reveal local chemistry, bonding, and elemental composition. For air-sensitive materials, however, transferring specimens

between instruments can alter the very surfaces and interfaces under investigation, making reliable correlation especially challenging.

This presentation describes recent developments enabling vacuum-protected transfer between electron microscopy and surface analysis instrumentation. The approach combines SEM-based imaging and site selection with subsequent XPS analysis using a two-way vacuum transfer capability, allowing selected regions to be analyzed while minimizing atmospheric exposure. The ability to move samples between instruments under controlled conditions supports iterative, correlative investigation without compromising reactive surfaces.

Examples will be presented from air-sensitive materials, including Li-ion battery components, where preservation of native surface chemistry is critical for interpreting degradation, interphase formation, and chemistry-structure relationships. These results illustrate how vacuum-integrated correlation of imaging and spectroscopy can provide a complete and more reliable picture of materials that are difficult to characterize using isolated techniques alone.

4:00pm BT-ThA-8 Correlating Surface Chemistry and Subsurface Structure in Battery Degradation Layers via XPS, HAXPES, AES, and TOF-SIMS, Sarah Zaccarine, Physical Electronics USA; Isaiah Oladeji, Sisom Thin Films; Jacob Schmidt, Juergen Scherer, Amy Ferryman, Physical Electronics USA

Understanding the formation and evolution of degradation layers at battery interfaces is critical for improving electrochemical performance, safety, and lifetime. These interphases are chemically complex and highly heterogeneous, requiring complementary, surface-sensitive analytical techniques to fully resolve their structure and composition. In this work, X-ray Photoelectron Spectroscopy (XPS), Auger Electron Spectroscopy (AES), and Time-of-Flight Secondary Ion Mass Spectrometry (TOF-SIMS) are combined to investigate both surface and subsurface features of degradation layers in advanced battery materials.

XPS provides quantitative elemental and chemical state information from the top ~1–10 nm, enabling identification of key inorganic and organic species within interphase layers and their evolution during electrochemical cycling. The use of hard X-ray photoelectron spectroscopy (HAXPES) further extends the probing depth into the subsurface (up to 30 nm), allowing non-destructive characterization of buried interfaces while maintaining chemical state sensitivity. AES offers high spatial resolution at the nanoscale, making it particularly effective for resolving compositional variations across interfaces and within localized degradation features. TOF-SIMS complements these approaches with ultra-high sensitivity to trace species and molecular fragments, along with 3D chemical imaging and sputter depth profiling capabilities, that reveal the layered structure and distribution of degradation products below the immediate surface.

Overall, the synergistic application of XPS, AES, and TOF-SIMS provides a comprehensive toolkit for correlating surface chemistry, subsurface composition, and interfacial structure in battery degradation layers. These insights are essential for guiding the design of more stable electrode-electrolyte interfaces and enabling next-generation energy storage technologies.

4:15pm BT-ThA-9 Airless Transfer for Battery Materials: Preserving Native Surface Chemistry for Analysis, Forrest Nichols, Dan Sullivan, Eurofins EAG

Analysis of materials that react with atmospheric chemicals, such as batteries, require airless preparation and airless transfer into the materials analysis tools. In this paper we describe the sample preparation to remove a cathode from a Li ion battery in a glove box in a dry room and placed into an airless transfer box. The sample is airlessly transferred into an XPS tool for analysis. This is followed by exposure of the sample to air for one minute and then another XPS analysis. The sample is then exposed to air for two days and a final analysis is performed. The change in the surface composition and chemistry is investigated as a function of air exposure time. Airless transfer enables accurate surface analysis of battery materials by preserving native chemistry.

