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 ($LiBO_x$), 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^- -> 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_x) 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 ~0.35 Ω·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} Ω.m** in the early cycles, resistivity increases by several orders of magnitude, reaching **1-7 Ω.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 columbic 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 $Li_xSi_yO_z$ 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 $Li_xSi_yO_z$ 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 $Li_xSi_yO_z$ 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) PillarHall[3] 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

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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 Li_2S 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 Li_2S 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 Li_2S nanocrystalline between (002)-dominated graphitic layers. Our approach directs an alternative Li_2S 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 μL 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 mhA/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 lab 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 (~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 $LiCoO_2$ (cathode), LiPON (solid electrolyte), Sn (anode), and a multilayer stack combining these materials were deposited on silicon and silicondioxide 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.

Chemical Analysis and Imaging at Interfaces
Room Ballroom A - Session CA-ThP

Chemical Analysis and Imaging at Interfaces Poster Session

CA-ThP-1 Synthesis and Characterization of Bayerite sub-Micrometer Particles, ***Mackenzie Savage***, *Xiao-Ying Yu,* Oak Ridge National Laboratory

The Hanford Site in Washington state houses the largest collection of nuclear waste in the United States, including 56 million gallons of mixed waste that must be processed for vitrification by the Department of Energy (DOE). However, the waste contains many insoluble micro- and nanoparticles that have unpredictable rheology due to their complex chemistry and are difficult to model. Aluminum hydroxides and oxyhydroxides such as bayerite make up a significant portion of these particles, and they must be filtered out before vitrification, as excess amounts can degrade the quality of the glass. Supply chain issues and a lack of information on their aqueous behavior has led to this study of aluminum (oxy)hydroxide nanoparticles. This research synthesizes bayerite micro- and nanoparticles using a hydrothermal synthesis process, namely by titrating and heating an aluminum hydroxide gel precursor solution. The resultant crystal structure of the largest particles was confirmed via powder x-ray diffraction, and elemental analysis confirmed that the particles were composed of bayerite. Scanning electron microscopy showed that particle sizes ranged from 70 nm needles to 4 μm particles in a bimodal distribution depending on the crystallization interval, with particles plateauing in size after about 48 h. Future work is needed to refine synthesis methods to reduce impurities and to obtain images of smaller particles at higher resolutions. Raman and Fourier Transform Infrared (FTIR) spectroscopy are also expected to confirm crystal structure of smaller particles. In situ characterization of particles in solution is necessary to observe the aqueous behavior of bayerite nanoparticles and how their rheology at varying pH might affect processing of Hanford waste.

CA-ThP-2 Enhanced Nanoprojectile Secondary Ion Mass Spectrometry (NP-SIMS): Correlation Among Spatially Resolved Secondary Ions, ***Pierre Hirchenhahn***, *Stanislav Verkhoturov,* Texas A&M University*; Michael J. Eller,* University of Mississippi*; Emile A. Schweikert,* Texas A&M University

Nanoprojectile secondary ion mass spectrometry (NP-SIMS) consists in bombarding an analyte with a heavy projectile in an event-by-event fashion. Each impact is resolved in space and time. Thus, the secondary ions (SIs) from each impact can be mass analyzed and recorded as individual mass spectra. The crater size is in the range of 10-15 nm wide and depth. These dimensions enable molecular analysis with unparalleled

lateral resolution. Yet more information can still be extracted from the SI, by considering their anisotropic distribution. The eject have distinct spatial distributions set by their axial and radial energies. These parameters in turn reflect the molecular environment, the formation-ejection mode and metastable decay. They can be accessed with the temporally and spatially correlated detection of SIs. We demonstrate spatially resolved SI analysis on a homogeneous surface of Tris(8-hydroxyquinolinato)aluminium (Alq_3) sublimated on a silicon substrate. The NP-SIMS measurement was performed on a custom-made system equipped with a fullerene effusion source accelerated at 50 keV coupled to a linear time-of-flight mass analyzer with a 64 anodes detector. We identify analyte-specific moieties, SIs synthesized by impact chemistry by their distinct spatial distributions. The latter can be crucial for enhanced accurate identification of SIs in surfaces of unknown composition.

CA-ThP-3 Studying Neutron Irradiation Effects on Lithium Aluminate Pellets Using Tof-Sims, ***Xiao-Ying Yu,*** *Jiyoung Son,* Oak Ridge National Laboratory*; Tanguy Terlier,* Rice University*; Gabriel Parker,* Oak Ridge National Laboratory*; David Senor,* Pacific Northwest National Laboratory

We studied tritium breeding lithium aluminate pellets in the Tritium-Producing Burnable Absorber Rod.The selected irradiated pellets have three different thickness, namely standard, thin, and thick wall thickness, which relates to their tritium producing and retention capacities.Irradiated materials were reduced to small sizes using scanning electron microscopy (SEM) – focused ion beam (FIB).Light isotopes, such as deuterium, tritium, and lithium, are detected using highly sensitive time-of-flight secondary ion mass spectrometry (ToF-SIMS).We show that the standard and thin thickness pellets are better in retaining tritium than the thick pellet in tritium production.The thinner wall pellet retains more tritium than the standard or thick ones.Isotope exchange reactions take place and tritiated hydrocarbons exist in all pellets.As the first study of irradiated pellets using ToF-SIMS, this work provides a new way to determine isotopic abundance of irradiated materials, improving understanding of tritium breeding in fission and fusion reactors.

