

Plasma Science and Technology Room 315 - Session PS+AP-MoM

Plasma Processes for Advanced Interconnects

Moderators: John Arnold, IBM, Takeshi Ohmori, Hitachi

10:00am **PS+AP-MoM-1 Interconnect for 7A and Beyond**, **Scott Allen**, IBM Research Division, Albany, NY **INVITED**

BEOL scaling continues to challenge the limits of plasma etch process capability. Conventional approaches have shown to have limited extendibility for next generation devices. Challenges with the traditional damascene integration and superior resistance scaling with alternate conductors make transition to subtractive patterning more attractive. Capacitance control using new dielectric materials or schemes requires innovation and examination. Additionally, High-NA EUV and other new patterning techniques provide pathways forward but require significant development. Advances in etch tooling and stack materials are providing options to meet these challenges. Novel use of known techniques can be integrated with these new tools and materials to enable semiconductor technology at 7A and beyond.

10:30am **PS+AP-MoM-3 Epitaxial conductors to replace Cu in advanced interconnect metallization**, **Jean-Philippe Soulie**, **François Chanceler**, **Steven Brems**, **Chen Wu**, **Johan Swerts**, **Seongho Park**, **Christoph Adelman**, IMEC, Belgium **INVITED**

Cu interconnects face a steep resistance and reliability penalty when line widths approach dimension of 10nm: electron scattering at surfaces and grain boundaries amplifies the resistivity, while diffusion barriers and liners consume an increasing fraction of the available cross-section and are increasingly difficult to scale[1]. In this context, epitaxial conductors offer a fundamentally different route to sustain conductivity at nanoscale dimensions by eliminating large-angle grain boundaries, reducing microstructural variability, and improving pattern-transfer fidelity during direct metal etch. Here, we summarize our recent progress on epitaxial conductors aimed at replacing Cu in advanced BEOL metallization, with a focus on CMOS-compatible epitaxial Ru and metallic delafossite PtCoO_2 .

For Ru, we demonstrate honeycomb epitaxy on $\text{AlN}(0001)/\text{Si}(111)$ using standard PVD at a BEOL-relevant thermal budget, scalable to 300-mm wafers[2]. Structural characterization confirms coherent epitaxial alignment and low mosaicity. Electrical transport measurements reveal bulk-like resistivity ($7.8\mu\Omega\text{cm}$ at 30nm), resulting in a markedly weaker thickness-dependent resistivity increase than polycrystalline Ru and Cu in the ultrathin regime. These findings highlight the advantage of suppressing grain-boundary scattering through epitaxy.

Beyond elemental metals, we show that metallic delafossite PtCoO_2 can deliver even lower resistivity at nanoscale thicknesses when epitaxy is stabilized. Using a multilayer PVD approach combined with repeated oxidation anneals, epitaxial PtCoO_2 is stabilized down to 4 nm and exhibits an unusual thickness-independent resistivity of $4.8\mu\Omega\text{cm}$ over the 8 to 17nm range[3]. This behaviour is enabled by solid-phase epitaxial regrowth, film densification, and defect annihilation, underscoring the critical role of interface chemistry and oxygen control in complex oxide conductors.

Looking forward, the integration of epitaxial conductors into realistic interconnect architectures will require scalable strategies compatible with amorphous BEOL dielectrics. Layer-transfer approaches, such as wafer-to-wafer bonding followed by substrate removal, emerge as a universally applicable pathway for epitaxial conductor integration, enabling the decoupling of epitaxial growth from backend processing. Continued progress in epitaxial growth, interface engineering, and transfer integration positions epitaxial Ru and delafossite oxides as strong contenders for future liner-light or linerless interconnect technologies beyond Cu.

[1]J-Ph. Soulié, J. Appl. Phys. 136(17)171101(2024). [2]C. Adelman, 2026 ECTC(2026), Orlando(USA). [3]J-Ph. Soulié, 2026 IEEE IITC(2026), San Jose (USA)

11:00am **PS+AP-MoM-5 Reactive High Power Impulse Magnetron Sputtering with Positive Cathode Reversal Using a Rotary Magnetron**, **Nicholas Connolly**¹, **Collin Jeckell**, University of Illinois Urbana-Champaign; **Rajib Paul**, **Brian Jurczyk**, Starfire Industries, LLC; **David Ruzic**, University of Illinois Urbana-Champaign

This work investigates the novel sputtering technology of HiPIMS with positive cathode reversal (Positive Kick™), herein referred to as HiPIMS+CR, on rotary magnetrons for reactive sputtering. As compared to DC, HiPIMS provides the advantage of higher ion fractions at the target[1] and at the substrate[2]. As compared to standard HiPIMS, positive cathode reversal allows for control of the ion energy at the substrate, allowing for tuning of film properties such as stress as well as increased deposition rate overall[3]. In this work, the deposited film is aluminum nitride (AlN), which has applications in resonator devices, piezoelectric devices, and as a thermal spreader for heat management[4]. Many large-area industrial coating processes utilize rotary magnetrons instead of planar magnetrons, as they offer increased target utilization and higher power throughput due to better heat removal. It has been shown with DC that rotary magnetrons offer some benefits and differing deposition physics when compared to planar targets because of the target shape and rotation[5]. This work characterizes HiPIMS+CR applied to a rotary magnetron with target length of 430mm and target outer diameter of 145mm, identifying where the rotary configuration may offer benefit through a research-scale system.

HiPIMS+CR AlN reactive sputtering in the rotary magnetron configuration is characterized by both plasma diagnostics and film characteristics. To characterize the plasma, a Hidden Analytical PSM probe was used to gather time and energy resolved spectra of each ion species at the substrate location versus various plasma parameters. Whether the target was rotating or not rotating affected both the Al^+ and N^+ ion counts without changing the Ar^+ and N_2^+ counts (see Figures 1 and 2), where the N^+/Al^+ ratio increased by over 50% by simply rotating the target and keeping all other process parameters constant. This indicates target rotation does affect the system and adds another variable to adjust film properties such as stoichiometry. Hysteresis curves were generated at various target rotation speeds to understand how the poisoning point was affected by rotation speed in HiPIMS+CR.

AlN film characterization will be presented across varying HiPIMS+CR process recipe parameters, as well as target rotation speed, for the critical characteristics of the applications of AlN. Characteristics measured include film stoichiometry (including oxygen contamination), crystal structure and orientation measured with X-ray diffraction (XRD), and film density measured with X-ray reflectivity (XRR). Thermal conductivity measurements are also made using time-domain thermoreflectance (TDTR).

11:15am **PS+AP-MoM-6 Two-Step Etching for Suppressing Etch-Depth Variation in Tin Etch-Back**, **Yudai Mashiko**, **Makoto Satake**, **Taku Iwase**, **Naoyuki Kofuji**, **Yasushi Sonoda**, Hitachi, Ltd., Japan

In the fabrication of advanced semiconductor devices, uniform TiN etch-back—in which TiN is filled into deep trenches—is essential for contact-plug formation. However, seams are unintentionally formed during the filling process. Consequently, the local etching rate increases due to seam widening, resulting in etch-depth variation between the area around the seam and the surrounding region [1].

In our previous study [2, 3], we reported that radical etching was superior to plasma etching because, in the latter, energetic ions accelerated by self-bias voltage exacerbated seam widening. However, even with conventional radical etching, it remains difficult to suppress the direct exposure of radicals to the seam region. Therefore, we propose a two-step etching process that employs a deposition-film formation step prior to etching. In this method, a deposition film is first formed to serve as a mediating layer, after which TiN is etched by radicals while maintaining this film. This approach is designed to prevent the direct exposure of radicals to the seam.

The mechanism underlying seam-widening suppression was investigated using etch-profile simulations. The deposition film on the TiN surface significantly shortens the effective mean free path of the radicals, thereby preventing their direct exposure to the TiN surface. Consequently, the radical flux reaching the seam interior is markedly reduced, shifting the etching process from a reaction-limited to a supply-limited regime. This transition ensures that etching is governed by the arrival rate of radicals rather than their reactivity, which the simulations confirmed effectively suppresses seam widening compared with conventional radical etching.

¹ PSTD Coburn & Winters Student Award Finalist

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Based on these findings, the two-step process was experimentally demonstrated. A SiCl_x deposition film was formed using $\text{Cl}_2/\text{SiCl}_4$ -based plasma, followed by TiN etching with Cl_2 -based plasma. Cross-sectional observations confirmed that the standard deviation of the etch-depth variation was significantly reduced from 3.8 nm (conventional) to 1.5 nm. These results demonstrate that the proposed two-step etching is an effective approach for achieving uniform TiN etch-back.

[1] G. Vereecke et al., *Microelectron Eng.* 200 (2018).

[2] Y. Mashiko, et al., *DPS 45th international symposium* (2024)

[3] T. Iwase, et al., *Proc. SPIE PC13429* (2025)

11:30am **PS+AP-MoM-7 Roughness Generation Mechanisms in Ru Etching Using O_2/Cl_2 -Based Plasma for Advanced Interconnects**, *Miyako Matsui, Tomoyasu Shohjoh, Taiga Kasai*, Hitachi Ltd., Japan; *Kiyohiko Sato, Makoto Miura*, Hitachi High-Tech Corporation, Japan; *Kenichi Kuwahara*, Hitachi High-Tech Corporation, Jamaica **INVITED**

Continued device miniaturization necessitates further reduction of metal pitch through advanced patterning technologies such as extreme ultraviolet lithography. As metal pitches shrink, alternative metal interconnects are required to replace Cu in order to achieve tighter pitches in the back end of line. Ru is a candidate for alternative interconnects at metal pitches of 20 nm and below, because Ru interconnects are expected to exhibit lower effective resistance than Cu interconnects at such small dimensions. In addition, Ru can be etched directly, enabling new scaling boosters such as semi-damascene patterning. To realize such patterning technologies, Ru patterns must be etched vertically with high selectivity to hard masks, while roughness and other forms of damage must be suppressed to reduce interconnect resistance.

In this work, we investigated the mechanisms of roughness generation during Ru etching and achieved low-roughness etching of fine Ru patterns using an O_2/Cl_2 -based plasma generated by a microwave electron cyclotron resonance etching system. The roughness of Ru line patterns is primarily governed by the transfer of mask pattern roughness, as well as by micromasking effects caused by non-volatile byproducts formed on the sidewalls and by the Ru grain structure.

To analyze the mechanisms of roughness generation in Ru etching, the line edge roughness (LER) of Ru lines and Si_3N_4 masks was evaluated before and after Ru etching using a high-voltage CD-SEM, which simultaneously evaluates Si_3N_4 mask roughness from secondary-electron images and Ru line roughness from backscattered-electron images. Ru etching was performed on 32 nm-pitch Ru patterns with Si_3N_4 mask line widths varied from 23 nm to 10 nm. We found that Ru line roughness is influenced not only by Si_3N_4 mask roughness but also by non-volatile RuO_x and RuCl_x species non-uniformly formed on the Ru sidewalls. In addition, Ru sidewall etching tends to proceed preferentially along grain boundaries. The roughness of Ru sidewalls depends on the balance between ion flux, which generates non-volatile RuO_x and RuCl_x on the sidewalls, and radical flux, which isotropically etches Ru.

To reduce the LER of Ru lines, we proposed adding a passivation gas to the O_2/Cl_2 plasma to form a uniform protective layer on the pattern sidewalls. This uniform protection layer enables stable etching of fine patterns even when the balance between ion flux and radical flux penetrating into the trenches varies to some extent with changes in aspect ratio during Ru etching.

Atomic Scale Processing Mini-Symposium Room 316 - Session AP+EL+EM+PS+TF-TuM

Area Selective Processing and Patterning

Moderators: Greg Parsons, North Carolina State University, Silvia Armini, IMEC, Belgium

8:00am **AP+EL+EM+PS+TF-TuM-1 ASCEND: A Novel Solution-Based Method To Control Deposit Composition and Placement**, *Jevalyne Vienes, Amy Walker*, University of Texas at Dallas

We introduce a novel selective deposition process called ASCEND, or “Additive and Surface Control of Electroless Nanostructure Deposition.” ASCEND integrates mechanistically-informed and well-controlled extensions of electroless deposition (ELD) and chemical bath deposition (CBD). It uses hybrid ELD-CBD conditions and patterned surfaces to deposit strongly adherent metallic and metal chalcogenide films and nanowires. It is inexpensive, easily scaled, and compatible with technologically relevant substrates. We illustrate ASCEND using Cu and CuS deposition. First, we employ ethanolamine additives in Cu ELD using dimethylamine borane (DMAB) on $-OH$ and $-CH_3$ terminated alkanethiolate self-assembled monolayers (SAMs) either to deposit nanowires or perform area-selective deposition. Using monoethanolamine or triethanolamine copper nanowires are formed at the junction of patterned $-OH/-CH_3$ terminated SAMs. In contrast using diethanolamine, area selective deposition is observed in the $-CH_3$ terminated SAM area. Using TOF SIMS data we show that monoethanolamine and triethanolamine adsorb vertically (or at an angle) with the amine group interacting with the $-CH_3$ terminated SAM and leaving the hydroxyl groups free to interact with the bath components. This causes repulsion of the DMAB reducing agent, hindering deposition on the $-CH_3$ terminated SAM areas leading to nanowire formation. In contrast, diethanolamine adsorbs flat on $-CH_3$ terminated SAMs and no nanowire formation is observed; rather area-selective deposition occurs.

In the second experiment we control the deposit composition in Cu ELD using thiourea, which is a common bath additive, on $-OH$, $-COOH$ and $-CH_3$ terminated SAMs. At $< 10^{-2}$ M thiourea, Cu films are deposited on all substrates. At $\geq 10^{-2}$ M the deposited layers are copper sulfides. At pH 5 and pH 9, the film is composed of CuS for all functionalized SAMs studied, while at pH 12 the deposit is Cu_2S . We attribute this to the role of thiourea in the bath. At low concentrations, thiourea acts as a complexing and reducing agent, while at high concentrations ($> 10^{-2}$ M) the dominant reaction is thiourea hydrolysis leading to the formation of copper sulfides. Finally, exploiting this understanding we demonstrate a simple method to deposit layered Cu/Cu_xS and CuS nanowires on patterned SAM substrates by changing the TU concentration in the bath.

8:15am **AP+EL+EM+PS+TF-TuM-2 Using Acetylacetates to Control Chemical Reactivity and Physical Properties of Surfaces**, *Andrew Teplyakov*, University of Delaware

Over the last few years, the applications of various acetyl acetates in tuning both chemical reactivity of surfaces in deposition and etching processes and physical properties of the interfaces and thin films have received a substantial attention both from fundamental perspective and from applications point of view. This presentation will analyze the chemistry of several acetylacetates, including the ligands formed on surfaces by acetylacetone (acacH), hexafluoroacetylacetone (hfacH) and 1,1,1-trifluoroacetylacetone (tfacH) and the implications of this chemistry in area-selective atomic layer deposition (AS-ALD), atomic layer etching (ALE), and tuning optoelectronic properties of oxide surfaces and thin films. The effects of the gas-phase keto-enol equilibrium and the acid/base properties of oxide surfaces on the adsorption mechanisms will be discussed. The applications of this knowledge to design non-growth and growth surfaces for ALD and to propose new chemistries for ALE of 2D materials will be demonstrated.

8:30am **AP+EL+EM+PS+TF-TuM-3 Atomic Layer Etching for Advanced Logic Devices**, *Masanaga Fukasawa*, National Institute of Advanced Industrial Science and Technology (AIST), Japan

INVITED

The dimensions of advanced logic devices require atomic-level control of variability, and atomic-layer etching (ALE) is a promising technique to meet this requirement. By separating adsorption and desorption, ALE improves controllability compared with conventional RIE. Currently, however, the long process time—leading to increased manufacturing cost—remains a major challenge, and thus industrial adoption has been limited. Nevertheless, in next-generation highly scaled 3D transistors such as CFETs, Tuesday Morning, November 10, 2026

further improvements in etch selectivity, mitigation of pattern loading, and reduction of etch-induced damage are required. As a result, the importance of ALE is expected to increase [1].

To further improve etch selectivity, we have developed a technique that integrates area-selective deposition (ASD) and ALE within a single etching chamber to achieve ultra-high etch selectivity [2]. In atomic-scale processes such as ALD and ALE, understanding surface reactions and process performance at the atomic level enables more precise process design through the combination of individual reaction steps. Moreover, this approach is beginning to be adopted not only for ASD + ALE but also for ASD + RIE [3] in various process modules. In particular, with High-NA EUV lithography reducing resist thicknesses to below 30 nm, extremely high etch selectivity becomes even more critical. Thus, integration schemes that combine ASD with RIE/ALE will play an increasingly important role.

Regarding low-damage processing, although several studies have reported blanket-level evaluations, examples that demonstrate practical-level performance have been limited. In this work, we applied ALE or RIE to the bottom-break step of contact hole etching and compared the resulting electrical characteristics of contact chains. The results clearly show that ALE provides superior performance [4].

ALE delivers the controllability, reproducibility, and integrability required for next-generation semiconductor technologies, and elevates semiconductor fabrication to a new level of precision. I believe ALE will become increasingly essential in advanced logic device manufacturing.

Acknowledgement: This paper is based on results obtained from the project, “Research and Development Project of the Enhanced Infrastructures for Post-5G Information and Communication Systems” (JPNP20017), subsidized by New Energy and Industrial Technology Development Organization (NEDO).

[1] M Honda *et al.*, *J. Phys. D: Appl. Phys.* **50**, 234002 (2017). [2] M. Fukasawa *et al.*, *JAP.* **64**, 06SP17 (2025).

[3] M. Matsui *et al.*, *JVST A* **41**, 063002 (2023). [4] A. Hirata *et al.*, *Proc. of IEEE IITC* (2026), San Jose, accepted for oral presentation.

9:00am **AP+EL+EM+PS+TF-TuM-5 Band Practice: Tuning Gold's Workfunction Through NHC d-Band Interactions**, *Sean Barry*, Carleton University, Canada; *Wai Tung Shiu, Paul Ragogna*, Western University, Canada

N-heterocyclic carbenes (NHCs) have emerged as robust small-molecule inhibitors (SMIs) for modifying metal surfaces, yet a quantitative understanding of their influence on surface electronic structure remains limited. Here, we investigate the modulation of the d-band center of a gold surface by NHC adsorption using a combination of solution- and vapour-phase deposition. Four structurally distinct NHCs were deposited on Au substrates, and the resulting interfaces were characterized using X-ray photoelectron spectroscopy (XPS), ultraviolet photoelectron spectroscopy (UPS), and time-of-flight secondary ion mass spectrometry (ToF-SIMS).

