

ALD Applications

Room Ybor Salons I-IV - Session AA-WeA

Novel ALD Applications

Moderators: Jessica Jones, Argonne National Laboratory, Austin Minnich, Caltech

1:30pm AA-WeA-1 Nanolaminate Bragg Reflectors for Acoustic Phonons in the > 100 GHz Range, John Murphy, Jeremy Robinson, Maxim Zalalutdinov, Kyle Munson, Jeffrey Woodward, U.S. Naval Research Laboratory

Nanolaminates grown by atomic layer deposition (ALD) offer a well-controlled platform capable of confining longitudinal acoustic (LA) phonons in > 100 GHz regime through engineered Bragg reflection. LA phonons in this frequency range strongly influence thermal and electronic behavior and require individual layers of Bragg reflectors to be < 10 nm thick with sub-nm roughness, while providing high contrast in acoustic impedance. ALD is unique in its ability to produce nanolaminate Bragg reflectors through sequential growth of ultra-thin, uniform, and low-roughness amorphous multilayers; with tailorable acoustic impedances through material selection. An ALD-based approach also allows conformal coating on planar and three-dimensional geometries where current state-of-the-art GaAs/AlAs, or other epitaxially grown superlattices cannot be realized. Additionally, the semi-surface-agnostic nature of the amorphous oxide ALD growth provides a route for integrating phononic mirrors, cavities, and filters into a variety of a fabrication flows in addition to well-established devices platforms, opening opportunities for next-generation acousto-optic and acousto-electronic systems compatible with current and emerging MEMS architectures.

In this work, we report results for $\text{Al}_2\text{O}_3/\text{HfO}_2$ nanolaminates grown via ALD using layer design guided by transfer-matrix simulations and targeting LA phonons in the frequency range 100-500 GHz. Effective sound speeds and mass densities of both single-layer films and stacked nanolaminates are extracted via ultrafast pump-probe measurements and x-ray reflectivity, respectively, allowing elastic properties to be correlated with ALD growth conditions. We compare the simulated spectral response for phonon lifetime with pump-probe measurements using ultrathin optoacoustic transducer layers that act both as acoustic cavities and a source of optically generated strain pulses. Exploiting this architecture, we are able to validate simulated reflection response with thin optoacoustic transducer layers deposited directly onto nanolaminate films. These results establish ALD nanolaminates as a tunable, integrable platform for GHz-sub-THz phonon control and phononic component design beyond conventional epitaxial III-V and nitride superlattice systems.

1:45pm AA-WeA-2 Next Generation ALD Functionalization of Lead-free MCPs for the Photomultiplier Tube: HRPPD, Stefan Cwik, Melvin, J. Aviles, Stephen M. Clarke, Matthew Grden, Cole J. Hamel, Alexey V. Vyashenko, Mark A. Popecki, Incom Inc.; Jeffrey W. Elam, Argonne National Laboratory, USA; Michael J. Minot, Incom Inc.

Microchannel plates (MCPs) find application in image intensifier tubes for night vision goggles, in spectrometers, and in photo multiplier tubes (PMTs) for time-of-flight (TOF) measurements based on their high resolution, ultrafast timing (10-50 ps) and high gain ($>10^4$). Conventional lead glass MCPs have been the industry standard since the 1960s. The manufacture includes a hydrogen firing step in which lead oxide gets reduced to bring the plate to target resistance and to simultaneously generate the electron emission layer limiting the adjustability of the MCP properties to target applications.

Incom Inc. has commercialized the ALD-GCA-MCP technology, applying ALD technology from Argonne (ANL) on Incom's glass capillary array (GCA) substrates. The physically and chemically robust silicate glass allows the fabrication of large area GCAs up to $20 \times 20 \text{ cm}^2$ and the high substrate resistance enables functionalization by ALD. ALD functionalization of the GCA comprises resistive and emissive layers. The resistive layer is a tunable nanocomposite of conductive and insulating materials and defines most electrical characteristics of the MCP since it serves as a strip layer to recharge the emissive layer during operation. The emissive layer is composed of a high secondary electron yield material like MgO or Al_2O_3 to maximize gain for intensifier applications.

Incom's 1st generation MCPs rely on the combination of Incom proprietary Chem1 resistive coating and MgO emissive coating, which provide high gain stability and durability for open MCP applications in space-flight applications. However, for application in sealed MCP-PMTs, high gain in dry UHV and minimum out-gassing are critical to limit the effects of after-pulsing. This maximizes the device lifetime, crucial for long-term applications such as TOF experimentation in Electron Ion Collider programs.