Advances in Battery Engineering, Interface Design, and Characterization Mini-Symposium

Room Ballroom A - Session BT+AS+CA+TF-ThP

Advances in Battery Engineering, Interface Design, and Characterization Mini-Symposium Poster Session

BT+AS+CA+TF-ThP-1 Orientated Deposition of Li₂S for Fast-Charging Lithium-Sulfur Batteries, Jeong-Hoon Yu, *Jong-Sung Yu*, DGIST, Republic of Korea

Precipitation/dissolution of insulating Li₂S has long been recognized as the rate-determining step in lithium-sulfur (Li-S) batteries, which dramatically undermines sulfur utilization at elevated charging rates. Herein, we present an orientated Li₂S deposition strategy to achieve extreme fast charging (XFC, ≤ 15 min) through synergistic control of porosity, electronic conductivity, and anchoring sites of electrode substrate. Via magnesiothermic reduction of a zeolitic imidazolate framework (ZIF), a nitrogen-doped and hierarchical porous carbon with highly graphitic phase was developed. This design effectively reduces interfacial resistance and ensures efficient sequestration of polysulfides during deposition, leading to (110)-preferred growth of Li₂S nanocrystalline between (002)-dominated graphitic layers. Our approach directs an alternative Li₂S deposition pathway to the commonly reported lateral growth and 3D thickening growth mode, ameliorating the electrode passivation. Therefore, a Li-S cell capable of charging/discharging at 5C (12 min) while maintaining excellent cycling stability (82% capacity retention) for 1000 cycles is demonstrated. Even under high S loading (8.3 mg cm⁻²) and low electrolyte/sulfur ratio (3.8 μ L mg⁻¹), the sulfur cathode still delivers a high areal capacity of >7 mAh cm⁻² for 80 cycles.

BT+AS+CA+TF-ThP-2 High Voltage Li-ion Battery Cathodes: Surface Fluorination and Transition Metal Doping of Lithium Cobalt Oxide (LCO), *Falak Sher*, Carlos Yescas, Timothy Spila, Arghya Patra, Ben Zahiri, Paul Braun, University of Illinois at Urbana-Champaign, USA

Lithium cobalt oxide cathode (LCO) is extensively used as a cathode material in lithium-ion batteries for portable electronics. It is among the highest-performing cathode materials because of its structural stability, long cycle life, and high volumetric density at a cut-off voltage of 4.2 V (vs. Li/Li⁺). Significant energy density increases would be provided by lifting the cut-off voltage from 4.2 V to 4.6 V (vs. Li/Li⁺), increasing the specific capacity from 140 mAh/g to 200 mAh/g. However, at high-voltages, the cathode material undergoes severe challenges such as unstable cathode-electrolyte interface (CEI), undesirable permanent phase transformations, and oxygen loss which limit energy density and cycle life. To improve the high-voltage stability and electrochemical performance of LCO, we demonstrate plasma-assisted surface modification and fluorine, and transition metal doping. Incorporation of fluorine, in combination with transition elemental doping, protects the surface by reconstructing the layered structure of LCO into a short-range disordered rocksalt structure. Once protected, the LCO can be cycled to higher voltages, resulting in enhanced energy density and cycle-life. Time-of-Flight Secondary Ion Mass Spectroscopy (ToF-SIMS) analysis of both untreated and plasma engineered LCO confirms fluorination of the surface of plasma treated LCO. X-ray Fluorescence (XRF) and ToF-SIMS analyses of the plasma modified and pristine LCO show the incorporation of transition metals in the surface. High resolution STEM indicates that the surface is reconstructed from a layered LCO structure to a local disordered rocksalt structure. SEM images of the pristine and plasma treated LCO before and after cycling clearly show the increased stability of the plasma-treated material. Surface modifications. At higher cycling voltages (4.6 V vs Li/Li⁺) plasma modified LCO cathodes show significantly improved electrochemical results including longer cycle life, higher capacity retention, and higher energy density.

BT+AS+CA+TF-ThP-3 Development and Characterization of Sputtered Thin-Film Solid Electrolyte for Microbattery Applications, *Ananya Bansal*, Ramesh Chandra, Indian Institute of Technology Roorkee, India

Thin-film solid electrolytes are gaining significant attention for next-generation microbatteries due to their high safety, compact size, and compatibility with on-chip electronic devices. In this work, thin-film solid electrolyte was fabricated using RF magnetron sputtering, a scalable technique that enables precise control over film thickness, composition, and uniformity. The deposited films were characterized using X-ray diffraction (XRD), scanning electron microscopy (SEM), and X-ray

photoelectron spectroscopy (XPS) to investigate their structural, surface, and chemical properties. Electrochemical impedance spectroscopy (EIS) was employed to evaluate the ionic conductivity, activation energy, and interfacial behavior of the thin films. The influence of sputtering parameters on the microstructure and electrochemical performance of the electrolyte films is discussed. The results demonstrate that sputtered thin-film solid electrolytes exhibit good film uniformity, dense morphology, and high ionic transport conductivity ($\sim 10^{-3}$ S/cm) suitable for microbattery integration. This study highlights the potential of sputtering-based thin-film electrolytes for the development of compact, reliable, and high-performance all-solid-state microbatteries for wearable electronics, IoT devices, and miniaturized energy storage systems.