CA-ThP-4 In-Plasma XPS: A New Operando Metrology For Process Development and Control, ***Andrei Kolmakov,*** NIST

Modern near-ambient-pressure X-ray photoelectron spectroscopy (NAP or AP-XPS) instruments now cover the pressure range typical of standard plasma applications, expanding the capabilities of XPS into plasma environments. We recently demonstrated that XPS spectra can be successfully collected in these conditions, extending the application of XPS to plasma-surface interactions [1]. We highlighted the influence of plasma chamber wall reactions on sample surface chemistry and showed that plasma-XPS can capture plasma chemistry in the gas phase [2]. Plasma-induced charging and damage of wafers is a well-known challenge in semiconductor fabrication [3]. We apply plasma-XPS to model poorly conducting samples and observe anomalous XPS binding energy shifts due to sample charging during plasma exposure. We propose mechanisms that explain these shifts. Additionally, we observed plasma-induced binding-energy shifts and peak splitting in the XPS spectra of the plasma gas phase, which were related to the local plasma potential and its time alterations [4]. Overall, plasma-XPS metrology offers an opportunity to record real-time surface chemistry on plasma-exposed surfaces, along with wafer charge and plasma diagnostics, and has significant potential to advance semiconductor process development and control, as well as defect-mitigation strategies.

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CA-ThP-5 A High-Throughput Multimodal Workflow for Characterizing Soil Mineral–Organic Interfaces with XPS, FTIR, SEM/EDS and ToF-SIMS, ***Vaithiyalingam Shutthanandan,*** *Ajay Karakoti,* Pacific Northwest National Laboratory*; Catherine Pettinger,* University of Wisconsin-Madison*; Kavin Thangaraj,* Washington State University*; Odeta Qafoku, Mark Engelhard, Bhuvana Modachur Sivakumar, Zihua Zhu, Ravi Kukkadapu,* Pacific Northwest National Laboratory*; Erica Majumder,* University of Wisconsin - Madison

Understanding how soil carbon is stabilized as mineral–associated organic matter (MAOM) at the molecular scale is critical for more accurately representing soil processes in Earth system models. In particular, quantifying the chemistry and spatial organization of organic matter (OM) at mineral interfaces including ion–binding mechanisms, the distribution and density of reactive sites, and the arrangement of functional groups which provides key insight into the persistence, transformation, and loss of soil carbon. To address these questions, we developed a high–throughput, multimodal imaging and spectroscopy workflow that characterizes mineral–organic interfaces in terms of their elemental composition, molecular structure, and micro– to nanoscale topology. This approach integrates X–ray photoelectron spectroscopy (XPS), Fourier–transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), and energy–dispersive X–ray spectroscopy (EDS), yielding complementary information on bonding environments, functional group chemistry, surface morphology, and mineral composition. We implemented a custom high–throughput sample preparation method based on a dot–blot style deposition system, enabling rapid and reproducible preparation of many small soil aliquots or mineral–OM extracts on a single substrate. This configuration allows all four analytical techniques to probe the same set of spots, ensuring direct comparability of chemical and structural measurements across methods. With this platform, we can analyze approximately 50 samples per day, supporting systematic, large–scale characterization of MAOM across environmental gradients. In this study, samples are drawn from the MONet project, providing a broad and spatially diverse soil inventory for establishing quantitative relationships between mineral surface properties, organic matter chemistry, and carbon stabilization.

CA-ThP-6 Influence of Surface Preparation on Au/P-Type Hgcdte Interfaces Investigated by Xps Depth Profiling, ***Alexandra Colas--Reuillon,*** *Clément Lobre, Eugénie Martinez, Steven Bel, Marc Veillerot, Sarah Petit, Olivier Gravrand,* CEA-Leti, France

P-type metal contacts on semiconductors remain a major technological challenge, particularly in Mercury Cadmium Telluride ($Hg_{1-x}Cd_xTe$, MCT), a reference material for cooled infrared detectors [1]. In the context of the SWaP-C (Size, Weight, Power, and Cost) approach, current developments aim to increase the operating temperature of infrared devices. However, higher temperatures are associated with anomalous pixel responses and increased noise levels, partly attributed to non-ideal Schottky-type behavior at the metal/p-type MCT interface.

Among the different strategies proposed to improve contact quality, surface preparation prior to metallization plays a critical role through the reduction of interface defects, passivation of dangling bonds, and control of interfacial oxides. Despite previous XPS studies, the mechanisms governing contact formation remain insufficiently understood. In this work, we investigate the influence of surface preparation on metal/MCT interfacial reactivity using X-ray Photoelectron Spectroscopy (XPS) combined with gentle Ar^+ sputter depth profiling.

