

## Thin Films

### Room 115 - Session TF-WeA

#### Vapor Synthesis of Hybrid, Organic, and Polymeric Materials (VSHOP III)

**Moderators:** David Bergsman, University of Washington, Rong Yang, Cornell University

#### 2:15pm TF-WeA-1 Chemical Vapor Deposition of Polymers for Lithium Ion Batteries, *Kenneth Lau*, Rowan University **INVITED**

The lithium ion battery (LIB) technology is rapidly dominating the energy storage market due to its competitive cost and energy capacity. However, due to growing market demands, there is a continued push to enhance LIB energy and power density. As such, there is significant research into high capacity, high voltage cathode materials based on transition metal oxides that provide more lithium ion intercalation. However, at high voltages and high reactivity, the intercalation process becomes unstable and the transition metals tend to leach out into the electrolyte, leading to degrading battery performance with repeated charge-discharge cycling. Our work aims to protect and stabilize LIB cathode materials by conformally coating individual cathode particles with conducting polymers by oxidative chemical vapor deposition (oCVD). The hypothesis is that the protective conducting polymer helps to stabilize the cathode-electrolyte interface against degradation processes. In a second approach towards increasing LIB energy and power density, there is increasing research in moving away from 2D planar sandwich cells to pursuing 3D LIB architectures in which the electrodes are intentionally constructed into microstructured geometries (e.g. micropillars) to provide higher surface area and shorter lithium ion diffusion pathways. Our work aims to create 3D LIB anode microstructures through the carbonization of aromatic polymers grown by initiated chemical vapor deposition (iCVD). The hypothesis is that the simplified iCVD process scheme allows facile microstructural formation of polymer geometries that lead to viable microstructured carbon anodes. In this talk, we will highlight our recent work in using oCVD and iCVD to create polymers relevant for enhancing the performance of LIBs, particularly in improving capacity retention. We will present experimental correlations of how oCVD and iCVD processing chemistries and conditions impact material structure and device behavior, knowledge that has helped to understand the role of different dynamic processes, including adsorption, nucleation and growth kinetics.

#### 2:45pm TF-WeA-3 Exploring the Influence of Reduced Vacuum Conditions on Structural and Chemical Variations in Hybrid Perovskite Films Deposited by Resonant Infrared Matrix-Assisted Pulsed Laser Evaporation, *Joshua Ayeeni*, A. Stiff-Roberts, Duke University

This study examines how gas kinetics impact the structural properties and chemical environment of phenylethylammonium lead iodide ((PEA)<sub>2</sub>PbI<sub>4</sub>) films deposited by resonant infrared matrix-assisted pulsed laser evaporation (RIR-MAPLE) under various background pressures (BGPs) of inert gas. (PEA)<sub>2</sub>PbI<sub>4</sub> is a promising hybrid perovskite material for optoelectronic applications such as LEDs [1-2], yet less extensive research exists on vacuum deposition compared to solution deposition. RIR-MAPLE uses a low-energy Er:YAG laser at 2.94 μm to reduce material degradation by resonant absorption of a host matrix [3]. Hybrid perovskite materials are typically grown under an active vacuum in RIR-MAPLE. Thus, understanding film deposition in a reduced vacuum with a BGP of inert gas is critical to determining industrial scalability.

Films were deposited from active vacuum (10<sup>-5</sup> Torr) to 500 mTorr in a nitrogen atmosphere. Standard film characterization such as AFM, SEM, XRD, PL, and UV-Vis absorbance do not reveal significant changes in film properties with increasing BGP. X-ray photoelectron spectroscopy (XPS) was used to characterize the chemical composition of film surfaces. Pristine films show two characteristic Pb peaks from the spin-orbit splitting at ~138.0 eV for 4f<sub>7/2</sub> and ~143 eV for 4f<sub>5/2</sub>, corresponding to a fully coordinated Pb<sup>2+</sup> in the perovskite system. Variations in the binding energies (BEs) and elemental ratio are observed for different BGPs, implying different chemical environments for the Pb element. At high BGPs, peak splitting is observed for Pb 4f, I 3d, and C 1s. Also, the I/Pb ratio determined from XPS exhibits marked differences with BGP. For BGP below 250 mTorr, the decrease in the I/Pb ratio relative to the stoichiometric ratio suggests iodine vacancies [4]. Conversely, at higher BGPs, high iodine content indicates an iodine-rich phase is present in the films.

