

Thin Films

Room 317 - Session TF-TuM

Fundamentals of TF II & Flash Session

Moderators: Prof. Elton Graugnard, Boise State University, Dr. Richard Vanfleet, Brigham Young University

8:00am TF-TuM-1 Argon and Nitrogen Background Atmospheres as Growth Modulators in RIR-MAPLE Deposited MAPbI₃ Thin Films, Joshua Ayeni, Adrienne Stiff-Roberts, Duke University

Controlling material delivery during pulsed laser deposition (PLD) is critical for hybrid perovskites, where volatile organic components make film growth highly sensitive to deposition conditions [1]. Resonant infrared matrix-assisted pulsed laser evaporation (RIR-MAPLE) provides a solvent-mediated, low-damage deposition route for delicate hybrid materials. During RIR-MAPLE, inert background gases can alter plume expansion through collisional scattering and thermalization, affecting precursor transport and crystallization. Although RIR-MAPLE has been applied to diverse material systems [2,3], the comparative effects of inert gases on three-dimensional perovskite growth remain largely unexplored.

In this study, methylammonium lead iodide (MAPbI₃) thin films were deposited under controlled argon (Ar) and nitrogen (N₂) environments to investigate how inert atmospheres regulate plume propagation, precursor incorporation, and film evolution during RIR-MAPLE growth. MAPbI₃ possesses strong optical absorption, long carrier diffusion length, high mobility, and tunable bandgap energy, making it promising for photovoltaic and optoelectronic applications [4,5].

Depositions were performed using a resonant 2.94 μm Er:YAG laser, enabling gentle matrix sublimation and transfer of intact material toward the substrate for nucleation and crystallization. By varying gas species and pressure, distinct growth behaviors emerged due to atmosphere-dependent plume scattering and thermalization. Structural characterization revealed differences in crystallinity, orientation, and crystallite development between Ar- and N₂-grown films, indicating that the background atmosphere strongly influences growth kinetics.

Compared to Ar, N₂ environments promoted improved crystalline evolution (~35–43 nm) and increased film thickness (~150–260 nm), consistent with enhanced precursor retention and reduced loss of volatile species near the substrate. In contrast, Ar atmospheres produced thinner films (~100–147 nm) and stronger evidence of scattering-induced disruption of precursor transport. Optical measurements showed preservation of the characteristic MAPbI₃ absorption edge and photoluminescence response across both atmospheres, while subtle spectral shifts reflected atmosphere-dependent variations in structural order and defect-mediated recombination pathways.

Overall, the results demonstrate that inert background environments in RIR-MAPLE act as active growth regulators rather than passive chamber conditions. These findings establish the role of inert gases and pressure in tuning growth dynamics and support scalable RIR-MAPLE deposition of high-quality hybrid perovskite thin films.

8:15am TF-TuM-2 High Throughput Thin-Film Ion Kinetic Testing Enabled Through Atmospheric-Pressure Spatial ALD, Daniel MacAyeal, Ellen Owens, Kaydence Delgado, Alexander Kozen, University of Vermont

Research in atmospheric pressure spatial atomic layer deposition (AP-SALD) has enabled new capabilities in quickly manufacturing thin metal-oxide coatings for photovoltaics, electrochemical interfaces, and low-cost transparent conductors for flexible electronic devices. Spatial ALD can also be used for rapid materials testing and processing through deposition of high-throughput combinatorial libraries. Here, we utilize this approach to investigate AP-SALD of Nb₂O₅ / TiO₂ mixed metal oxide (MMO) bronzes, which are known to exhibit fast ion transport. Discrete regions of each mixed metal oxide can be varied by an arbitrary material property or manufacturing parameter of our choosing. We construct TiO₂ / Nb₂O₅ bronze test structures with varied alloy composition, precursor flow parameters, nanolaminate thickness, and overall region composition. We use spectroscopic ellipsometry, Raman spectroscopy, AFM, and XRD to evaluate materials structure, composition, and morphology as a function of the aforementioned parameters. We demonstrate the capabilities of AP-SALD and highlight new accelerated methods for high-throughput complex oxide materials development.