Herein, we report the performance of Incom's new proprietary halide-free Chem5-MgO MCP technology in sealed MCP-PMTs such as Incom's 10x10 cm HRPPD, which was developed to optimize the application specific MCP characteristics. The Chem5-MgO performance under dry UHV conditions is compared with MCPs based on Chem1-MgO chemistry used in HRPPDs. Notably, the higher gain provided by Chem5 allows lower voltage operation ($\Delta V=200\text{V}$) thereby reducing electric fields in the PMT and minimizing after-pulsing. Large area MCPs with the advanced Chem5-MgO coatings are also being incorporated into Incom's LAPPD, the world's largest planar MCP-PMT.

This work was supported by the US Department of Energy (NNSA), DE-SC0018778

2:00pm AA-WeA-3 Growing Grass for the Stars: Conformal Nanostructured Ar Coatings for Astronomical Micro-Optics, Ishan Rana, Suvrath Mahadevan, Pennsylvania State University

Many Astronomical Instruments require high precision micro-optics with complex free form geometries. A new and attractive choice to fabricate such microoptics is the IPX clear photoresin, this resin offers very high internal transmission (>99.9%) in the visible and near infra-red wavelength range and has excellent shape fidelity. However, fresnel reflections at the air-polymer interface introduce significant photon loss (~4%). This is detrimental for photon starved fields like astronomy. To reduce photon loss we need to apply antireflective coatings on the microlenses. However, due to the complex shape of the microlenses we need highly conformal coatings. We use Atomic Layer Deposition to create these AR coatings.

In our project, we study the application of grass-like anti reflective coatings on IPX clear micro-optics. These coatings are created by first depositing alumina using thermal ALD on the microlenses and then generating nanostructures on the surface of the alumina by treating the samples in de-ionized water. The treated samples with nanostructured surfaces can then again be coated with various other materials like SiO_2 , TiO_2 , and HfO_2 to create a multilayer grass like coating. These coatings mimic a gradient change in refractive index, suppressing surface reflections. Such coatings can improve average transmission from 91.9% up to 99%

While in previous work ALD has been used to coat micro-optics, we demonstrate here for the first time application of ALD on micro-optics with nanostructured surfaces and the antireflective performance of such coatings, as well as discuss the challenges and process steps of such applications.

2:15pm AA-WeA-4 Electrochemical Oxidation of Perfluorobutanoic Acid using ALD Thin Film Electrocatalysts Deposited on Reactive Electrochemical Membranes, Saurabh N Misal, Argonne National Laboratory, USA; Atefeh Nadeali, Brian P Chaplin, University of Illinois at Chicago; Jeffrey W Elam, Argonne National Laboratory, USA

Electrochemical oxidation of per- and polyfluoroalkyl substances (PFAS) using Magnéli-phase titanium suboxide ($\text{Ti}_n\text{O}_{2n-1}$) reactive electrochemical membranes (REMs) is a promising and effective approach for mineralizing concentrated PFAS wastes. Still, it can lead to the accumulation of short-chain PFAS as byproducts. Perfluorobutanoic acid (PFBA) is one such short-chain product observed during PFAS oxidation and is also commonly detected in groundwater and industrial wastewater. Due to its low hydrophobicity, low polarizability, and high mobility, PFBA adsorbs poorly at the anode surface, leading to sluggish kinetics. In this work, we focused on the synthesis of an electrocatalyst using atomic layer deposition (ALD) on REMs to further enhance the removal and defluorination of PFBA during oxidation. In the first part of the study, thin films of SnO_2 , Sb_2O_5 , and Pd were deposited on REMs. For the ALD process, the reactor temperature, precursor dose time, and number of cycles were optimized for high-aspect-ratio (~5000) REMs. The synthesized electrocatalytic REMs were characterized using X-ray photoelectron spectroscopy to confirm oxidation states of the electrocatalyst. Moreover, top surface and cross-sections of the REMs were mapped using scanning electron microscopy with energy-dispersive X-ray spectroscopy. In the second part of the study, the synthesized REMs were tested for the electrochemical oxidation of ~21 mg L^{-1} PFBA in a flow-through reactor at a constant potential and flux. The permeate samples collected from oxidation experiments were analyzed using ion chromatography. At $150 \text{ L m}^{-2} \text{ h}^{-1}$ permeate flux and a constant potential of 3.6 V/SHE, ~80% PFBA was removed with ~100% fluorine recovery using thin-film-deposited REM. The analysis showed the presence of trifluoroacetic acid and perfluoropropanoic acid in $\mu\text{g L}^{-1}$ levels (10–20% product yield), along with the formation of fluoride ions in mg L^{-1} levels (80–90% fluoride yield). In the final part of the study, insulating TiO_2 films were deposited on the synthesized electrocatalytic REM by ALD to suppress

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oxygen evolution side reaction, and electrochemical oxidation of PFBA was tested using the synthesized thin-film-deposited REM.

