

AI/ML for Scientific Discovery Room 125 - Session AIML-WeM

AI/ML for Scientific Discovery

Moderators: Alain Diebold, University at Albany-SUNY, Erica Douglas, Sandia National Laboratories

8:00am AIML-WeM-1 "Beyond Fingerprinting": Rapid Process Exploration and Optimization via High-Throughput and Machine Learning, Brad Boyce, Sandia National Laboratories, USA; R. Dingreville, J. Coleman, E. Fowler, C. Martinez, Sandia National Labs; D. Adams, Sandia National Laboratories

INVITED

Material properties are governed by composition and associated microstructure dictated by the thermodynamics and kinetics of manufacturing processes. Often, the connectivity between process conditions and the resulting structure and properties is complex, evading full predictivity via high-fidelity modeling. In this work, we are exploring three manufacturing processes where material properties are difficult to predict directly from process settings: physical vapor deposition, electroplating and additive manufacturing (laser powder bed fusion). Each of the three processes offer unique challenges and opportunities. Across these three exemplars, we are augmenting traditional process-structure-property investigations with an accelerated workflow to detect material structure/composition, prognose associated properties, and adapt the associated process to achieve improved product outcomes. This accelerated detect-prognose-adapt cycle is aided by four key elements: (1) automated combinatorial synthesis to enable rapid parameter sweeps, (2) high-throughput evaluation of both conventional and surrogate indicators of material chemistry, structure, and properties, (3) unsupervised learning algorithms to unravel correlations beyond expert cognition, and (4) Bayesian optimization strategies to efficiently explore and exploit high-dimensional process parameter space. In each of these four domains, we take advantage of previously developed capabilities, or where such capabilities are insufficient, we develop novel techniques. For example, in the domain of electroplating synthesis, we have employed an existing robotic pipetting system for formulation of solution chemistries while developing a custom 12-cell parallel electroplating system that enables hundreds of unique conditions to be explored in about a day. While we consider purely data-driven ML algorithms for some correlation analysis, a more interpretable and robust solution includes physical models based on established governing equations. In this regard, we have developed a physics-informed multimodal autoencoder that fuses data from multiple characterization modalities alongside physical models to provide a deeper fingerprint of material state, enabling cluster disentanglement and cross-modal inference. SNL is managed and operated by NTESS under DOE NNSA contract DE-NA0003525.

8:30am AIML-WeM-3 Simulations of Epitaxial Inorganic Interfaces Using DFT with Machine-Learned Hubbard U Corrections, Noa Marom, Carnegie Mellon University

INVITED

Epitaxial inorganic interfaces lie at the heart of semiconductor, spintronic, and quantum devices. At an interface between two dissimilar materials physical properties and functionalities may arise, which do not exist in any of the isolated components in the bulk. To predict the structure of domain-matched epitaxial interfaces, we use a combination of lattice matching and surface matching algorithms implemented in the OGRE Python package [J. Chem. Phys., 155, 034111 (2021); J. Phys. Condensed Matter, 34, 233002 (2022)]. To study the electronic and magnetic properties of interfaces we use density functional theory (DFT). Within DFT, the many-body interactions between electrons are described by approximate exchange-correlation functionals. The accuracy of the results hinges on an appropriate choice of functional. We have developed a method of machine learning the Hubbard U correction added to a DFT functional by Bayesian optimization (BO) [npj Computational Materials 6, 180 (2020)]. The DFT+U(BO) method balances accuracy with computational cost, enabling unprecedented simulations of large models of surfaces and interfaces of interest for applications in quantum computing. Examples include InAs and InSb surfaces [Advanced Quantum Technologies, 5, 2100033 (2022)], which are the substrates of choice for superconductor/semiconductor Majorana devices; the HgTe/CdTe and InAs/GaSb interfaces [Phys. Rev. Mater. 5, 084204 (2021)], in which a 2D topological insulator phase may arise; the EuS/InAs interface [Phys. Rev. Mater. 5, 064606 (2021)], proposed as a candidate for the realization of a ferromagnet-semiconductor-superconductor Majorana devices without an external magnetic field; and

CdTe as a tunnel barrier to control the coupling strength at the interface between InSb and α -Sn [ACS Applied Materials & Interfaces 15, 16288 (2023)].

9:00am AIML-WeM-5 On-The-Fly Analysis of RHEED Images During Deposition Using Artificial Intelligence, Tiffany Kaspar, J. Pope, S. Akers, H. Sprueill, A. Ter-Petrosyan, D. Hopkins, E. King, J. Drgona, Pacific Northwest National Laboratory

Modern synthesis methods enable the fabrication of an ever-expanding array of novel, non-equilibrium, and/or metastable materials and composites that may possess unique and desirable functionality. Thin film deposition by molecular beam epitaxy (MBE) can produce atomically precise (or nearly so) materials with a wide range of functional electronic, magnetic, ferroelectric/multiferroic, optical, and/or ion-conducting properties. We are working to employ artificial intelligence (AI)-accelerated analysis of in situ and ex situ data streams for on-the-fly feedback control of the MBE deposition process that will enable targeted synthesis of novel materials with desired structure, chemical stability, and functional properties. Here we present a machine-learning-enabled framework for analysis of reflection high energy electron diffraction (RHEED) pattern images in real time (one image per second). Our approach utilizes pre-trained neural networks for image preprocessing, statistical analysis to identify change points in the images over time, and network graph analysis methods to precisely identify and classify changes. We demonstrate this framework using RHEED images collected from the deposition of epitaxial oxide thin films such as anatase TiO₂ on SrTiO₃(001). Advantages and disadvantages of our approach will be discussed, as well as its potential use as the basis for on-the-fly feedback control of deposition parameters.

