AI/ML/Autonomous Experimentation for Thin Films Processing
Room Ballroom A - Session AIML-ThP
Poster Session

AIML-ThP-1 Ai-Based Real-Time Ashing Rate Estimation and Anomaly Detection Using Equipment Sensor and OES Data in Semiconductor Manufacturing, *Taekyung Ha,* ***You Tack Suh****,* PSK, Republic of Korea

The semiconductor industry is currently undergoing rapid transformation driven by the expansion of artificial intelligence (AI) technologies. AI is not only increasing the demand for advanced semiconductor devices, but is also becoming a key enabler of innovation in semiconductor manufacturing processes. As semiconductor devices become more sophisticated with higher integration density and smaller process nodes, wafer fabrication costs continue to rise significantly. Consequently, unexpected process failures or abnormal equipment conditions can result in substantial financial losses and reduced production yield.

Despite the increasing complexity of semiconductor manufacturing, many plasma-based processes such as etching and ashing still lack direct in-situ methods to determine whether the process is proceeding normally in real time. In current manufacturing environments, abnormal process conditions are typically monitored through post-process sampling inspections and offline metrology techniques. However, these approaches have inherent limitations, including delayed feedback, increased inspection costs, and the risk of missing transient process anomalies.

To address these challenges, this study proposes an AI-based process monitoring framework for semiconductor ashing equipment. The proposed approach utilizes both equipment sensor signals and Optical Emission Spectroscopy (OES) measurement data collected during the ashing process to estimate the ashing rate and detect abnormal process conditions in real time. By integrating multiple process-related data sources, the model aims to improve process visibility and enable early detection of equipment or process deviations.

For the development of the predictive models, machine learning methodologies including XGBoost and Long Short-Term Memory (LSTM)-based neural network algorithms were investigated and compared. Experimental results demonstrated that the XGBoost-based models achieved the best overall performance among the evaluated approaches. The proposed model achieved an ashing rate estimation accuracy of 89% and a process anomaly detection rate of 95%, indicating strong potential for practical deployment in semiconductor manufacturing environments.

The results of this study suggest that AI-driven virtual metrology and anomaly detection technologies can significantly enhance process stability, reduce inspection dependency, and minimize yield loss in advanced semiconductor fabrication processes.

AIML-ThP-2 ASSD Student Award Finalist Poster: Leveraging Convolutional Neural Networks (CNN) for the Real-Time Classification of Melt-Fraction of Phase Change Materials (PCMs) Using Infra-Red (I.R.) Imaging, ***Nishit Pachpande****, Anusree Sen, Debjyoti Banerjee,* Texas A&M University

Phase Change Materials (PCMs) are attractive candidates for incorporation in the Latent Heat Thermal Energy Storage (TES/ LHTES) Systems due to their high latent-heat capacity. Despite their attractive performance, PCMs (especially salt hydrates) often pose reliability issues. Salt hydrates (as PCMs) suffer from debilitating complications due to supercooling issues, i.e. the tendency of PCMs to stay in liquid phase at a temperature lower than the melting temperature (during the solidification portions of thermal cycles involving repeated melting and freezing). Also, the traditional methods for analyzing the thermal properties of PCMs are often time-consuming, expensive and require specialized protocols (where the measured properties are dependent on the measurement techniques).

Machine Learning (ML) techniques can be leveraged to address these issues by enhancing the reliability of PCMs with minimal effect on their performance.The objective of this study is to predict the amount of energy stored in a PCM-based TES using ML techniques (CNN model). An experimental apparatus which was developed in this investigation for performing experimental validations of the computational predictions. These model predictions were also compared with that of a pre-trained model ("EfficientNetB0"/ TensorFlow). The PCM (PureTemp 29^{TM}) was melted in a vertically graduated cylinder using a nichrome coil. Digital image acquisition (GoPro HERO8) of the melting PCM was performed for obtaining the corresponding melt-fraction values based on the height of meniscus in the melt pool. For training and testing the CNN model ~1660 I.R. images were acquired at 1-minute intervals (using FLIR One camera) during melting. The classification model was trained for classification classes corresponding to 10 melt-fraction bands (0-10%, 11-20%, ..., 91-100%). The CNN model predictions yielded a test accuracy of 93.98%, thus performing better than the pre-trained model (93.73%). These results demonstrate the efficacy of implementing deep learning based image analyses models as a fast, low-cost, reliable, accurate, robust, resilient and scalable tool for the real-time monitoring, prediction and characterization of PCM based TES systems.