All NHCs formed stable monolayers on Au, with surface coverages that depended on both molecular structure and deposition method, ranging from 1.88 to 3.56 NHC/nm². UPS measurements revealed significant reductions in the Au work function (up to ~1.2 eV), with vapour-deposited films exhibiting larger shifts, consistent with differences in interfacial structure and electron redistribution. Analysis of the valence band spectra shows a systematic lowering of the Au d-band center upon NHC adsorption, indicating increased filling of antibonding states and weakened adsorbate-metal interactions.

Three primary factors governing d-band modulation were molecular orientation and the formation of Au(NHC)₂ adatom complexes, which lead to enhanced electron donation. Surface coverage plays a critical role, with higher coverages producing stronger modulation due to increased adsorbate-metal interactions. Finally, ligand electronics, particularly backbone substitution, influence the NHC's σ -donating ability and thus the extent of electronic perturbation at the interface.

This presentation will discuss the structure-function relationships of NHC substitution and coverage as well as absorption interactions.

9:15am **AP+EL+EM+PS+TF-TuM-6 Inhibition of SiO₂ by plasma treatments for bottom-up gap-fill applications**, *Aurelia Trevisan, Adrie Mackus, Erwin Kessels*, Eindhoven University of Technology, The Netherlands

As the features in electronic devices become smaller, top-down processes based on lithography need eventually to be complemented by bottom-up processes such as area selective atomic layer deposition (AS-ALD). Although

ALD allows for conformal deposition on 3D features, it is challenging to fill high aspect ratio (AR) trenches without the formation of defects such as voids and seams in the middle. To obtain a defectless bottom-up gap-fill of a trench by ALD, the growth per cycle (GPC) of the employed ALD process should be lower at the top of the trenches than at the bottom. Different approaches that result in a decreased GPC on the top of AR trenches for seamless gap-fill have been studied employing molecular inhibition species¹ and NH₃ plasma pre-treatments.²

In this work, we investigated the mechanisms of inhibition of SiO₂ by NH₃, N₂/H₂ and SF₆/H₂ plasma exposures aiming at the application of such plasma pre-treatment for achieving gap-fill with SiO₂. We exposed the SiO₂ surface to NH₃, N₂/H₂ and SF₆/H₂ plasma at different conditions and we studied the surface chemistry by reflection-absorption infrared spectroscopy (RAIRS). For a NH₃ plasma exposure, we found that the substitution of the OH groups with NH₂ groups is almost complete³ at low NH₃ pressure from the comparison of the areas of the OH peak (~3740 cm⁻¹) in the RAIRS spectra acquired after the plasma exposures at different NH₃ pressures. Next, to investigate the inhibition of the SiO₂ surface by plasma, RAIRS spectra were also obtained after dosing the Si precursor, bis(diethylamino)silane (BDEAS), with and without the plasma pre-treatment step. From the IR spectra, we found that the amount of adsorbed BDEAS molecules on SiO₂ pre-treated with NH₃ plasma (60s, 0.01 mbar) is reduced by ~82%.

A NH₃ plasma pre-treatment was added before the dose of the precursor to every cycle of the SiO₂ PE-ALD process. The effect of the NH₃ plasma pre-treatment on the growth was investigated by conducting spectroscopic ellipsometry (SE) after every cycle. When the NH₃ plasma pre-treatment was added, a 53% reduction of the GPC was obtained. We expect this reduction to be enough to achieve gap-fill of high AR trenches, which will be studied by cross-sectional transmission electron microscopy.

References

- 1 C. T. Nguyen *et al.*, *Nature Communications* **13**, 7597 (2022)
- 2 Y. Choi *et al.*, *Scientific Reports* **12**, 15756 (2022)
- 3 L. T. Zhuravlev, *Colloids and Surfaces A: Physicochemical and Engineering Aspects* **173**, 1-38 (2000)

9:30am **AP+EL+EM+PS+TF-TuM-7 Precursor Isomerization-Enabled Etching Amplification: A Novel Route to Area Selective Deposition of SiOC Films, Xiaocheng Huang, Junjie Zhao**, Zhejiang University, China

Self-aligned patterning via area selective deposition (ASD) is a promising approach to mitigate overlay variations challenges in photolithography for advanced nanomanufacturing. However, it remains difficult to achieve area selective deposition when the adsorption and/or film growth on the target non-growth surface (NGS) exceeds that on the target growth surface (GS). We developed an etching amplification strategy based on precursor isomerization for selective deposition of SiOC when adsorption and/or film growth was higher on the target NGS than on the target GS. *In-situ* DRIFTS demonstrated that V₃D₃ cyclosiloxane undergoes ring-opening on CoO_x surfaces. This ring-opening reaction significantly enhances the etching rate under O₂/Ar plasma by 243% compared to the pristine pV₃D₃, as confirmed by *in-situ* QCM measurements. *In-situ* QCM during ASD cycles showed higher mass gain on CoO_x than on SiO₂ during V₃D₃ deposition steps; however, the subsequent etching step completely removed the adsorbed species from the NGS due to etching amplification. By integrating deposition and etching processes, we achieved selective deposition of a ca. 18.36 nm thick SiOC film on pre-patterned Co/SiO₂ substrates with nearly 100% selectivity. This work sheds light upon etching amplification by precursor isomerization for developing highly selective ASD processes.

9:45am **AP+EL+EM+PS+TF-TuM-8 A Novel Hybrid Approach to Area-Selective Deposition: Combining Two Strategies for Blocking Growth, Jay Swarup, James Jensen, Gokul Rajagopal, James Engstrom**, Cornell University

Area-selective atomic layer deposition (AS-ALD) has emerged as a promising bottom-up nanofabrication strategy for next-generation semiconductor manufacturing, where continued device scaling makes conventional lithographic patterning increasingly difficult and costly. Selectivity can be achieved by passivating non-growth surfaces (NGS) with blocking molecules while permitting growth on designated growth surfaces (GS), and it depends critically on precursor chemistry, blocking molecule identity and application method, and reactor temperature. Here we systematically investigate two blocking strategies for AS-ALD of Al₂O₃ targeting selective growth on Cu (GS) with suppression on SiO₂ (NGS) using BDMADA-Al, a bulky, non-pyrophoric precursor [1]. We employ *in situ* real

time quartz crystal microbalance (QCM) techniques, coupled with selected *ex situ* techniques including X-ray photoelectron spectroscopy (XPS). The first strategy employs sequential dosing of a small molecule, while the second strategy introduces a co-adsorbate dosed simultaneously with the precursor to enable competitive adsorption at NGS reactive sites. In the first strategy, dimethylamido trimethylsilane (DMATMS) was applied repetitively in an ABC-type cycle (*T* = 285 °C), which produced modest blocking efficacy, where detectable growth from QCM could be delayed for the first ~ 20 cycles, while Al is clearly observed with XPS after 40 cycles. In the second strategy we also employ an ABC-type cycle, but also introduce a co-adsorbate dosed simultaneously with BDMADA-Al at *T* = 120 °C. We examined six different co-adsorbates, all possessing reactive functional groups, which revealed stark differences in blocking efficacy depending on the co-adsorbate used. Three co-adsorbates did not enhance blocking but, rather, produced linear (with exposure time) CVD-like growth during the combined precursor/co-adsorbate pulse. In contrast, three co-adsorbates yielded *complete blocking* of growth on SiO₂ for at least 40 cycles of ALD, confirmed by both QCM and XPS. In order to verify the operative mechanism of competitive adsorption we varied both the partial pressure of the co-adsorbate and the reactor temperature. We found that by either decreasing the partial pressure of the co-adsorbate, or increasing the temperature led to less effective blocking of growth, confirming the mechanism based on competitive adsorption. These results demonstrate that an approach that combines two strategies can produce synergy and a more effective method to enable area selective deposition.

[1] J. V. Swarup, H.-R. Chuang, J. T. Jensen, J. Gao, A. L. You and J. R. Engstrom, *J. Vac. Sci. Technol. A* **43**, 022404 (2025).

11:00am **AP+EL+EM+PS+TF-TuM-13 Probing Reactivity at Heterointerfacial Sites Using Area Selective Atomic Layer Deposition, Shani Monadeev, Oz M. Gazit, Alexander Stook**, Technion Israel Institute of Technology, Israel

Background
Heterointerfaces are a sub-class of defects arising from atomic mismatches across material boundaries. These coordinatively unsaturated sites modulate the electronic and geometric structure of catalysts to facilitate specific reactive adsorption, activation, and transformation of reactant molecules.¹ In electronic devices the critical heterointerfaces are “buried” between the layers,² whereas for catalysts, the reaction performance can depend on both the buried interfaces (i.e. metal support interactions) and on the exposed heterointerfacial (defect) sites, interacting with substrate and product molecules, see **Scheme 1**.

Results and discussion

In the current work, we use a bottom-up area selective atomic layer deposition (AS-ALD) to form supported thin layer raft-like oxides. To facilitate the AS geometry, we developed a new approach, which uses NaCl nanocrystals as masking domains during the ALD process. This unique approach allows us to apply this methodology onto silicon wafers and on different surface area particulate silica support materials. The HRSEM-EDS (Energy Dispersive Spectroscopy) mapping in **Figure 1** shows an example of a well-defined multilayer structure, formed following the sequential deposition of TiO₂ (~20 nm) and subsequently Al₂O₃ (~20 nm) layers onto an NaCl-mask before and after mask removal. Leveraging on the strength of AS-ALD we study the structure, binding energy, strain and more of the exposed heterointerface, aiming to decouple those from the exposed basal plan and bulk effects. We compare our AS-ALD materials to materials obtained using a top-down deposition of thin nanosheets of layer double hydroxides. All materials are extensively characterized using XPS, DRIFT-IR, HRSEM and HRTEM, grazing angle-WAXS and probe molecules TPD-MS. The functionality of the heterointerfacial sites is tested using Aldol type condensation reactions for the conversion of biomass derived platform molecules as a target and probe reaction.

References

- (1) Xie, C., et al., *ACS Catal.* 2020, 10 (19).
- (2) Christensen, D. V., et al., *APL Materials* 2018, 7 (1), 013101.

11:15am **AP+EL+EM+PS+TF-TuM-14 Toward Autonomous Discovery of UV-Activated Small-Molecule Inhibitors for Area-Selective Atomic Layer Deposition, Lucas R. Kuehnle, 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 MoS₂ 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.

11:30am AP+EL+EM+PS+TF-TuM-15 Beyond Two-Color Selectivity: Area Selective Deposition on Multi-Oxide Surfaces Using Small Molecule Inhibitors, Benjamin Sanhueza, Marc J. M. Merckx, Wilhelmus M. M. Kessels, Eindhoven University of Technology, Netherlands; Tania E. Sandoval, Universidad Tecnica Federico Santa Maria, Chile; Adriaan J.M. Mackus, Eindhoven University of Technology, Netherlands

Area-selective deposition (ASD) using small molecule inhibitors (SMIs) emerges as a promising bottom-up alternative, enabling selective deposition on growth areas (GAs) while inhibiting the non-growth areas (NGAs). Although ASD has shown strong potential, most studies are limited to two-surface-selectivity, which does not capture the multi-material complexity of modern semiconductor devices.^[1] In this context, we investigate the selectivity of SiO₂ ASD in a multi-oxide system by evaluating the role of different SMIs and their impact on inhibition selectivity, with relevance on 3D memory^[2] and 3D logic devices.^[3]

Here, we employ a previously developed SiO₂ ASD process on Al₂O₃ and SiO₂ surfaces,^[4] using the bis(diethylamino)silane (BDEAS) precursor, H₂ and O₂ plasma as co-reactants, and different SMIs. Building on this process, we investigate multi-surface selectivity by introducing ZrO₂ as an additional third oxide surface.

Among the SMIs investigated, acetylacetone (Hacac) is presented as a representative case. *In situ* reflection-absorption infrared spectroscopy (RAIRS) shows that Hacac reacts with ZrO₂, achieving surface saturation and up to 96% blocking of a single BDEAS dose, which is comparable to that observed on Al₂O₃ as an NGA. Preliminary results further indicate that the H₂ plasma pre-treatment increases the density of isolated and vicinal -OH groups, enhancing Hacac coverage and improving blocking up to 99.5%, demonstrating the strong influence of this plasma pre-treatment on the ZrO₂ surface.

In situ ellipsometry measurements show that ZrO₂ acts as an effective NGA, exhibiting a nucleation delay of up to 28 cycles (~2.6 nm of selective SiO₂ deposition). Remarkably, similar selectivity is achieved on both Al₂O₃ and ZrO₂ surfaces, indicating consistent inhibition behavior across different oxides.^{[1], [5]}

In addition to the results of this case study, it will be discussed that the choice of SMIs can tune inhibition behavior enabling different growth across surfaces. In this way, the loss of selectivity can be preferentially initiated on one NGA prior to another through SMI selection, enabling controlled thickness variations across different materials.

References

- [1]: Lee et al., *Chem. Mater.* 37 (2025) 1741–1758.
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- [3]: Wang, *Micromachines* 15 (2) (2024) 269
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11:45am AP+EL+EM+PS+TF-TuM-16 Surface Hydroxyl Topology and Relative Orientation Effects on Surface-Inhibitor Reactivity: Revisiting Chemical Passivation in Alumina and Zirconia., Lucas Lodeiro, Nicolás Rozas-Castro, Universidad Tecnica Federico Santa Maria, Chile; Dennis Hausmann, Rachel Nye de Castro, Lam Research Corporation; Adriaan Mackus, Eindhoven University of Technology, Netherlands; Tania Sandoval, Universidad Tecnica Federico Santa Maria, Chile

Hydroxylated metal oxide surfaces are paramount for catalysis and thin-film processing. In this context the reduction of surface reactivity via molecular inhibitors or chemical passivation is a common and necessary practice. Small-molecule inhibitors (SMIs) such as acetylacetone (Hacac) and acetic acid (HAc) are widely employed to inhibit growth by reactive site consumption on hydroxylated oxides.^[1] Despite their ubiquitousness, the influence of **surface site configuration** on inhibitor reactivity remains unexplored.

Experimental evidence that chemically distinct hydroxyl motifs exhibit different reactivities toward SMIs is available.^[2] In this work, we revisit the chemical passivation of hydroxylated alumina and zirconia by SMIs, with emphasis on the role of surface site configuration, defined as a combination of Local Topology and Relative Orientation of hydroxyl groups on metal oxide surfaces. Rather than treating hydroxylated sites as a whole entity, we examine how geometrically and electronically distinct surface motifs modulate inhibitor binding modes and reactivity.

Considering that systematic exploration of all possible site configurations rapidly becomes challenging due to the large chemical space, we further investigate whether simple surface descriptors, derived from local electronic structure and geometry, can provide predictive insight into surface reactivity toward SMIs. Our goal is to identify descriptor trends (e.g., charge distribution, coordination, or accessibility metrics) that correlate with inhibitor adsorption behavior, enabling informed prioritization of surface motifs prior to exhaustive site screening.

By linking surface site configuration, inhibitor-surface reactivity and descriptor-based analysis, this work aims to understand the nuances in the chemical passivation of metal oxide surfaces, and provides guidance to design descriptor-informed screening strategies.

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12:00pm AP+EL+EM+PS+TF-TuM-17 Can pFA Invert Selectivity? The Chemistry of TiO₂ Nucleation on Chlorinated and Aminated Si(111) Surfaces During Thermal Atomic Layer Deposition, Tyler Parke, Andrew Teplyakov, University of Delaware

Recently, the near-atomic level precision offered by atomic layer deposition (ALD) has been exploited by developing area-selective schemes which can be used to fabricate complex three-dimensional nanostructures, such as modification of a substrate's surface functionality to induce a "selectivity inversion" between growth surfaces (GS) and non-growth surfaces (NGS). This is a newly developing technique, which has only been demonstrated thus far with few vapor-phase treatments, and which requires a molecular level understanding of the chemistry between surface and modifying reactant, as well as between modified surface and precursor. Here, single-crystal Si(111) surfaces terminated with hydrogen [H-Si(111)] or chlorine [Cl-Si(111)] monolayers, which are typically NGS's in thermal TiO₂ processes, are proposed as potential substrates to modify with a basic amine, para-fluoroaniline (pFA), and invert their selectivity. Amination with pFA is demonstrated to selectively modify the H-Si(111). Thermal TiO₂ ALD is subsequently attempted to test the aminated surface's capability as a GS or NGS. The fundamental chemistry of the interaction between the pFA and either surface, as well as between the aminated surface and ALD precursors TiCl₄ and tetrakis(dimethylamido)titanium (TDMAT), are probed with a combination of X-ray photoelectron spectroscopy (XPS), Fourier transform infrared spectroscopy (FT-IR), density functional theory (DFT), and time of flight secondary ion mass spectrometry (ToF-SIMS) in order to determine the structure of the aminated surface and understand its effect on reactivity under ALD conditions.

Atomic Scale Processing Mini-Symposium Room 316 - Session AP1+EL+MS+PS+TF-TuA

Advanced Atomic Scale Processing through Modeling and Simulation

Moderators: Robert Bruce, IBM Research, T. J. Watson Research Center, Eric Joseph, IBM T.J. Watson Research Center

2:15pm **AP1+EL+MS+PS+TF-TuA-1 Design and performance of AI agents interfacing with atomic layer deposition tools, Angel Yanguas-Gil**, Argonne National Laboratory

INVITED

In recent years there has been an increased interest in exploring the integration of generative AI in combination with experimental tools to create multipurpose autonomous systems that can solve complex research problems. This vision relies on AI agents based on LLMs that can autonomously interact with experiments to solve multiple tasks (e.g. "Grow 20 nm of tin oxide" or "selectively grow 10 nm of Cu on Co but not on silicon oxide"). However, in order to be useful, these systems have to be compatible with state of the art manufacturing tools and they have to perform reliably well in a broad enough set of materials synthesis tasks.

In this presentation, I will focus on these two aspects in the context of atomic scale processing. First, I will describe how we have augmented an existing ALD tool so that it can be integrated with generative AI models. Our approach relies on defining a Python interface and API that abstract all the specific details of the hardware from the AI agent, while making it compatible with standard approaches for tool use with LLMs. I will also describe how we have implemented AI agents that can communicate with this ALD tool as well as with existing data, such as data logs and background data on ALD and area selective deposition.

In the second part of the presentation, I will introduce a general methodology to evaluate the performance of AI agents on atomic scale processing tasks. We have applied it to evaluate the performance of state of the art models in instruction, process discovery, and materials discovery challenges in the context of atomic scale processing, primarily ALD and selective growth. In all these cases the output of the challenge is a specific instruction to run one or more processes in an ALD tool. Our results show that state of the art models excel at instruction tasks, but they struggle on process and materials discovery tasks where they involve processes and materials that are underrepresented in the literature, pointing to potential limitations and areas of improvement for future AI models specialized in materials synthesis and atomic scale processing.