8:30am TF-TuM-3 Accelerated Approaches to Atmospheric Pressure ALD Process Development Using Predefined Test Structures, Ellen Owens, Holly Wilson, Dan MacAyeal, Alex Zaccardi, Drew Ledger, Alexander Kozen, University of Vermont

Process development and associated materials screening for atomic layer deposition (ALD) processes can be long and arduous, particularly for compositionally complex ternary quaternary, and pentanary films. Beyond conventional ALD, Atmospheric pressure spatial ALD (AP-SALD) is an emerging and powerful tool that can produce discrete arrays of deposited material on a single wafer to increase the rate of material optimization of thin-film materials. In this talk, we discuss strategies to dramatically decrease process development and materials characterization timelines for ALD films using atmospheric pressure spatial ALD (AP-SALD) onto prepatterned arrays of test structures and devices.

We use TiO₂ as a prototype material to demonstrate the high throughput capabilities of AP-SALD process by deposition onto interdigitated electrodes and evaluate the electrical, optical, and structural properties of these films.

We vary combinatorial parameters including precursor, carrier, and sheath gas flow rates, nozzle to substrate gaps, deposition temperatures, and post deposition processes, allowing us to quickly access a wide parameter space on a single substrate.

We evaluate the spatially-resolved deposition characteristics of TiO₂ using profilometry, spectroscopic ellipsometry, and electrical measurements including IV, CV, linear & nonlinear impedance, and Hall effect measurements.

8:45am TF-TuM-4 Facile Optimization of Combinatorial Sputtering Processes with Arbitrary Numbers of Components for Targeted Compositions, Shelby Fields, U.S. Naval Research Laboratory

Combinatorial thin films and coatings, which display analogue compositions that vary as a function of position, enable high-throughput materials discovery and optimization. For example, when paired with mappable characterization, whole explorations of composition effects on materials properties may be contained within a single synthesis step. However, the applicability of combinatorial films and coatings to many materials challenges is hindered by irreproducibility and constrained processing spaces, which reduce the extent to which these experiments map on to end-use technology. To address this deficiency, this presentation introduces a process development method that enables the reproducible synthesis of n -component sputtered combinatorial thin films with targeted compositions at specific coordinates. It is determined that areal mass density profiles of individually sputtered species are well described by two-dimensional Gaussian peak shapes, the amplitude of which possesses a strongly linear relationship with applied target power. In addition, areal mass density profiles are shown to be noncovariant during co-deposition. Based upon these characteristics, an optimization expression is derived that computes the power required to obtain a targeted atomic concentration at a specific coordinate. It is shown that a minimum of two depositions at different powers are sufficient produce an optimized process model, which is subsequently enabled to suggest powers to obtain any composition at a targeted coordinate, permitting the high throughput exploration of entire composition spaces. Furthermore, this optimized process allows for the substitution of substrates of different diameter and material following calibration, is compatible with both direct current and radio-frequency magnetron sputtering, and can suggest powers to obtain identical compositions at different growth rates. As a proof-of-concept, a Cr_{0.5}Fe_{0.5}Mo_{0.5}Nb_{0.5}Ta_{0.5} alloy thin film is synthesized on a 6"-diameter Si wafer with an equiatomic composition at the center that matches well with simulation following calibration. This capability is further demonstrated through the synthesis of alloy combinatorial wafers using reactive and rf sputtering processes for separate materials exploration investigations. In total, this optimization method enables the rapid and reproducible synthesis of combinatorial films with arbitrary numbers of components, which better connects this high throughput experimental method with down-stream applications and technology.

9:00am TF-TuM-5 Combining Pyroelectric Calorimetry and Multiscale Modeling to Untangle the Interplay of Precursor Delivery, Kinetics, and Byproducts in ALD ZrO₂, Ashley Bielinski, Anik Biswas, Cong Liu, Alex Martinson, Argonne National Laboratory