KEYWORDS: latent heat thermal energy storage, TES, LHTES, machine learning, ML, artificial intelligence, AI, thermal management

AIML-ThP-3 Machine Learning-Assisted Pulsed Laser Deposition Based on Plasma Optical Emission Signatures, ***Dorien Carpenter****, Roman Luckett, Zahra Nasiri, Shiva Gupta, Rodion Mayatskiy,* University of Alabama at Birmingham*; Sumner Harris,* Center for Nanophase Materials Sciences, Oak Ridge National Laboratory*; Renato Camata,* University of Alabama at Birmingham

Growth of thin films by pulsed laser deposition (PLD) is governed by the formation, transport, and condensation of a transient laser-generated plasma plume. During ablation, the target is converted into a complex mixture of atoms, ions, molecules, clusters, droplets, and particulates, whose relative populations depend on target chemistry, constituent vapor pressures, and laser-material coupling. As the plume expands, its ionization state, temperature, particle flux, and kinetic energy distribution are further shaped by laser-plasma interactions, ablation geometry, and collisions with background gas. These coupled processes determine the composition, arrival rate, and energy of growth precursors reaching the substrate, thereby strongly influencing nucleation, phase formation, crystallinity, morphology, and ultimately thin film properties.

Optical emission spectra (OES) acquired during PLD encode rich information about the plasma processes that mediate thin film growth. Because OES can provide real-time, noninvasive measurements of plume composition, excitation, and plasma evolution, it offers actionable data streams that machine-learning workflows can harness to guide thin film growth on synthesis-relevant timescales. This could enable autonomous control of PLD and open new pathways for fabricating nonequilibrium materials that remain difficult to realize using conventional approaches.

In this work, we explore how representations of plasma temperature (*T*) and ionization fraction (*Xe*) can be constructed using Gaussian Process Bayesian Optimization (GPBO) from a limited number of experiments. These quantities are extracted from OES of Fe-rich plumes. A GP regression model is trained on progressively accumulating experimental data to generate surrogate models of the underlying *T* and *Xe* as a function of laser fluence and spot size. We assess the impact of different acquisition functions and GP kernels against baseline random sampling. Model performance is evaluated using synthetic datasets from coupled laser ablation-fluid dynamics simulations, where *T* and *Xe* serve as ground-truth quantities.

We discuss results for PLD of iron, over a fluence range of 2-10 J/cm^2 and spot area of 0.2-13 mm^2 obtained from 1000 independent GPBO trials initialized with random three-point seed pairs of fluence, spot area, and *T* or *Xe*. We quantify the rate of convergence of the optimization to regions of interest in the (*T*, *Xe*) space, with respect to the choice of kernel and acquisition function. We then show how the surrogate models for *T* and *Xe* evolve in time and how they can be used to guide PLD to little explored plume ionization and temperature conditions for thin film growth.

AIML-ThP-4 Role of Oxide Phase and Surface Facets on Self-Limiting Thermal Atomic Layer Etch in High-k Oxides, ***Michael Nolan****, Rita Mullins,* Tyndall National Institute, University College Cork, Ireland

Thermal Atomic Layer Etching (ALE) is of high interest for its potential to deliver atomic level control over the etch of many materials and shows potential for use in future CMOS nodes with requirements for sub-nm levels of control on complex structures. It is performed using sequential surface modification and volatile release reactions. For metal oxides, HF fluorinates the initial surface to form a MF4 layer (M = metal) which undergoes ligand-exchange with precursors such as TiCl4 or SiCl4, which volatilizes the MF4 layer. The question of the role of the phase and surface facets in a deposited high-k metal oxide film has received little attention but can be addressed with first principles atomistic simulations. In this contribution we use density functional theory and molecular dynamics simulations to explore the effect of the phase and surfaces of HfO2 and

ZrO2 on the HF modification half-cycle of ALE. The models used in this study representing polycrystalline materials are the (111) and (001) surface facets of monoclinic, orthorhombic and tetragonal HfO2 and ZrO2. Our thermodynamic analysis shows that for polycrystalline HfO2 and ZrO2, the HF pulse reacts in a self-limiting manner, and is preferred up to processing temperatures that are sensitive to the phase and surface. Models of HF coverage are used to compute calculated theoretical etch rates for the different oxide phases and surface facets and these show a strong dependence on both the crystal phase and the surface so that if different phases and facets are present an uneven etch profile will be seen. The stability, geometry and surface atomic coordination environments drive this dependence.