This research is based upon work supported by Laboratory Directed Research and Development (LDRD) funding from Argonne National Laboratory, provided by the Director, Office of Science, of the U.S. Department of Energy under Contract No. DE-AC02-06CH11357.

2:45pm **AP1+EL+MS+PS+TF-TuA-3 A Multiscale Framework for ALD/ALE Process and Precursor Development Using Neural-Network-Potential Atomistic Simulations and COSMO-SAC Thermodynamic Modeling, Yukihiro Shimogaki**, The University of Tokyo, Japan

Atomic layer deposition (ALD) is indispensable for gate-all-around transistors, high-aspect-ratio DRAM capacitors, and 3D NAND, where conformality and precise thickness control are both required. Atomic layer etching (ALE), the reverse of ALD, matters for low-damage, selective integration, while area-selective deposition (ASD) is attractive but suffers "selectivity loss": unwanted nuclei form on non-growth areas and degrade selectivity over time. We present a multiscale framework coupling COSMO-SAC thermodynamic modeling for precursor down-selection with neural-network-potential (NNP) atomistic simulations for reaction-mechanism analysis; an integrated ALD+ALE scheme designed within it enables highly selective, robust ASD.

An ideal precursor reaches saturation adsorption and is delivered stably; a vapor pressure ≥ 100 Pa below 100 °C, preferably ~ 1000 Pa, broadens the process window. Conventional COSMO-SAC is often inaccurate for metal-containing organometallics. By tuning dispersion-related parameters we obtained good agreement between predicted and measured vapor pressures, enabling efficient down-selection. For Co precursors the framework predicted a high vapor pressure for CpCoCOTFE; the synthesized compound showed ~ 1060 Pa at 85 °C. The same volatility prediction directly guides ALE, where formation and removal of volatile products are central.

Surface-reaction analysis has long been limited by the cost of reaction-path and vibrational searches. NNP-based simulations (e.g., Matlantis) now

provide near-DFT accuracy at large scale, making ALD/ALE surface mechanisms tractable. For the Co precursor, NNP indicated that controlling surface hydrogen coverage prior to dosing suppresses carbon incorporation from side reactions; this was confirmed experimentally, showing precursor design (vapor pressure) and reaction design (surface state) must be co-optimized.

For ASD with CCTBA, NNP molecular dynamics showed facile reaction on Cu but low reactivity on SiO₂, and selective growth on Cu was experimentally confirmed. Residual Co nuclei on SiO₂ degrade selectivity; we designed an ALE path in which Co is chlorinated by SO₂Cl₂ and reacts with Hf₄C to form volatile products whose volatility is supported by COSMO-SAC. The resulting ALD (growth) + ALE (nuclei removal) scheme restores robust selectivity. Finally, sub-monolayer growth per cycle (~ 0.1 ML) is rationalized by steric hindrance, and NNP-MD with kinetic Monte Carlo captures these collective adsorption dynamics. This COSMO-SAC + NNP framework offers a unified route to co-develop ALD/ALE processes (Atomic Layer Process, ALP) and precursors and to overcome ASD selectivity loss.

3:00pm **AP1+EL+MS+PS+TF-TuA-4 Re-designing ALD Experiments for AI-ready Data: The IGZO Case Study, Eleni Poupaki, Cas Visser, Freek Klabbers, Adrie Mackus, Erwin Kessels**, Eindhoven University of Technology, The Netherlands

The AtomicLimits ALD database [1] is a community-driven repository of ALD processes from the literature. The next step is to populate it with more data suitable for artificial intelligence (AI) and machine learning (ML) models. [2] Recent efforts have used large language models for structured data extraction from publications. [3] However, literature data remains fundamentally limited for data-driven modeling: it is incomplete, often missing key process parameters, biased toward successful experiments, and inconsistently reported. ML can accelerate ALD development by predicting film properties, guiding experiment design, and identifying optimal recipes. Unlocking this potential requires not only more data, but experiments designed to produce data suitable for ML. We address this challenge using ALD of InGaZnO (IGZO) as a case study. IGZO is a quaternary oxide semiconductor that has emerged as an alternative to crystalline silicon channels, used in displays and increasingly explored for DRAM. Amorphous IGZO exhibits high electron mobility, ultralow leakage, and a low thermal budget. [4,5] ALD is well-suited for IGZO, due to its sequential, self-limiting surface reactions, enabling precise control of thickness and composition. [6] For multicomponent materials like IGZO, composition is controlled via the supercycle approach, with subcycles of In₂O₃, Ga₂O₃, and ZnO. Their relative ratios and sequence strongly influence the resulting film properties [7], creating a vast, interdependent parameter space. In this work, we develop a protocol for generating AI-ready ALD experimental data, demonstrated on IGZO. The protocol proceeds stepwise from binary to ternary to quaternary processes, with explicit criteria at each stage: binary processes are characterized for saturation and nucleation, and the ternaries (GaZnO, InZnO, InGaO) are screened to identify supercycle criteria. Using these findings, we explore the supercycle parameter space, focusing on cycle ratio and mixing, with consistent characterization. We discuss preliminary trends in crystallinity and electrical properties across the explored space, contrasting with conventional optimization-driven experimentation. Finally, we outline how the resulting datasets enable ML-guided exploration of the IGZO supercycle space, paving the way toward AI-assisted ALD process optimization and a richer experimental foundation for community resources such as AtomicLimits.

[1] 10.6100/alddbdatabase

[2] Westermayr et al., Chem. Mater. 2026.

[3] Saddrudin, Poupaki et al., JVSTA 2026.

[4] Song et al., Nat. Electron. 2025.

[5] Okajima et al., IEDM 2025.

[6] Sheng et al., ACS AMI 2019.

[7] Oh et al., Ceram. Int. 2025.

3:15pm **AP1+EL+MS+PS+TF-TuA-5 Atomistic Modeling of Atomic Layer Etching and Deposition: HF/SiO₂ ALE and TMA/Al₂O₃ ALD, Fedor Goumans**, Software for Chemistry & Materials, Netherlands

Atomic layer processes rely on surface reactions that are sequential, self-limiting, and highly sensitive to local chemical structure. Understanding these reactions at the atomistic level is essential for improving atomic layer deposition, atomic layer etching, and area-selective processing, but many key reaction steps occur at buried, transient, or chemically heterogeneous interfaces that are difficult to characterize directly. In this presentation, we

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discuss how reactive atomistic simulations can support the interpretation and design of atomic layer processes, with examples from HF-based SiO₂ etching and TMA/Al₂O₃ deposition.

Reactive force fields and machine-learned interatomic potentials provide complementary routes for exploring surface chemistry over length and time scales beyond conventional first-principles molecular dynamics. Using these approaches, one can follow bond breaking and formation, surface hydroxylation, ligand exchange, fluorination, densification, and volatile product formation under process-relevant conditions. For SiO₂ etching, simulations can help identify how HF exposure modifies hydroxylated and strained oxide surfaces, how local bonding environments influence fluorination and material removal, and which surface motifs may control etch initiation. For Al₂O₃ deposition, simulations of TMA reactions with hydroxylated alumina surfaces can reveal the molecular origin of saturation behavior, steric effects, methane elimination, and the evolution of reactive surface sites across ALD cycles.

The broader goal is to connect the relevant calculated parameters such as sticking rate, thermal accommodation coefficients, and atomistic reaction mechanisms with experimentally observable trends in growth, etch rate, selectivity, and film quality. We highlight practical considerations for building reliable reactive models, including training data selection, validation against quantum chemical calculations, and the use of accelerated workflows. These examples illustrate how atomistic modeling can complement experiments by exposing reaction pathways, identifying limiting surface structures, and guiding the development of more predictive models for ALD and ALE process optimization.

4:00pm AP1+EL+MS+PS+TF-TuA-8 Characterizing Directionality, Energy and Spatial Uniformity of Plasma Generated Neutral Beams for Low-Damage Material Processing, *Gonçalo A. Cardoso, Mark J. Kushner*, University of Michigan, Ann Arbor

Ion bombardment onto biased wafers during plasma etching can result in charge buildup within features that can deflect incoming ion trajectories, resulting in feature defects such as bowing or notching, or lead to device damage. UV photons emitted from the plasma might also cause surface defects such as dangling bonds [1]. These issues are exacerbated as device dimensions are scaled down, due to larger electric fields for a given charging potential and increased surface-to-volume ratio. This is particularly the case for the fabrication of 2-dimensional materials consisting of nearly monolayer thickness. Energetic neutral beams have been proposed as an alternative, low-damage and charge-free technique for etching and deposition, and particularly so for 2D materials. Such beams can be produced through ion acceleration and subsequent neutralization, either through charge-exchange collisions (volume neutralization) or by reflection from surfaces (surface neutralization) [2, 3]. The primary technical challenges for neutral beam sources involve optimization of directionality, energy and spatial uniformity of the energetic neutral fluxes striking the substrate.

In this paper, we discuss the results of a computational investigation of a neutral beam source consisting of an inductively coupled plasma separated from the wafer by a radio frequency (RF) biased grid powered at 10 MHz. Typical conditions for this source are pressures of tens of mTorr, gas mixtures containing Ar, O₂ and Cl₂ and biases on the grid of hundreds of volts. This source has been simulated using the Hybrid Plasma Equipment Model (HPEM). The Plasma Chemistry Monte Carlo Module (PCMCM) in the HPEM tracks the motion of ions (and neutrals) as they are accelerated by electric fields, collide with background gas particles and neutralize by reflecting from the grid. The PCMCM records the resulting neutral energy and angular distributions (NEADs) at the wafer. This work investigates the characteristics of these NEADs and how they are influenced by grid surface roughness (non-specular scattering), inelastic scattering from the grid, gas-phase charge-exchange reactions and pressure gradients across the grid.

This work was supported by the US Department of Energy, Office of Science, Fusion Energy Sciences (FES) and Basic Energy Sciences (BES) as part of the Extreme Lithography & Materials Innovation Center (ELMIC), a Microelectronics Science Research Center (MSRC), through the Plasma-enabled 2D Materials project (DEAC02-09CH11466); and by FES (SC-00274510).

4:15pm AP1+EL+MS+PS+TF-TuA-9 Plasma Etching of Diamond: Molecular Dynamics Studies and Comparison to Experiments, *Louis Hoffenberg*, Princeton University; *Justin Boles*, University of Houston; *Jack Draney*, Princeton University; *Igor Kaganovich*, Princeton Plasma Physics Laboratory; *Vincent Donnelly*, University of Houston; *David Graves*, Princeton University

Nitrogen-vacancy (NV) centers in diamond are promising for multiple applications in quantum information processing and sensing, including in the fields of high-power/high-frequency electronics, quantum computing, and quantum sensing.[1] Diamond NV centers near surfaces can locally detect and measure physical quantities such as magnetic and electric fields with unprecedented sensitivity. However, these devices are currently limited by near-surface (<10 nm) defects that compromise charge stability and spin coherence.[2] Plasma etching of diamond has been widely employed to process and pattern diamond, but there is limited understanding of the mechanisms of plasma etching. In this work, classical molecular dynamics (MD) simulations are used to simulate O₂/Ar plasma etching of diamond, with comparisons to experiment. We explore both conventional reactive ion etching (RIE) and atomic layer etching (ALE) conditions. The experimentally validated MD simulations are used to identify fundamental etch mechanisms. Based on the MD simulations, we propose a parameterized reduced order model of diamond plasma etching that relates etch yield to ion flux, ion energy, and ion composition, as well as neutral O* radical to ion flux ratio.

References:

[1] Janitz et al. "Diamond surface engineering for molecular sensing with nitrogen-vacancy centers." *J. Mat. Chem. C* 10.37 (2022): 13533-13569.

[2] Sangtawesin et al. "Origins of diamond surface noise probed by correlating single-spin measurements with surface spectroscopy." *Phys. Rev. X* 9.3 (2019): 031052.

4:30pm AP1+EL+MS+PS+TF-TuA-10 Predictive Screening of Sustainable Etchants via DFT Thermodynamics, Machine-Learned Vapour Pressures, and Infrared Spectra, *Christopher Pashartis, Philippe Bezaud, Konstantina Philippidou, Geoffrey Pourtois*, IMEC Belgium; *Hideharu Shimizu, Yoshiaki Nakoyama*, NSC, Belgium

Fluorinated etchants are heavily used in semiconductor processes for their high etch rates and material selectivity. Typical etchants such as CF₄ and C₄F₈ have global-warming potentials in the range of 7,400-13,900 over 100 years and atmospheric lifetimes exceeding hundreds of years, raising concern about the by-products of such processes. While abatement downstream is standard practice, it runs continuously and is never fully effective. Therefore, a more impactful path to reducing the climate footprint of fabrication is to identify replacement etchants that are intrinsically more sustainable while remaining competitive in performance.

Experimentally screening new etchants includes sourcing gases, fine-tuning reactor conditions, characterising effluent; it can be very slow and time consuming. We present a predictive screening workflow built on Density Functional Theory (DFT) rather than specialised plasma simulations: DFT-derived Gibbs free energies are used to solve the closed-system equilibrium for each etchant-substrate pair, giving both the predicted etch capability and the by-product distribution as a function of temperature, pressure, and stoichiometry. To translate predicted species into global warming potential, we couple this with two further ingredients: a graph neural network trained on open-source vapour pressure data to determine which by-products are most likely to be gaseous, and DFT-computed infrared spectra to estimate their radiative efficiency. We will present results aggregated across more than ten candidate etchants and three substrates (Si, SiO₂, Si₃N₄), demonstrating how the combined thermodynamics-volatility-impact screen can rank chemistries by their projected greenhouse gas burden.

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Atomic Scale Processing Mini-Symposium

Room 316 - Session AP2+EL+PS+TF-TuA

Advancing X-Ray Photoelectron Spectroscopy to enable Atomic Scale Processing

Moderators: Robert Bruce, IBM Research, T. J. Watson Research Center,
Eric Joseph, IBM T.J. Watson Research Center

4:45pm **AP2+EL+PS+TF-TuA-11 Understanding the Chemical Reaction Mechanisms of Oxide, Nitride and Metal ALD: Insights from Operando X-Ray Photoelectron Spectroscopy, Joachim Schnadt, Lund University, Sweden**
INVITED

Over the past decades, atomic layer deposition (ALD) has emerged as a leading thin-film deposition technique, particularly within semiconductor technology. The reason is that ALD provides excellent thickness control down to the atomic scale, as a result of the use of sequences of self-limiting surface reactions. However, continued device miniaturization imposes more and more stringent demands on process control. Interfaces need to be engineered with extremely high precision, and lateral dimension control grows in importance. This implies that the exact surface chemistry underlying ALD – and this chemistry is both process and surface specific – needs to be understood in true detail. Experimental *operando* techniques provide an avenue towards the monitoring and characterization of the ALD surface chemistry in a time-resolved manner; these techniques can be applied during the deposition and at measurement rates that allow to resolve the time evolution of surface species and surface structure. Ambient pressure X-ray photoelectron spectroscopy (APXPS) is such an *operando* tool. In contrast to conventional X-ray photoelectron spectroscopy, it is carried out at pressures up to the millibar range, i.e. exactly the operational pressure regime of ALD. Time-resolved APXPS provides precise insight into the occurrence and time evolution of surface and subsurface species and their chemical state and provides information that can be used to formulate chemical reaction mechanisms in the different stages of deposition. In my presentation, I will discuss the capabilities of *operando* APXPS for research into ALD, using examples of research into the ALD of metal oxides and nitrides from transition metal amido or metal tetraisopropoxide complexes and water or ammonia. I will also highlight the first APXPS study into Pt metal ALD. Due attention will be given to instrumental aspects, foreseen future developments and a discussion of what can and what cannot be learned from APXPS.

5:15pm **AP2+EL+PS+TF-TuA-13 In vacuo XPS for Studying ALD Processes, Matthias Minjauw, Tippi Verhelle, Jin Li, Jolien Dendooven, Christophe Detavernier, Ghent University, Belgium**
INVITED

X-ray photoelectron spectroscopy (XPS) is a well-established technique for probing near-surface composition and chemistry. When employed as an in vacuo technique—i.e., without intermittent air exposure—it becomes a powerful tool for investigating surface chemistry during atomic layer deposition (ALD) processes. Three case studies will be presented: (1) the well-known TMA-based chemistry for ALD of alumina, (2) metal ALD with a focus on Pt growth (as reported by Li et al., J. Phys. Chem. C 2024, doi:10.1021/acs.jpcc.3c07568), and (3) ALD on lithium foil substrates (as reported by Verhelle et al., J. Mater. Chem. A 2026, doi:10.1039/D6TA00751A).

Atomic Scale Processing Mini-Symposium Room 316 - Session AP+EL+PS+TF-WeM

Thermal and Plasma Enhanced Atomic Layer Etching

Moderators: Eric A. Joseph, IBM Research Division, T.J. Watson Research Center, Erwin Kessels, Eindhoven University of Technology, the Netherlands

8:00am **AP+EL+PS+TF-WeM-1** **Cyclic dielectric etching challenges for next-generation FDSOI logic and quantum devices**, *François Boulard*, CEA-LETI, France; *Ramy Ayoub*, CEA-LETI, France, Lebanon; *Emmanuel Petitprez*, *Stéphane Pocas*, *Nicolas Gauthier*, *Benoit Martin*, *Pierre Brianceau*, *Antoine Ronco*, CEA-LETI, France; *Bernard Pelissier*, LTM-CNRS, France; *Nicolas Posseme*, CEA-LETI, France

INVITED

As logic devices dimensions scale down⁽¹⁾ and quantum gates advance in scalability and environment resilience⁽²⁾, the demand for gentle, selective, and highly controlled fabrication processes has become critical. Atomic-scale processing—particularly cyclic approaches like Atomic Layer Etching (ALE) and quasi-ALE—has emerged as a potential solution. By leveraging fluorocarbon-based chemistries in sequential deposition/removal cycles, these methods have already demonstrated superior performances in terms of SiO₂ to Si₃N₄ selectivity, such as in Self-Aligned Contact (SAC) etching⁽³⁾. While the fundamental mechanisms of these cyclic processes are increasingly well understood⁽⁴⁾, their industrial applicability—especially for advanced integration schemes—still faces challenges in versatility, robustness, and throughput. In this presentation, we will highlight recent advances in adapting cyclic etching strategies to selectively etch dielectric materials (SiO₂, SiON, Si₃N₄) for two cutting-edge applications: Contact multipatterning in next-generation FDSOI CMOS logic devices and intertwined gate patterning for cryogenic FDSOI quantum chips. This study explore the critical role of process parameters, including fluorocarbon precursor selection, multi-step cyclic strategies, substrate temperature and plasma power modulation, and their collective influence on etch per cycle stability, selectivity, uniformity, throughput, and pattern transfer fidelity.