Therefore, this study will explore how BGP affects gas diffusion, adsorption kinetics, and nucleation processes to provide better insight into gas-phase kinetics and film growth mechanisms. By combining experimental results with theory to understand the role of BGP in compositional changes, better control of stoichiometry, purity, bonding, or doping by RIR-MAPLE could be enabled.

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#### References

- [1] G. Tanbo et al., *J All & Comp* 940, 168894 (2023)
- [2] W. Dou et al., *Nanomaterials*, 12, 2556 (2022)
- [3] A. D. Stiff-Roberts & W. Ge. *Appl. Phys. Rev.* 4 041303 (2017)
- [4] H. T. Chen et al. *Adv. Optical Mater.*, 8, 1901390 (2020)

#### 3:00pm TF-WeA-4 Low Surface Energy for Tuning the Surface Repellency through Initiated Chemical Vapor Deposition, *Syed Ibrahim Gnani Peer Mohamed*, G. Kaufman, D. Egbunmi, M. Bavarian, J. Shield, C. Zuhlke, S. Nejadi, University of Nebraska Lincoln

Surface-wetting properties are crucial in various applications, such as microfluidics, water treatment, drag reduction, immersion cooling, degassing, and self-cleaning surfaces. For instance, traditional additive manufacturing techniques have been used to fabricate omniphobic surfaces, but concerns about durability and scalability remain. This study presents a novel approach to ultra-low surface tension repellency through a two-stage fabrication method, which integrates scalable techniques to restructure the surface and initiated chemical vapor deposition (iCVD). By applying this method to bulk aluminum, we achieved robust, hierarchal roughened surfaces that repel ultra-low surface tension liquids, including n-hexane ( $\gamma = 18.2$  mN/m). This approach offers flexibility in substrate material and shows promising results for scalability and durability. Surface and subsurface chemical analysis revealed the preservation of perfluoroalkyl side groups crucial for imparting superhydrophobic functionality. Wetting characteristic assessments highlighted the impact of hierarchal roughness on repellency, with the PTFE-co-PPFDA coating outperforming PPFDA for lower surface tension liquids. We present data on the coating at a high value of surface availability and note the transition in properties of the coated materials as a function of surface availability. Importantly, this study's findings have practical implications, providing insights into the design and fabrication of hierarchal roughened surfaces for ultra-low surface tension repellency, three different applications of these surfaces will be discussed for the very-low surface energy samples that show surface energy in the range of 12 mN/m.

#### 3:15pm TF-WeA-5 From iCVD Thin Films to 3D Aeropolymers for Biomedical Applications, *Torge Hartig*, J. Paulsen, W. Reichstein, M. Hauck, J. Piehl, Kiel University, Germany; M. Taale, Universität Heidelberg, Germany; T. Strunskus, Kiel University, Germany; C. Selhuber-Unkel, Universität Heidelberg, Germany; G. Schnell, University of Rostock, Germany; A. Amin, National Research Center, Giza, Egypt; R. Adelung, Kiel University, Germany; B. Freedman, Harvard University; F. Schütt, F. Faupel, S. Schröder, Kiel University, Germany

With rising requirements for functional polymer surfaces, wet chemical approaches meet their limits. Representing an all-dry technology initiated Chemical Vapor Deposition (iCVD) thin films are highlighted regarding conformal coating of nanoscale features, tailoring of chemical functional groups, as well as crosslinking via copolymerization. The conformal nature of the iCVD process is used in PFAS-free superhydrophobic coatings based on femtosecond-laser processed substrates.