9:15am AIML-WeM-6 An Unsupervised Machine Learning Approach for the Identification of Adsorbates on a Semiconductor Surface: BCl₃ Adsorption on Si(100), Azadeh Farzaneh, University of Maryland, College Park; C. Wang, S. Kalinin, University of Tennessee Knoxville; R. Butera, Laboratory for Physical Sciences

A more thorough understanding of the reaction of molecular precursors on crystalline and amorphous surfaces will play a significant role in the optimization of industrially relevant processes, such as chemical vapor deposition and atomic layer deposition. Here, we explore an unsupervised machine learning approach to identify reaction products related to molecular precursor adsorption on a semiconductor surface and provide a general framework for analyzing surface species. In particular, we focus our investigations on the adsorption of BCl₃ on Si(100) using scanning tunneling microscopy (STM). We designed an unsupervised workflow that results in the identification of distinct surface moieties and their relative concentrations following the initial adsorption of BCl₃ and subsequent decomposition reactions on the surface. While previous methods have relied on manual cropping of STM images based on defect coordinates, our workflow isolates surface features from the base lattice to generate a training dataset. Two key components of the Si(100) surface are taken into account for isolating surface features: (1) steps and (2) orientation of Si dimer rows. This unsupervised method eliminates the need for manual labeling an untenable amount of surface features, thereby removing any label bias introduced by the operator. It circumvents the bottleneck of machine learning workflows when experimental conditions change and new labeled data is required. We optimize the performance of the unsupervised neural networks by selecting the proper number of feature classes that minimize the image-to-image identification error of distinct surface features in a given experimental data set. This methodology can be generalized and extended to other material systems to provide insight into reactions on surfaces.

9:30am AIML-WeM-7 Quantum and Classical Supervised Learning Analysis of Synthesis-Structure Relationships in Epitaxially-Grown Semiconductors, Andrew Messecar, Western Michigan University; S. Durbin, University of Hawai'i at Mānoa; R. Makin, Western Michigan University

The design of material synthesis experiments occurs within highly multidimensional processing spaces that are defined by many design parameters. Identifying the optimal values for each synthesis parameter is often performed through an expensive, Edisonian, trial-and-error approach to experiment design. Considerable interest exists in the development of machine learning-based approaches for the rapid and accurate identification of optimal materials designs and synthesis conditions yielding material samples that display target properties of interest. In this work, data detailing hundreds of plasma-assisted molecular beam epitaxy

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(PAMBE) growth trials each of ZnO and various nitride semiconductors have been organized into separate, composition-specific data sets. For each growth record, the complete set of experiment parameters (substrate temperature, effusion cell temperatures, growth duration, etc.) are associated with binary measures of crystallinity as well as surface morphology as determined by *in-situ* reflection high-energy electron diffraction (RHEED) patterns. A Bragg-Williams measure of lattice disorder (S^2) is included as an additional, continuous figure of merit for investigation. Quantum and conventional machine learning algorithms – including logistic regression, tree-based algorithms, and quantum support vector machines – are trained on the data to investigate which growth parameters are most statistically significant for influencing crystallinity, surface morphology, and S^2 . When predicting the occurrence of monocrystalline GaN PAMBE, we show that supervised machine learning algorithms for quantum computers can display significant advantage over their classical machine learning counterparts. The class conditional probabilities of obtaining single crystalline and atomically flat thin film crystals are predicted across processing spaces of the two PAMBE operating parameters determined to be most statistically important. S^2 is also forecasted across the same growth spaces. These predictions are compared to conventional experimental wisdom as well as the results described within published literature regarding the PAMBE growth of these materials. The predictions indicate that different growth conditions are of interest depending on whether a single crystalline sample, a flat surface, or a well-ordered lattice is desired. The superior generalization performance displayed by the quantum machine learning algorithms when predicting GaN crystallinity implies possible advantage gained via quantum algorithms when studying synthesis-structure relationships in other material systems.

9:45am **AIML-WeM-8 'DECIEDD with CARE' - Building an Autonomous Ecosystem for the Discovery and Optimization of Metal Nanoparticle Inks**, *J. Elliott Fowler*, Sandia National Laboratories; *N. Trask*, University of Pennsylvania; *M. Kottwitz*, *N. Bell*, *A. Hesu*, *A. Roth*, Sandia National Laboratories; *J. Hanna*, University of Wisconsin - Madison; *J. Foster*, University of Texas at Austin; *J. Boissiere*, Sandia National Laboratories

The end-to-end design and manufacturing of printed circuit boards substrates, a ubiquitous and critical technology in energy storage, communication, and defense systems, is poised to undergo a transformation following developments in additive manufacturing within the last decade. These advancements include droplet-on-demand inkjet printing of conductive inks—suspensions of metallic nanoparticles, graphene, carbon nanotubes, etc.—onto dielectric substrates. Despite extensive research, few printed commercial inks possess the conductivity and robustness desired by high-reliability design agencies. A major contributor to the limited availability of viable inks is the enormous parameter space of processing conditions and material structure, property, and performance criteria that must be balanced during development.

For this reason, Sandia National Laboratories, together with university partners, has engaged in the design and implementation of an autonomous materials discovery platform to efficiently (1) synthesize Cu, Ag, and Au nanoparticles, (2) formulate those nanoparticles into inks, and (3) print those inks to form devices. At each step of the process, characterization data of the structure, properties, and performance is provided to a machine learning algorithm utilizing a self-consistent and scalable/tunable data schema and data management application. Initial campaigns have utilized off-the-shelf machine learning methods to autonomously optimize the size and dispersity of silver nanoparticle via manipulating the stoichiometric ratio of mono-, di- and tri- functionalized carboxylic acid ligands, amongst other variables. Concurrently, development of bespoke solutions such as multifidelity reinforcement learning and scientific machine learning continues to address the challenges of relatively sparse data sets, multimodality and fidelity, and the need to identify underlying process-structure-property-performance relationships.