BT+AS+CA+TF-ThP-4 Fabrication and Characterization of the Thin Film Solid State Li and Na Ion Batteries, *SATILMIS BUDAK*, Zhigang Xiao, Mebougna Drabo, Aschalew Kassu, Richard Lagle, Jamar Dozier, Kendall Montgomery, Kelvin Perkins, Alabama A&M University

In this project, thin films of LiCoO₂ (cathode), LiPON (solid electrolyte), Sn (anode), and a multilayer stack combining these materials were deposited on silicon and silicon dioxide substrates using electron-beam evaporation. Samples were annealed at 50°C, 100°C, and 150°C, with unannealed films serving as a baseline. Electrical properties were observed using a Van der Pauw measurement system to obtain resistivity, mobility, carrier density, Hall coefficient, and sheet resistance, and were complemented by current-voltage (I-V) characterization for each material and for the multilayer stack. Further analysis involved Seebeck analysis and Scanning Electron Microscopy/Energy-Dispersive X-ray Spectroscopy (SEM/EDS) to observe sample thermoelectric properties and microstructure. Similar studies are being under study for Na ion batteries. Finding will be shared during the meeting.

Advances in Battery Engineering, Interface Design, and Characterization Mini-Symposium Room 319 - Session BT+AS+CA+TF-FrM

Solid-State Batteries

Moderators: Prof. Alexander Kozen, University of Vermont, Prof. Chuan-Fu Lin, Catholic University of America

8:15am BT+AS+CA+TF-FrM-1 Grain Structure Control of Evaporated Li Metal Films for Optimized Solid State Batteries, Andrew Westover, Peyton Carden, Oak Ridge National Laboratory, USA **INVITED**

Lithium metal is a critical anode material for next-generation, high-energy batteries. To realize these batteries, the lithium metal must be between thick, depending on the cathode chemistry. Furthermore, lithium metal must have controlled surface chemistry and high purity for optimal performance. Thermal evaporation is one of the most effective methods for producing thin lithium metal films that meet these criteria. In this work, we present our efforts to understand how to produce lithium metal films as thin as , and we explore the ability to fabricate lithium metal thin films with controlled microstructures. We have developed a process to modify the as-deposited lithium metal grain size in evaporated films from . Using these thin films with standard liquid electrolytes, localized high-concentration electrolytes, and solid electrolytes lead to significant differences in the cycling performance of lithium metal films. This work demonstrates the critical importance of microstructure control for realizing next-generation, high-energy lithium metal batteries.

This work was funded by the United States Department of Energy Transportation Technology Office Battery Materials Research Program and German-US Collaboration on Energy Storage under Simon Thompson and Tien Duong.

8:45am BT+AS+CA+TF-FrM-3 Unravelling the Mechanism of Al₂O₃ Atomic Layer Deposition on Li₆PS₅Cl for All-Solid-State Batteries, Kyobin Park, Donghyeon Kang, Taewoo Kim, Vepa Rozyyev, Anil Mane, Hacksung Kim, Francisco Vargas, Zachary Hood, Peter Zapol, Justin Connell, Jeffrey Elam, Argonne National Laboratory

Sulfide superionic conductors with the argyrodite structure (e.g., Li₆PS₅Cl, LPSCI) are extremely promising for all solid-state batteries, but poor atmospheric stability and high interfacial reactivity limit their widespread adoption. Coating LPSCI powders with ultrathin, metal oxide coatings using atomic layer deposition (ALD) mitigates these problems, protecting LPSCI against atmospheric degradation¹ and reducing reactivity with Li metal, yielding more stable cycling². Despite significant promise, the ALD mechanism is unknown, hampering the development of new coating chemistries.