To understand the impact of certain functional groups on the bio-interface-performance, tailored surface chemistry coatings as well as nanocomposite coatings are compared regarding applications for human fibroblasts and respiratory viruses.

Furthermore, the conformal nature of iCVD coatings is used to fabricate freestanding 3D thin film scaffolds. For this, sacrificial porous tetrapodal ZnO is coated and etched wet-chemically resulting in so-called aeropolymers of more than 99% empty space. The used aero-hydrogels have striking compressive properties that can be tailored by crosslinking ratios. The scaffolds mimic the extracellular matrix shape allowing the use in 3D cell culture for muscle cell applications. To include electrical conductivity in the aero-hydrogels multilayers of graphene and iCVD hydrogels are fabricated resulting in optimized cell interaction.

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**3:30pm TF-WeA-6 Investigating PEDTT Thin Films: Comparing Synthesis Methods and Properties for Electronic Applications, Siamak Nejati, S. Gnani Peer Mohamed, L. Okpaire, V. Medic, M. Bavarian, University of Nebraska-Lincoln, USA**

This study explores the properties and characteristics of poly(3,4-ethylenedithiathiophene) (PEDTT) thin films synthesized using oxidative molecular layer deposition (oMLD) and oxidative chemical vapor deposition (oCVD). The investigation focuses on conductivity, defect-free film formation, and the influence of structural variations on the overall properties of these films. PEDTT, notable for its solvent-free synthesis, is examined for its potential applications in electronic devices, drawing comparisons with poly(3,4-ethylenedioxythiophene) (PEDOT). A comparative analysis of the oMLD and oCVD techniques highlights their efficiency, scalability, and resultant film properties. oMLD offers precise, controlled layer-by-layer deposition, while oCVD provides versatility with the thermal decomposition of organic precursors. This study aims to elucidate the relationship between synthesis methods and thin film properties through detailed experimentation. A key aspect of this research is understanding the mechanisms governing PEDTT's electrical conductivity, examining molecular structure, doping levels, and processing parameters. The study also evaluates PEDTT's ability to form defect-free films, emphasizing factors influencing film uniformity and integrity. Structural differences between PEDTT and PEDOT, particularly the roles of sulfur and oxygen atoms, are analyzed to reveal their impact on electronic properties and chemical reactivity. Furthermore, the structural features of PEDTT, including steric repulsion and twisted conformations, are shown to affect its porosity and surface area, with implications for surface modifications and interfacial interactions. The research delves into the deposition processes, focusing on the flow dynamics and vapor pressure of the ethylenedithiathiophene (EDTT) precursor, to achieve a comprehensive understanding of thin film fabrication. This investigation provides valuable insights into the interplay between synthesis methods, molecular structure, and thin film properties of PEDTT-based materials. By uncovering the mechanisms behind conductivity, defect-free film formation, and structural characteristics, this research advances the field of electroactive polymers and their applications in emerging technologies.

**4:15pm TF-WeA-9 Exploring New Avenues Resulting from the Rational Process Design of Sequential Infiltration Synthesis, Nari Jeon, Chungnam National University, Republic of Korea** **INVITED**

Sequential infiltration synthesis (SIS) has been demonstrated as a novel approach for fabricating inorganic-organic hybrid materials and inorganic porous materials, thereby offering precise control over nano- to microscale morphology. This method relies on the infiltration of vapor-phase precursors into a polymer matrix, followed by the reaction of these precursors with coreactants in the matrix. SIS-derived thin films and nanostructures show promise for applications in lithography, photovoltaics, tribology, and water remediation. Despite the continuous expansion of the material library prepared through SIS, a comprehensive understanding of the growth mechanism of SIS is limited. During SIS, precursor diffusion and reactions occur concurrently in the polymer matrix. SIS-derived thin films exhibit various compositional and structural complexities. This study investigates the complex relationship between the key physical/chemical phenomena governing the SIS process and the structural/compositional distribution of SIS-prepared films using *in situ* and *ex situ* characterization techniques. Moreover, it explores the potential of using organic coreactants in SIS as a new means of controlling the compositional distribution of inorganic-organic hybrid thin films and the pore morphology of inorganic porous thin films. Finally, future research directions are provided to help establish SIS as a mainstream technique in the fields of atomic layer deposition (ALD) and ALD-derived processing.