AIML-ThP-5 Automated Experimentation Reveals Co-Spacer Design Rules for Targeted Phase Selection in Quasi-2D Halide Perovskites, ***Elham Foadian***, *Mahshid Ahmadi,* University of Tennessee Knoxville

Targeted phase control in quasi-2D halide perovskites (HPs) is essential for tailoring their optoelectronic properties, yet remains challenging since phase formation is governed by the coupled effects of spacer chemistry, precursor assembly, and crystallization kinetics. Despite rapid advances in quasi-2D HPs, the identification of spacer design rules still relies largely on manual trial-and-error synthesis, limiting mechanistic understanding and reproducible phase selection. Here, we present a multimodal, closed-loop autonomous experimentation workflow for targeted phase selection in quasi-2D HPs using co-space engineering. We investigate a ternary 3D:2D compositional space composed of 3D $FAPbI_3$ and two Dion–Jacobson co-spacers, 1,4-butanediammonium (BDA) and 3-(aminomethyl)piperidinium (3AMP), to identify compositions that converge toward a stable 3D-like phase. In situ photoluminescence (PL) spectroscopy reveals that BDA promotes rapid crystallization and early formation of 3D-like emissive domains, whereas 3AMP slows crystallization and favors the persistence of lower-n quasi-2D phases. By systematically regulating the BDA:3AMP co-spacer ratio, the crystallization pathway can be tuned to balance fast framework formation with sufficient structural relaxation, enabling convergence toward the target phase. These kinetic insights are integrated with automated high-throughput synthesis, time-dependent PL characterization, X-ray diffraction similarity scoring, and Gaussian process–Bayesian optimization to navigate phase homogeneity and stability. This work establishes co-spacer engineering as a design strategy for targeted phase selection in kinetically governed quasi-2D HPs.

AIML-ThP-6 Toward Autonomous Discovery of UV-Activated Small-Molecule Inhibitors for Area-Selective Atomic Layer Deposition, ***Lucas R. Kuehnel***, *Campbell A. Sweet, Anthony A. Khoury, Erick A. Gutierrez-Monje, Matthias J. Young,* University of Missouri-Columbia

Recent work from our group has demonstrated functional group lithography to nucleate atomic layer deposition (ALD) for patterned deposition on non-growth surfaces like MoS_2 and highly oriented pyrolytic graphite. An analogous approach could enable patterned deposition on growth surfaces through UV-activated inhibition to further develop the dry functional group lithography tool set. However, it is unclear what chemistries might enable UV-activated inhibition. To efficiently search chemical space for UV-activated inhibitors, we developed a high-throughput screening framework for machine-learning-guided discovery. As a model system, we focus here on identifying inhibitors for ZnO ALD from diethylzinc and water on alumina surfaces. Quartz crystal microbalance measurements are used to monitor adsorption, desorption, and film growth dynamics, enabling quantitative assessment of nucleation delay and related mass change metrics. These experimentally derived metrics are used as targets in an initial exploratory active learning phase to generate a diverse seed dataset. We employ a suite of machine learning models for predicting vapor pressure, melting point, boiling point, pK_a, and oxidation potential to screen candidate molecules for physical processing feasibility within the constraints of the experimental system. These experimental constraints, imposed by a high-throughput design, place inherent biases on the searchable chemical space. For example, choosing to eliminate precursor heating enables more rapid testing, but favors smaller, more volatile molecules. Practically speaking, careful consideration of the imposed constraints can favor the discovery of molecules that are more likely to be industrially viable. The success of this discovery framework would provide a first step toward establishing dry functional group lithography as a tool for nanofabrication.