In this study, we elucidate the mechanism for Al₂O₃ ALD on LPSCI using trimethyl aluminum (TMA) and H₂O by combining *in situ* Fourier transform infrared spectroscopy measurements and *ex situ* solid-state magic angle spinning nuclear magnetic resonance, UV Raman spectroscopy, and X-ray photoelectron spectroscopy measurements with density functional theory calculations. We determine that ALD Al₂O₃ nucleates promptly via TMA reaction with native -OH, -SH, and PS₃-OH groups to form transient C-Al-O(S) species that are rapidly hydrolyzed during the subsequent H₂O exposure. This reversible transformation maintains surface nucleophilicity and prevents sulfide decomposition. The resulting layer-by-layer growth leads to highly conformal Al₂O₃ coatings on LPSCI.

This detailed understanding of ALD surface reactions provides critical insights guiding the selection of future ALD chemistries with greater enhancement in cycling performance.

1. *Acs Materials Letters* **2024**, 6, (12), 5409-5417.
1. *Advanced Materials* **2023**, 35, (21), 13.

9:00am BT+AS+CA+TF-FrM-4 Engineered Cathode Chemomechanics Enables Ultra-Low Stack Pressure Solid-State Batteries, Paul Braun, University of Illinois Urbana-Champaign

In solid-state batteries, stresses resulting from electrode material chemomechanics are strongly coupled to solid electrolyte-electrode interface failures. Such failures are significant barriers to realization of practical Li metal solid-state batteries. While high external pressures can reduce these failures, the required pressures are impractical, and thus a different approach to reducing interface failures is required. We show that control of cathode chemomechanical stress provides an important path for

realization of solid-state batteries which operate at commercially relevant low (e.g., <1 MPa) stack pressures. Using a series of cathodes with different chemomechanics we provide experimental evidence of the role of electrode chemomechanics in solid-state battery failure. Our model systems reveal that electrode chemomechanics significantly alter Li metal plating and stripping behavior at low stack pressure. We utilize these learnings to build long cycle-life solid-state batteries with practical areal capacity (5 mAh/cm²) operating under a 1 MPa stack pressure and at room temperature. Our findings highlight the importance of controlling positive electrode chemomechanics to realize low stack pressure solid-state batteries.

9:15am BT+AS+CA+TF-FrM-5 Pulsed Laser Deposition of Solid Electrolytes: How Limited Thermal-Budget Processing Relates to Li-ion Conductivity, Nicola Perry, University of Illinois; En Ju Cho, Kai-Wei Lan, Nicola Perry, University of Illinois Urbana-Champaign **INVITED**

Near-room-temperature, thin-film deposition of air-sensitive Li-ion conductors can enable multilayer solid electrolytes and coatings for high-energy-density batteries and microdevices, compatible with thermally sensitive components. We investigate the capabilities of pulsed laser deposition for fabrication of high-quality layers of low-voltage- and high-voltage-stable solid-electrolyte compositions, including low-melting antiperovskite hydroxyhalides and higher-melting nasicon-structured oxides that contain glass-forming elements. The influence of growth parameters such as temperature, pressure, and laser fluence on stoichiometry, density, roughness, crystallinity, and conductivity is explored. Grazing-incidence nuclear reaction analysis paired with Rutherford backscattering spectrometry provides insight into light-element stoichiometry (e.g. Li, Cl, P) and the expected loss of Li (e.g.) with increasing temperature (given its volatility) and growth pressure (given its mass that enables scattering). Strategies to control stoichiometry of multiple light elements and deposition rates, through composite targets and sequential multi-target deposition, are demonstrated. X-ray reflectometry, grazing-incidence diffraction, and ellipsometry shape the understanding of structural dependence on growth parameters. We also examine the benefit of integrated impedance spectroscopy in the PLD chamber during and after growth to provide insights into transport behavior in a clean (air-free) environment as a function of temperature. We find most compositions benefit from vacuum deposition, but while the antiperovskite hydroxyhalides form single-phase, highly conductive films at room temperature, with phase decomposition and conductivity degradation above ~60 C, the nasicon-structured electrolytes benefit from post-deposition anneals to improve crystallinity and conductivity.