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11:00am **AIML-WeM-13 Machine Learning for Accelerating Atomic Layer Deposition Process Optimization**, *Minjong Lee*, *D. Kim*, *T. Chu*, *D. Le*, *J. Kim*, *D. Narayan*, *J. Kim*, University of Texas at Dallas

In the era of Industry 4.0, artificial intelligence (AI) and machine learning (ML) are revolutionizing manufacturing processes, enhancing flexibility and efficiency while minimizing process errors [1]. ML integration is particularly valuable in semiconductor fabrication, where current techniques often require over 30 steps to deposit a single high-quality film layer. Out of semiconductor fabrication techniques, atomic layer deposition (ALD) stands

out as a promising deposition method due to its precision in atomic-scale film engineering and compatibility with advanced 3D architectures. However, ALD involves numerous critical parameters affecting film quality, and comprehensive exploration of all conditions is economically unfeasible, with each experimental run costing over a thousand dollars per wafer, plus additional labor and analysis costs [1]. This limitation constrains inspection ranges of ALD conditions, hindering the development of accurate, atomic-scale process models. The incorporation of AI/ML technologies into ALD processes is thus beneficial in improving cost-efficiency for process optimization by potentially decreasing the volume of data necessary.

This study presents a deep neural network (DNN) framework for ML-driven ALD processes, focusing on optimizing hafnium oxide film deposition. While previous ML applications in ALD processes have primarily focused on monitoring film thickness [2], we investigate the film quality, particularly density, which is crucial property for film performance. We incorporate film density using the wet etch rate (WER) test into our DNN framework, leveraging the principle that higher density films have more tightly packed atoms (i.e., slow WER). In this presentation, we report that the DNN case studies involve four ALD process parameters (deposition temperatures, precursor temperatures, and sample locations (x , y)) and three film properties (thickness (T_{ox}), refractive index (RI), and WER). The DNN system demonstrates significant advantages in terms of high prediction accuracy, exceeding 90% for all film properties within prediction ranges of ± 1.0 nm, ± 0.04 , and ± 0.9 Å/min for T_{ox} , RI, and WER, respectively. We also investigate prediction maps of film properties for further efficiency toward “digital twin” of ALD process.

[1] K. J. Kanarik et al., *Nature*, 616, 707 (2023).

[2] A. Arunachalam et al., *JVST A* 40, 012405 (2022).

11:15am **AIML-WeM-14 Utilizing a Machine Learning Potential for Investigating Defects in Hexagonal Boron Nitride**, *John Janisch*, *D. Le*, *T. Rahman*, University of Central Florida

Defects in materials can bring rise to many unique properties and behaviors, and engineering these defects to appear in desirable ways is of utmost importance in maximizing these properties and behaviors. We need atomistic simulations with high accuracy to study not only the defect itself, but also the area around the defects that are affected by its presence. This influence can range from small, local areas near single atom defects, such as a vacancy, to much larger, sprawling areas near large defects, such as grain boundaries. It is not feasible to study large systems with typical high-accuracy methods such as Density Functional Theory (DFT) due to prohibitively long calculation times but utilizing Machine Learning we can construct Machine Learning Potentials (MLP) and overcome these issues. Here, we will present the performance of a MLP for Hexagonal Boron Nitride with and without defects, demonstrating the DFT-level accuracy prediction of material properties such as phonon dispersion as well as defect properties such as vacancy structures and the motion of grain boundaries.

11:30am **AIML-WeM-15 Accelerating Innovation: Using AI for Process Pathfinding**, *L. Medina*, SandBox Semiconductor; *Mokbel Karam*, *S. Sirard*, *M. Chopra*, SandBox Semiconductor

While AI is becoming more common in high volume manufacturing, it remains underleveraged in R&D and technology development settings. In these research environments, where data sets are often sparse and the process requirements constantly changing, it is challenging to establish robust data pipelines to take advantage of traditional AI/ML approaches. In this work, we show how AI can be used to provide key process and pathfinding insights for even small datasets, using a gate-all-around etch (GAA) as a case study. Using the software platform SandBox Studio™ AI, we demonstrate how physics-enabled AI can be used to (1) improve process metrology, (2) generate predictive models of the process space, (3) quickly rule out insufficient process regimes and target more viable spaces, and (4) evolve with pathfinding development cycles with novel process parameters permutations. We first collect a limited set of metrology data from disparate sources and use it to generate a high accuracy predictive model of the process space for the GAA etch. We specifically target common metrology sources which are non-destructive, and cost-effective, including Optical Critical Dimension (OCD) scatterometry, ellipsometry, and CD-SEM. We then illustrate how an AI-based model can be used to capture the experimental process space accurately and efficiently. Next, we demonstrate a search strategy for identifying an optimal set of process conditions subject to a defined set of constraints. We highlight that entire process regimes can be visualized, searched, and/or ruled out using the predictive model. Lastly, representative of an R&D environment, we

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illustrate how the model can be updated to predict outcomes for new process parameters.

11:45am **AIML-WeM-16 AI-Driven Synthesis of Thin Films with Pulsed Laser Deposition**, *Sumner Harris*, Oak Ridge National Laboratory; *A. Biswas*, University of Tennessee, Oak Ridge National Laboratory; *C. Rouleau*, A. Puzetky, S. Yun, R. Vasudevan, D. Geohagan, K. Xiao, Oak Ridge National Laboratory

Traditional methods for synthesizing thin films have typically involved slow, sequential processes requiring significant human intervention, with material optimization often relying on a mix of expertise and chance discoveries. Recent technological progress in autonomous synthesis experiments which combine automated synthesis and characterization with artificial intelligence (AI) has enabled rapid exploration of large parameter spaces, promising to greatly accelerate and advance our understanding of synthesis science. In this presentation, I will highlight the development of two flexible, autonomy-enabled pulsed laser deposition (PLD) platforms: one incorporating real-time, in situ gas-phase and optical diagnostics, and the other featuring in vacuo robotic transfer for subsequent characterization. I will detail how we merged in situ, real-time diagnostics and characterization with high-throughput methodologies and cloud connectivity to execute an autonomous synthesis experiment using PLD. We synthesized ultrathin WSe₂ films via co-ablation of two targets, employing real-time laser reflectivity for precise thickness control, and achieved a tenfold increase in throughput over conventional PLD workflows. Bayesian optimization with Gaussian process regression, utilizing in situ Raman spectroscopy, directed the synthesis process and autonomously identified the optimal growth windows after sampling 0.25% of a 4D parameter space. Furthermore, the Gaussian process surrogate model predicted process-property relationships, revealing two distinct growth regimes and providing a broader understanding of the synthesis space than would be feasible in traditional PLD workflows. Our platforms and methodologies enable the autonomous synthesis of any material that can be grown by PLD, promising to greatly accelerate thin film synthesis with automated, AI-driven workflows.