Gold thin-film contacts (~10 nm) were deposited on arsenic-doped p-type $Hg_{1-x}Cd_xTe$ (x = 0.29) with a carrier concentration of ~10^{18} cm^{-3}. Two surface preparation routes were compared prior to metallization: (i) a deoxidized surface obtained by dry plasma treatment followed by wet chemical etching, and (ii) a deliberately oxidized surface prepared by plasma treatment only. Depth profiles were acquired by XPS (Al Kα source, Quantes, ULVAC-PH) combined with low-energy Ar^+ sputtering. Quantification was corrected using sputter sensitivity factors determined from a non-metallized reference MCT sample.

The measurements revealed strong preferential sputtering effects following the sequence Hg >> Te > Cd, attributed to the fragility of Hg–Te bonds. After correction, the Au/MCT interface formed on the intentionally oxidized surface appeared significantly sharper than that obtained on the deoxidized surface. Surface deoxidation promotes Au diffusion into the MCT as well as reciprocal interdiffusion processes. In contrast, the plasma-grown oxide layer, richer in HgO and CdTeO species than the native oxide formed after air exposure, likely stabilizes the surface and acts as a barrier against Au interdiffusion during deposition.

Such diffusion phenomena may strongly affect the electrical quality of the contacts through the formation of interfacial defects, local stoichiometry changes, and Au-induced doping effects.

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CA-ThP-7 Multi-modal analysis of NIST standard Iron Ore for establishment of the LIOS database, ***Gabriel Parker***, *Mackenzie Savage,* Oak Ridge National Laboratory, USA*; Michael Mulholland,* Idaho National Laboratory*; Xiao-Ying Yu,* Oak Ridge National Laboratory, USA

Abstract: The US Department of Energy (DOE) recently announced $155 million to advance American industrial innovation toward enhancing the capabilities of US national laboratories to develop technologies which will improve technological innovation, reduce costs, and increase prosperity for American workers and consumers. The DOE Office of Critical Minerals and Energy Innovation (CMEI) invested in new programs to address critical industrial challenges in areas such as energy intensive industry, cross sector technologies, data center collaboratives, and national library for iron ore and scrap. This library for iron ore and scrap (LIOS) project focuses on the iron ore and steel scrap to develop a national resource center and to deliver advanced characterization of iron ore and steel scrap samples of shared or donated by users for boosting American industrial innovations. The ORNL team's efforts are to develop analytical methods using X-ray diffractometry (XRD), inductive coupled plasma mass spectrometry (ICP-MS), and scanning electron microscopy (SEM) with energy dispersive x-ray spectroscopy (EDS) and electron backscatter detection (EBSD) to provide detailed characterization of the natural and processed/recycled feedstocks. In the first step, several iron ore reference materials available from National Institute of Standards (NIST) will be used to establish the analytical benchmarking for XRD, ICP-MS, and SEM/EBSD. Additionally, time-of-flight secondary ion mass spectrometry and atom probe tomography will be used as complementary techniques to verify composition and microstructures at the nanoscale. Our results provide the necessary baseline measurement to build the foundation for a resource library, which will provide important technical information for industrial needs.

Keywords: iron ore, SEM/EBSD, ICP-MS, XRD, ToF-SIMS, chemical imaging

CA-ThP-8 Exploring Radiolytic Chemistry of Tin Oxide Photoresists for Extreme Ultra-Violet (EUV) Photolithography Using Plasma-XPS, ***Trey Diulus***, NIST-Gaithersburg*; Priyanka Ketkar, Conan Weiland, Cherno Jaye,* NIST*; J. Anibal Boscoboinik, Ashley Head,* Brookhaven National Laboratory*; Andrei Kolmakov, Daniel Sunday, R. Joseph Kline,* NIST-Gaithersburg

Recent developments in semiconductor nanomanufacturing towards next generation extreme ultraviolet (EUV) lithography now allows for fabrication of leading edge microchips with even smaller features. EUV lithography utilizes higher photon energy than the previous industry standard, which allows for patterning with higher resolution. This is compromised by low photoabsorption cross-sections for elements commonly found in conventional polymer-based photoresists. As a result, tin oxide organometallic nanoclusters are being optimized as photoresist materials, as Sn has a cross section 30x higher than carbon. Unfortunately, this transition to metal oxides yields a different radiation mechanism that results in a solubility contrast compared to their polymer counterparts, while further contrasting in post exposure processing. To identify chemical changes upon EUV exposure, we prepared spin coated thin films of a well-known Sn_{12} dodecamer cluster and treated the films with flood exposures for a range of EUV dosages to correlate changes in solubility with structural changes in the resist, including loss of ligands and cross-linking between clusters. In addition to EUV exposure, we also exposed samples to O_2 and H_2 plasmas using an AC-driven cold cathode 22 kHz plasma to simulate basic post exposure etch processing. Recently, Plasma-XPS has been developed where x-ray photoelectron spectroscopy (XPS) can be collected during exposure to plasma, allowing for studies of chemical changes in operando. We additionally utilize x-ray absorption spectroscopy (XAS) to examine the electronic structure of the elemental components ex situ, after EUV and/or plasma exposure. XPS before and after EUV irradiation shows a decrease in C 1s intensity along with an increase in the O 1s corresponding to the metal oxide, which is expected as butyl ligands are cleaved and the clusters cross-link. Comparing the C and O K-edges for the unexposed versus EUV exposed samples display a clear change suggestive of an increase in carbonyl chemistry. Furthermore, exposure to H_2 plasma seems to further pattern the film, likely due to UV emission from the hydrogen Lyman series lines. Overall, we provide a more detailed understanding of the radiation induced chemistry that we anticipate will allow for the design of more efficient EUV photoresists and can lead to advances in EUV lithography.