By bridging fundamental insights with integration-driven optimization, this work clears the path to highly scalable, atomic-precision etching—a key enabler for both classical and quantum semiconductor technologies.

The research work presented in this paper was carried out in the framework of the FAMES Pilot Line of the Chips JU, funded by Horizon Europe grant 101182279 and the ANR NextGen project ANR-22-NEXTG001 of the France 2030 initiative. Part of this work, carried out on the Platform for Nanocharacterisation (PFNC), was supported by the “Recherche Technologique de Base” and “France 2030 - ANR-22-PEEL-0014” programs of the French National Research Agency (ANR).

(1)C. Fenouillet-Beranger, et.al., Solid-State Electronics, 2026, 231, 109264, <https://doi.org/10.1016/j.sse.2025.109264>

(2)B. Bertrand, et.al., International Electron Devices Meeting, IEDM 2023, DOI: 10.1109/IEDM45741.2023.10413763

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(4)R.J. Gasvoda, J. Vac. Sci. Technol. A 38, 050803 (2020); <https://doi.org/10.1116/6.0000395>

8:30am **AP+EL+PS+TF-WeM-3** **Atomic Layer Etching of TiN using O₂/Ar Plasma and SF₆/H₂/Ar Plasma Half-Cycles**, *Kazuya Tajima*, Hitachi High-Tech Corporation, Japan; *Andrew P. Kaye*, Colorado School of Mines; *Kazumasa Okuma*, *Hiroaki Ishimura*, Hitachi High-Tech Corporation, Japan; *Sumit Agarwal*, Colorado School of Mines

Titanium nitride (TiN) has become a widely adopted material for both logic and memory devices due to its appropriate work function with high-k gate stacks and excellent barrier properties against fluorine and oxygen diffusion. Atomic layer etching (ALE) of TiN was performed by a combination of surface modification by oxidation during the O₂/Ar plasma half-cycle and release of volatile etch products in the SF₆/H₂/Ar plasma half-cycle. [1,2] For the ALE experiments, we used low-resistivity TiN deposited by atomic layer deposition. In the O₂/Ar plasma half-cycle, TiN was oxidized to TiO_x with O radicals. For the SF₆/H₂/Ar plasma cycle, the SF₆-to-H₂ flow ratio was tuned to minimize continuous etching of TiN. Quadrupole mass spectrometry (QMS) was used to identify conditions that maximize the production of HF in the SF₆/H₂/Ar plasma, which acts as the etchant for surface modified TiO_x layer, and does not spontaneously etch TiN. Using QMS, we also identified the primary reaction products produced in each half-cycle. Our data show that NO was the primary nitrogen-containing

product in the Ar/O₂ plasma half-cycle. TiF₂⁺ and TiF⁺ ions were detected by the QMS in the SF₆/H₂/Ar plasma half-cycle, which most likely originate due to cracking of TiF₄ parent molecules in the ionizer. The likely O-containing product during the SF₆/H₂/Ar plasma half-cycle is H₂O, which could not be detected due to the background signal in the QMS. In addition, we also detected SO⁺ and SO₂⁺ ions during both half-cycles due to the presence of S on the substrate and/or the chamber walls.

The etching process was performed at 200 °C. While the oxidation of TiN was quasi-self-limiting during the O₂/Ar plasma half-cycle, subsequent etching in the SF₆/H₂/Ar plasma half-cycle was not self-limiting. The synergy parameter, S, [3] is an indicator of the ideality of the ALE process. We show that the ALE end point could be determined by the time decay of the reaction products measured by the QMS, and the synergy parameter could be optimized by limiting the duration of the SF₆/H₂/Ar plasma half-cycle. We were able to obtain a synergy parameter of ~82% under optimized conditions.

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8:45am **AP+EL+PS+TF-WeM-4** **Atomic Layer Etch Process for InGaAs Using (CH₄+H₂)/Ar+ Plasma**, *Burak Okur*, *Ryan Walsh*, *J. Russell Renzas*, University of Nevada, Reno

Precise, damage-free etching of InGaAs with Angstrom-scale control is a critical requirement for next-generation III-V semiconductor device fabrication, including gate recess etching in InGaAs HEMTs, which require very precise etching of ~20 nm of InGaAs. Conventional plasma etching is insufficiently precise for this application and introduces subsurface damage and surface roughness, motivating the development of atomic layer etching (ALE) processes that achieve controlled material removal through sequential, self-limiting surface reactions.

In this work, we investigate a plasma-based ALE process for InGaAs using (CH₄+H₂)/Ar⁺ chemistry, consisting of alternating surface modification and removal half-cycles. The expected volatile products are In(CH₃)₃, Ga(CH₃)₃, and AsH₃. The effects of chamber conditioning, process pressure, and Ar flow rate are systematically investigated to achieve self-limiting ALE behavior. Real-time optical emission spectroscopy (OES) measurements are used to monitor plasma species and investigate the role of residual CH-related species in the transition between self-limiting ALE and RIE-like behavior. For a process at 10 mTorr with 200 W ICP power, 5 sccm Ar, a 1 sec dose of 5 sccm CH₄ and 20 sccm H₂, and 5 sec etch time, an ALE window is present from 10-15 W RF bias, with EPC of 0.25 nm/cycle. The etched surfaces are characterized using scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS), atomic force microscopy (AFM), and profilometry to evaluate etch per cycle (EPC), surface morphology, roughness evolution, and compositional uniformity of the InGaAs surface. Synergy is also measured.

9:00am **AP+EL+PS+TF-WeM-5** **O₂/CF₄/Ar+ 3-Step Plasma Atomic Layer Etch Process for Aluminum**, *Ryan Walsh*, *Burak Okur*, *J. Russell Renzas*, University of Nevada, Reno

Aluminum is a light, conductive metal used in an incredible assortment of device fabrication processes, including in superconducting devices, where performance is highly sensitive to material impurities, interface damage, and critical dimensions. Most aluminum reactive ion etch processes use Cl₂ or BCl₃, which have the dual disadvantages of toxicity and post-etch corrosion. Fluorine is not typically used as an etchant for Al films because of the high melting point of AlF₃. It is known, however, that fluorinated AlO_x has a lower sputter threshold than untreated AlO_x[1]. We present a novel 3-step plasma Atomic Layer Etch (ALE) process for Al using O₂, CF₄, and Ar⁺. This room-temperature cyclical process starts by oxidizing Al in O₂ in Step A. Step B consists of a tetrafluoromethane dose to create an aluminum oxyfluoride modified layer. In Step C, this modified layer is removed via argon ion bombardment, using the oxide as an etch stop for the removal of the more easily sputtered oxyfluoride layer.

EPC of 0.08 nm/cycle and synergy of >99% were achieved with 100 W ICP power and 7 W table bias during step C. Etch control is governed by the thickness of the grown oxide and the depth of fluorine diffusion. We also present AFM, SEM, and XPS to show the effect of this process on the Al surface. This process could provide an avenue toward improved performance in aluminum-based superconducting devices.

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9:15am **AP+EL+PS+TF-WeM-6 Directional Atomic Layer Etching of Lithium Niobate for Nonlinear Integrated Nanophotonics**, *Ivy Chen¹*, California Institute of Technology; *Frank Greer*, Jet Propulsion Laboratory (NASA/JPL); *Austin Minnich*, California Institute of Technology

Atomic layer etching (ALE) is of intense interest in microelectronics, photonics, and other fields owing to increasing demands for nanofabrication with nanometer-scale precision, aspect-ratio independent etching, and sub-nanometer surface roughness. Nonlinear integrated nanophotonic devices typically employ complex oxides like lithium niobate for which dry etching processes perform poorly. Fundamental improvements in nanofabrication processes are vital to realize the full potential of nonlinear integrated nanophotonics for on-chip quantum information processing and beyond. Here, we report a directional ALE process for LN consisting of sequential exposures of HBr/BCl₃/Ar plasma for surface modification and Ar plasma for removal. We observe an etch rate of 1.04 ± 0.01 nm/cycle with a synergy of 84.6% at a temperature of 0°C. At higher process temperatures, the HBr chemistry is found to decrease redeposition compared to F- and Cl-based plasmas, which we attribute to the higher vapor pressures of Br-based products. A grating pattern etched entirely by the process (total etch depth of 220 nm) exhibits no aspect-ratio dependent etching (ARDE) down to the smallest tested gap of 150 nm, in contrast to ion milling in which ARDE manifests even at 300 nm gaps for the same etch depth. Additionally, we present results on applying our ALE processes to TFLN devices to obtain higher aspect ratios, smoother sidewalls, and precise etch depths, yielding enhanced performance metrics for coupler gratings, ring resonators, and spontaneous parametric downconverters for on-chip quantum information processing.

9:30am **AP+EL+PS+TF-WeM-7 Native Oxide Removal and Passivation of Niobium for Superconducting Devices**, *Jessica Jones*, Argonne National Laboratory; *Jeffrey Elam*, Argonne National Laboratory, USA

Superconducting materials are foundational to many quantum technologies, yet surface and interfacial disorder remain important barriers to device performance and scalability. In particular, native oxide formation on superconducting metals is believed to contribute to loss and decoherence, creating a need for processing methods that provide precise surface control while maintaining the integrity of the underlying material. Niobium is an attractive superconducting material because of its relatively high critical temperature and broad relevance to quantum circuit platforms. However, complex native oxide formation and near-surface chemical disorder complicate its integration into devices such as Josephson junctions and other superconducting circuit elements. Here, we investigate an atomic-scale surface treatment and passivation approach for niobium thin films aimed at improving control over the metal/oxide interface. Our results show that thermal atomic layer etching can effectively remove native surface oxides from niobium while preserving the underlying metallic surface. Subsequent atomic layer deposition prevents reformation of the native oxide layer on the surface. Together, these findings establish a promising pathway for interface control in superconducting niobium and support the development of improved fabrication strategies for quantum circuit technologies.

9:45am **AP+EL+PS+TF-WeM-8 Thermal Atomic Layer Etching of the Semiconductor Oxide Channel Materials by Sequential Surface Modification and Volatile Release Reactions**, *Muhammad Hamza Ansar*, *Steven George*, University of Colorado Boulder

Indium gallium oxide (IGO) and Indium gallium zinc oxide (IGZO) can be used as channel materials in metal oxide semiconductor thin-film transistors (TFTs). These TFTs have high carrier mobility, low leakage current and a wider bandgap compared with silicon. Etching the channel material to a precise and controlled thickness is important for device performance. Thermal atomic layer etching (ALE) of these metal oxide semiconductors is possible using surface modification and volatile release reactions.

In this study, the thermal ALE was performed with sequential exposures of boron trichloride (BCl₃) and hydrogen fluoride (HF). In situ spectroscopic ellipsometry (SE) measurements on flat films determined etch rates for IGO of 2.1 Å/cycle at 225°C and 3.0 Å/cycle at 255°C. The etch rate for IGZO was 2.5 Å/cycle at 205°C and 5.6 Å/cycle at 255°C. The etch rates were observed to be nearly self-limiting versus the BCl₃ and HF exposures.

Quadrupole mass spectrometry (QMS) investigations identified the volatile products during the BCl₃ and HF exposures on In₂O₃, Ga₂O₃ and ZnO powders. The initial BCl₃ exposure reacted with the In₂O₃, Ga₂O₃ and ZnO powders and produced InCl₃, GaCl₃ and ZnCl₂, respectively. These products

are consistent with conversion to B₂O₃. The next HF exposure then produced BF₃ and H₂O from the spontaneous etching of B₂O₃. The HF then proceeded to yield additional H₂O from the fluorination of the metal oxide. The subsequent BCl₃ exposure first yielded BF₃ from ligand-exchange with the metal fluoride. The BCl₃ exposure then proceeded to produce metal chloride by conversion of the underlying metal oxide. The QMS results for Ga₂O₃ powder are displayed in **Figure 1**.

Additional studies on flat films explored the effect of IGO composition on the etch rate. **Figure 2** shows that the IGO etch rates were 2.8, 3.6, and 4.2 Å/cycle at In:Ga ratios of 1:1, 2:1 and 4:1, respectively at 230 °C. The etch rate then decreased to 0.8 Å/cycle at an In:Ga ratio of 8:1. The thermal ALE was also observed to smooth the initial roughness of the IGZO surface.

11:00am **AP+EL+PS+TF-WeM-13 Isotropic Atomic Layer Etching via Thermal-Cyclic Processing: Broad Material Capabilities with a Focus on Work-Function Metal Films**, *Kazunori Shinoda*, Hitachi High-Tech Corporation, Japan; *Thi-Thuy-Nga Nguyen*, *Shunya Hirai*, Nagoya University, Japan; *Nobuya Miyoshi*, Hitachi, Ltd., Japan; *Masaru Izawa*, Hitachi High-Tech Corporation, Japan; *Kenji Ishikawa*, *Masaru Hori*, Nagoya University, Japan

INVITED

An isotropic atomic layer etching (ALE) process based on thermal-cyclic processing (thermal-cyclic ALE), consisting of repeated plasma exposure and infrared lamp heating, has been developed for advanced semiconductor manufacturing applications. This approach enables highly controllable and selective isotropic material removal through self-limiting surface reactions driven by cyclic thermal activation. A key advantage of this approach is the independent optimization of the adsorption and desorption temperatures, which enables enhanced material selectivity, improved surface smoothness, and sufficient etch per cycle (EPC). To realize this process at a manufacturing scale, a dedicated 300-mm wafer tool, referred to as a Dry Chemical Removal (DCR), has been developed and commercialized. Using this DCR tool, isotropic ALE processes have been developed for a wide range of materials used in semiconductor device fabrication, including oxides, nitrides, carbides, metals, and compound films. Furthermore, surface reaction mechanisms during the ALE steps have been systematically investigated using in-situ X-ray photoelectron spectroscopy (XPS), providing direct insight into adsorption and desorption steps governing the self-limiting etching behavior. These analyses reveal material-dependent reaction pathways and clarify the roles of plasma-induced surface modification and thermally activated removal of the modified surfaces. This invited presentation provides a comprehensive overview of the thermal-cyclic ALE technology, covering its historical development, process fundamentals, tool design, and mechanistic understanding across multiple material systems. In particular, recent progress in the isotropic ALE of work-function metal films, such as TiC and TiN, will be highlighted, with an emphasis on surface reaction mechanisms investigated by in-situ XPS. These materials are of increasing importance for advanced semiconductor devices, where precise thickness and surface control are critical. Process characteristics and surface chemistry insights obtained by in-situ XPS will be discussed.

11:30am **AP+EL+PS+TF-WeM-15 Selective Thermal Chemical Vapor Etching (CVE) and Atomic Layer Etching (ALE) of Ge Versus Si**, *Micah Duffield*, *Marcel Junige*, *Steven George*, University of Colorado at Boulder

The fabrication of gate-all-around nanosheet transistors employs multi-layer stacks of crystalline SiGe and Si. Completely removing sacrificial SiGe and precisely recessing SiGe may be accomplished by chemical vapor etching (CVE) and atomic layer etching (ALE), respectively. Facile Si CVE has been shown previously using anhydrous HF vapor above 150°C. This Si CVE is ceased by co-dosing H₂O+HF. The present work explored selective CVE and ALE pathways for Ge compared with Si using HF only or with oxidants.

Quadrupole mass spectrometry (QMS) measurements identified the volatile etch products during different reactant gas exposures at ramped or isothermal temperatures. Exposing Ge nanopowder to HF during an up-ramp from 25 to 400°C discovered that facile Ge CVE produced volatile GeF₄ at 50-75°C and then turned off due to non-volatile GeF₂ polymer formation. The GeF₂ polymer could then be volatilized at >300°C. Additional experiments at varied ramp rates, a reversed down-ramp, and isothermal HF exposures corroborated this non-volatile GeF₂ polymer formation.

Exposing a mixture of Ge & Si nanopowders to HF during a temperature ramp showed that the GeF₄ etch product in turn etched Si, producing volatile SiF₄ at 75-110°C (**Figure 1a**). Co-dosing H₂O+HF still etched Ge (or GeO₂) and produced volatile GeF₄. However, co-dosing H₂O+HF eliminated the parasitic Si etch pathway via GeF₄ (**Figure 1b**). Introduced H₂O oxidized

Si and formed an ultra-thin chemical oxide. This SiO_2 was not etched by GeF_4 .

Repeatedly exposing GeO_2 nanopowder to HF at 100°C revealed that GeO_2 etched spontaneously without the self-termination observed for Ge. This finding inspired a unique Ge ALE mechanism: A first oxidant exposure may oxidize Ge to form an ultra-thin GeO_2 surface layer. A subsequent HF exposure may volatilize this GeO_2 surface layer but self-terminate on the underlying Ge due to GeF_2 polymer buildup. The following oxidant exposure may remove the GeF_2 polymer and again oxidize Ge.

Exposing Ge nanopowder to a sequence of oxidant and HF exposures at 100 and 150°C , respectively, revealed that volatile GeF_4 was released after each oxidant exposure in a self-terminating fashion. To refresh the Ge surface after HF exposures, H_2O was ineffective, whereas O_2 was effective, and O_3 was even more effective than O_2 (**Figure 2**). To verify these QMS findings, *in situ* spectroscopic ellipsometry (ISE) studies were employed on Ge thin film samples. Ge ALE using an alternating O_2 /HF sequence at 150°C exhibited an etch per cycle of $1.38 \text{ \AA}$, near-ideal ALE synergy of 97%, and selectivity versus Si (**Figure 3**).

11:45am **AP+EL+PS+TF-WeM-16 Controlled SiO_2 Etch Rates Using a HF/ H_2O Liquid-Layer in a Vacuum Environment: Dependence on H_2O and HF Pressures, Samantha Rau**, University of Colorado at Boulder; Antonio Rotondaro, Hanna Paddubrouskaya, Kate Abel, Tokyo Electron America, Inc.; Steven George, University of Colorado at Boulder

Our earlier research demonstrated SiO_2 etching by HF using a H_2O liquid-layer in a vacuum environment. The current work characterized the dependence of SiO_2 etch rate on the H_2O and HF pressures. We have observed a dramatic increase in SiO_2 etch rates at higher H_2O and HF pressures. This behavior suggests that the thickness of the HF/ H_2O liquid-layer may be increasing rapidly at higher pressures.

SiO_2 etch rates may be sensitive to the HF/ H_2O liquid-layer thickness when the thickness is close to the size the hydrated F^- , HF_2^- and SiF_6^{2-} ions that are involved during SiO_2 etching. For example, the size of the hydrated F^- ion is $7 \text{ \AA}$. Lower SiO_2 etch rates may result from incomplete hydration of these ions. SiO_2 etch rates may approach the SiO_2 etch rates observed in solution for thicker liquid-layers.