Thermal atomic layer deposition (ALD) processes are typically described as alternating sequences of self-limiting surface reactions. When we inspect the dynamics of individual reactions as they approach saturation, we see that in addition to simple mechanisms of ligand exchange reactions, this

self-limiting nature also depends on an interplay between precursor delivery, reaction kinetics, and the role of reaction byproducts. An investigation of these dynamic processes can provide insights into the results of ALD processes ranging from fundamental reaction thermodynamics and kinetics to reactor design. However, these reactions happen quickly, often at rates faster than the sampling frequency of common in situ measurement techniques. Pyroelectric calorimetry provides in situ measurements of the heat generated and transferred from surface reactions as well as precursor and byproduct flow with sufficient time resolution to measure these dynamics. Here we present how experimental calorimetry measurements can be combined with multiscale computational modeling to untangle the influence of precursor flow dynamics and reaction kinetics for the reaction between tetrakis(dimethylamido)zirconium(IV) (TDMAZr) and water to form ZrO_2 . By combining experimental measurements that represent the true conditions of realistic samples surfaces, first principles modeling of reaction mechanisms, and reactor-scale modeling of precursor flow and byproduct generation, we provide insight into the complexities of ALD surface reactions, the non-idealities of which have broader implications for the development of site- and area-selective ALD.

9:15am TF-TuM-6 Flow Imaging for Validated Mass Transport Models of an Atomic Layer Deposition Chamber, Berc Kalanyan, Hyunwoo Yang, Vladimir Khromchenko, James Maslar, National Institute of Standards and Technology (NIST)

Digital twins of semiconductor unit processes could provide value in process development, optimization, and real-time control applications. For a vapor phase deposition process, a digital twin would entail multiple linked models representing reactive transport processes at disparate length scales from the process equipment level to the device structures on the wafer. However, widespread development and validation of such models require non-proprietary process data, which is not readily available. To address this need, NIST is generating process data and validated models for atomic layer deposition (ALD) processes. An important component of this effort is *in situ* metrology development to access key process parameters such as partial pressures, flow rates, temperature, and mass uptake as a function of space and time. In this talk we will focus on 1) mass transport measurements within a research-grade ALD reactor and 2) the use of transport data to validate flow simulations. To obtain the process data, we use absorption imaging of precursor flow as a function of process conditions, *e.g.*, gas flow rate, chamber pressure, and temperature. Two precursors selected for this investigation are molybdenum pentachloride (MoCl_5) and molybdenum oxytetrachloride (MoOCl_4). Precursor flow was visualized at about 100 frames per second in the ultraviolet-visible spectral region using a CMOS camera and a light emitting diode source. Simulations of flow in this chamber were performed using commercial computational fluid dynamics (CFD) packages. Simulations were validated using the time-dependent, pathlength-integrated precursor concentration obtained from the absorption imaging measurements and the time-dependent total pressure measured at selected locations in the deposition system. Carrier gas flow rates and system pressures that resulted in quiescent and recirculating flow regimes were simulated using a single model. The spatial distribution of precursor in the chamber was found to be driven by continuum flow of the carrier gas. Flow imaging collected from two orthogonal axes was used to assess asymmetry in flow fields resulting from injector geometry.

References

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- (2) Alghamdi, A.; Ou, F.; Kalanyan, B.; Maslar, J. E.; Christofides, P. D. A CFD-Based Digital Twin Framework for Transient MoCl_5 Transport in an Experimental Atomic Layer Deposition Process. *Digital Chemical Engineering* **2026**, *19*, 100304. <https://doi.org/10.1016/j.dche.2026.100304>.

9:30am TF-TuM-7 Mgo ALD Coatings for Improvement of Secondary Electron Emission, Sun Gil Kim, Min-Seop Song, Hyun-Mi Kim, Korea Electronics Technology Institute, Republic of Korea; Jeonggil Na, Kyunghwan Jeong, JJ CNS, Republic of Korea; Hyeonkeun Kim, Korea Electronics Technology Institute, Republic of Korea

Microchannel Plates (MCP) are used to amplify photoelectron, ion, or electron signals in spectrometers, electron microscopes, and night vision devices. Conventional MCPs are fabricated from lead glass, where a SiO_2 emissive layer is formed during glass fabrication, facilitating electron

multiplication. Despite materials such as MgO and Al_2O_3 exhibiting superior secondary electron emission (SEE) properties compared to SiO_2 , conventional deposition techniques such as physical vapor deposition (PVD) and chemical vapor deposition (CVD) have limitations in achieving uniform coating inside MCP channels. In contrast, atomic layer deposition (ALD) enables uniform coating of materials with excellent SEE properties even within the narrow MCP channels, thereby enhancing MCP performance. In this study, we developed the ALD process to deposit Al_2O_3 as a resistive layer and MgO as an emissive layer inside MCP channels.