CA-ThP-10 ToF-SIMS Analysis of Surface Deposited Reaction Species in Selectively Passivized MXene Catalysts, ***Tobias Misicko***, *Gabriel Parker,* Oak Ridge National Laboratory, USA*; Yang Xiao,* Louisiana Tech University*; Xiao-Ying Yu,* Oak Ridge National Laboratory, USA

Heterogeneous catalysis is controlled by the chemistry of the outermost atomic layers of catalyst surfaces and interfaces, where active metals, promoters, supports, and adsorbed reactants interact to form the catalytic system. A key deactivation pathway is coking, leading to deposition of carbonaceous species onto active surfaces, which causes a progressive loss of catalytic activity over time. Selective passivation of heterogeneous catalysts, the intentional neutralization of unselective and unproductive sites[1], has been applied to platinum catalysts using MXene.MXene is a class of two-dimensional (2D) metal carbides with the empirical formula of $M_{n+1}X_nT_x$, where M is an early transition metal, X is a carbon or nitrogen, and T is a surface functional group (such as F^- or OH^-). In prior work,[2,3] platinum was deposited onto Mo_2TiC_2 MXene via incipient wetness impregnation to yield a 0.5 wt.% Pt/Mo_2TiC_2 nanolayer catalyst. This catalyst demonstrated excellent activity, with turnover frequencies (TOFs) of 0.4–1.2 s^{-1} for the conversion of methane[2] and ethane[3]. It also exhibited high selectivity, >98% toward C_2 products in non-oxidative coupling of methane (NOCM) and >95% toward ethylene in catalytic dehydrogenation of ethane, along with robust stability, showing no loss of activity over 72 h and 24 h time-on-stream (TOS) for NOCM and ethane dehydrogenation, respectively. This stability is attributed to the catalyst's strong coke resistance. Following kinetic performance evaluation, time-of-flight secondary ion mass spectrometry (ToF-SIMS) measurements were conducted to probe surface-deposited species formed during the catalytic conversion of short-chain alkanes. Surface spectral analysis, depth profiling, and mass spectral imaging were performed to elucidate the identities and spatial distribution of carbonaceous species on the 0.5 wt.% Pt/Mo_2TiC_2 surface. Surface spectral analysis revealed the presence of many hydrocarbon species, including known coke precursors such as tropylium (C_7H_7).

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CA-ThP-11 Multimodal Raman and Luminescence Techniques for Defect Characterization in Wide Band Gap Semiconductors., ***Praveena Manimunda***, HORIBA

Wide band gap semiconductors such as SiC, Ga_2O_3, and GaN, are essential for next–generation power electronics, optoelectronics, and quantum technologies, yet their performance is strongly influenced by strain and crystallographic defects introduced during growth and processing. In this work, we demonstrate how Raman spectroscopy, Photoluminescence (PL), Time–Resolved PL (TRPL), and Cathodoluminescence (CL) provide a comprehensive, non–destructive characterization platform for evaluating these materials with high spatial and spectral resolution. Case studies on 4H–SiC and SnO_2 doped Ga_2O_3 nanowires illustrate how this multimodal approach enables the identification of defect–related optical signatures and microstructural non–uniformities that impact material quality and device reliability. Raman spectroscopy reveals crystal quality and strain distributions, while PL and TRPL provide insight into impurity levels, recombination dynamics, and defect–related transitions. CL further correlates luminescence features with microstructural variations at the microscale. Together, these results highlight how integrated optical and electron–beam characterization supports optimization of epitaxial growth, improves device yield, and accelerates materials development across high–performance wide band gap semiconductor technologies.

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 Li_6PS_5Cl 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., Li_6PS_5Cl, LPSCl) are extremely promising for all solid-state batteries, but poor atmospheric stability and high interfacial reactivity limit their widespread adoption. Coating LPSCl powders with ultrathin, metal oxide coatings using atomic layer deposition (ALD) mitigates these problems, protecting LPSCl against atmospheric degradation[1] and reducing reactivity with Li metal, yielding more stable cycling[2]. Despite significant promise, the ALD mechanism is unknown, hampering the development of new coating chemistries.

In this study, we elucidate the mechanism for Al_2O_3 ALD on LPSCl using trimethyl aluminum (TMA) and H_2O 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 Al_2O_3 nucleates promptly via TMA reaction with native -OH, -SH, and PS_3-OH groups to form transient C-Al-O(S) species that are rapidly hydrolyzed during the subsequent H_2O exposure. This reversible transformation maintains surface nucleophilicity and prevents sulfide decomposition. The resulting layer-by-layer growth leads to highly conformal Al_2O_3 coatings on LPSCl.

