AI/ML/Autonomous Experimentation for Thin Films Processing
Room 303 - Session AIML-WeM
AI/ML for Process Monitoring, Optimization, and Autonomous Synthesis: MBE, ALD/MLD, PLD, and IR Imaging

Moderators: Renato Camata, University of Alabama at Birmingham, **Michael Nolan**, Tyndall National Institute, University College Cork

11:00am **AIML-WeM-13 AI/ML Techniques for Molecular Beam Epitaxy**, ***Stephanie Law***, Pennsylvania State University **INVITED**

MBE synthesis offers precise control over film thickness, composition, and crystallinity by tuning a range of growth parameters. While this tunability enables tailored synthesis, it also introduces significant complexity, resulting in multidimensional growth spaces. Identifying optimal growth parameters within these complex spaces typically involves trial-and-error experimentation, in which growers use a combination of intuition and grid search methods to guide the choice of growth parameters. An alternative way to navigate complex growth spaces is through the use of Bayesian Optimization (BO) or AI-driven hypothesis generation, which can explore parameter space more efficiently and find global optimal growth parameters. By leveraging probabilistic models such as Gaussian Process Regression (GPR), BO can statistically select the most informative experiments, balancing exploration of new growth parameter spaces with exploitation of known good parameters. AI-guided hypothesis generation can be used to read the extant literature and generate experimental campaigns. In this work, we apply BO to efficiently explore the growth space for In_2Se_3 thin films on Al_2O_3 substrates using MBE. By integrating prior experimental data and employing GPR, we employed a data-driven synthesis campaign that significantly reduced the number of required experiments. Leveraging BO resulted in an increase in the fraction of γ-In_2Se_3 from ~50% to >90% in fewer than 10 samples. To further understand the influence of individual growth parameters on model predictions, we used SHapley Additive exPlanations (SHAP), w hich indicated that the indium flux is the most important parameter in predicting the film polymorph fraction, pointing toward defects as a method of stabilizing the polymorph. We also employed AI-guided experimental design to explore growth parameter space for In(Ga)Sb quantum dots. This approach led us to a clear understanding of how QD morphology and density depends on growth parameters, and how QD photoluminescence is correlated with both. These findings highlight the potential of data-driven optimization in complex materials synthesis and further solidify the growing body of evidence supporting the use of ML for efficient thin film experimental design.

11:30am **AIML-WeM-15 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.

11:45am **AIML-WeM-16 ASSD Student Award Finalist Presentation: 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*** [1], *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

[1] **ASSD Student Award Finalist**

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