Investigations were performed to determine the thresholds for HF/ H_2O condensation versus H_2O and HF pressure. Spectroscopic ellipsometry was employed to measure the liquid-layer thickness, the mean squared error (MSE) in the thickness model, and the transmitted light intensity. Condensation was assumed to occur when the increase in liquid-layer thickness, the increase in MSE, and the decrease in light intensity all occurred rapidly at higher H_2O and HF pressures. Figure 1 shows that rapid changes in the liquid-layer thickness, MSE and transmitted light intensity are observed at a total pressure of 22.9 Torr (18 Torr H_2O and 4.9 Torr HF) at 30.4°C .

Experiments were conducted at initial H_2O pressures from 0-35 Torr and increasing HF pressures from 0-40 Torr to determine the threshold H_2O and HF pressures for condensation at 30.4°C . The resulting condensation points were then compared with the measured SiO_2 etch rates at various H_2O and HF pressures in Figure 2. Below the condensation curve in the ultrathin liquid layers, there were lower SiO_2 etch rates ranging from $\sim 1\text{-}100 \text{ \AA}$ per 5 s HF exposure. These lower SiO_2 etch rates may result from incomplete hydration of the F^- , HF_2^- and SiF_6^{2-} ions. Above the condensation line, the measured SiO_2 etch rates of $\sim 100\text{-}1000 \text{ \AA}$ per 5 s HF exposure were comparable with SiO_2 etch rates in bulk HF solutions with HF wt% values of 25-40%.

12:00pm **AP+EL+PS+TF-WeM-17 Thermal Selective Etching of $\text{Si}_{1-x}\text{Ge}_x$ in XeF_2 , Daniel Cho**, Yi Chen, UCLA; John Hoang, Nick Altieri, Ji Zhu, Samantha Tan, Lam Research Corporation; Jane P. Chang, UCLA

In GAAFET fabrication, $\text{Si}_{1-x}\text{Ge}_x$ is etched selectively from Si. Wet, plasma, and thermal chemistries have been investigated to etch $\text{Si}_{1-x}\text{Ge}_x$ with a range of selectivity [1]. Thermal fluorine-based etch processes, including F_2 and XeF_2 , show high selectivity with etch rates exhibiting a volcano-shaped dependence on x [2, 3].

In this study, we report $\text{Si}_{1-x}\text{Ge}_x$ ($x = 0$ to 1) etch by a XeF_2 beam. XeF_2 was sublimed into an expansion chamber to a target pressure, then pulsed into the reaction chamber. In each pulse, ΔP_{XeF_2} was proportional to the XeF_2 dose. *Ex situ* x-ray photoelectron spectroscopy (XPS) identified the chemical bonding states on the $\text{Si}_{1-x}\text{Ge}_x$ surface from XeF_2 , while scanning electron microscopy (SEM) measured the etch depth for etch rate (ER) calculations.

XeF_2 exposure on as-received $\text{Si}_{1-x}\text{Ge}_x$ samples showed no etch, confirmed by XPS and SEM. This was attributed to a native oxide which prevented XeF_2

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from interacting with the underlying $\text{Si}_{1-x}\text{Ge}_x$. Prior to XeF_2 exposure, *in situ* Ar ion sputtering was used to remove the native oxide for all sequential etch conditions.

XPS results of XeF_2 -processed $\text{Si}_{0.75}\text{Ge}_{0.25}$, Si, and Ge identified Ge-F, Si-F, and C-F bonding in the F 1s region at binding energies of 685.0 eV, 687.1 eV, and 689.6 eV, respectively, consistent with literature values [4-6]. Ge-F, Si-F, and C-F made up 20.8%, 37.4%, and 41.8% of the total fluorine content on the XeF_2 -processed $\text{Si}_{0.75}\text{Ge}_{0.25}$. Quantitative analysis of fluorination by *ex situ* XPS was limited by air exposure between etch and measurement.

Synergistic etching, with Ar ions and XeF_2 simultaneously exposed, showed a Si ER of 6 nm/pulse at an ion beam voltage of 200 V and $\Delta P_{\text{XeF}_2} = 0.5$ Torr, showing a higher ER than the sequential Si ER of 0.4 nm/pulse at the same ion beam voltage and ΔP_{XeF_2} .

The peak sequential $\text{Si}_{1-x}\text{Ge}_x$ ERs for the following Ar ion beam voltage and ΔP_{XeF_2} conditions, 100 V and 0.5 Torr, 200 V and 0.5 Torr, and 100 V and 0.2 Torr, were 7.8 nm/pulse, 9.9 nm/pulse, and 4.1 nm/pulse, respectively. For all conditions, ER increased from $x = 0$ to 0.1, followed by a decrease or plateau past $x = 0.15$. Higher pretreatment ion beam voltage and higher ΔP_{XeF_2} both led to an etch rate increase for $x = 0$ to 0.25. The peak $\text{Si}_{1-x}\text{Ge}_x/\text{Si}$ etch selectivity ranged from 20 to 30 under all conditions.

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Atomic Scale Processing Mini-Symposium Room 316 - Session AP+2D+EL+EM+PS+TF-WeA

Atomic Scale Processing to Enable 2D Materials

Moderators: Robert Bruce, IBM Research, T. J. Watson Research Center, Cristina Satriano, University of Catania

2:15pm **AP+2D+EL+EM+PS+TF-WeA-1 Chemistry (and Some Physics) of Atomic Layer Deposition of Two-Dimensional Metal Dichalcogenides, Miika Mattinen**, Aalto University, Finland **INVITED**

Two-dimensional (2D) materials are of interest for a range of applications from microelectronics to sensing to energy storage and production. One of the most prominent group of 2D materials are metal dichalcogenides, some of which are semiconductors, such as the prototypical MoS_2 , while others are (semi)metals such as TiS_2 and TaS_2 .¹ Deposition of 2D materials with desired properties on large, often temperature-sensitive, and complexly shaped substrates is a key challenge for practical applications. Atomic layer deposition (ALD) can fulfill these requirements, but unlocking the full potential of ALD requires understanding of precursor chemistry, nucleation, substrates, and film characteristics, among other factors.^{1,2}

I will discuss chemistries for deposition of a range of 2D metal dichalcogenides including MoS_2 ,³ SnS_2 ,⁴ ReS_2 ,⁵ WS_2 ,⁶ HfS_2 and ZrS_2 ⁷ using thermal ALD. I will cover precursor challenges related to reactivity, thermal stability, and etching reactions. Controlling crystallinity and morphology and continuity at low thicknesses are also key challenges encountered in ALD of 2D materials. To this end, I highlight the role of substrate choice and the use of a two-step SnS_2 process where the deposited amorphous film is crystallized with mild-post deposition annealing.^{4,8,9}

Plasma-enhanced ALD (PEALD) can help overcome some of the challenges encountered in thermal ALD, such as limited reactivity of many precursors. For example, few thermal chemistries are available for direct deposition on low-cost plastic substrates for flexible applications ($T \leq 150^\circ\text{C}$). To this end, I will discuss PEALD of TMDCs including MoS_2 , TiS_2 , and WS_2 at record-low temperatures down to 100°C by controlling plasma chemistry.¹⁰ I will also describe advanced ABC type processes with two plasma steps, where both the plasma chemistry (reactive radicals) and physics (low-energy ions) are used to control film growth and properties.¹¹

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2:45pm **AP+2D+EL+EM+PS+TF-WeA-3 First-Principles Survey of Molybdenum ALD Precursors for MoS_2 Thin Films, Ian Kerby, Ashlee Riedinger, Lan Li**, Boise State University

Transition metal dichalcogenides, such as molybdenum disulfide (MoS_2), are promising candidates for various semiconductor applications. A crucial requirement for these applications is the controlled and conformal deposition of a monolayer film. Chemical vapor deposition (CVD) and atomic layer deposition (ALD) techniques are key to achieving thin films at commercial scale. Towards this end, the reactions of a variety of precursor chemicals with another variety of surfaces need to be understood. A reaction between a surface and a precursor can be difficult to study in-situ and ex-situ, making computational modeling a valuable tool for addressing this challenge. The work presented here is a survey of common and proposed molybdenum precursors for use in atomic layer deposition of MoS_2 films. Our work in modeling nucleation of these precursors includes various surfaces with the primary aim of determining the role of surface morphology and chemistry during deposition. We have conducted thermodynamic surveys using density functional theory (DFT) for spontaneity of proposed reaction pathways for the precursors to interact and nucleate on the surfaces. To generate and account for the inherent variability in the amorphous substrate, we have utilized ab-initio molecular dynamics (AIMD), enabling a more comprehensive exploration of the

complex energy landscape. We have also investigated the formation of reaction residues on surfaces. Our studies lay the groundwork for future kinetic analyses. The stable intermediate states identified along the reaction pathways can be used in nudged elastic band (NEB) calculations to determine the associated energy barriers during nucleation. These insights may further help guide and inform experimental efforts.

3:00pm **AP+2D+EL+EM+PS+TF-WeA-4 Thermal and Plasma-Enhanced ALD of MoS_2 using $\text{MoO}_2(\text{THD})_2$, Ryan Weiland**, University of Michigan, Ann Arbor; Ian Campbell, IMEC; Pierre Morin, Benjamin Groven, IMEC Belgium; Ageeth Bol, University of Michigan, Ann Arbor

Field effect transistors (FETs) are foundational components of modern electronics. For these applications, scalability is as important as device performance. Two-dimensional MoS_2 exhibits a direct band gap and enables high fidelity electrostatic control due to its reduced dimensionality and the absence of dangling-bonds at the surface. MoS_2 is therefore an ideal channel material for future FETs. Atomic layer deposition (ALD) is a promising technique for scalable, conformal, and back-end-of-line compatible MoS_2 deposition. While promising, the electrical performance of ALD grown 2D MoS_2 does not meet expectations when compared to the electrical properties of mechanically exfoliated MoS_2 , due to its polycrystalline nature. Polycrystalline MoS_2 contains grain boundaries that act as scattering centers, trap-rich regions, and structural discontinuities, that degrade its desirable electronic properties. In this work, ALD processes were developed using a novel molybdenum precursor, $\text{MoO}_2(\text{THD})_2$. $\text{MoO}_2(\text{THD})_2$ contains bulky organic ligands, which could impose steric hindrance during surface reactions, thus limiting surface chemisorption density and favoring formation of larger crystal grains.

First, $\text{MoO}_2(\text{THD})_2$ volatility and decomposition behavior were studied by thermogravimetric analysis (TGA). TGA showed sublimation beginning at 120°C with 100% mass loss at 300°C . Next, plasma and thermal ALD processes were developed at 400°C . A mixed $\text{H}_2\text{S}/\text{Ar}$ co-reactant was used for both plasma and thermal ALD. The effects of precursor and co-reactant dosing times, co-reactant ratios, as well as purge conditions were systematically investigated using in situ ellipsometry. The growth per ALD cycle (GPC) was measured to be $0.07 \text{ \AA/cycle}$ for the thermal recipe and $0.15 \text{ \AA/cycle}$ for the plasma process. The resulting films were characterized by X-ray photoelectron spectroscopy (XPS), Raman spectroscopy, and atomic force microscopy (AFM). XPS indicates near-stoichiometric MoS_2 . Raman spectra confirm MoS_2 vibrational modes and film crystallinity for both processes, while AFM reveals smooth, continuous films without out-of-plane features, with an RMS roughness of $\sim 0.5 \text{ nm}$.

Overall, this work establishes $\text{MoO}_2(\text{THD})_2$ -enabled thermal and plasma-enhanced ALD at 400°C as a scalable route to smooth, continuous, and crystalline MoS_2 thin films. These results lay the groundwork for integrating conformal, large-grain MoS_2 channels into back-end-of-line compatible process flows, with ongoing grain-size quantification and electrical testing expected to link microstructural improvements to device performance.

3:15pm **AP+2D+EL+EM+PS+TF-WeA-5 Low Temperature Magnetized Plasmas for Processing of Two-Dimensional Materials, Yevgeny Raites**, Princeton Plasma Physics Laboratory; Stanislav Musikhin, Yerbolat Usenov, Princeton University Plasma Physics Lab; Satya Butler, Saien Xie, Princeton University; Nirbhav Chopra, Princeton University Plasma Physics Lab **INVITED**

Low-temperature magnetized plasmas generated by electron beams or non-thermal electrons offer new opportunities for atomic-scale materials processing relevant to microelectronics and quantum technologies.¹⁻³ Operating at low pressures ($0.1\text{--}10 \text{ mTorr}$), the applied magnetic field enables spatial separation between a high-density plasma generation region ($\sim 10^{11}\text{--}10^{12} \text{ cm}^{-3}$, electron energies $>10 \text{ eV}$) and a peripheral processing region characterized by lower plasma density ($<10^9 \text{ cm}^{-3}$) and electron temperatures ($<1 \text{ eV}$).⁴ Substrates placed in this region are primarily exposed to low-energy ion fluxes ($\lesssim$ a few eV), enabling damageless processing of two-dimensional materials or processing with a controllable defect formation of such materials. However, plasma instabilities can drive anomalous cross-field transport and increase ion fluxes and energies at the surface.⁵ We present experimental results on instability mitigation in ExB plasma systems⁴ and demonstrate improved low-damage processing of graphene and transition metal dichalcogenides. Quantitative defect characterization in plasma-treated monolayered WS_2 films is presented and benchmarked against conventional RF inductively coupled plasma processing. A focus will also be made on the effect of vacuum conditions in the plasma reactor chamber on the defect formation

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(e.g. sulfur vacancies). Applications to hydrogen and nitrogen surface passivation of diamond for quantum devices are also discussed.²

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4:15pm **AP+2D+EL+EM+PS+TF-WeA-9 Impact of Fluorination and Oxygenation Sources on the Thermal Atomic Layer Etching of Crystalline MoS₂**, *Spencer Smith, Steven M. Hues, Elton Graugnard*, Boise State University

Atomic layer etching (ALE) has emerged as a transformative technique for atomic-scale processing of two-dimensional (2D) materials, including molybdenum disulfide (MoS₂), a promising semiconductor due to its high carrier mobility in monolayer form. Precise etching of MoS₂ films offers a pathway for tuning electrical and optical properties through controlled thickness. Previous studies have explored alternative fluorination and oxygenation chemistries for thermal ALE of amorphous MoS₂, including oxygen sources such as H₂O and O₃ and fluorine sources including HF/pyridine and MoF₆. In this work, we investigate the application of these alternative chemistries to the thermal ALE of MOCVD-grown and ALD-grown MoS₂ films. Preliminary work on ALE of crystalline MoS₂ indicates that etching occurs at defects and grain boundaries. This study aims to correlate etching behavior with changes in surface topography and defect density. Etching effectiveness will be characterized by atomic force microscopy, Raman spectroscopy, and photoluminescence spectroscopy. This study seeks to further expand the capabilities of ALE for precise processing of 2D materials relevant to next-generation semiconductor devices.

4:30pm **AP+2D+EL+EM+PS+TF-WeA-10 Atomic Layer Etching of Tungsten Disulfide in O₂/Ar and BCl₃/Ar Remote Plasmas**, *Jeremy J. Mettler*, University of Houston; *Jaehong (Russell) Kwon, David. B. Graves*, Princeton University; *Vincent M. Donnelly*, University of Houston

Transition-metal dichalcogenides (TMDs) are a class of 2D materials consisting of covalently bonded sheets. Multi-layers are held together by cross-plane van der Waals forces. While these materials hold promise in future semiconductor applications, processes for manufacturing using TMDs are in the early stages of development. One challenge is obtaining uniform, monolayer growth of TMD films over large areas. Chemical vapor deposition results in the formation of multilayer islands. Etching of TMD films will also be required, if for no other reason than to thin multilayers to single layers. Atomic layer etching (ALE) is likely to be called upon for etching steps. TMDs are easily damaged by high temperature and energetic species bombardment, making thermal or plasma-assisted etching challenging. We have explored ALE of WS₂, a promising TMD 2D material. A remote plasma supplied reactive radicals. A switchable magnetic field allow low energetic ion bombardment to be turned on or off while having little effect on radical density. ALE of WS₂ was achieved in a two-step process. Vacuum-transfer x-ray photoelectron spectroscopy (XPS) was used to characterize the chemical composition and thickness of the remaining film at various stages in the process. Sulfur-terminated multilayer films of WS₂ were exposed to O atoms downstream of an Ar/O₂ inductively coupled plasma (ICP). Without ion bombardment thermal O atoms removed S from the surface and formed a tungsten oxide layer 1-2 monolayers thick, with a composition of roughly WO₄. These results agree favorably with parallel molecular dynamics simulations. In the presence of low energy ions (~15 eV) at a flux of ~3 x 10¹⁵cm⁻², the etching rate increased several-fold and the W-oxide stoichiometry was not changed. The Ar/O₂ plasma was followed by Ar/BCl₃ plasma to selectively etch the tungsten oxide layer relative to the underlying WS₂. This step required low energy ion bombardment to remove the WO₄ film and expose the next WS₂ layer. A detailed analysis of the film at various stages in the ALE process was carried out by using the high-resolution W(4f), S(2p), B(1s) and Cl(2p) in the film as well as the Si(2p)

from the underlying substrate and O(1s) in the WS₂ film and underlying interfacial native oxide layer on Si. A complete picture of the stoichiometry of the film during ALE was obtained and will be presented.

This material is based upon work supported by the U.S. Department of Energy, Office of Science, Fusion Energy Sciences and Basic Energy Sciences, as part of the Extreme Lithography & Materials Innovation Center (ELMIC), a Microelectronics Science Research Center (MSRC), under contract No. DEAC02-09CH11466.

4:45pm **AP+2D+EL+EM+PS+TF-WeA-11 Investigation of Layer-by-Layer Etching of MoS₂ by Atomic Oxygen and XeF₂**, *Sei-won Chung, Daniel Cho, Jane Chang*, University of California, Los Angeles

As semiconductor devices continuously scale down, the demand for channel materials of field effect transistors (FETs) with high mobility at low dimensions has increased. 2D transition metal dichalcogenides (TMDs) have gained interest due to relatively consistent mobility at 5 nm of body thickness compared to conventional silicon channel. However, non-uniform large area synthesis has been a challenge in integration. To improve device performance, non-uniform adlayers should be removed layer-by-layer to realize a uniform and large-area TMD films.