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

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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/cm^2) 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 $LiNi_xMn_yCo_zO_2$ (NMC)/$Li_7La_3Zr_{2-x}Ta_xO_{12}$ (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.

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 (V_2O_5) 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 V_2O_5, 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 V_2O_5. We wanted to address the roughness of the LiPON/V_2O_5 interface by altering the fabrication steps to form a smooth interface, but unintentionally reduced the V_2O_5 to a VO_2 phase.

This smooth interface was achieved by sputtering amorphous V_2O_5 thin films in Ar/O_2 environment, then coated with LiPON, and subjected to post-deposition annealing in N_2 gas environment at 300 ◦C. The N_2 gas environment for the post-deposition annealing would crystallize the V_2O_5 and preserve the LiPON composition. We tracked the formation of a VO_2 phase, which was not observed for N_2 annealed V_2O_5 without LiPON, which remains VO_2 free. We used electrochemical cycling to probe the presence and evolution of the VO_2 phase, illustrating VO_2 reaction peaks instead of characteristic crystalline V_2O_5 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 V_2O_5 phases. Systematic variation of LiPON sputtering parameters and annealing conditions, while holding LiPON thickness constant, reveal processing regimes that drive VO_2 formation versus preserving V_2O_{-5}. Ultimately, preserving the morphological benefits of a smooth LiPON/ V_2O_5 interface while constraining VO_2 formation, providing guidance for designing desired application-specific vanadium-oxide phases.

Chemical Analysis and Imaging at Interfaces
Room 321 - Session CA1-FrM

Chemical Analysis and Imaging at Interfaces Oral Session I

Moderators: Dr. Andrei Kolmakov, National Institute of Standards and Technology (NIST), **Gabriel Parker**, Oak Ridge National Laboratory

8:15am **CA1-FrM-1 Semiconductor Manufacturing Metrology**, ***Alexander Liddle***, Canon Nanotechnologies Inc. **INVITED**

The old adage from optics that, if it can be measured, it can be made, is nowhere more true than for semiconductor manufacturing.Leading-edge devices contain nanoscale features, and require sub-nanometer dimensional, compositional, and interfacial control over length scales up to the diameter of a 300 mm wafer.Every step of the fabrication process, from deposition, to lithography, to etch, depends on sophisticated metrology, often in-line and at speed, to achieve the level of precision and accuracy needed to flawlessly produce the billions of transistors that make up today's high-performance integrated circuits.In this talk, I will provide a high-level overview of some of the outstanding measurement challenges that must be overcome in order to maintain the semiconductor industry's extraordinary progress in manipulating and controlling matter at the atomic scale.In particular, I will focus on the metrology needed to enable the transition to highly three-dimensional structures, such as gate-all-around (GAA) transistors, high-capacity NAND flash memory, and hybrid-bonded high-bandwidth memory.

8:45am **CA1-FrM-3 High Performance Computing and Artificial Intelligence Enabled Materials Characterization and Experimental Automation**, ***Mathew Cherukara***, Argonne National Laboratory **INVITED**

The capabilities provided by next generation light sources along with the development of new characterization techniques and detector advances are revolutionizing materials characterization (metrology) by providing the ability to perform scale-bridging, multi-modal materials characterization under *in-situ* and *operando* conditions. For example, providing the ability to image in 3D large fields of view (~mm^3) at high resolution (<10 nm), while simultaneously acquiring information about structure, strain, elemental composition, oxidation state, photovoltaic response etc.

However, these novel capabilities dramatically increase the complexity and volume of data generated. Conventional data processing and analysis methods become infeasible in the face of such large and varied data streams. The use of AI/ML methods is becoming indispensable for real-time analysis, data abstraction and decision making at advanced, high-data rate instruments. I will describe how high-performance computing (HPC) along with AI on edge devices enables real-time data analysis and self-driving experiments, creating the next generation of AI-powered materials characterization tools.

As instrument and analysis workflows increase in complexity, large language models (LLM) have the potential to assist and enhance the productivity of scientists. I will describe early experiments with AI-powered scientific co-pilots that can provide assistance through every stage of an experiment; planning, execution, analysis and even instrument operation.

9:15am **CA1-FrM-5 Challenges in Real-Time Plasma-Surface Diagnostics**, ***Vincent Donnelly***, University of Houston **INVITED**

An array of quantitative surface diagnostic methods has been used over the past 65 years to gain detailed insights into the physics and chemistry of surfaces. These include Auger electron spectroscopy (AES), x-ray photoelectron spectroscopy (XPS), low-energy electron diffraction and similar methods. These techniques required high- or ultrahigh vacuum to detect electrons without collisions with background gas, as well as to keep surfaces clean of unwanted contaminants. Similar constraints are imposed by mass spectrometry methods such as temperature programmed desorption (TPD) and secondary ion mass spectrometry (SIMS). When the gas pressure exceeds a level where the collision mean-free-path becomes comparable to a characteristic distance (e.g. 1 mTorr at ~5 cm), the primary information carried by the electron energy, and neutral or ion desorption product is degraded or lost. The problem is exacerbated in a plasma, where large electron and ion densities, electric fields and magnetic fields dilute and distort the surface analysis signals. Despite these issues, considerable advances have been made in the application of conventional surface analysis methods for real-time probing of plasma-surface interactions. This talk will review some of this work from our laboratory and elsewhere, where AES, XPS and TPD/SIMS-like desorption spectroscopy have been used to obtain information such as adsorbate coverages, reactive sticking coefficients, surface catalyzed reactions, and primary desorption products, all while operating a plasma. Examples will include operando XPS, spinning wall AES and desorption mass spectroscopy, ion-desorption optical emission spectroscopy and laser-desorption spectroscopy.