**4:45pm TF-WeA-11 Vapor Phase Infiltration of ZnO into Photopolymerizable Polyacrylates: Patterning and the Importance of Free Volume, Mark Losego, Georgia Tech; L. Demelius, Graz University of Technology, Austria; A. Coclite, University of Bari, Italy**

Vapor phase infiltration (VPI) exposes polymers to inorganic vapors that sorb into the polymer to transform it into an organic-inorganic hybrid material with properties unique from its parent polymer. This talk will discuss our work investigating VPI of ZnO into photopolymerized polyacrylates. Photopolymerizable polymers are easily micropatterned with standard photolithography or advanced two-photon lithography processes. VPI offers a way to transform these micro-patterned polymeric structures into more functional hybrid materials or even fully inorganic structures upon combusive removal of the polymer. However, this transformation from polymer to hybrid to inorganic requires control and understanding of

the inorganic loading during the VPI process. Here, we will show how the addition of "soft" acrylate segments containing ethoxylated chains can increase the free volume of these network polymers and permit greater VPI uptake of inorganic precursors, leading to more inorganic loading. The increased free volume of these polymers is confirmed with thermal expansion measurements. These more "flexible" polymer networks increase both the VPI kinetics and the total inorganic mass loading. In the compositional depth profiles, we can also detect a transition from a diffusion-limited process to a reaction limited process. We will also demonstrate the basic ability to photo-pattern these polyacrylates and then VPI the structures to make micro-patterned hybrid materials.

**5:00pm TF-WeA-12 Effect of Alumina Priming on Structural and Electrical Properties of Zn Nanostructures Synthesized via Vapor-Phase Infiltration Into Self-Assembled Block Copolymer Thin Films, C. Nam, Won-Il Lee, A. Subramanian, Stony Brook University/Brookhaven National Laboratory; K. Kisslinger, Brookhaven National Laboratory; N. Tiwale, Brookhaven National Laboratory**

Alumina priming is a technique that can improve the fidelity of the vapor-phase infiltration (VPI) of weakly reactive organometallic precursors into polymer templates, such as self-assembled block copolymer (BCP) thin films. The process begins with the infiltration of trimethylaluminum (TMA), a strong Lewis acid that can diffuse and bind to Lewis-basic groups within polymers. Upon exposure to water vapor, TMA forms molecular alumina terminated with hydroxyl groups. This priming step promotes subsequent infiltration of weakly reactive organometallic precursors of target inorganic materials by activating the polymer matrix. While alumina priming expands the range of inorganics applicable to VPI, the insulating properties of bulk alumina can negatively affect the electrical properties of the final inorganic nanostructures produced by VPI. In this study, we explore the impact of alumina priming on the structural and electrical properties of zinc oxide nanowires derived from the VPI of diethylzinc (DEZ) and water into self-assembled poly(styrene-block-methyl methacrylate) (PS-*b*-PMMA) BCP thin films. We demonstrate the ability to easily adjust the characteristic dimensions, chemical composition, and electrical conductivity of the resulting aluminum-doped ZnO (AZO) nanowires by varying the TMA exposure time during a single-cycle alumina priming. Increasing the TMA exposure duration enhances not only ZnO infiltration fidelity but also ZnO electrical conductivity with the minimized resistivity of 14.3 k $\Omega$ ·cm with an Al concentration of 6.02 % at a TMA exposure time of 100 s. Simultaneously, the variation of the TMA exposure time provides control over the final AZO nanowire feature sizes with width and height in the ranges of 26 – 33 nm and 23 – 47 nm, respectively. These findings provide a guideline for controlling the composition, dimensions, and electrical properties of alumina-primed metal oxide nanostructures based on VPI in polymer templates.