To investigate layer-by-layer etching of MoS₂, thermochemical analysis suggests thermodynamic feasibility of using oxygen and fluorine chemistries for MoS₂ etching. Individual and synergistic effects of Ar⁺, O radical (produced by a coaxial waveguide microwave source) and XeF₂ (3Torr) were studied in the molecular beam where each species can be independently controlled in flux and energy. The as-received MoS₂ has S/Mo and Si/Mo ratios of 1.6 and 1.0. Neither O nor XeF₂ spontaneously reacted with the as-received MoS₂. Ar⁺ ion beam (200V, 0.5mA, 10s) was used to remove surface contamination prior to etching, resulting S/Mo and Si/Mo ratios of 0.94 and 0.18 respectively. Following in-situ Ar⁺ ion beam cleaning, O radical was shown to oxidize MoS₂ decreasing S/Mo from 0.94 to 0.46, while XeF₂ removes MoS₂ increasing Si/Mo from 0.18 to 7.1. It showed surface fluorination (~3%), corresponding to formation of MoOF_x and CF_x.

Finally, the sequential process consisting of Ar⁺ ion bombardment, O radical introduction, and XeF₂ vapor exposure (0.5T, 10 pulses) was applied which could enable surface activation, self-limiting oxidation and removal of modified layer by forming oxyfluorides. After XeF₂, O atomic composition decreased and S atomic composition increased indicating removal of MoO₃ with surface fluorination (~3%) of MoF_x and MoOF_x. Increasing number of pulses to 20 increases S/Mo from 1.1 to 1.4 while reducing the MoO₃ content. This research reveals the individual and combination effects of Ar⁺ ion beam, O radicals and XeF₂ on MoS₂ etching and potentially applied to investigating mechanism of layer-by-layer etching of MoS₂.

This research is supported by the U.S. DOE, Office of Science, as part of the ELMIC MSRC through the Plasma-enabled 2D Materials project at PPPL, under contract No. DEAC02-09CH11466.

5:00pm **AP+2D+EL+EM+PS+TF-WeA-12 Width Reduction and Edge Smoothing of 2D MoS₂ Nanoribbons by Lateral Thermal Atomic Layer Etching**, *Janine Wyand¹*, University of Colorado at Boulder; *Tara Pena, Eric Pop*, Stanford University; *Steven George*, University of Colorado at Boulder

Atomic layer etching (ALE) of 2D MoS₂ nanoribbons is important for the fabrication of MoS₂ channel transistors. E-beam lithography and conventional dry etching processes face problems when etching MoS₂ films. These disadvantages include delamination for nanoribbon widths < 25 nm and inability to control line edge roughness. ALE methods may bypass these limitations and produce narrower width MoS₂ channels with smoother edges that may have better electrostatic control and on/off switching.

Narrowing the width of 2D MoS₂ nanoribbons requires lateral etching in the 2D plane. Our earlier work on the thermal ALE of 2D MoS₂ showed that sequential exposures of O₃ and SOCl₂ produced lateral etching by either “outside-in” or “inside-out” mechanisms. In this study, the “outside-in” lateral etching of 2D MoS₂ crystalline nanoribbons was utilized to reduce the widths of 2D MoS₂ nanoribbons. O₃ (ozone) was used for MoS₂ oxidation to MoO₃ and SOCl₂ (thionyl chloride) was used for the volatilization of MoO₃ as MoO₂Cl₂. The studies were performed using high quality 2D MoS₂ nanoribbons on silicon coupons. The MoS₂ nanoribbons were prepared from monolayer 2D MoS₂ films using e-beam lithography and dry etching.

¹ TFD James Harper Award Finalist

Wednesday Afternoon, November 11, 2026

The etching of the 2D MoS₂ nanoribbons was examined by atomic force microscopy (AFM) at 150°C. The lateral etching could be quantified by returning to the same nanoribbon after various numbers of etching cycles and measuring the nanoribbon width using AFM. **Figure 1** shows that the width of the nanoribbons decreases progressively versus number of etching cycles. The AFM images indicate that the etching cycles also smooth the nanoribbon line edge roughness.

The AFM images were consistent with lateral etching at the edges of the 2D MoS₂ nanoribbons. The AFM measurements determined that the 2D lateral etching rate was ~3.9 Å per etching cycle at 150°C as displayed in **Figure 2**. This lateral etch rate represents ~1 MoS₂ units removed per etching cycle at the step edge of the 2D MoS₂ crystalline domains. Additional AFM measurements revealed that nanoribbon widths < 25 nm were possible using this thermal ALE process.

Atomic Scale Processing Mini-Symposium Room 316 - Session AP+EL+MS+PS+TF-ThM

Advancing Spectroscopic Ellipsometry and Other in-Situ Techniques to Enable Atomic Scale Processing

Moderators: Marcel Junige, University of Colorado at Boulder, Adrie Mackus, Eindhoven University, Netherlands

8:00am **AP+EL+MS+PS+TF-ThM-1 Atomic Processes in Surface-Limited ALD Growth Studied by Real-Time Spectroscopic Ellipsometry, Eva Schubert, Ufuk Kilic, Yousra Traouli, Mathias Schubert, University of Nebraska - Lincoln** **INVITED**

We employ in-situ spectroscopic ellipsometry (SE) to investigate the growth dynamics of ultrathin transition metal oxide films (ZnO , WO_3 , TiO_2 , and Ga_2O_3) during plasma-enhanced atomic layer deposition (PE-ALD). To analyze the dynamic optical response, we introduce a dual-box regression model in which the first box represents surface roughness using an effective medium approximation (EMA), while the second box captures cyclic variations in subsurface layer thickness associated with molecular rearrangements occurring during each ALD cycle. This approach provides a time-resolved and quantitative description of both surface roughness evolution and subsurface film growth dynamics, enabling accurate characterization of layer-by-layer deposition processes.

Accurate extraction of film thickness, density, and roughness at elevated substrate temperatures requires precise knowledge of the temperature-dependent dielectric function (TDF), which can differ substantially from room-temperature values. In this work, the TDFs of the transition metal oxides were determined through multi-sample analysis using optical data obtained from films of varying thicknesses measured at different stages of the growth process. The incorporation of experimentally verified dielectric functions enables the dual-box model to reliably describe the evolving optical response during high-temperature deposition, allowing detailed monitoring of sub-monolayer coverage, interface formation, and roughness evolution throughout the ALD process.

Post-deposition structural and chemical characterization using scanning electron microscopy (SEM), atomic force microscopy (AFM), X-ray diffraction (XRD), and X-ray photoelectron spectroscopy (XPS) corroborates the in-situ optical measurements and provides complementary insight into film crystallinity, morphology, and composition.

The capability of in-situ ellipsometry as a powerful tool for investigating growth dynamics will also be demonstrated for multilayer fabrication, ultra-precise thickness control, and monitoring of phase transitions in materials synthesized using physical and chemical vapor deposition techniques.

8:30am **AP+EL+MS+PS+TF-ThM-3 Tracking Critical Points: In Situ Ellipsometry for Rapid Assessment of Atomic Layer Processes on III-V Interfaces, John Murphy, Glenn Jernigan, Michael Johnson, David Boris, Scott Walton, Jill Nolde, U.S. Naval Research Laboratory**

Plasma-enhanced atomic layer processing (PE-ALD, ALE) on semiconductor interfaces, especially III-V semiconductor interfaces, requires precise control of plasma-surface interactions to achieve oxide-free interfaces and low-defect dielectric interfaces. Conventional post-process characterization (XPS, AFM, CV profiling) is time-consuming and inefficient for navigating large, nonlinear plasma process parameter spaces. In this work, we demonstrate an *in situ* spectroscopic ellipsometry (SE) methodology that provides a real-time, model-light assessment of III-V surface quality by tracking high-energy critical-point amplitudes directly from the pseudodielectric function, without constructing full multilayer optical models. Notably, critical points in the 4–5 eV range have photon penetration depths of ≈ 10 nm in III-V semiconductors, making them highly sensitive to near-surface chemistry.

This methodology is illustrated on InAs (100) surfaces during remote Ar/H₂ plasma treatments, aimed at reducing native oxides. In the case of InAs (100) surfaces the E_0' critical point (≈ 4.4 eV) is used as the primary surface-quality metric during plasma treatments. While second-derivative analysis of the directly measured pseudodielectric function provides temperature dependent energy positions for critical points and the magnitude of which are calibrated using chemically clean reference surfaces.

Variations in the normalized E_0' amplitude are monitored as function of various substrate temperatures (100–300 °C), plasma powers (100–500 W), and H₂ flow fractions (1.25–6.25%) to capture the interplay between oxide removal, metallic species formation, and plasma-induced disorder with

greater sensitivity and immediacy than conventional XPS metrics. These variations can be used to rapidly define practical processing windows and enable efficient down-selection within large plasma parameter spaces. The results highlight a generally useful strategy for integrating in situ critical-point tracking into atomic layer processing of III-V materials and suggest straightforward extensions to other compound semiconductor and dielectric growth environments.

8:45am **AP+EL+MS+PS+TF-ThM-4 Intelligent Sustainable ALD Research Platform Integrating On-Demand Precursor Delivery with *in situ* Imaging Ellipsometry, Takeshi Momose, Hiroshi Nishizato, Kumamoto University, Japan; Yugo Nakaya, HORIBA STEC, Co., Ltd. / Kumamoto University, Japan; Kanta Ishida, Shota Oda, Kinichi Nasu, Kumamoto University, Japan; Lianhua Jin, University of Yamanashi, Japan; Hiroshi Okajima, Kumamoto University, Japan**

The development of efficient and sustainable processes in atomic layer deposition (ALD) is becoming increasingly important for the fabrication of next-generation three-dimensional (3D) semiconductor devices. However, conventional run/vent precursor delivery systems are inherently inefficient, with precursor dosing durations reported to be only 1–10% of the total cycle time, resulting in over 90% of the precursor being discarded without contributing to film growth. Furthermore, the development of the ALD process, especially the conformality of high-aspect-ratio (HAR) features, typically relies on time-consuming offline characterizations. In this study, we developed an integrated intelligent ALD research platform that combines on-demand precursor delivery with *in situ* imaging ellipsometry for real-time conformality analysis.

The platform integrates two key technologies. First, to achieve stable and precise on-demand precursor delivery, we developed an in-house piezoelectric-valve system. By applying a machine learning (ML) and control engineering approach, we achieved feedforward valve control, thereby successfully compensating for the inherent hysteresis and nonlinear flow responses of the valve. This allowed the achievement of ideal step-wise gas pulses with a settling time of only 60 ms, compared with 2 s in the uncontrolled condition. This precise control significantly enhances the precursor utilization efficiency while maintaining rapid pulse operation. Second, an in-house lateral high-aspect-ratio (LHAR, AR = 300) test structure combined with imaging ellipsometry was employed to quantitatively monitor the temporal evolution of the precursor penetration depth (PD) during ALD. Sample imaging was enabled by installing an Offner optical system on our custom-built ellipsometer, which allowed us to determine the PD formed in a groove on the Si substrate. As this is currently in an offline setting, we plan to install it in an ALD chamber with on-demand precursor delivery for real-time PD monitoring.

The integration of these technologies enables the instantaneous evaluation of the effect of precise changes in the precursor dose and pulse duration on conformality within HAR features. Initial experiments using trimethylaluminum (TMA) demonstrated that PD evolution in LHAR structures could be tracked. The proposed platform concept leads to a foundation for data-driven, low-waste, and rapidly adaptive ALD process development, contributing to reduced precursor consumption, shorter development cycles, and more sustainable atomic-scale manufacturing.

9:00am **AP+EL+MS+PS+TF-ThM-5 Real-Time Insights into Plasma-Based Atomic-Scale Processing via Ellipsometry, Gottlieb Oehrlein, University of Maryland College Park** **INVITED**

Single-wavelength ellipsometry (SWE) has been employed as an attractive diagnostic technique for real-time, non-invasive monitoring of thin-film evolution during semiconductor processing. Characterized by high temporal resolution and sub-angstrom sensitivity, SWE allows for the precise detection of surface modifications without disrupting the plasma environment. These aspects, combined with relative ease of integration into complex process chambers, make SWE a premier tool for tracking atomic-scale processing. Because of its rapid sampling capabilities, SWE is an invaluable tool for probing the primary kinetics of plasma-surface interactions. We will discuss several case studies where ellipsometric monitoring provides critical insights into complex surface mechanisms, including the achievement of high material etching selectivity for dielectric materials, semiconductors and metals, atomic layer etching (ALE), and the dynamics of electron-beam enhanced remote plasma processing.

While ellipsometry excels at tracking processes including endpoints across diverse materials, optical data alone cannot resolve complex surface chemistry. This talk emphasizes the methodological importance of coupling ellipsometric data with chemical analysis, e.g. X-ray Photoelectron Spectroscopy (XPS) or other materials characterization techniques, to

construct robust, chemically informative surface models. Integrating these complementary diagnostics bridges the gap between optical observations and fundamental chemical mechanisms.

Acknowledgements

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9:30am **AP+EL+MS+PS+TF-ThM-7 Spectroscopic Ellipsometry for Screening Chemical and Microstructural Properties of MoS₂ Thin Films Grown by Plasma Enhanced ALD**, *Douglas Heine*, University of Michigan, Ann Arbor; *Paula Arellano*, University of Michigan; *Ageeth Bol*, University of Michigan, Ann Arbor

The two-dimensional layered semiconductor MoS₂ has attracted significant interest for applications in next-generation electronics, photonics and electrocatalysis due to its favorable electronic and optical properties. Plasma-enhanced atomic layer deposition (PEALD) provides a scalable route for the growth of MoS₂ thin films, offering excellent thickness control, conformality, and relatively low deposition temperatures, which are advantageous for integration into device architectures. However, PEALD grown MoS₂ films often exhibit complex microstructures, consisting of small and misaligned grains. Such structural disorder can adversely affect device performance due to the strong optical and electronic anisotropy of MoS₂⁽¹⁾. In this presentation, we report the development of a model that rapidly estimates electronic and microstructural properties of PEALD-grown MoS₂ thin films from ellipsometry data. The influence of misaligned, out-of-plane oriented grains (“fins”) is described using a Bruggeman effective medium approach for intrinsically anisotropic inclusions⁽²⁾, while local structural disorder is incorporated through modest variations in the intrinsic optical constants. The model is applied to a broad set of films grown under different deposition conditions, and the extracted parameters are correlated with independently measured film properties, including composition, crystallinity and roughness. By enabling rapid insight into the film microstructure and electronic properties, this ellipsometry-based analysis facilitates the development of PEALD processes that yield higher quality MoS₂ films.

(1) Toolbox of Advanced Atomic Layer Deposition Processes for Tailoring Large-Area MoS₂ Thin Films at 150 °C. M. Mattinen et al. ACS Applied Materials & Interfaces, 2023 15 (29), 35565-35579.

(2) Bruggeman formalism versus “Bruggeman formalism”: particulate composite materials comprising oriented ellipsoidal particles. T. G. Mackay and A. Lakhtakia. Journal of Nanophotonics, 2012 6 (1), 069501-069501.

9:45am **AP+EL+MS+PS+TF-ThM-8 Monitoring Electron Beam-Assisted Atomic Layer Etching via Real-Time Ellipsometry and in-Situ XPS: Insights on the Early Stages**, *Vennela Vuruputuri*, Department of Physics, and Institute for Research in Electronics and Applied Physics, University of Maryland; *Robert Bruce*, IBM TJ Watson Research Center; *Nathan Marchack*, IBM TJ Watson Center; *Marinus Hopstaken*, IBM TJ Watson Research Center; *Gottlieb Oehrlein*, Department of Material Science and Engineering, and Institute for Research in Electronics and Applied Physics, University of Maryland

Electron beam assisted etching of plasma functionalized materials has emerged as a novel approach to processing damage sensitive materials. This method employs synergistic interactions between low energy electrons and reactive neutral species to enable material removal while mitigating ion bombardment induced damage and achieving higher atomic precision. While this seems to be a promising approach, the influence of electron irradiation on surface chemistry, defect formation and bond scission are not well understood. Clarifying these mechanisms is essential to establishing electron beam-assisted atomic layer etching (EB-ALE) as a viable method for nanoscale fabrication. In this work, we investigate the EB-ALE of chlorinated single crystal silicon (c-Si) surfaces prepared using a remote plasma source with Ar/Cl₂ gas mixtures. The study examines the dependence of surface coverage, material modification and etching behavior on key parameters, including neutral exposure, and flood gun parameters like electron energy and dose delivery. ALE relies on self-limiting surface reactions, and one key question is if the EB-ALE process can maintain a constant etch depth per cycle as surface roughness, defect or byproduct accumulation evolve. The surface evolution is monitored in real time using in-situ ellipsometry, and it is further characterized using X-ray photoelectron spectroscopy (XPS) and atomic force microscopy (AFM) to analyze changes in chemical composition and surface roughness respectively. Together, these measurements will provide a framework to better understand the mechanisms governing EB-

ALE and ensure predictive control over low-damage atomic scale processing.

Acknowledgements

This material is based upon work supported by the National Science Foundation under the award number NSF CMMI – 2350338.

11:00am **AP+EL+MS+PS+TF-ThM-13 Building Intelligence Through in Situ Techniques for Atomic Layer Processes**, *Parag Banerjee*, University of Central Florida

INVITED

Note: This abstract was initially for TF, but I request that the talk be moved to Gary W Rubloff (GWR) Symposium as it is much more topically relevant.

Atomic layer processing, which consists of atomic layer deposition (ALD) and atomic layer etching (ALE), provides the ultimate processing toolbox for dimensional and compositional control of thin films. In this talk, I will first provide a historical perspective of how specifically, *in situ* techniques implemented for atomic layer processes have impacted our understanding of surface physical and chemical mechanisms that impart atomic layer processes their singular deposition and/or etching characteristics. For example, early work using *in situ* techniques such as, quadrupole mass spectrometry (QMS), Fourier transform infrared spectroscopy (FTIR) and quartz crystal microbalance (QCM) laid the foundation for understanding of the popular ALD chemistries of Al₂O₃ and ZnO. Next, I will showcase the use of *in situ* QMS and spectroscopic ellipsometry (SE) to rapidly screen new precursor molecules and deposit materials for a myriad of applications ranging from lasing fibers to passivation contacts for solar cells. The use of tandem *in situ* techniques will be highlighted which allows for unambiguous determination of both chemical and physical aspects of growth mechanisms during atomic layer processes. Last, due to the massive amounts of data obtained during *in situ* monitoring of processes, machine learning (ML) algorithms can now be successfully used to interpret process performance against process parameters, determining end points and establishing process – property causality. In summary, this presentation highlights the evolution of atomic layer diagnostics from foundational surface chemistry to modern, AI-driven process optimization.