Film thickness and density were analyzed using an ellipsometer and X-ray reflectometry (XRR). Additionally, X-ray photoelectron spectroscopy (XPS) was employed to examine the elemental composition and Mg/O ratio across different film thicknesses. The crystal structure was characterized using X-ray diffraction (XRD), while high-resolution transmission electron microscopy (HR-TEM) was utilized to investigate the microstructure of the deposited films. Moreover, the SEE coefficient of MgO thin films under various process conditions was measured using a γ -focused ion beam (γ -FIB) system, and based on these results, the optimal process parameters and film thickness were determined.

This study is expected to serve as a key reference for material selection in emissive and resistive layers of MCP. Future research will explore various oxide thin-film combinations and novel materials, not only to enhance MCP gain but also to improve MCP lifetime and noise characteristics, thereby contributing to overall performance advancements. Moreover, the findings can be applied across various industrial and research fields, including time-of-flight mass spectrometry (ToF-MS) and image intensifiers.

9:45am TF-TuM-8 Autodoping of Thin-Film Ionic Materials Using Atomic Layer Deposition (ALD), Steven Douglass, Daniela Fontecha, Gary Rubloff, Sang Bok Lee, Keith Gregorczyk, University of Maryland, College Park

Recently, our group has shown that semiconductor manufacturing techniques can be leveraged to add electrochemical functionality and make devices outside of the traditional semiconductor portfolio. These devices include solid-state microbatteries and super capacitors, as well as electrochemical random-access memory (ECRAM). In the production of these iontronic devices (devices where ions operate as the main carriers of charge), notable intermixing of ionic material layers has occurred between, for example, the vanadium pentoxide (V_2O_5) cathode and the lithium phosphorus oxynitride (LiPON) electrolyte in solid-state microbatteries. This autodoping has been termed “autolithiation” and is believed to occur in two stages: before LiPON film growth and during LiPON film growth. It was first observed as a physical process during sputtering of LiPON onto a V_2O_5 substrate.

Since, we have observed a similar phenomenon while depositing ALD LiPON onto V_2O_5 , with the lithium source being lithium *tert*-butoxide (LiO^tBu). In this talk we discuss how autolithiation during ALD depends on temperature in its initial stage. At 300°C , there was no lithiation after 20 pulses of LiO^tBu . However, one pulse at 350°C and 400°C yielded lithiation, followed by a linear trend of increased lithiation state with increased pulsing. Figure 1 displays the trend of lithium inserted through LiO^tBu pulsing at different temperatures, calculated from electrochemical delithiation of the samples. XPS demonstrates that the $\text{V}^{4+}:\text{V}^{5+}$ oxidation state ratio grows quicker with increased temperature. Raman spectroscopy shows evolution of the bulk away from $\alpha\text{-V}_2\text{O}_5$ with higher temperatures and more LiO^tBu exposure. This presentation discusses characterization and quantification of ALD autolithiation of V_2O_5 before film growth. Future work on the subject will be focused on deconstructing autolithiation after film growth.

11:00am TF-TuM-13 Use of Xps-Crem for Detecting Hydration-Driven Changes in the Electrical Conductivity of $\text{La}_2\text{Ce}_2\text{O}_7$ Thin Films, Ayelet Vilan, Weizmann Institute of Science, Israel

Chemically Resolved Electrical Measurements (CREM)¹ harnesses the charging-like effects in X-ray Photoelectron Spectroscopy (XPS) to extract sample's electrical properties. Here, we apply CREM to investigate a novel proton-conducting material comprising hydrated $\text{La}_2\text{Ce}_2\text{O}_7$.² Conventional electrical measurements show that the activation energy for conductance decreases from ~ 1.07 eV in the as-deposited film to ~ 0.7 eV after hydration. We attribute this reduction to a change in the dominant transport carriers: electron conduction via oxygen vacancies in the as-deposited film and proton conduction in the hydrated film.