12:00pm **AIML-WeM-17 Active-Learning Based Structure Discovery in STM**, *Ganesh Narasimha*, Oak Ridge National Laboratory; *S. Hus*, Oak Ridge National Lab (ORNL); *A. Biswas*, Oak Ridge National Laboratory, USA; *D. Kong*, University of Virginia, USA; *Z. Gai*, R. Vasudevan, Oak Ridge National Laboratory, USA; *M. Ziatdinov*, Pacific Northwest National Laboratory

Scanning tunneling microscopy (STM) is a widely used tool for atomically-resolved imaging of materials and their surface energetics. However, the optimization of the imaging conditions is a time-consuming process due to the extremely sensitive tip-surface interaction. Additionally, conventional experimentation involves sequential imaging procedures, and the material-property correlations are usually deciphered by an operator based on auxiliary spectroscopic information. This limits the experimental throughput. Here we show a Bayesian optimization-based framework to improve imaging conditions in real time [1]. Further, we demonstrate a characterization technique using a probabilistic deep learning framework to automatically correlate structure-property relationships in a Europium-based semimetal, EuZn₂As₂ [2]. The data-driven inference is dynamically incorporated to drive STM exploration in regions that optimize a given material property. This framework employs a sparse sampling approach to efficiently construct the property space using minimal measurements, as little as 1 % of the data required in conventional hyperspectral imaging methods. We further demonstrate property-guided sample exploration using a multiscale-process implementation for the autonomous discovery of structural origins of an observed material property. Our findings reveal correlations of the electronic properties unique to local defect density, surface terminations, and point defects [3]. The deep learning framework offers future implications to study and induce dynamic processes such as molecular manipulations to assemble artificial quantum structures.

References:

1. Narasimha, G., Hus, S., Biswas, A., Vasudevan, R., & Ziatdinov, M. (2024). Autonomous convergence of STM control parameters using Bayesian optimization. *APL Machine Learning*, 2(1).
2. Blawat, J. et al. Unusual Electrical and Magnetic Properties in Layered EuZn₂As₂. *Advanced Quantum Technologies* 5, 2200012 (2022).
3. Narasimha, G., Kong, D., Regmi, P., Jin, R., Gai, Z., Vasudevan, R., & Ziatdinov, M. (2024). Multiscale structure-property discovery via active learning in scanning tunneling microscopy. *arXiv preprint arXiv:2404.07074*.

Electronic Materials and Photonics

Room 114 - Session EM+AIML+AP+QS+TF-WeM

Ferroelectrics and Memory Devices

Moderators: *Samantha Jaszewski*, Sandia National Labs, *Erin Cleveland*, Laboratory of Physical Sciences

8:00am **EM+AIML+AP+QS+TF-WeM-1 A Scalable Ferroelectric Non-Volatile Memory Operating at High Temperature**, *Dhiren Pradhan*, Department of Electrical and Systems Engineering, University of Pennsylvania; *D. Moore*, 2Materials and Manufacturing Directorate, Air Force Research Laboratory, Wright-Patterson AFB; *G. Kim*, Department of Engineering Chemistry, Chungbuk National University, Cheongju, Republic of Korea; *Y. He*, Department of Electrical and Systems Engineering, University of Pennsylvania; *P. Musavigharavi*, Department of Materials Science and Engineering, University of Central Florida; *K. Kim*, *N. Sharma*, *Z. Han*, *X. Du*, Department of Electrical and Systems Engineering, University of Pennsylvania; *V. Puli*, Materials and Manufacturing Directorate, Air Force Research Laboratory, Wright-Patterson AFB; *E. Stach*, Department of Materials Science and Engineering, University of Pennsylvania; *W. Kennedy*, *N. Glavin*, Materials and Manufacturing Directorate, Air Force Research Laboratory, Wright-Patterson AFB; *R. Olsson III*, *D. Jariwala*, Department of Electrical and Systems Engineering, University of Pennsylvania

Non-volatile memory (NVM) devices that reliably operate at temperatures above 300 °C are currently non-existent and remain a critically unmet challenge in the development of high-temperature (T) resilient electronics. There are numerous emerging harsh environment applications including aerospace, space exploration, oil and gas exploration, nuclear plants, mining and others that require complex, in-situ computing and sensing capabilities, for which high temperature NVM is critical. Current Silicon (Si)-based micro(nano)electronics, utilizing complementary metal oxide semiconductor (CMOS) technology, encounter reliability challenges above 200 °C and cannot retain their functional properties at high temperatures. Ferroelectric Al_xSc_{1-x}N exhibits strong potential for utilization in NVM devices operating at very high temperatures (> 500 °C) given its stable and high remnant polarization (P_R) above 100 $\mu\text{C}/\text{cm}^2$ with demonstrated ferroelectric transition temperature (T_C) > 1000 °C. Here, we demonstrate an Al_{0.68}Sc_{0.32}N ferroelectric diode based NVM device that can reliably operate with clear ferroelectric switching up to 600 °C with distinguishable On and Off states. The coercive field (E_C) from the Triangle Wave I-V measurements is found to be -5.84 (E_{C-}) and +5.98 (E_{C+}) (+/- 0.1) MV/cm at room temperature (RT) and found to decrease with increasing temperature up to 600 °C. The devices exhibit high remnant polarizations (> 100 $\mu\text{C}/\text{cm}^2$) which are stable at high temperatures. At 600 °C, our devices show 1 million read cycles with On-Off ratio above 1 for > 60 hours. Finally, the operating voltages of our AlScN ferrodiodes are < 15 V at 600 °C which is compatible with Silicon Carbide (SiC) based high temperature logic technology, thereby making our demonstration a major step towards commercialization of NVM integrated high-T computers. NVM characteristics of engineered ferrodiodes with higher On-Off ratios at > 600 °C will also be presented in the meeting.