AIML-ThP-7 Toward Autonomous Screening of oMLD Thin Films for Next-Generation Lithium-ion Battery Materials, ***Campbell A. Sweet***, *Kim A. Gerhard, Blaine D. Kelly, Lucas R. Kuehnel, Matthias J. Young,* University of Missouri

Organic conductive polymers with high lithium-ion redox capacities are promising as sustainable positive electrode materials and as performance-enhancing coatings for established cathodes such as lithium iron phosphate. Oxidative Molecular Layer Deposition (oMLD) enables the delivery of conformal organic conductive polymer thin films on porous powder substrates with precise thickness control, making it a promising platform for this application domain. However, exploration of oMLD chemistries remains limited because the experimental workflows are time-intensive and require substantial manual intervention. Given the large chemical design space of organic precursors, traditional oMLD workflows are not readily scalable for rapid materials discovery. Here, we discuss progress to date on establishing a rapid and autonomous workflow for screening oMLD thin films as organic positive electrode coating materials for lithium-ion batteries. We specifically describe our efforts to accelerate deposition, automate precursor handling, and streamline electrochemical characterization and analysis. Workflow analysis revealed key bottlenecks including low precursor volatility, slow performance characterization, and inefficient chemical data mapping and search. We report progress toward autonomous workflow operation using LLM agents to manage repetitive and cumbersome tasks. These advances provide a framework for accelerating oMLD-driven materials discovery and may extend to other challenging-to-accelerate workflows.

AIML-ThP-8 Small Science Agentic Models for Automated Perovskite Thin Film Exploration via Monte Carlo Decision Trees and Delayed Bayesian Optimization Feedback, *Elham Foadian,* ***Sheryl Sanchez***, *Mahshid Ahmadi,* University of Tennessee Knoxville

Autonomous materials discovery requires decision frameworks capable of handling process-dependent outcomes, delayed feedback, and sparse experimental data. We introduce Small Science Agentic Models (SSAMs), a distributed architecture for self-driving laboratories in which scientific reasoning is embedded across narrow, physically interpretable models tied directly to the experimental workflow. Implemented as an event-driven, agent-based closed loop, the SSAM framework integrates hypothesis generation, protocol construction, characterization-derived state representation, machine-learning decision-making, and orchestration, treating each experiment as an evolving process trajectory rather than an isolated parameter evaluation. We apply this framework to discover an effective Dion–Jacobson (DJ) organic spacer for stabilizing the photoactive 3D-like phase of $FAPbI_3$. Through agent-guided hypothesis formation employing Socratic reasoning and Tree-of-Thought branching, furan-2,5-diyldimethanammonium (FuDMA) was identified as a previously unexplored DJ spacer compatible with the perovskite lattice. The binary $FAPbI_3$/$FuDMAPbI_4$ compositional system was then explored autonomously using a Monte Carlo decision tree with delayed-reward updating. Over ten iterations, the system achieved 95% predictive agreement between predicted and measured film quality, identified the highest-performing sample within three iterations, and converged toward focused exploitation by approximately seven iterations. Optimal film formation was localized to FA based compositions of 60–80% within a tightly constrained spin-coating and annealing regime. Complementary Nuclear Magnetic Resonance Spectroscopy (NMR), Fourier Transform Infrared Spectroscopy (FTIR), and time-resolved photoluminescence (TRPL) analysis revealed two composition-dependent FuDMA interaction regimes. At intermediate spacer concentrations, FuDMA acts as a lattice-stabilizing additive that extends carrier lifetimes and suppresses non-radiative recombination, while at higher fractions it drives quasi-2D phase formation accompanied by accelerated fast-decay dynamics. These results establish SSAMs as a transparent, workflow-grounded approach to trajectory-aware autonomous experimentation in dynamic materials systems.