**5:15pm TF-WeA-13 Spectroscopic Study of the Inorganic Structure in PIM-1/Metal Oxyhydroxide Hybrid Membranes Synthesized via Vapor Phase Infiltration, Benjamin Jean, I. Slagle, F. Alamgir, M. Losego, Georgia Tech**

Polymers of intrinsic microporosity (PIMs) have well-defined microporosity that provide excellent chemical separation ability. However, the chemical stability of these polymers in organic solvents limit their use in organic separations. Vapor phase infiltration (VPI) can be used to transform PIM membranes into organic-inorganic hybrid membranes with superior chemical stability and separation performance. This talk will address our recent efforts to better understand the physicochemical structure of VPI-synthesized hybrid PIM-1 membranes and the importance of the inorganic's network structure within the hybrid to affect the membrane's chemical stability and separation performance. We use a combination of spectroscopic techniques including advanced X-ray absorption spectroscopies (XANES and EXAFS) to study the structure of inorganic clusters in VPI treated PIM-1 hybrid membranes. In PIM-1/ZnO<sub>x</sub>H<sub>y</sub> hybrids, XPS and FTIR show the inorganic structure is primarily a hydroxide structure. EXAFS indicates that these inorganics readily form several metal atom clusters as well. The degree of inorganic clustering can be controlled by processing conditions such as exposure time and number of cycles. The membrane solvent stability appears to be most influenced by the degree of clustering. For PIM-1/AlO<sub>x</sub>H<sub>y</sub> hybrid membranes, XPS and FTIR in conjunction with DFT calculations indicate that the final inorganic species is an oxyhydroxide species. Solid state NMR and XANES provide additional evidence that these aluminum hydroxide clusters network with each other into larger chains or sheets. The degree of inorganic clustering can be controlled by processing conditions such as exposure time and number of cycles. The membrane material properties such as membrane selectivity and solvent stability can be modified significantly based on the degree of

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clustering. This talk will discuss these findings and provide guidance for how to design these materials to achieve desired properties like solvent stability and membrane selectivity.

**5:30pm TF-WeA-14 Nanoporous TiO<sub>2</sub> Coating Designed by Swelling-Assisted Sequential Infiltration Synthesis (SIS) of Block Copolymer for Self-Cleaning Application, Khalil Omotosho, D. Berman, University of North Texas**

Highly porous photoinduced self-cleaning coatings are pivotal across a broad range of applications like surfaces for optical coatings, solar panels, mirrors, etc. In this work, we described a new technique to design nanoporous photocatalytic self-cleaning TiO<sub>2</sub> coating with very high hydrophilicity. This coating was created by the swelling-assisted sequential infiltration synthesis (SIS) of a block copolymer template (PS75-b-P4VP25), followed by the elimination of the polymer via oxidative thermal annealing in a furnace at 450 °C for 4 h. The optimization of the infiltration process was realized with the quartz crystal microbalance (QCM), by estimating the mass of infiltrated precursor molecules as a function of the number of SIS cycles. SEM images show that the adopted swelling-assisted SIS approach is suitable for designing smooth uniform TiO<sub>2</sub> film with an interconnected network of pores. The films exhibited good crystallinity in the anatase phase. Hydrophilic property of the film was improved upon 1 h Ultraviolet (UV) exposure, leading to unmeasurable contact angle values upon contact with water droplets. The photocatalytic self-cleaning performance of the synthesized porous TiO<sub>2</sub> nanostructures were tested by measuring the photocatalytic degradation of methylene blue (MB). They displayed promising photocatalytic activity, with ~92% of the MB degraded after 180 min of UV irradiation, thus demonstrating comparable level of photoactivity with commercial anatase TiO<sub>2</sub> nanoparticles of the same quantity. This result highlights a new robust approach for designing hydrophilic self-cleaning coatings with controlled porosity and composition.