^aDhiren K. Pradhan and David C. Moore contributed equally to this work.

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8:15am **EM+AIML+AP+QS+TF-WeM-2 Oxygen Diffusion Coefficients in Ferroelectric Hafnium Zirconium Oxide Thin Films**, *Jon Ihlefeld*, *L. Shvilberg*, University of Virginia; *C. Zhou*, North Carolina State University

Just over a decade ago, ferroelectricity – the presence of a permanent reorientable dipole – was reported in doped hafnium oxide thin films. This report generated a great deal of excitement as the inherent silicon compatibility of HfO₂, coupled with the extreme thinness of the films that exhibited the ferroelectric response promised to overcome a number of technological hurdles limiting utilization of ferroelectrics in microelectronics. While the material is moving toward mass production, there are lingering issues of insufficient endurance and limited retention. These issues are commonly attributed to oxygen point defects, including the drift of these defects in poled devices. As such, knowledge of oxygen transport in the ferroelectric phase is vital toward overcoming the current shortcomings. In this presentation, we will show the results of experiments using ¹⁸O tracers to evaluate the diffusion coefficient of oxygen in the ferroelectric phase. Hafnium zirconium oxide films containing ¹⁶O were prepared via plasma-enhanced atomic layer deposition followed by post-metallization annealing to form the ferroelectric phase. Following removal of the metal layer, an ¹⁸O-containing hafnium zirconium oxide film was

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deposited via thermal atomic layer deposition with ^{18}O provided from a H_2^{18}O source. Tracer anneals were then performed and the ^{18}O position evaluated with secondary ion mass spectrometry. The results will show that the oxygen diffusion coefficients in the ferroelectric phase are extremely low, with extrapolated room temperature values of only 10^{-26} cm^2/sec derived. The activation energy for oxygen diffusion was calculated to be 1 eV, which is intermediate the values calculated for the equilibrium monoclinic phase and amorphous films. These results indicate that oxygen vacancies may be relatively immobile in ferroelectric hafnia devices and that other charged defects may be the primary source of degradation.

9:00am **EM+AIML+AP+QS+TF-WeM-5 Iridium Etching: Exploring Reactive Ion Etching Parameters for Efficient Electrode Fabrication in Ferroelectric Memory**, Yanan Li, P. Bezar, S. Kundu, F. Lazzarino, X. Piao, Y. Canvel, IMEC Belgium

Non-volatile Ferroelectric lead zirconium titanate (PZT) are interesting candidates for future memory applications but the fatigue resistance of the electrode material from the capacitors is a challenge. Iridium (Ir) is being investigated as electrode material for its superior characteristic. Thus, a patterning process must be developed. Due to the low volatility of the etch products, etching Ir is typically performed by ion beam etching (IBE). The low-throughput, relative scarcity of IBE chambers in the industry, as well as the limited tunability of the sputtering process are motivations for the development of a plasma-based etching approach.

In this work, we conducted experiments with TiN as a hard mask, following the process flow shown in Figure 1. Preliminary data indicates that Ir can be etched using both fluorine-based and chlorine-based gases. We identify and highlight the primary parameters affecting the Ir etch rate in the RIE process, focusing on gas flow rates, power settings, pressure, and substrate temperature. We also compare the relative contributions of physical and chemical reactions to the etch rate of Ir.

XSEM pictures for those experiments are shown in figures 2 and 3. It is observed that selectivity with a TiN hard-mask is a challenge when using these chemistries. Sidewall residues have also been observed in conditions where ion sputtering is dominant. Therefore, optimization of the etch processes based upon an understanding of the etch mechanisms in place is necessary.

9:15am **EM+AIML+AP+QS+TF-WeM-6 Investigations in Current Transport Mechanisms of Multi-Resistance State Hafnia Zirconia Ferroelectric Tunnel Junctions**, Troy Tharpe, Sandia National Laboratories; M. Lenox, University of Virginia; S. Jaszewski, G. Esteves, Sandia National Laboratories; J. Ihlefeld, University of Virginia; M. Henry, Sandia National Laboratories

Since the discovery of ferroelectricity in doped hafnia (HfO_2) and alloyed hafnia zirconia thin films (Hf,ZrO_2) over a decade ago, fluorite-structure binary oxides have garnered great interest for use within ferroelectric memory devices to realize compute-in-memory (CIM) and neuromorphic applications. With conformal atomic layer deposition (ALD) techniques, process temperatures below 400°C , coercive fields close to 1 MV/cm , and ferroelectricity down to $\sim 1\text{ nm}$, hafnia thin films are ideal candidates for back-end-of-line (BEOL) integration with complementary metal oxide semiconductor (CMOS) circuits. Leveraging these qualities, recent research has extensively focused on charge-based hafnia devices, such as ferroelectric random access memory (FeRAM) and ferroelectric tunnel junctions (FTJs). FTJs are realized by sandwiching a 4-7nm ferroelectric between electrodes to form a metal-ferroelectric-metal (MFM) structure with a voltage-controlled resistance modulated by polarization. Thinner than FeRAM and able to generate multistate resistances, FTJs are poised to enable energy efficient CIM devices and artificial intelligence (AI) hardware accelerators with improved performance and small form factor.