11:30am **AP+EL+MS+PS+TF-ThM-15 Real Time Spectroscopic Ellipsometry and Passivation Studies of Ultra-Thin a-Si:H Films for C-Si / a-Si:H Heterojunction Solar Cells**, *Venkanna Kanneboina*, *Prabin Dulal*, *Bishal Bishal Shrestha*, *Madan K. Mainali*, *Balaji Ramanujam*, *Ambalanath Shan*, *Nikolas J. Podraza*, University of Toledo

High passivation quality and excellent electronic properties of ultrathin hydrogenated amorphous silicon (a-Si:H) layers are essential to fabricate high efficiency crystalline silicon (c-Si) / a-Si:H heterojunction intrinsic thin layer (HIT) solar cells. The quality of the surface passivation of both undoped and doped a-Si:H layers deposited on c-Si are studied using minority carrier lifetime (τ_{eff}) measurements. Stacks of undoped a-Si:H / n-type c-Si / undoped a-Si:H, n-type a-Si:H / undoped a-Si:H / n-type c-Si / undoped a-Si:H / n-type a-Si:H, n-type a-Si:H / undoped a-Si:H / n-type c-Si / undoped a-Si:H / p-type a-Si:H are fabricated by radio frequency plasma enhanced chemical vapor deposition (RF-PECVD). Real time spectroscopic ellipsometry (RTSE) is performed to monitor growth during the deposition of the stacks to determine the structural and optical properties of the undoped and n- and p-type doped a-Si:H. To prevent epitaxial growth on the c-Si wafers, the first layer of undoped a-Si:H is deposited at relatively low temperature of 120°C without hydrogen dilution followed by a second layer of higher quality undoped a-Si:H with hydrogen dilution deposited at 160°C. The influence of additional hydrogen plasma treatment (HPT) on the passivation quality of a-Si:H layers on c-Si is studied by subjecting the undoped a-Si:H on both sides of the c-Si wafers to a hydrogen plasma. Passivation studies are performed on stacks of undoped a-Si:H deposited on both sides of c-Si, and then with an additional stack of doped a-Si:H. The symmetrically deposited bilayers of undoped a-Si:H effectively passivated the c-Si. The best $\tau_{\text{eff}} = 11.61$ ms is obtained with HPT on undoped a-Si:H bilayers for 30 seconds at 200°C. The sample is exposed to atmosphere for about 30 min before it is loading into PECVD chamber and the measured τ_{eff} is 8.25 ms prior to deposition of the n-type a-Si:H. The τ_{eff} is recovered about 3 ms with doped n-type a-Si:H stack, whereas 1 ms is recovered with the n- and p-type a-Si:H stack. The passivation quality of single layer of ~6 nm undoped a-Si:H is also studied with variation of temperature from 120 – 200°C. In this case, the τ_{eff} decreased from 3.85 to 1.6 ms as the temperature increased from 120 – 200°C. High efficiency c-Si / a-Si:H HIT solar cells are fabricated using these optimized, high quality ultrathin a-Si:H layers.

Public Affairs release approval # _____

11:45am **AP+EL+MS+PS+TF-ThM-16 Quantifying Interface Formation in InP/Al₂O₃: ALD Films Probed by XPS and MIS C-V**, *Fabiano Borges*, University of Campinas (Unicamp) and Federal Institute of Education, Science and Technology of São Paulo (IFSP), Brazil; *Cassio Almeida*, Semiconductor Laboratory (LabSem), Center for Telecommunication Studies (CETUC), Pontifical Catholic University of Rio de Janeiro (PUC-Rio), Brazil; *Ângela Albuquerque*, Brazilian Nanotechnology National Laboratory (LNNano), Brazilian Center for Research in Energy and Materials (CNPENM), Brazil; *Gustavo Vieira*, Institute for Advanced Studies (IEAv), Department of Aerospace Science and Technology (DCTA), Brazilian Air Force, Brazil; *José Diniz*, University of Campinas (UNICAMP), Brazil

Abstract

Surface passivation of n-type InP was evaluated by X-ray Photoelectron Spectroscopy (XPS) depth-profile analysis after depositing an Al₂O₃ layer [1] with and without plasma treatments (oxidation (O2), nitriding (N2), oxidation followed by nitriding (O2N2), and nitriding followed by oxidation (N2O2)). To compare samples, we use the atomic fractions of Al2p, In3d, P2p, O1s, N1s, and C1s and two metrics [2]: top-film O/Al ratio at the first depth, with values near 1.5 indicating stoichiometric Al₂O₃; (ii) interface depth, the etch time where the sum of In+P≥20%.

The data show that O2 yields a stoichiometric surface (O/Al = 1.37) and the deepest interface (75s), consistent with a thicker interfacial oxide. N2 presents a very low O/Al (0.12) with detectable N and no interface onset within 120s, indicating a dense nitride/oxy-nitride barrier. N2O2 is intermediate (O/Al=1.42; interface 50s) with no residual N, suggesting oxidation of the nitride. The control sample shows oxidation (O/Al=0.51; interface 45s), while O2+sputtering produces the lowest O/Al (0.14) and the shallowest interface (35s), indicative of a less oxidized/less dense film.

Electrical C-V corroborates the chemical trends: the O2 sample exhibits a median flat-band voltage $V_{fb}=0.00V$ (-0.30V for the control) and a donor density of the same order (2×10^{19} vs $1 \times 10^{19} \text{ cm}^{-3}$), indicating a flatter band alignment without doping degradation. Altogether, O2 delivers the most stoichiometric Al₂O₃, a thicker/interfacial oxide and superior C-V metrics, pointing to improved passivation of InP compared with ALD on untreated surfaces; N2 forms a robust nitride that retards sputter breakthrough; and ALD delivers denser, more stoichiometric Al₂O₃ than sputtering.

References

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[2] A. G. Shard, "A step-by-step guide to X-ray photoelectron spectroscopy (XPS)," *J. Appl. Phys.*, vol. 131, no. 6, p. 061101, 2022.

Acknowledgments

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12:00pm **AP+EL+MS+PS+TF-ThM-17 In Situ Scatterometry Monitoring of Selectivity in 3D Patterns during Atomic-Scale Processing**, *Marcel Junige*, *Steven M. George*, University of Colorado Boulder

Spectroscopic ellipsometry (SE) is a non-destructive and non-invasive technique uniquely suited to monitor thin-film thickness evolution with sub-monolayer precision. While ellipsometry has been employed widely across both research and industry for many years, standard ellipsometry is limited to planar, mainly isotropic thin films. In contrast, 3D structures are inherently anisotropic because of their periodicity in one or more directions. This anisotropy requires utilizing Mueller-matrix (MM) SE. This technique is also known as scatterometry when applied to simple line gratings or periodic 3D structures. Scatterometry has been employed commercially for industrial semiconductor production since ~2003, whereas its employment for research and development of processing technologies is lacking behind.

Most research involving scatterometry has focused on instrumentation and data analysis. MM-SE-based scatterometry has been demonstrated for multi-layered Si| SiGe nanosheet structures. However, a series of these samples had to be prepared and taken out of the reactor at different times to characterize the evolution of selective SiGe etching. Therefore,

scatterometry has been limited primarily to *ex situ* application under static conditions before and after processing.

The work presented herein takes this analysis approach one step further by employing MM-SE-based scatterometry *in situ* to monitor 3D patterns during atomic-scale processing. We utilized line gratings that are commercially available from Chipmetrics as Area Selective Deposition Test Chips. We positioned these line gratings in conical mount to access cross-polarization information in the off-diagonal MM elements. We verified that focusing probes were not needed during MM-SE data acquisition with our iSE (J.A. Woollam Co.) instrument mounted on the vacuum reactor. We combined these *in situ* MM-SE measurements with *ex situ* atomic force microscopy (AFM) imaging before and after processing. We studied selective etching of SiN_x and SiO₂ versus Si non-etch, as well as selective etching of SiN_x versus SiO₂ non-etch. These studies demonstrate a feasible characterization of etch selectivity in 3D patterns.

Verifying selectivity in complex 3D device architectures is necessary after developing atomic-scale processes on individual planar samples. This MM-SE-based scatterometry metrology advancement enables the required *in situ* analysis of selective deposition or etching in 3D test structures. The novel data streams are useful to accelerate lab-to-fab technology transfer toward high-volume manufacturing, and to pave the way for future digital-twin technologies that monitor device production in line.

Atomic Scale Processing Mini-Symposium Room 316 - Session AP+EL+MS+PS+TF-ThA

Advancing Atomic Scale Processing Through Novel Sources and Pulsing Methods

Moderator: Han-Bo-Ram Lee, Incheon National University

2:15pm AP+EL+MS+PS+TF-ThA-1 High-Throughput Silicon ALE via Rapid Matchless Power Modulation, Banks Peete, North Carolina State University; **Jeremy Mettler,** University of Houston; **Paul Melnik,** EHT Semi; **Shreyansh Rastogi, Zuhair Khan,** University of Houston; **Chris Bowman, James Prager, Timothy Ziembra,** EHT Semi; **Steven Shannon,** North Carolina State University; **Vincent Donnelly,** University of Houston

By leveraging rapid, multi-level power modulation, we have expanded the self-limiting window of continuous gas flow atomic layer etching (ALE) of silicon to higher chlorine flow rates enabling much quicker ALE cycle periods. ALE offers superb controllability, but it is limited in speed by the time it takes to purge and switch gases from the chamber. Previously, a continuous flow of argon and chlorine gases were injected into continuously-powered, inductively-coupled plasma (ICP) while the bias power to the substrate stage was pulsed on for 0.2 s and off for 1 s. When the chlorine flow was kept in a narrow window, the silicon chloride surface layer was removed by argon ions faster than the surface can re-chlorinate. At higher chlorine flow rates, this process collapsed into traditional reactive ion etching.

In this work, we expand this “ALE window” by modulating the ICP power in sync with the bias-on period. This was accomplished using a matchless power generator (EHT Semi “Orion”), capable of rapidly changing power levels by an order of magnitude in microseconds. The matchless capabilities ensure that power is still delivered efficiently during the large change in plasma impedance. Time-resolved hairpin probe measurements confirm electron density transition times between power states in the hundreds of microseconds time-scale, far faster than would be possible with a traditional matching network based power delivery system. By operating the ICP at 300W during the bias-off period and at higher powers (600 W to 1.5 kW) during the bias-on period, the ALE window can be expanded to higher chlorine flow rates. During the bias off phase, the higher chlorine flow rates re-chlorinate the surface faster, decreasing the total ALE cycle time. Time-resolved optical emission spectroscopy of the SiCl (280.75 nm) line was used as a real-time monitor of the relative etching rates. At higher chlorine flows, the line exhibited a “spike and decay” profile indicative of ALE, unlike the previous work with constant 300 W power, which showed a square wave-like response to the bias turning on that is associated with simple pulsed reactive ion etching. This confirms that syncing the bias with a higher level ICP power enables higher chlorine flow rates while still outperforming re-chlorination.

2:30pm AP+EL+MS+PS+TF-ThA-2 Etching Commercially-Relevant Materials Using Tailored Waveform Biasing, Benjamin Harris, Daryl White, Kate Stokes, Matthew Loveday, James Ellis, Geoffrey Hassall, Oxford Instruments Plasma Technology, UK

Control of the ion energy distribution function (IEDF) in a plasma is essential for optimising etch processes, such as atomic layer etching (ALE) and reactive ion etching (RIE). If the ion energy is too low, ions will fail to etch the surface of a material. If the ion energy is too high, ions may induce subsurface damage by sputtering material below the surface layer. Between these two boundaries lies the optimal range for ion energies, where ion utilisation is maximised and subsurface damage is minimised. Historically, a radio-frequency, sinusoidal bias is applied to the wafer table to control the IEDF near the wafer surface. This produces bimodal IEDFs with characteristic widths on the order of tens of eV, which is often wider than the optimal window for etching industrially-relevant materials [1]. Tailored waveform biasing (TWB) provides an alternative to radio-frequency biasing that offers enhanced control over the width and mean energy of the IEDF.

In this study, TWB is used to sputter etch commercially-relevant materials. A retarding field energy analyser is used to measure IEDFs, and on-wafer outcomes are investigated with a suite of metrology tools, including atomic force microscopy and scanning electron microscopy. The results are compared with radio-frequency biasing, showing the benefits of TWB for enhanced process control.

[1] Faraz, T., Verstappen, Y.G., Verheijen, M.A., Chittock, N.J., Lopez, J.E., Heijdra, E., van Gennip, W.J., Kessels, W.M. and Mackus, A.J., 2020. Precise

ion energy control with tailored waveform biasing for atomic scale processing. *Journal of Applied Physics*, 128(21).

2:45pm AP+EL+MS+PS+TF-ThA-3 Precise Ion Energy Control Using Advanced Pulse Modulation for Thin Film Deposition and ALD, Yejin Shin, WONIK IPS CO., LTD., Republic of Korea; **Junghoon Kim, Tae Cho,** WONIK IPS CO., LTD.; **Hyun-Joung Woo, YongBaek Jeon, GyuTai Kim, TaeJoon Kim,** WONIK IPS CO., LTD., Republic of Korea

As semiconductor device dimensions continue to shrink, advanced thin film deposition processes increasingly require precise control of ion energy and ion flux to achieve void-free filling in narrow-pitch and high aspect ratio structures. In particular, lateral gap-fill processes for next-generation semiconductor architectures demand highly controlled plasma-surface interactions to improve bottom coverage, suppress seam and void formation, and maintain film uniformity within confined geometries. Conventional RF plasma technology often provides limited flexibility for controlling ion bombardment conditions, motivating the development of advanced waveform engineering approaches.

In this work, an advanced pulse modulation was adapted to precisely control ion energy characteristics during RF plasma deposition processes through tailored voltage waveform. To enable this capability, the bottom components of a system were extensively modified, including the integration of a redesigned junction module and an optimized RF grounding architecture. The modified structure was specifically designed to improve transient voltage response, reduce parasitic effects, and support consistent pulse modulation under plasma load. Particularly, the hybrid junction module was designed to have minimized reflected power fluctuation and enabled more stable waveform delivery to the substrate electrode, which is critical for precise ion energy control during deposition processes. These hardware enhancements enabled more accurate control of sheath voltage evolution and allowed dynamic tuning of ion bombardment energy at the substrate surface. Using the developed platform, various pulse modulation conditions were investigated to evaluate their influence on plasma stability, ion energy distribution, and deposition characteristics. Experimental observations which were made using the advanced PEALD tool manufactured by Wonik IPS expects that advanced pulse shaping can significantly influence ion acceleration dynamics and plasma-surface interactions without substantially perturbing bulk plasma stability. In particular, the ability to manipulate transient sheath behavior enabled improved control of ion-assisted deposition compared with conventional continuous-wave RFs as a 13.56 or 27.1MHz.

This work establishes a hardware and process framework for future investigation of advanced deposition applications, focused on evaluating the effectiveness of this approach for next-generation conformal film deposition. The presented approach provides a promising pathway toward plasma processing for advanced semiconductor manufacturing applications requiring precise control of plasma-surface interactions.

3:00pm AP+EL+MS+PS+TF-ThA-4 Characterizing Inductively Coupled Plasmas in Ar/N₂/H₂ Mixtures for Plasma Enhanced Atomic Layer Deposition of Crystalline Films, David Boris, Jeffrey Woodward, Virginia Wheeler, Michael Johnson, Mackenzie Meyer, Scott Walton, U.S. Naval Research Laboratory

Low temperature plasmas containing mixtures of Argon, Nitrogen, and Hydrogen are widely used in the plasma enhanced atomic layer deposition of crystalline metal nitrides (e.g. AlN) at low temperatures (<500C). Generally, the addition of H₂ is beneficial in that it facilitates the removal of precursor ligands and leads to films with low carbon content (<1%). In addition, if the process conditions are properly chosen, highly crystalline metal-nitride films can be grown in Ar/N₂/H₂ mixtures. However, the effects of H₂ addition on the downstream plasma properties near the substrate are not well understood in remote, inductively coupled plasma (ICP) geometries. As such, a better understanding of the downstream plasma properties in this gas chemistry will be the focus of this presentation.

In this work, we use a combination of Langmuir probes, a retarding field energy analyzer, and optical emission spectroscopy (OES) to examine the effects of varying process parameters on the physical characteristics of Ar/N₂/H₂ plasmas generated in a remote, ICP geometry. In particular, a range of applied RF powers, gas flows, and pressures are explored with a focus on the resulting changes in atomic species density, plasma density, plasma potential, and the energy and flux of ions at the substrate. Of particular interest is the effect H₂ has on the ion flux and ion energy distribution at the substrate. These changes in plasma properties are then tied to changes in the characteristics of AlN thin films grown via plasma-

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enhanced ALD using a remote ICP employing Ar/N₂/H₂ gas mixtures. This work was supported by the NRL Base program through the Office of Naval Research.

3:15pm AP+EL+MS+PS+TF-ThA-5 Direct Atomic Printing of High-K Dielectric Thin Film Oxide Materials, *Tesfalem Welearegay, Nandan Singh Ruhela, Simone Santucci, Mira Baraket, Maksym Plakhotnyuk*, ATLANT 3D Nanosystems, Denmark

The rapid scaling up of CMOS microfabrication combined with growing demand for wide- and ultrawide-bandgap power electronics (GaN, SiC, β -Ga₂O₃), and the integration of high memory and neuromorphic devices have placed high-k dielectric thin films at the centre of modern semiconductor technology. High-k dielectric oxide thin films such as HfO₂, ZrO₂, Al₂O₃, Ta₂O₅, Nb₂O₅, and Ga₂O₃ are predominantly as-deposited by conventional atomic layer deposition (ALD) and spatial ALD techniques. Although ALD offers sub-nanometre control and excellent conformality, its low throughput, vacuum-based batch operation, high precursor consumption, and lack of in-line patterning have become major bottlenecks for the development and scale-up of next-generation high-k materials. These limitations become significant when device roadmap require sub-nm equivalent oxide thickness, and ultralow defect density to fine tune the film's intrinsic properties required for rapid materials screening with high throughput and precision..

We, therefore, present Direct Atomic Layer Processing (DALP[®]) technique — a spatial, atmospheric-pressure ALD technology platform that enables direct atomic printing of oxide thin films with monolayer-level precision and higher throughput while significantly reducing precursor waste. By spatially separating the precursor and oxidant zones under inert-gas exposures, DALP[®] technology combines the self-limiting surface chemistry of ALD with the speed and selective-area capability of a direct-write process. The platform offers unique versatility in fine-tuning the film composition, property and and structure, thereby enabling to control over the targeted properties of the film. The capability is demonstrated on two selected high-k oxide materials. GaOx deposited from its spatial precursor Galai™ (air liquide) with H₂O at relatively low temperatures of 150–240 °C under open atmosphere conditions. The as-deposited oxide films follows an ALD-like deposition window at 180–210 °C with growth per pass (GPP) of 0.35–0.5 Å/pass. Moreover, NbOx (Nb₂O₅) thin films were grown from its spatial precursor Nautilus 2™ (Air liquide) with H₂O at relatively low-temperature ALD window of 180 - 240 °C with GPP of 0.41 - 0.43 Å/pass, producing high-purity amorphous films suitable for different applications including DRAM capacitors, Mott memristors, and high-index optical coatings. These results establish DALP[®] technology as a versatile, sustainable, and high-throughput platform for the rapid development and manufacturing of high-k dielectric thin films for power electronics, memory, and quantum-device applications.