AIML-ThP-9 Revolutionizing Sem Image Analysis: Deep Learning vs. Classical Restoration for Enhancement and Segmentation, ***Amanda Georgina Nieto Sánchez***, Universidad Anáhuac México*; Leon Hamui,* School of Engineering, Universidad Anáhuac México

Scanning electron microscopy (SEM) images frequently suffer from noise, blur, low contrast, and acquisition artifacts that complicate automated microstructure analysis and segmentation. While classical restoration approaches such as Wiener filtering, Richardson–Lucy deconvolution, CLAHE enhancement, and unsharp masking are commonly used, their impact on downstream AI-based segmentation tasks remains insufficiently explored.In this work, we investigate a deep learning-based image

restoration framework for SEM post-processing using convolutional autoencoders trained with synthetically degraded SEM datasets. The degradation pipeline incorporates Gaussian blur, additive and multiplicative noise, and contrast perturbations designed to emulate realistic SEM acquisition conditions. The restored images are subsequently evaluated both through image-quality metrics and automated segmentation performance.A comparative study was performed between the proposed convolutional autoencoder and four classical restoration techniques: Wiener filtering, Richardson–Lucy deconvolution, CLAHE, and unsharp masking. Restoration quality was quantified using PSNR and SSIM metrics. In addition, segmentation performance was evaluated using U-Net architectures trained separately on degraded and restored SEM images.Although classical metrics showed moderate variations between restoration methods, segmentation-oriented evaluation revealed significant differences in model behavior. Quantitative analysis demonstrated that restoration-assisted segmentation reduced false positive regions, decreased over-segmentation, and lowered fragmentation of detected microstructures. Specifically, restored-image segmentation reduced the false-positive component ratio from 0.36 to 0.29 and decreased fragmentation metrics relative to models trained on degraded data.The results suggest that deep learning-based SEM restoration may provide advantages not fully captured by traditional image-quality metrics alone, particularly when evaluated within automated materials characterization workflows. This approach demonstrates the potential of AI-assisted SEM post-processing for improving robustness and reliability in microstructure segmentation tasks.

AIML-ThP-10 Enabling AI-Driven Materials Discovery Through Data Infrastructure*, **Akshay Talekar**,* UL Research Institutes

Advances in machine learning have expanded the role of AI in materials discovery, but model development is rarely the primary constraint in practice. Instead, progress is limited by fragmented data ecosystems, inconsistent metadata, and weak integration between computational and experimental workflows.

This talk frames materials discovery as a data-centric systems problem, where the ability to ingest, standardize, and operationalize heterogeneous data ultimately determines the effectiveness of downstream learning. Emphasis is placed on the role of data architecture, provenance, and interoperability in enabling reproducible and scalable experimentation.

More broadly, this perspective highlights the importance of tightly integrated data and learning pipelines that support continuous iteration under real-world constraints. Addressing these challenges is essential for transitioning AI from isolated modeling efforts to robust, production-scale capabilities within experimental science.

AIML-ThP-11 Statistical Analysis of Pressure-Growth Correlations in Temporal ALD and MLD toward the Identification of Process Deviations*, Andrew Ball, Joelle Scott, Jay Werner, **David Bergsman**,* University of Washington

In the development of many new vacuum deposition-based processes, such as in atomic layer deposition (ALD) and molecular layer deposition (MLD), process conditions are optimized through correlation with deposition outputs, such as film thickness and growth per cycle (GPC). Other data, such as reactant dose pressure and exposure, can also be used in process optimization, either quantitatively (e.g., by correlating growth per cycle with reactant exposure) or qualitatively (e.g., by identifying erroneous dose conditions or reactant depletion). However, while there are many robust tools informing process optimization, there remains a need for tools that can identify deviations within a deposition run.

In this work, we introduce a diagnostic approach for processing and analyzing reactor parameters in temporal ALD/MLD processes. By linking reactor pressure data (via LabVIEW JSON output) with a Python-based processing pipeline, we are able to correlate steady-state and maximum chamber pressure of a given step in the deposition process with the overall process growth-per-cycle (GPC). Through this correlation, we identify processing steps and pressures that most influence the GPC of the process, which can help identify areas for process improvement. False positives were filtered from the list of correlations using a perturbation stress test that involved repeatedly injecting 15% Gaussian noise across 1,000 iterations of a given step. To test this pipeline, we analyzed the pressure and GPC data from several processes in development in our lab as case studies. Results of these analyses suggest that this approach is able to identify subtle pressure drifts as well as processing steps that may be contributing to effects like CVD. This work provides a framework for cleaning data for potential use in interpretable machine learning models like Random Forest and Gradient Boosting, enabling autonomous process optimization with suitable, high-training data, while maintaining the transparency needed for physical reactor troubleshooting.

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