9:45am BT+AS+CA+TF-FrM-7 Open-Circuit Energy Band Alignment at NMC/LLZTO and LLZTO/Li Interfaces for Solid-State Microbattery Applications, Austine Amisi, Raquel-Garza Hernandez, Fabian Ambriz-Vargas, Centro de Investigaciones en Óptica, Mexico

Stable electrode/electrolyte interfaces are critical for the electrochemical performance and long-term reliability of solid-state microbatteries. This work reconstructs the open-circuit energy band diagram of an all-solid-state LiNi_{0.8}Mn_{0.2}Co_{0.2}O₂ (NMC)/Li₇La₃Zr_{2-x}Ta_xO₁₂ (LLZTO)/Li microbattery stack through band alignment analysis of the NMC/LLZTO cathode–electrolyte and LLZTO/Li electrolyte–anode heterojunctions. Thin-film bilayers were fabricated by RF magnetron sputtering and thermal evaporation. Valence and conduction band offsets (VBOs and CBOs) were experimentally determined using the Kraut method through conventional and angle-resolved X-ray photoelectron spectroscopy (XPS/ARXPS). A VBO of +3.13 ± 0.1 eV and a CBO of -6.00 ± 0.1 eV were measured at the NMC/LLZTO interface, with a large CBO that suggests the formation of an electronically insulating interface that suppresses electrolyte decomposition at the cathode during charging. In contrast, a VBO of +1.92 ± 0.1 eV and a near-zero CBO of +0.08 ± 0.1 eV were determined at the LLZTO/Li heterojunction, indicating an electronically conductive interface that promotes electron accumulation and facilitates LLZTO degradation upon contact with the anode. An electrochemical potential window of 6.00 ± 0.1 eV and an open-circuit voltage of 3.29 ± 0.1 eV were obtained from the reconstructed full-stack band diagram. These results establish a direct link between interfacial band alignment and potential degradation mechanisms and provide critical design guidelines for advancing the operational stability of LLZTO-based solid-state microbatteries.

Friday Morning, November 13, 2026

10:00am **BT+AS+CA+TF-FrM-8 LiPON-Enabled Reduction of V₂O₅ during Fabrication of Thin-Film Ionic Devices**, *Leopoldo Tapia-Aracayo, David Stewart, Gary Rubloff*, University of Maryland College Park

Vanadium (V) Oxide (V₂O₅) is a well-researched layered oxide with high capacity (375 mAh/g), often cited for its applications in energy storage, electrochromic, and neuromorphic applications. In the field of thin-film ionic devices, we can sputter Lithium Phosphorus Oxynitride (LiPON) on top of crystalline V₂O₅, creating our electrolyte/electrode stack. This sequence simplifies the fabrication of thin-film ionic batteries as sputtering LiPON can insert Lithium up to 2 Li⁺ per V₂O₅. We wanted to address the roughness of the LiPON/V₂O₅ interface by altering the fabrication steps to form a smooth interface, but unintentionally reduced the V₂O₅ to a VO₂ phase.

This smooth interface was achieved by sputtering amorphous V₂O₅ thin films in Ar/O₂ environment, then coated with LiPON, and subjected to post-deposition annealing in N₂ gas environment at 300 °C. The N₂ gas environment for the post-deposition annealing would crystallize the V₂O₅ and preserve the LiPON composition. We tracked the formation of a VO₂ phase, which was not observed for N₂ annealed V₂O₅ without LiPON, which remains VO₂ free. We used electrochemical cycling to probe the presence and evolution of the VO₂ phase, illustrating VO₂ reaction peaks instead of characteristic crystalline V₂O₅ peaks in the 2.2 V – 4.0 V vs Li⁺/Li window. (Seen in Figure 1). We will discuss how this behavior may be caused by LiPON-enabled reduction pathways at the buried interface. We confirm this reduction is limited to the interface as analysis via Raman identifies lithiated V₂O₅ phases. Systematic variation of LiPON sputtering parameters and annealing conditions, while holding LiPON thickness constant, reveal processing regimes that drive VO₂ formation versus preserving V₂O₅. Ultimately, preserving the morphological benefits of a smooth LiPON/ V₂O₅ interface while constraining VO₂ formation, providing guidance for designing desired application-specific vanadium-oxide phases.

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