In this work, we study FTJs with 7 nm thick $\text{Hf}_{0.5}\text{Zr}_{0.5}\text{O}_2$ deposited by plasma enhanced ALD, and asymmetric 20 nm niobium (Nb) / 115 nm niobium nitride (NbN) electrodes deposited by magnetron and reactive sputter, respectively. Film ferroelectricity is stabilized by subsequent anneal at 565°C for 90s in argon ambient. Fig. 1 (a) depicts an optical image of fabricated HZO FTJs while Fig. 1 (b) illustrates a cross section of device layers. Current density vs voltage (J-V) sweeps are taken at 294, 323, 348, 373, and 398 K for HZO FTJs with diameters varying from $74\ \mu\text{m}$ to $117\ \mu\text{m}$. Fig. 2 (a) shows these J-V sweeps for a $100\ \mu\text{m}$ diameter device, after application of 10 wakeup cycles. Fig. 2 (b) shows high resistance state (HRS) and low resistance state (LRS) trends across temperature for this same device at 0.2V and 0.3V. Fig. 3 (a, b) shows average and outlier resistance ratio (RR) temperature dependence for 4 devices at 0.2 V and 0.3V, respectively. Fig. 3 (c) depicts pulsed hysteresis curves for a $99\ \mu\text{m}$ diameter device at 294 K and 398 K. Device resistance is read at 0.2V and a pulse width of 100ms,

following a write pulse progressing from 1.5V to 1.3V and back with 100mV step and 100ms pulse width. The nonlinear HRS, LRS and RR temperature trends indicate a complex conduction system within HZO FTJs, highlighting the need for continued investigation of current transport mechanics for the realization of ferroelectric CIM devices and multistate AI accelerators.

9:30am **EM+AIML+AP+QS+TF-WeM-7 Correlation between Elastic Modulus and Biaxial Stress in Hafnium Zirconium Oxide (HZO) Thin Films**, Megan Lenox, University of Virginia; S. Jaszewski, Sandia National Laboratories; S. Fields, Naval Research Laboratory; A. Salanova, M. Islam, M. Hoque, University of Virginia; J. Maria, Penn State University; P. Hopkins, J. Ihlefeld, University of Virginia

The discovery of ferroelectricity in hafnium oxide based thin films has catalyzed significant research focused on understanding the ferroelectric property origins when fabricated in conventional metal-ferroelectric-metal geometries. Studies have revealed that electrode material selection impacts oxygen vacancies, interfacial layers, and biaxial stress, all noted responsible ferroelectric mechanisms. The coefficient of thermal expansion (CTE) incongruity between the hafnia and the electrode material induces an in-plane tensile stress following post-metallization annealing. However, recent work has shown that while the electrode material CTE does have an effect, the overall strain resulting from the device is primarily from the CTE of the silicon substrate and densification of the hafnia film during crystallization. This notwithstanding, comparisons between electrode materials have shown significant differences in ferroelectric remanent polarization (P_r) behavior. This work describes these polarization differences through the lens of the elastic modulus of the electrode material. TaN/HZO/TaN/M devices, where M is platinum, TaN, iridium, tungsten, and ruthenium, were fabricated using plasma enhanced atomic layer deposition and sputtering for the hafnia and metal layers, respectively. Wafer flexure measurements done using stylus profilometry revealed each metal electrode material was compressive as deposited. Two-dimensional X-ray diffraction, utilized to derive the $\sin^2(\psi)$ in-plane biaxial stress in the HZO, revealed a strong correlation between stress and electrode elastic modulus (E). Further, Polarization-electric field ($P(E)$) measurements at 2.5 MV cm^{-1} field also showed dependence of P_r on measured E. Conversely, no correlation exists between the electrode CTE and P_r or biaxial stress, respectively. Increasing modulus results in a greater resistance to deformation of the electrode, which when deposited prior to annealing the HZO to crystallize from the amorphous state, restricts the out-of-plane expansion of the HZO, promoting the stabilization of the ferroelectric orthorhombic phase, in a phenomenon known as the "capping effect". This work further promotes the acceleration integration of HZO into MFM devices, such as a non-volatile memory devices.

9:45am **EM+AIML+AP+QS+TF-WeM-8 Ferroelectric Behavior of $\epsilon\text{-WO}_3$** , Mohammad Mahafuzur Rahaman, A. Annerino, J. Shell, P. Gouma, The Ohio State University

Designing novel ferroelectric polymorphs having fast and low energy dipole switching responses is crucial for developing next generation's neuromorphic devices. Reported here a unique binary ferroelectric polymorph called $\epsilon\text{-WO}_3$ and its dipole response to electro-optical stimuli, contributed from the stabilization of ϵ -phase nanoparticles of $\sim 20\text{ nm}$ grains at RT, previously was at -43°C in bulk, by our group using FSP. The epsilon phase of the material is confirmed from XRD and Raman analysis. The nano-domains of ferroelectric $\epsilon\text{-WO}_3$ shows switching behavior within few KV/cm electric field. The ferroelectric nanodomains interact with light and shows blue coloration under electro-optical stimuli. The response of these nanodomains with visible light and its switching behavior with electric field is studied via C vs V measurement. Furthermore, the origin of the observed "ferro-chromic effect", the unique, rapid and reversible coloration of the $\epsilon\text{-WO}_3$ thin films upon application of a voltage in the absence of an electrolyte has been deduced from X-ray Photoelectron Spectroscopy. An important finding for all solid-state single layer ubiquitous displays, energy-saving windows and next generation's non-volatile memory device for neuromorphic computation.

11:00am **EM+AIML+AP+QS+TF-WeM-13 Innovations in DARPA's Optimum Processing Technology Inside Memory Arrays (OPTIMA) Program**, Todd Bauer, DARPA

Fast, compact, and power-efficient compute-in-memory (CIM) accelerators can move machine learning from data centers to edge compute devices, enabling training and inference to be done where the training data is collected. However, conventional accelerators that use vonNeumann architecture have poor area and computational power efficiency and

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long execution latency. CIM architectures with Multiply Accumulate Macros (MAMs) can address the power and performance limitations of approaches that use von Neumann hardware architectures. To date these MAM implementations have been hindered by the large physical size of memory elements and the high-power consumption of supporting circuitry. The Defense Advanced Research Program Agency's Optimum Processing Technology Inside Memory Arrays (OPTIMA) program seeks to develop area- and power-efficient high-performance MAMs within innovative signal processing circuits. The key technical challenges that performers are addressing include 1) developing area-efficient, multi-bit memory elements (i.e. 8 bits of storage in a 1T-1C structure) that can be incorporated into compact multiply compute elements (MCEs) and 2) achieving compact, scalable, and power-efficient MAM circuits. This presentation will provide an overview of the OPTIMA program goals and approaches to achieving those goals.