9:45am **CA1-FrM-7 Gaede Langmuir Award Talk: Investigation of Solid-Solid and Solid-Liquid Interfaces under Electrical Potential via XPS and EIS**, ***Sefik Süzer***[1], ÜNİVERSİTELER MAH, Turkey **INVITED**

X-ray Photoelectron Spectroscopy (XPS) and Electrochemical Impedance Spectroscopy (EIS) are well established analytical tools for interrogating surface and interface chemistry of mostly solid materials and their interfaces. Herein, we report using both techniques together, our observations on slow dynamics of devices with Semi-Conducting n- and/or p-doped Si Electrodes, having also ~3-4 nm oxide layer on top, separated by a Porous Polyethylene Membrane (PEM) soaked by an Ionic-Liquid as the electrolyte. As such, the device has two different interfaces: (i) Ionic Liquid / Silicon, and (ii) Silicon / Silicon Oxide. Using XPS, time dependent shifts of well-separated Si2p peaks of the semi-metallic (Si^0) as well as the oxide (Si^{X+}), together with O1s peak have been recorded under external electrical bias, to reflect the local electrical potential developments. For the electrolyte (Ionic Liquid), C1s, O1s and F1s peaks have been used to record the electrical potential developments at different locations. Complementary Electrochemical Impedance Spectroscopic measurements have also been recorded on the very same device within the XPS instrument before and after XPS measurements to follow the various changes, in addition to similar measurements under ambient conditions. Electrochemical findings, regarding the two interfaces, obtained from measurements of these two powerful spectroscopic techniques will be presented and discussed.

[1] **Gaede-Langmuir Award Winner**

Chemical Analysis and Imaging at Interfaces
Room 321 - Session CA2-FrM

Chemical Analysis and Imaging at Interfaces Oral Session II

Moderators: Carles Corbella, NIST-Gaithersburg, **Xiao-Ying Yu**, ORNL

10:15am **CA2-FrM-9 in Situ Molecular Imaging of Ion Clusters Reveals the Acid Gas Capture Capacity and Mechanism of Water-Lean Ionic Liquids**, ***Xiao-Ying Yu***, Oak Ridge National Laboratory; *Difan Zhang,* Oak Ridge Natinal Laboratory; *Zihua Zhu,* Pacific Northwest National Laboratory; *Gabriel Parker, Roger Rousseau,* Oak Ridge Natinal Laboratory

Water-lean solvents are a promising technology for capturing acid gases like carbon dioxide (CO_2).In situ liquid time-of-flight secondary ionization mass spectroscopy (ToF-SIMS) is used to study a representative solvent N-(2-ethoxyethyl)-3-morpholinopropan-1-amine (2-EEMPA) with different CO_2 loadings to reveal the complex solvent structure upon CO_2 capture.Characteristic peaks of 2-EEMPA, such as m/z^- 215 $C_{11}H_{23}N_2O_2^-$ (deprotonated 2-EEMPA) and m/z^+ 217 $C_{11}H_{25}N_2O_2^+$ (protonated 2-EEMPA), are detected due to acid gas uptake.Also, solvent molecules and carboxylate ion pairs, such as m/z^- 259 $C_{12}H_{23}N_2O_4^-$ [(deprotonated 2-EEMPA$\cdots CO_2$)] and m/z^+ 261 $C_{12}H_{25}N_2O_4^+$ (protonated 2-EEMPA$\cdots CO_2$), are observed.Interestingly, more than one CO_2 molecule can be captured per each solvent molecule as evidenced in SIMS mass spectra, for example, m/z^- 321 $C_{13}H_{25}N_2O_7$ [(deprotonated 2-EEMPA)$\cdots 2CO_2\cdots H_2O$], m/z^+ 305 $C_{13}H_{25}N_2O_4^+$ [(protonated 2-EEMPA)$\cdots 2CO_2$], m/z^- 389 $C_{17}H_{29}N_2O_8$ [(deprotonated 2-EEMPA)$\cdots 3CO_2\cdots 3CH_2$], and m/z^+ 373 $C_{16}H_{25}N_2O_8^+$ [(protonated 2-EEMPA)$\cdots 3CO_2\cdots 2C$].However, the monomer of 2-EEMPA and CO_2 seems to be most prevalent.Furthermore, solvent clusters are detected in loaded solvents, for instance m/z^+ 433 $C_{22}H_{49}N_4O_4^+$ [(2-EEMPA)$_2\cdots$H] and m/z^+ 646 [(2-EEMPA)$_3$-2H], while capturing CO_2 at different amounts.Relative abundance of cluster ions provides a semi-qualitative venue to assess the free energies gas capture energetics, indicating the relative stability trend within the same solvent system, previously impossible.These observed ion clusters are verified with molecular modeling, where dimer, trimer, and cluster ions are validated for their presence either due to weak molecular interactions or hydrogen bonds.In situ molecular imaging of ionic liquids and molecular modeling reveals that the acid gas capture mechanism by ionic liquids includes both physical adsorption and chemical bonding with multiple reaction pathways, engaging cluster formation and alteration of solvent structures.