2:30pm AA-WeA-5 Development of an Atomic Layer Deposition System for Tritium Permeation Barriers on Arbitrary Geometries, Zachary Robinson, University of Rochester; *Jeffrey Woodward*, NRL; *Alexander Kozen*, University of Vermont; *Tyler Liao*, University of Rochester; *Soren Bentley*, UKAEA, UK; *Luke Herter*, *Rashad Ahmadov*, *Josh Ruby*, *Mark Wittman*, *Matthew Sharpe*, University of Rochester

Atomic layer deposition (ALD) is an ideal technique for deposition of films on arbitrarily shaped containers such as those used to contain tritium for fusion applications. In this work we present the design, construction, and initial experimental results from a custom ALD system built to deposit hydrogen isotope permeation barriers. This challenge is motivated by the extreme difficulty of producing and containing tritium while mitigating losses. The ALD films developed in this project could be used to filter, purify, store, and transport tritium, increasing the efficiency of Fusion fuel management.

The primary benefit of our ALD system is the ability to deposit films on arbitrarily shaped surfaces, such as the interior walls of tubing and canisters used to contain tritium. In our initial experiments, we deposited thermal ALD alumina films on both silicon wafers and planar copper foil substrates. Characterization with ellipsometry yielded ALD growth rates of $\sim 1.1 \text{ \AA/cycle}$ for temperatures between 100 °C and 210 °C on the Si witness samples. X-ray photoelectron spectroscopy (XPS) on both the Cu foils and Si substrates indicates stoichiometric Al_2O_3 . To quantify the permeation reduction factor (PRF) of the ALD films, the permeability of deuterium through 25 μm Cu-foils coated with 10 nm of alumina was measured. It was found that thin ALD films have a PRF of around 25 at permeation temperatures between 275 °C and 350 °C.

Following our initial characterization of the system, a 1 L type 316 stainless steel (SS316) canister was installed in our ALD reactor, with 10 silicon witness samples mounted throughout the interior volume of the canister. ALD process conditions were systematically varied, and the uniformity of the alumina films throughout the volume of the canister was optimized such that the inlet side of the reactor was about 10% thicker than the outlet. All of the witness samples had sub-nm roughness. Experiments were also performed on highly polished SS316 to compare film properties between the Si and the stainless substrates. XPS indicates that an aluminum hydroxide forms on SS316 for growth temperatures below 200 °C. Experiments are currently underway to fill a canister with tritium and study the residual gases that exist in the canister as a function of time.

2:45pm AA-WeA-6 Argon Ion Implantation in ALD PbTe Thin Films for Phonon Engineering, Haifeng Cong, Helmut Baumgart, Old Dominion University

Lead telluride (PbTe) is a promising thermoelectric material due to its high Seebeck coefficient and large figure of merit (ZT) at elevated temperatures. While atomic layer deposition (ALD) enables high-quality PbTe thin films, strategies to enhance thermoelectric performance via phonon engineering remain limited. In this study, PbTe thin films were grown on silicon substrates with native oxide using thermal ALD, employing $\text{Pb}(\text{C}_{11}\text{H}_{19}\text{O}_2)_2$ and $(\text{Me}_3\text{Si})_2\text{Te}$ as precursors. Films were deposited at 130–170 °C under 500 mTorr, following surface functionalization to ensure reproducible chemisorption. To engineer phonon transport, argon ions were implanted at 180 keV with a fluence of $1 \times 10^{15} \text{ cm}^{-2}$ and subsequently annealed to generate controlled lattice defect clusters. The effects of implantation were characterized using X-ray diffraction (XRD) for crystal structure, FE-SEM and AFM for morphology and roughness, TEM to directly observe defect profiles, and EDS for composition and stoichiometry. Seebeck coefficient, Hall effect, and thermal conductivity measurements were conducted to assess the impact of defects on thermoelectric performance. Results demonstrate that argon implantation introduces well-distributed lattice defects, effectively scattering phonons and reducing thermal conductivity without significant degradation of electrical properties, leading to enhanced ZT. These findings highlight defect engineering via inert ion implantation as an effective route to improve the thermoelectric performance of ALD PbTe thin films.