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11:30am **EM+AIML+AP+QS+TF-WeM-15 A Transition Toward Solid-State in TiO₂ Protonic ECRAM**, *John Hoerauf*, University of Maryland, College Park; *M. Schroeder*, Army Research Laboratory; *D. Stewart*, *G. Rubloff*, University of Maryland, College Park

AI and inference learning energy demands are on pace to surpass global energy production¹, but analog in-memory computing hardware can reduce the energy required by up to six orders of magnitude². Electrochemical RAM (ECRAM) is a new and promising transistor technology to realize physical neuromorphic analog in-memory computing circuits, achieved on the device level by modifying a thin-film battery stack to measure the impedance of a selected electrode. The impedance is controlled by changing the state of charge of the battery, electrochemically doping the selected electrode with the electrochemically active species. ECRAM that utilizes protons as the electrochemically active species is compatible with existing CMOS devices, has faster programming speed and increased device durability compared to more established Lithium ion ECRAM. As a less well studied system, it is helpful to understand the insertion of protons in and out of the electrode of interest using a more traditional liquid cell before advancing to a solid-state system. In this presentation, the liquid cell electrochemical characteristics and degradation mechanisms in anatase TiO₂ are discussed with and without a capping Nafion film. It is observed that the anatase TiO₂ electrode's typically quick degradation is suppressed by adding a spin-cast Nafion film, increasing cyclability in an aqueous acetate buffer solution by >10x cycles and altering the H⁺ insertion kinetics. Subsequently, TiO₂ is used in an all-solid-state three electrode transistor by splitting the bottom current collector into a source-drain configuration and using PdH₂ as the counter electrode and H⁺ reservoir. Results toward novel solid state anatase TiO₂ based protonic ECRAM are discussed with a focus on device state modulation by electrochemical doping.

[1] B. Bailey, "AI Power Consumption Exploding," Semiconductor Engineering. Accessed: May 09, 2024. [Online]. Available: <https://semiengineering.com/ai-power-consumption-exploding/>

[2] E. J. Fuller et al., "Li-Ion Synaptic Transistor for Low Power Analog Computing," *Advanced Materials*, vol. 29, no. 4, p. 1604310, 2017

11:45am **EM+AIML+AP+QS+TF-WeM-16 Effects of Gamma Radiation on the Structural and Ferroelectric Properties of Hafnium Zirconium Oxide Capacitors**, *Samantha Jaszewski*, Sandia National Laboratories; *M. Lenox*, *J. Ihlefeld*, University of Virginia; *M. Henry*, Sandia National Laboratories

Ferroelectric hafnium oxide (HfO₂) presents opportunities for technological developments in microelectronics, such as scaling of ferroelectric random-access memory (FeRAM) and new devices such as ferroelectric field-effect transistors (FeFETs) and ferroelectric tunnel junctions (FTJs), that were not previously possible with conventional ferroelectrics. This is due to its compatibility with silicon and ability to exhibit a ferroelectric response in films as thin as 1 nm. Understanding the interaction between radiation and HfO₂-based ferroelectrics is necessary before this material can be utilized in devices facing radiation-hostile environments. In this work, the effects of varying doses of gamma radiation (1 to 8 Mrad) on the structural and electrical properties of metal-ferroelectric-metal capacitors fabricated with 17 nm thick hafnium zirconium oxide (HZO) layers is investigated. Additionally different electrode materials, titanium nitride and tungsten, will be compared. Polarization-electric field, capacitance-voltage, and leakage current measurements were made after electric field cycling with voltages ranging from 2.6 to 4 V. It will be shown that the devices experience decreased endurance and a shift in the coercive voltage that scales with the applied gamma dose and depends on the electrode

material. Synchrotron nano-Fourier transform infrared spectroscopy measurements demonstrated that no significant phase changes occur after radiation in these films. This work advances the understanding of the interaction between radiation and HfO₂-based ferroelectrics in order to probe the fundamental limits of radiation tolerance in this material.

12:00pm **EM+AIML+AP+QS+TF-WeM-17 Reconfigurable Ferroelectric Field-Effect Transistor Arrays from SWCNTs**, *Dongjoon Rhee*, *K. Kim*, *S. Song*, University of Pennsylvania; *L. Peng*, Peking University, China; *J. Kang*, Sungkyunkwan University (SKKU), Republic of Korea; *R. Olsson III*, *D. Jariwala*, University of Pennsylvania

Ferroelectric field-effect transistor (FeFET) is a promising nonvolatile memory device due to its simple and compact device structure for high-density integration, fast switching speed, and non-destructive readout. Recent progress in FeFETs based on two-dimensional (2D) semiconductor channels and ferroelectric Al_{0.68}Sc_{0.32}N (AlScN) has enabled high-performance nonvolatile memory devices with remarkably high ON-state currents, large current ON/OFF ratio, and large memory windows. However, the wafer-scale synthesis of these 2D semiconductors typically demands growth temperatures exceeding 500 °C, rendering the synthesis process incompatible with back-end-of-line (BEOL) processing and necessitating a subsequent transfer step. Solution-based assembly of semiconducting single-walled carbon nanotube (SWCNT) has shown promise as a strategy to fabricate high-quality semiconducting channels at room temperature, but their integration with AlScN for FeFETs has not yet been achieved. In this work, we present a large array of FeFETs utilizing a dense monolayer film of highly aligned semiconducting SWCNTs and ferroelectric AlScN. Our SWCNT FeFETs can be engineered from *p*-type to ambipolar by changing the contact metals at the metal-semiconducting interface. The ambipolar FeFETs showed high electron and hole current densities, both exceeding 300 μA μm⁻¹, along with stable memory retention over 10⁴ s and endurance greater than 10⁴ cycles. Our devices can also function as reconfigurable *p*- and *n*-FETs by switching the polarization direction of AlScN, potentially enabling multifunctional logic and memory applications at the circuit level.