As temperature decreases from 473 to 360 K, both films become less conductive; however, hydrated $\text{La}_2\text{Ce}_2\text{O}_7$ exhibits weaker temperature dependence: At 360 K, its conductance is approximately five times higher than that of the as-deposited film. Measurements below this temperature

failed, because the films become too resistive for conventional measurements, preventing reliable room-temperature characterization.

To overcome this limitation, we employed CREM within the XPS chamber to evaluate conductivity at room temperature under ultra-high vacuum. In brief, CREM modulates the electron flood gun while simultaneously measure (1) the net current through the sample and (2) the shifts in core-level binding energies, reflecting resistive potential drops. The ratio of current changes over energy shifts, provides an effective film's perpendicular conductivity.¹ CREM measurements (see figure) revealed that, at room temperature, hydrated $\text{La}_2\text{Ce}_2\text{O}_7$ was roughly three orders of magnitude more conductive than the as-deposited film, consistent with the expected exponential dependence on activation energy. Although XPS cannot directly detect hydrogen, the sustained high conductivity under vacuum supports stable proton incorporation within the lattice.

To directly probe proton transport, we deposited proton-sensitive WO_3 , which turns from transparent to opaque upon proton insertion (H_xWO_3), accompanied by reduction of W(VI) to W(V) . Devices comprising $\text{ITO/La}_2\text{Ce}_2\text{O}_7/\text{WO}_3$ exhibited bias-induced coloration only when $\text{La}_2\text{Ce}_2\text{O}_7$ was hydrated. Furthermore, CREM-driven proton injection revealed a significant shoulder of reduced-tungsten in the W 4f line, characteristic of W(V) , only when the underlying $\text{La}_2\text{Ce}_2\text{O}_7$ was hydrated. These results confirm proton-mediated transport.

The presentation will outline the CREM methodology and its application to XPS under varying electronic conditions.

References:

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- [2] Freidzon D. et al., Solid State Ionics **438** (2026) 117150;

11:15am TF-TuM-14 Influence of Substrate Interactions on the Growth of MoO_3 on Graphene/Ru(0001) and Highly-Oriented Pyrolytic Graphite, Buddhika Alupotha Gedara, Zdenek Dohnalek, Zbynek Novotny, Pacific Northwest National Laboratory

Two-dimensional transition-metal oxides (TMOs) are promising for electronic applications due to their stability and favorable electrical and optical properties. Among them, molybdenum trioxide (MoO_3) stands out for its high dielectric constant, wide band gap (2.9–3.2 eV), and catalytic activity, enabling diverse electronic and energy-related applications. In this work, we investigate the effects of substrate topography and charge transfer on MoO_3 growth on highly oriented pyrolytic graphite (HOPG) and graphene (Gr)/Ru(0001) using scanning tunneling microscopy and X-ray photoelectron spectroscopy (XPS). Small MoO_3 clusters formed on Gr/Ru(0001), whereas large, dendritic, and disordered MoO_3 clusters formed on HOPG following deposition at 300 K. Upon increasing the temperature to 500–700 K, more ordered, crystalline MoO_3 islands form on both substrates. Notably, a distinct height difference of MoO_3 is observed on these two substrates over the studied temperature range. The height of MoO_3 islands on Gr/Ru(0001) corresponds to a single layer of MoO_3 ($3.9 \pm 0.2 \text{ \AA}$), whereas the height of the MoO_3 islands on HOPG is consistent with the previously reported double layer ($6.8 \pm 0.1 \text{ \AA}$) [1, 2]. Single-layer MoO_3 on Gr/Ru(0001) comprises a planar MoO_2 sheet aligned parallel to the Gr/Ru(0001) surface, with Mo atoms terminated by additional oxygen atoms. We observed a brick-style pattern on the MoO_3 islands on Gr/Ru(0001) substrate and the formation of this characteristic pattern is due to the ordered removal of terminal oxygen atoms. Double-layer MoO_3 islands grow on HOPG with a staggered arrangement of corner- and edge-sharing MoO_6 octahedra [1, 2]. XPS data show that Mo in the islands on HOPG are predominantly in (6+) oxidation state while on Gr/Ru(0001) at least half is reduced to (5+) over the temperature range of 500–700 K. The formation of lower oxidation states is attributed to charge transfer from the substrate due to the electron chemical potential difference at the interface, and the presence of oxygen vacancies. Our results demonstrate that substrate-induced interfacial interactions play a key role in controlling the nucleation, growth mode, and structure of MoO_3 on graphene-based supports, providing a pathway to tailor 2D TMO heterostructures for electronic and catalytic applications.