10:30am **CA2-FrM-10 Modulating Active Site for Water Electrolysis**, ***Sen Zhang***, University of Virginia **INVITED**

This presentation focuses on modulating active sites for oxygen evolution reaction (OER) through precise control of single atom/cluster sites and nanocrystal surfaces and interfaces. Emphasis is placed on the use of in situ and operando spectroscopic techniques, including X-ray absorption spectroscopy (XAS), Raman spectroscopy, and surface sensitive IR, to directly probe dynamic surface reconstruction, oxidation state evolution, and formation of catalytically active species under working conditions. These real-time insights reveal how applied potential induces active-phase transformations and how tuning active site coordination environment can optimize adsorption energetics and reaction intermediates. By correlating spectroscopic signatures with electrochemical performance, we establish robust structure–activity relationships that guide rational active-site engineering for highly efficient and durable OER electrocatalysts.

10:45am **CA2-FrM-11 Synthesis and in Situ Characterization of Simulated Hanford Tank Waste Models in Aqueous Solution**, ***Tobias Misicko***, Oak Ridge National Laboratory, USA; *Mackenzie Savage,* University of Virginia; *Gabriel Parker, John Lasseter,* Oak Ridge National Laboratory, USA; *Kaleigh Louque, Yang Xiao,* Louisiana Tech University; *Xiao-Ying Yu,* Oak Ridge National Laboratory, USA

The nuclear waste stored at the US Department of Energy's Hanford Site as an environmental remediation facility presents many major challenges. Mainly, the remediation of aluminum-containing tank waste generated from the separation of aluminum cladding of spent fuel rods requires processing and its complex dissolution chemistry is still not well understood. These wastes typically consist of three aluminum oxide/oxy-hydroxide species, namely gibbsite (γ-Al(OH)$_3$),[1] boehmite (α-AlOOH),[2] and bayerite (α-Al(OH)$_3$).[3] Targeted remediation of these solids is complicated by their dissolution and stability in solution as well as a wide range of particle sizes. Physical property information on the tank waste is scarce due to computational simulation limits and the radioactivity of the waste preventing laboratory handling. Herein we report a versatile hydrothermal synthesis technique enabling tunable and scalable production of gibbsite, boehmite, and bayerite in submicrometer for use as simulant materials for in situ analysis and related experimental investigations. Synthesized samples were characterized ex situ using X-ray Diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), time-of-flight secondary ion mass spectrometry (ToF-SIMS), and physisorption to verify structural and chemical properties. *In situ* particle analysis and imaging were subsequently investigated using the System for Analysis at the Liquid-Vacuum Interface (SALVI) microfluidic platform, which enabled probing of the vacuum-liquid interface with SEM and ToF-SIMS.[4] The insights gained from this work are expected to inform the development of remediation approaches and formulate remediation methodologies that are safe for both site personnel and the environment.

References:

[1] Page, J.S., *et al.*, *Journal of Hazardous Materials,* 384 (**2020**) 121318

[2] Peterson, R.A., *et al., Separation Science and Technology,* 42 (**2007**) 1719-1730

[3] Herting, D.L., *et al., US DOE Office of Environmental Management,* (**2002**), OSTI ID: 808264

[4] Yang, L., *et al., Lab on a Chip*, 11 (**2011**), 2481-2484

11:00am **CA2-FrM-12 Accessing Surfaces and Interfaces in the Modern Scientific Era: An Overview of Important Instrumentation Advances**, ***Andrew Yost***, Scienta-Omicron Inc.; *Patrick Lömker, Takahiro Hashimoto,* Scienta-Omicron AB, Sweden; *Jürgen Köble, Andreas Janzen,* Scienta-Omicron GmbH, Germany; *Patrik Karlsson,* Scienta-Omicron AB, Sweden; *Daniel Beaton,* Scienta-Omicron Inc. **INVITED**

Scientific progress has repeatedly been driven by advances in experimental capability, with many of the most transformative discoveries in physics, chemistry, and materials science arising not solely from new theoretical understanding, but from the development of instrumentation capable of probing nature with greater sensitivity, spatial resolution, temporal resolution, and analytical precision. The ability to observe previously inaccessible phenomena frequently creates entirely new areas of research and enables scientists to ask questions that were once beyond experimental reach. In many cases, breakthroughs in instrumentation have reshaped our understanding of matter at fundamental length and energy scales while simultaneously enabling technological developments that extend far beyond the laboratory environment.