3:00pm AA-WeA-7 Multifunctional Hierarchically Restructured Antibacterial Neural Interfacing Electrodes via Plasma-Enhanced Atomic Layer Deposition, Shahram Amini, Drexel University; *Feng Gang*, *Henna Khosla*, Villanova University; *Wesley Seche*, *Kriti Panchal*, *Steve May*, *Ekaterina Pomerantseva*, Drexel University; *Jacob Elmer*, Villanova University; *Gregory Caputo*, *Jeffrey Hettinger*, Rowan University

Next-generation neural and cardiac implantable electrodes are increasingly constrained by the coupled requirements of miniaturization, electrochemical performance, and resistance to bacterial colonization. Hierarchical Surface Restructuring (HSR™) enables substantial amplification of electrochemically active surface area on metallic electrodes, thereby addressing charge-transfer limitations associated with geometric scaling. However, translating these surface architectures into long-term implantable devices requires additional surface-level functionality to mitigate infection risk without degrading electrochemical efficiency. In this work, plasma-enhanced atomic layer deposition (PEALD) is employed as a conformal, thickness-controlled surface-engineering strategy to functionalize HSR™ Pt–10Ir electrodes with ultrathin ZnO-based films. The self-limiting reaction chemistry of PEALD enables uniform coating of complex micro- and nanoscale features while preserving the underlying hierarchical morphology. By modulating plasma chemistry during deposition, ZnO films with distinct phase compositions are integrated onto HSR™ electrodes, enabling independent tuning of antibacterial and electrochemical responses. Antibacterial activity is demonstrated under dark, aerobic conditions, decoupling the observed bactericidal behavior from photocatalytic mechanisms and establishing controlled Zn^{2+} ion release as the dominant mode of action. Simultaneously, select ZnO–Zn nanocomposite coatings enhance electrochemical performance, exhibiting reduced impedance and increased charge storage capacity relative to uncoated HSR™ electrodes. Collectively, these results establish PEALD as a scalable and manufacturing-compatible approach for introducing multifunctionality onto hierarchically restructured neural interfacing electrodes, providing a practical pathway toward infection-resistant bioelectronic interfaces that maintain high electrochemical performance under clinically relevant conditions.

3:15pm AA-WeA-8 ALD Al_2O_3 on Nanocellulose Substrates – Tailoring Barrier and Wetting Properties for Food Packaging, Hugo Patureau, SIMAP, Grenoble-INP, CNRS, France; *Chloé Parisi*, *Julien Bras*, LGP2, France; *Erwan Gicquel*, CILKOA, France; *Frédéric Mercier*, SIMAP, Grenoble-INP, CNRS, France; *Elisabeth Blanquet*, SIMAP, Grenoble-INP, CNRS, France; *Arnaud Mantoux*, SIMAP, Grenoble-INP, CNRS, France

Cellulosic products are a promising alternative to plastics for food packaging due to being biodegradable and recyclable. Nanocellulose is especially promising in the food packaging industry due to high oxygen barrier performances. However, high moisture sensitivity leads to poor water vapor barriers while also compromising oxygen barrier performance under humid conditions. Recent studies have shown that Al_2O_3 deposited by atomic layer deposition (ALD) on cellulosic substrates increases water vapor and oxygen barrier performance and grants hydrophobic properties^{1–4}. However, the influence of ALD process parameters on the performances obtained remain insufficiently explored.

In this work, we demonstrate how deposition temperature, number of ALD cycles and different co-reactants (H_2O , O_2 plasma and ethanol) govern the water vapor barrier and wettability performance of Al_2O_3 films on different nanocellulose substrates. Findings demonstrate a significant increase in wettability using few ALD cycles of TMA and ethanol as co-reactant (figure 1), with correlations observed between wettability and surface energy, surface chemistry, and film morphology. Water vapor transmission rates (WVTR) were measured and also displayed a high dependence on deposition temperature, ALD cycles and co-reactants (figure 2). Experiments revealed a critical thickness of Al_2O_3 on nanocellulose, where no reduction of WVTR was observed beyond that point. The results highlight the critical role of ALD processing conditions in developing high performance nanocellulose food packaging.

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- (3) Mirvakili, M. N.; et al. *ACS Appl. Mater. Interfaces* 2016, 8, 13590–13600.
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