References

- [1] D.A. Kowalczyk et al., ACS Appl. Mater. Interfaces, **14** (2022) 44506–44515.
- [2] J.H. Kim et al., 2D Materials, **6** (2019) 015016.

11:30am TF-TuM-15 Atomic Growth Mode of Capping Layers for Surface Passivation of Nb, Van Do, Robert Burnley, Margaret Hall, Helena Lew-Kiedrowska, Sarah Willson, Steven Sibener, University of Chicago

Nb is the standard material for superconducting radio-frequency (SRF) cavities, but the accelerating performance of these cavities is limited by the spontaneous growth of Nb oxides on the surface. These oxides are insulating and dielectric in nature, which can lead to hot spots and vortex penetration, ultimately degrading cavity performance. To address this challenge, we are improving Nb surface stability through the implementation of two types of capping layers: an inert capping layer and sacrificial capping layer. A chemically inert Au film can suppress oxidation by protecting the underlying Nb surface from environmental exposure. In contrast, a sacrificial Zr layer, owing to its higher oxidation affinity, preferentially oxidizes and thereby shields the underlying Nb. In addition, Zr can intermix with Nb to form a substitutional Zr–Nb alloy with a predicted superconducting transition temperature of 17.7 K. Using scanning tunneling microscopy, we have visualized Au capping layers ranging from sub-monolayer coverage to 10 monolayers, as well as Zr capping layers from initial adsorption through alloy formation on the $(3 \times 1)\text{-O/Nb}(100)$ surface. We further evaluated the passivation efficiency of these modified surfaces by leaking oxygen and observing the chemical evolution using x-ray photoelectron spectroscopy. These results provide atomic mechanistic insight into surface treatment methods for enhancing the oxidation resistance and superconducting performance of Nb-based SRF cavities.

11:45am TF-TuM-16 Distal electrofabrication and Microfluidic Integration of Chitosan Anion-Exchange Membranes for Low-Cost Direct Methanol Fuel Cells, Phuc Long Duong, College of Engineering, Physics and Computing, Catholic University of America; Xiaolong Luo, Catholic University of America

Commercial adoption of hydrogen fuel cells remains limited by high material costs and the lack of a dedicated hydrogen infrastructure. Direct Methanol Fuel Cells (DMFCs) offer a compelling alternative by using an easily refuelable liquid fuel with high energy density. However, conventional DMFCs face significant economic and technical hurdles due to their reliance on expensive proton exchange membranes (e.g., Nafion) and noble-metal catalysts, as well as issues with methanol crossover and sluggish reaction kinetics.

This work demonstrates the design of a microfluidics DMFC (DMFC) that uses ultra-low-cost, non-platinum-group-metal (non-PGM) catalysts and a sustainable, biopolymer-based anion exchange membrane (AEM). We apply a patent-pending distal electrofabrication technique to deposit freestanding chitosan thin films directly within microfluidic channel networks. The electrofabrication provides precise control and utilizes the biopolymer's naturally low methanol permeability to significantly minimize fuel crossover.

Material characterization, including Atomic Force Microscopy (AFM), Scanning Electron Microscopy (SEM), and X-ray Diffraction (XRD), confirms our hypothesis that distal electrofabrication induces high molecular alignment and a distinct crystallographic ultrastructure compared to standard cast membranes. Furthermore, Electrochemical Impedance Spectroscopy (EIS) validates enhanced hydroxide-ion conductivity within the thin-film networks.

Initial device performance testing demonstrated a peak power density of 70 mW/cm^2 , with structural and crosslinking optimizations promising commercial adoption at 5%–10% the cost of existing commercial fuel cells. The success of this project may open a cost-competitive pathway for portable zero-emission energy solutions.

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