**5:45pm TF-WeA-15 Permanent Electric Polarization in Vapor Phase Deposited Polymer Thin Films, Stefan Schröder, T. Hartig, L. Schwäke, T. Strunskus, F. Faupel, Kiel University, Germany**

Dielectric materials with a permanent electric polarization, i.e. electrets, have been attracting great interest in application fields such as sensors/transducers, catalysis, cell growth and microfluidics. The increasing demand for device miniaturization and new application pathways, e.g. in organic electronics, creates the need for high precision thin films of such materials. However, since polymers exhibit the best electret properties, the production of precise, high-quality polymer electret films with sufficient dielectric breakdown strength on large-area substrates is a challenge for conventional wet chemical approaches. In this work, we present the latest results on electret thin film fabrication via initiated chemical vapor deposition (iCVD). The iCVD process is a single-step, solvent-free polymer thin film fabrication process capable to grow precise films of high dielectric breakdown strength on large-area substrates as well as complex geometries. Due to the CVD-typical growth, the process can be integrated into existing microelectronics process lines and enables e.g. the use of electret films in complex micro-electromechanical systems (MEMS). The electronic structure, trap landscape as well as polarization response of the electret films can be tailored by the deposition parameters and (co-)monomer combinations. First principle calculations assist in the search for suitable monomers which yield effective electret properties. The fabricated films are tested in different transducer and sensor devices, where they can provide new pathways for next-generation devices.

**6:00pm TF-WeA-16 Using ALD to Alter the Biological Response of Cellulose Fabrics, Li Zhang, M. Losego, Georgia Institute of Technology**

Atomic layer deposition (ALD) of metal oxides onto fabrics has previously been shown to alter the biological response various microbes have with a textile. For example, ALD ZnO<sub>x</sub> coatings have been shown to both accelerate bacterial growth ("feeds" the bacteria) at low coating thicknesses (< 3 nm) and kill the bacteria at higher thicknesses. In this study, we examine how ALD coatings can alter the enzymatic biodegradation of cotton fabrics. Specifically, we study how AlO<sub>x</sub> ALD coatings affect the cellulase enzyme *Trichoderma viride*'s ability to hydrolyze and degrade cotton fabrics. ALD coatings of AlO<sub>x</sub> are deposited using trimethyl aluminum (TMA) and H<sub>2</sub>O with cycle counts varying from 1 – 750 cycles (~0.12-93 nm). It was found that as little as one ALD cycle of AlO<sub>x</sub> could reduce the enzymatic degradation by as much as 5% over a period of 5 days. Deposition of 10 ALD cycles resulted in a further reduction of enzymatic degradation of around 8% over the same time period. However, additional layers of deposition, up to 750 cycles, did not significantly reduce the degradation rate further. To explain both the initial reduction in degradation and the plateauing in rate,

the chemical and physical structures of the coated fabrics were studied with X-ray photoelectron spectroscopy (XPS) and scanning electron microscopy (SEM). XPS indicates that after 3 days of enzymatic degradation the deposited alumina layer is etched away. This dissolution may be due to the alumina layer dissolving in the acetic acid buffer required for the enzyme or because the enzyme is hydrolyzing the surface glucose monomers bound to the alumina. In contrast, the alumina layer on the 10 cycle treated fabric remains intact throughout all 5 days of the enzymatic degradation. This indicates that, on the time-scale studied, ALD deposition of 10 or more cycles results in an alumina coating that is able to survive the enzymatic hydrolysis process resulting in this large disparity between the untreated, 1 cycle and 10 cycle samples. SEM images of the fabrics coated with more cycles indicate crack formation in the as-deposited state and after submerging the fabric in water for 2 days, the amount of cracks present increases significantly. In summary, the large difference in enzymatic degradation for low cycle count fabrics can be explained by the alumina layer surviving and the much smaller difference between higher cycle count treatments can be explained by the primary diffusion mechanism of the enzyme being through the cracks formed in the alumina layer, bypassing any changes in thickness of the alumina.

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