AI/ML for Scientific Discovery

Room Central Hall - Session AIML-ThP

AI/ML for Scientific Discovery Poster Session

AIML-ThP-1 High-Throughput Ab Initio Screening of MAB Phases: Phase Stability and Mechanical Property Relationships, *Nikola Koutna*, TU Wien, Austria; *L. Hultman*, Linköping Univ., IFM, Thin Film Physics Div., Sweden; *P. Mayrhofer*, TU Wien, Austria; *D. Sangiovanni*, Linköping Univ., IFM, Thin Film Physics Div., Sweden

MAB phases (MABs)—alternating atomically-thin ceramic and metallic-like layers—offer an interesting combination of mechanical, magnetocaloric, and catalytic properties, high-temperature oxidation resistance as well as damage tolerance, and have conquered a prominent role in the development of 2D materials. Despite their vast chemical and phase space, relatively few MABs have been achieved experimentally. In this poster I will present high-throughput ab initio screening of MABs that combine group 4–7 transition metals (M); Al, Si, Ga, Ge, or In (A); and boron (B). I will aim on revealing and understanding their phase stability trends and mechanical properties derived from elastic-constants-based descriptors. Considering the 1:1:1, 2:1:1, 2:1:2, 3:1:2, 3:1:3, and 3:1:4 M:A:B ratios and 10 competing phase prototypes for each elemental combination, the corresponding formation energy spectra of dynamically stable phases will be used to estimate the synthesizability of a single-phase MAB. Furthermore, the volumetric proximity of energetically-close MABs will allow identifying systems with possible transformation toughening abilities. The analysis of directional Cauchy pressures and Young's moduli will allow to analyze mechanical response parallel and normal to M–B/A layers. The poster will also suggest the most promising MAB candidates, including Nb₂AlB₄, Cr₂SiB₂, Mn₂SiB₂ or the already synthesised MoAlB.

AIML-ThP-2 Leak Detection Algorithm Through 2D Image Transformation of Multi-Wavelength Data from SPOES and Application of CNN, *Youngjun Yuk*, *k. Kim*, *H. Kim*, Tech University of Korea, Korea (Democratic People's Republic of)

In semiconductor processing, vacuum chambers are utilized for contamination prevention, plasma generation and so on. External air can enter through cracks in the chamber, which is referred to as a "leak." Leak can lead to a reduction in semiconductor yield. Therefore, it is essential to detect leaks in real-time. SPOES (Self-Plasma Optical Emission Spectroscopy) can analyze the gas composition inside the chamber in real-time through spectroscopic analysis and detect changes in gas composition instantaneously.

During the process of detecting leaks using SPOES, the DC offset can change due to various chamber conditions or noises such as thermal noise. If the offset changes, it becomes difficult to determine whether the increase in signal is due to a leak or a change in offset when only a single peak is used for leak detection. As a result, this can lead to the misclassification of leaks, thereby reducing the reliability of the system.

In this paper, to improve the misclassification problem, we propose a leak detection system with high reliability by using CNN classification algorithm with using 2D image transformation to monitor multi-wavelengths.

The most significant characteristics of the leak and offset change appear in the wavelengths where the signal changes. A leak signal affects only within specific wavelengths, whereas an offset change affects all wavelengths. A 2D image transform was applied to emphasize these characteristics, and by adding color changes, it became possible to simultaneously represent three dimensions of data: time, intensity, and wavelength. By utilizing this characteristic and monitoring both the wavelengths where leaks are detected and where leaks are not detected, we were able to collect SPOES data corresponding to leak, offset, and normal conditions, and clearly to identify the distinguishing features of each.

When CNN was applied to multi-wavelength data, higher accurate leak detect was achieved compared to applying machine learning to single-wavelength data. In an environment with 2.5 Torr N₂ gas flow and no offset variation, a leak of approximately 0.4 ppm was detected with a maximum validation accuracy of 97.46% when three types of filters and machine learning algorithms were applied to the single-wavelength data. In contrast, when CNN was applied for classification, the leak was detected with a validation accuracy of 99.5%.

Acknowledgement: This paper was funded by the MOTIE (1415181071) and KSRC (Korea Semiconductor Research Consortium) (20019500) support program for the development of future semiconductor devices.

AIML-ThP-4 Neo: An Artificial Intelligence-Based Framework to Address Reproducibility Challenges in Materials Characterization, *Min Long*, *M. Lau*, Boise State University; *A. Burleigh*, *J. Terry*, Illinois Institute of Technology

We address the reproducibility challenges of improper analysis of the large and growing materials characterization data from modern and next-generation instruments by developing an open-source, artificial intelligence (AI) based framework: Neo that can reliably, more efficiently, and automatically analyze the data from various types of measurements. The datasets are usually complex and often require significant human effort to interpret and extract meaningful physicochemical insights. AI techniques have the potential to improve the efficiency and accuracy of surface analysis by automating data analysis and interpretation. Thus, we adopted intelligent methods like genetic algorithms, differential evolution, and neuroevolution algorithms to efficiently find the physical and chemical properties of materials that lead to high-quality fits of the experimental spectra. A human analyst suggests a set of initial parameters potentially present in the sample, used as theoretical standards and passes it to the framework. The framework then searches the large multidimensional space of combinations of these materials to determine the set of structural paths using the theoretical standards that best reproduce the experimental data. Our AI-based framework integrates with knowledge by not only finding the best mathematical description of the data but also the most physical and chemically meaningful results to improve the interpretability of models. The framework has been applied to various spectroscopy measurements and created sub-packages, such as EXAFS-Neo for extended X-ray absorption fine structure (EXAFS) measurements, Nano-Neo for nanoindentation load-displacement curve analysis, XES-Neo for X-ray emission spectroscopy (XES) analysis. We are also planning to extend this frame to other measurements like X-ray photoelectron spectroscopy (XPS). Our published results show that the method can successfully provide refined structures in simple molecules, bulk crystals, and from an operando lithium-ion battery with much less human intervention in comparison with conventional methods.

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