In this talk, we take a brief look at several major advancements in scientific instrumentation spanning the past several decades through the present day and examine how these developments have transformed modern surface science research. The progression of increasingly sophisticated measurement tools has enabled researchers to move from macroscopic characterization toward atomic-scale and electronic-scale investigations, allowing direct access to the structural, chemical, and electronic properties of materials and interfaces.

Particular focus will be placed on techniques that have had substantial impact across both fundamental research and industrial applications. Topics will include angle-resolved photoemission spectroscopy (ARPES), which has provided unprecedented insight into electronic band structures and many-body interactions; scanning probe microscopy (SPM), which has enabled real-space imaging and manipulation at near-atomic and atomic length scales; x-ray photoemission spectroscopy (XPS), which has become an essential tool for understanding surface chemistry and composition; and time-of-flight photoemission electron microscopy (ToF-PEEM), which combines spectroscopic and imaging capabilities to reveal spatially resolved electronic and chemical information.

Both academia and industry have benefited significantly from these developments, which have contributed to technologies that influence everyday life, including semiconductor devices, energy systems, catalysis, advanced materials, and nanotechnology. By examining the historical progression of these techniques and their broader impact, this talk aims to illustrate the central role that instrumentation innovation continues to play in enabling scientific discovery and driving future technological advancement.

11:30am **CA2-FrM-14 Studying Electrified Catalysis at the Laboratory and at Synchrotron End Stations**, ***Patrick Lömker***, Scienta Omicron, Sweden; *Andrew Yost, Daniel Beaton, Xin Zhang,* Scienta Omicron

Catalysts are central to the transition from fossil-based to renewable energy systems, with both thermal and electrocatalysis playing key roles in enabling carbon-neutral production of chemicals and fuels. However, their investigation presents significant technical challenges. This contribution

discusses advanced sample environments for Dip+Pull methodologies and cell-based electrocatalytic systems.

In Dip+Pull catalysis, recent developments emphasize improved usability and experimental efficiency. Sample cell designs have evolved from in-line electrode configurations to on-axis arrangements, thereby reducing electrolyte transport distances. This approach has been successfully applied to a range of systems, including CO reduction catalysis and studies of battery materials and their degradation mechanisms.

The second part focuses on electrochemical cell geometries, particularly configurations in which the catalyst acts as a barrier between the liquid phase and the vacuum environment. Such setups enable the investigation of membrane transport properties and the operando surface states of platinum catalysts and their intermediates under oxygen reduction reaction conditions.

A unifying aspect of both approaches is the investigation of the electrochemical double layer, which plays a critical role in governing catalytic activity and interfacial processes. Its characterization under these experimental conditions is discussed in detail.

11:45am **CA2-FrM-15 Energy Flux from Non-Thermal Discharges Measured via Nanocalorimetry: Toward Plasma Standard Development***, **Carles Corbella,*** National Institute of Standards and Technology (NIST)/ University of Maryland, College Park*; Aritra Sil, Berc Kalanyan, James Maslar,* National Institute of Standards and Technology (NIST)*; Lakshmi Ravi Narayan,* National Institute of Standards and Technology (NIST)/ University of Maryland, College Park*; William Osborn, Feng Yi, Andrei Kolmakov,* National Institute of Standards and Technology (NIST)

The use of rapid thermal probes to characterize plasma processes has been under development for a few decades. Despite a growing interest in quantifying the energy fluxes from low-temperature discharges used for surface processing, the decoupling of the elementary contributions from the different plasma species remains a challenge. Primary heat fluxes carried by ions, electrons, photons, and neutrals, are usually convoluted with secondary fluxes from chemical reactions, phase transitions, and recombination processes in calorimetry measurements. Nanocalorimeter sensors, which in our design consist of a Pt thin film microresistor lithographically defined on a free-standing, ultrathin SiN_x membrane with a thermal mass down to nJ/K, constitute an alternative solution to obtain instantaneous energy fluxes given their sensitivity, selectivity, and short response time. In this presentation, the potential application of nanocalorimetry to elucidate and control fundamental surface plasma-induced processes is motivated by the current needs of the coatings industry, where substrate temperature is a critical parameter. Here, we will discuss nanocalorimetric methods to discriminate the individual thermal fluxes from plasma species, such as the use of catalytic coatings to detect plasma radicals, the application of DC bias voltages to select impinging charged particles, and the exploration of interactions with light using optical filters. The presented prototype has been tested under hydrogen, helium, argon, and oxygen RF glow discharges operated in both inductive and capacitive modes. The energy flux, including the dominant participation of energetic ions in Ar plasma, ranges from 10 W/m^2 up to 1700 W/m^2. The experiments were performed in a dedicated research setup equipped with a Langmuir probe, retarding field energy analyzer, optical emission spectroscopy, and quadrupole mass spectrometry, to support and validate the nanocalorimetry measurements. The main objective is to develop plasma energy flux metrology as a standard plasma diagnostics tool that quantifies the thermal effect on plasma-exposed materials across different plasma sources and applications. The relevance of this emerging metrology to the plasma-materials community is justified by the nanocalorimeters' real-time and sensitive performance, robustness, and integrability to other metrology platforms.

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