3:30pm AP+EL+MS+PS+TF-ThA-6 Plasma-Enhanced ALD of TaCxN_{1-x} Tailored for Superconducting Quantum Technologies Through Ion Energy and Other Plasma Control Knobs, *Silke Peeters¹, Arthur De Jong, Adrie Mackus, Erwin Kessels*, Eindhoven University of Technology, Netherlands; *Harm Knoops*, Oxford Instruments Plasma Technology, Netherlands

Tantalum nitride compounds are emerging materials for superconducting quantum circuits due to their reduced surface oxidation compared to Ta, which is used in state-of-the-art devices. However, the synthesis of electrically conductive tantalum nitride compounds is challenging. Moreover, these circuits require sharp material interfaces with minimal impurities to achieve state-of-the-art performance.

Ar-H₂ plasma has enabled the preparation of superconducting TaCxN_{1-x} through atomic layer deposition using the metalorganic Ta[N(CH₃)₂]₃[N(CH₃)₃] precursor. In previous work^{1,2}, we have shown that the quality of TaCxN_{1-x} and Nb_xTi_{1-x}N films can be substantially improved through ion-energy control, achieved via application of a radiofrequency (RF, 13.56 MHz) substrate bias during low-pressure, remote plasma exposure. In this work, we explore the novel method of tailored-waveform (TW, 200 kHz) biasing^{3,4} and variation of the Ar-H₂ plasma composition to tune film properties for application in superconducting quantum devices.

Compared to RF biasing, TW biasing offers more precise control over the ion energy. Consistent with earlier RF-bias results, application of TW biasing up to an ion energy of 140 eV enhances grain growth and grain boundary quality through increased adatom mobility and preferential removal of weakly-bonded surface species. As a result, room-temperature electrical resistivity is improved from $(9.6 \pm 0.5) \cdot 10^4 \mu\Omega \text{ cm}$ for an 11 nm film to $(3.8 \pm 0.2) \cdot 10^2 \mu\Omega \text{ cm}$ for a 7 nm film. A decrease of the H₂-dilution in the Ar-H₂

gas mixture supplied to the plasma further enhances TaCxN_{1-x} electrical conductivity and crystallinity, and promotes (111) texturing. A decrease in blistering, which can occur at high-stress, H-containing interfaces, is observed at the Si(100) substrate interface. The balance between Ar and H species significantly affects crystal grain growth. Ar species more effectively stimulate near-surface atomic rearrangement through physical bombardment, while H species act as reducing agents. However, H species can also reduce adatom mobility through over-passivation of dangling bonds and accumulate in the film.

This work shows that accurate understanding and control of plasma properties is necessary to tailor TaCxN_{1-x} films to superconducting quantum technologies. Using the developed toolbox, we demonstrate successful integration of TaCxN_{1-x} thin films in superconducting quantum technologies.

[1]S. A. Peeters et al. *Appl. Phys. Lett.* 123, 132603 (2023)

[2]S. A. Peeters et al. *AVS Quantum Sci.* 7, 026801 (2025)

[3]T. Faraz, A. A. De Jong et al. *J. Vac. Sci. Technol.* A 44, 033004 (2026)

[4]A. A. De Jong, S. A. Peeters et al. *to be published*

3:45pm AP+EL+MS+PS+TF-ThA-7 Direct Atomic Layer Processing (DALP[®]): Spatially Localized, Multi-Material Fabrication for Next-Generation Devices from Discovery to Manufacturing, *Mira Baraket*, ATLANT 3D Nanosystems, Denmark

Progress in next-generation advanced electronic and functional devices, based on complex heterostructures and advanced materials integration, is increasingly constrained by the rigidity of conventional thin-film processing and patterning workflows. While these approaches deliver high material quality and uniformity, they offer limited flexibility for spatially localized, multi-material fabrication, three-dimensional thickness engineering, and rapid experimentation at the nanoscale within a single process flow.

ATLANT 3D introduces Direct Atomic Layer Processing (DALP[®]), a nanofabrication technology enabling digitally controlled, spatially localized deposition of multiple materials with atomic-scale precision. DALP allows different materials to be deposited sequentially and locally in a unified workflow, enabling manufacturing of complex material stacks, heterostructures, interfaces, and thickness gradients without intermediate lithographic patterning steps.

This presentation describes the DALP process architecture and its role in both combinatorial materials discovery and targeted device manufacturing for next-generation devices. By enabling programmable material placement, controlled thickness variation, and repeatable execution within a single platform, DALP supports accelerated materials exploration while also enabling the direct production of device-ready structures as part of broader manufacturing flows. Representative examples include multi-material nanoscale structures for advanced semiconductor and functional material applications, where precise interface control, spatial selectivity, repeatability, and manufacturability are critical. DALP expands the accessible design space of nanoscale fabrication and provides a direct pathway from materials discovery to device-ready, manufacturable structures.

4:00pm AP+EL+MS+PS+TF-ThA-8 From Plasma to Process: Understanding Dielectric Barrier Discharges for Spatial Atomic Layer Deposition, *Ralph Houben, Antoine Salden, Jente Wubs, Richard Engeln, Erwin Kessels, Julian Held, Bart Macco*, Eindhoven University of Technology, The Netherlands

Atomic layer deposition (ALD) is a thin-film deposition technique based on sequential, self-limiting reactions. Plasma-enhanced ALD (PE-ALD) extends this concept by leveraging non-equilibrium plasmas to enable low-temperature processing, wider choice in materials, and enhanced film properties. Advancing PE-ALD requires a detailed understanding of plasma chemistry and the role of reactive species from the plasma in driving surface reactions.

Spatial ALD (SALD), in which precursor and reactant zones are separated in space rather than time, enables high-throughput and large-area processing, making it attractive for low-cost and high volume applications such as photovoltaics and batteries. SALD often operates at atmospheric pressure in linear slit geometries, making dielectric barrier discharges (DBDs) a natural plasma source. However, the high-pressure environment introduces strong collisionality that fundamentally alters plasma chemistry compared to low-pressure PE-ALD. For example, for O₂ plasmas, rapid three-body reactions favor ozone (O₃) formation over atomic oxygen (O). The gas temperature plays a crucial role here, as it directly affects the rate coefficients of the ozone-forming reactions. Furthermore, power dissipation occurs through filamentary microdischarges rather than a uniform glow —

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both aspects that have no direct analogue in conventional low-pressure PEALD.

The oxidative species composition governs surface chemistry in oxide ALD. Quantifying O_3 and O with absolute densities — and linking them to plasma parameters through models — is essential for process control, yet such data for atmospheric DBDs in an ALD context are scarce.

In this work, ozone densities have been determined using broadband UV absorption spectroscopy, yielding absolute concentrations on the order of 10^{15} cm^{-3} under typical operating conditions. The density depends on dissipated power, gas temperature, and O_2 input in the gas mixture. We further present cavity ring-down spectroscopy (CRDS) as technique for probing the forbidden transition of atomic oxygen near 630 nm, providing calibration-free, absolute quantification of atomic oxygen densities. These measurements are supported by plasma chemistry modeling, which shows that the O_2/N_2 ratio governs whether ozone or atomic oxygen dominates — a critical distinction for ALD surface reactivity.

Furthermore, the species generation predicted by the model is directly tied to how power is dissipated in the discharge. Power dissipation analysis reveals the filamentary nature of the discharge and how it governs species generation. These insights are essential for rationally designing plasma-assisted SALD processes.

4:15pm AP+EL+MS+PS+TF-ThA-9 Additive 2D and 3D Micropattern Fabrication with Ruthenium Enabled by Direct-Write Chemical Vapor Deposition, Koichi Tanaka, Argonne National Laboratory, USA; **Eeshan Ketkar, Swarnabha Chattaraj,** University of Chicago; **Samy Kouidri,** university of chicago; **Supratik Guha,** University of Chicago

In the last decade, additive direct-write approaches have been paid considerable attention as alternatives of subtractive, lithography-based processes. Among various direct-write approaches, direct-write chemical vapor deposition (DW-CVD), where precursor gases are directly injected towards the substrate surface through a thin nozzle, has certain advantages to achieve mask-less direct-write materials synthesis and fabrication of monolithic 3D structures. In this talk, we report on the development of DW-CVD system, demonstration of additive 2D and 3D patterning of ruthenium (Ru) and an analytical framework for the localized deposition through thin nozzles.

The DW-CVD system was developed combining a gas delivery system through thin nozzles with tip internal diameters of $2 \sim 30 \mu\text{m}$ and a 3-axis scanning sample stage in a high-vacuum chamber. Localized CVD of metallic Ru dots on Si(100) and TiN- and Au-coated surfaces with diameters of $2 \sim 30 \mu\text{m}$ was achieved using gas mixture of $\text{Ru}(\text{EtCp})_2$ vapor and oxygen (O_2) injected through the nozzles placed $1 \sim 3 \mu\text{m}$ away from the substrate surface at substrate temperatures ranging $360 \sim 420^\circ\text{C}$. By moving the substrate stage in plane, 2D patterns of Ru depositions were fabricated with linewidths of $10 \sim 30 \mu\text{m}$. Furthermore, monolithic, vertical growth of Ru micro-pillars was successfully demonstrated by scanning deposition along the out-of-plane direction.

Theoretically, the resolution of the DW-CVD is affected by multiple factors including the dimensions and geometrical placement of the nozzle tip, and growth temperatures, all of which potentially cause significant changes in materials transport phenomena. To understand the physics of such localized deposition, we developed an analytical framework considering Knudsen's gas flux distribution model and the migration length of molecules on the growing surface. In this talk, we present the highlights of the analytical approach focusing on aluminum (Al) depositions prepared by DW-CVD with dimethylethylaminealane (DMEAA).

Finally, we believe that our studies on the fabrication of 2D and 3D features by additive DW-CVD can contribute to the development of new fabrication techniques in microelectronics.

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Atomic Scale Processing Mini-Symposium Room Ballroom A - Session AP+EL+PS+TF-ThP

Atomic Scale Processing Mini-Symposium Poster Session

AP+EL+PS+TF-ThP-1 Design of Gas Flow Field for a Slip Flow ALD Processing Chamber, *Kyung-Hoon Yoo*, Korea Institute of Industrial Technology, Republic of Korea; *Geun-Soo Song*, The KUMYOUNG Inc., Republic of Korea; *Chun-Sik Kim*, TNG Co., Republic of Korea; *Jun-Young Hwang*, *Sang-Ho Lee*, *Jeong-Jin Kang*, *Shin-Ae Song*, *Kyung-Tae Kang*, Korea Institute of Industrial Technology, Republic of Korea; *Hyeong-Cheol Lee*, Hanyang University, Republic of Korea; *Kun-Hyung Lee*, SAMSUNG DISPLAY, Republic of Korea

As semiconductor feature sizes reach the nanometer scale, ALD processing equipment has become indispensable for atomic-layer control. However, conventional ALD processes face significant challenges regarding economic and environmental feasibility due to the excessive consumption of precursors and energy.^{1,2} To address these issues, it is essential to establish sustainable manufacturing technologies through the development of high-efficiency ALD processing chambers. In the present study, we propose a slipflow based ALD chamber designed to optimize the process space and minimize resource waste. The nitrogen flow fields within the chamber were numerically investigated using Computational Fluid Dynamics (CFD) for process gap sizes of 1, 10, and 100 mm at 400 °C. Under an inlet static pressure of 1 Torr and a mass flow rate of 4.233×10^{-5} kg/s, the Knudsen number (Kn) and flow Reynolds number (Re) were evaluated as 0.0137 and 0.822, respectively for the 10 mm gap. These values confirm that the flow resides within the slip flow regime, where rarefaction effects at the surface wall become significant. Accordingly, the steady-state compressible laminar flow field was simulated by solving the continuity, momentum, and energy equations.^{3,4}

Acknowledgment

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AP+EL+PS+TF-ThP-2 Nucleation and Evolution of Dislocations and Extended Faults in Complex Perovskite Oxides, *Rishi Raj*, *K. Andre Mkhoyan*, University of Minnesota, USA

The stoichiometric flexibility of a perovskite oxide (ABO₃) results in several unique properties due to the presence of three elements in the crystal structure. This allows it to host a variety of defects across all dimensions. 1D primitive edge dislocations with varying terminations, screw dislocations as well as 2D structural faults like Ruddlesden-Popper, elastic distortions, and BO₃ octahedral tilts are commonly observed and studied. Even though structure and electronic properties of some of these defects have been reported, the nucleation and growth of such defects and dislocations is still unclear. Analytical STEM study of perovskite oxide thin films of BaSnO₃ and SrSnO₃ show the existence of two kinds of edge dislocations: single ([001]/(100)-type) and dissociated ([001]/(110)-type) edge dislocation each with two half planes missing. Six different types of such dislocations are possible and observed due to the terminations of the perovskite half planes. Similarly, 2D Ruddlesden-Popper faults are also observed to result from missing planes of atoms, suggesting a correlation with 1D dislocations. Apart from such conventional topological defects, complex perovskite oxides also showcase several line defects and dislocations with reconstructed cores. This suggests we can selectively populate the material with defects by altering the terminations of crystal half planes. Strain tuning during growth by swapping substrates or decorating the surface morphology can effectively allow termination control. This would allow certain defect and dislocation structures to be more favorable than others. By establishing this correlation between 1D and 2D faults, this work

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provides a potential pathway to transition from passive extended defect management to intentional, termination-controlled defect engineering.

AP+EL+PS+TF-ThP-4 Low Temperature Atomic Layer Deposition of ZnO for Optoelectronic Applications, *Anil Yadav*, *Aditya Sadhanala*, Indian Institute of Science, India

This work demonstrates the ultra-low-temperature atomic layer deposition (ALD) of ZnO thin films at 50 °C and 70 °C using a 1 M diethylzinc (DEZ) solution in hexane and water as precursors. This was achieved in a custom-built ALD chamber with precise control over the process parameters. Despite the ultra-low deposition temperatures, the resulting ZnO films demonstrated excellent uniformity. This approach not only solves the problem of substrate compatibility by enabling ultra-low-temperature growth but also demonstrates the use of solvent-diluted pure precursors. The X-ray diffraction (XRD) patterns of as-deposited ZnO films deposited at 50 °C and 70 °C as a function of the number of ALD cycles from 50 to 250 were measured. As the ALD deposition cycles increased to 100, distinct diffraction peaks corresponding to (100), (002), and (101) planes emerged. When the deposition temperature increased from 50 °C to 70 °C, the (002) peak became more intense and sharper, indicating improved crystallinity.

To evaluate the electronic properties of ZnO thin films deposited at ultra-low temperatures of 50 °C and 70 °C, we integrated them as the active layer in field-effect transistors (FETs). The fabricated devices demonstrated exceptional electrical characteristics, exhibiting an I_{ON}/I_{OFF} ratio exceeding 10⁸ with the saturation mobility of 0.85 cm²/V.s. These results clearly confirm the applicability of solution-based precursors for ultra-low-temperature ALD depositions in electronic device applications.

AP+EL+PS+TF-ThP-5 Oxidation-Assisted SF₆ Remote Plasma Etching for Surface Roughness Reduction of TiN, *Min Kyun Sohn*, *Subin Heo*, *Seong Hyun Lee*, *Sun Kyu Jung*, *Jeong Woo Park*, *Dongwoo Suh*, Electronics and Telecommunications Research Institute, Republic of Korea

Titanium nitride (TiN) is widely used as a conductive diffusion barrier and work-function metal in advanced semiconductor devices. As device dimensions continue to scale down, controlling not only the etch amount but also the post-etch surface morphology becomes increasingly important, because nanoscale roughness can directly affect electrical reliability, interface quality, and subsequent film deposition. In the present study, we investigate the effect of SF₆ remote plasma-based etching on the surface morphology of TiN thin films, with particular emphasis on the role of a pre-oxidation step.

Three TiN surface conditions were compared: as-deposited TiN, TiN exposed to SF₆ radicals only, and oxidized TiN followed by SF₆ radical exposure. The etch per cycle (EPC) increased from 0.16 Å/cycle for the SF₆-only process to 0.38 Å/cycle for the oxidation-assisted SF₆ radical process, indicating that surface oxidation enhanced the fluorine-radical-driven removal of TiN. Atomic force microscopy measurements showed that the as-deposited TiN exhibited an RMS roughness of 1.313 nm, which modestly decreased to 1.117 nm after direct SF₆ radical exposure. In contrast, when the TiN surface was oxidized prior to SF₆ radical exposure, the RMS roughness further decreased to 0.850 nm, corresponding to an approximately 35% reduction compared with the as-deposited TiN surface, demonstrating a clear surface-smoothing behavior.

This difference suggests that the oxidation step plays a critical role in modifying the TiN surface before fluorine-radical exposure. The smoothing behavior is attributed to the formation of a TiO_x or TiO_xN_y surface layer during oxidation, followed by its conversion into volatile TiF_x species during SF₆ radical exposure. The higher EPC and lower RMS roughness obtained after oxidation-assisted SF₆ exposure suggest that the modified surface layer can be removed more efficiently and uniformly than the native TiN surface. This oxidation-assisted pathway provides a chemically controlled route for reducing TiN surface roughness without relying on ion-driven physical sputtering.

These results indicate that surface pre-oxidation can be an effective strategy for controlling the morphology of TiN during radical-based etching. The proposed oxidation-assisted SF₆ radical process offers a potential strategy for morphology-controlled TiN etching in nanoscale device integration.

AP+EL+PS+TF-ThP-6 Development of in-Situ Laser Diagnostics Measurements of CH₃ Radical Concentrations for Study of Thin Film Depositions Using Organo-Metallic Precursors, *Mruthunjaya Uddi*, Advanced Cooling Technologies Inc.; *Prawal Agarwal*, *Devon Jensen*, ACT; *Anatoli Morozov*, Princeton University; *Arthur Dogariu*, Texas A&M University

Many environmentally friendly non-halogen precursors contain CH₃ structure component. Plasma chemical vapor deposition of pure metallic thin films using these precursors is challenging and requires better understanding of the gas phase and surface reaction mechanisms. We present a novel, time and spatially resolved, in-situ femtosecond laser diagnostics method to measure CH₃ radicals in a non-equilibrium plasma environment. The CH₃ radicals are predissociated to CH₂ and H radicals using a 275 nm femtosecond laser pulse, followed by a 205 nm probe beam to measure the H radical concentrations. A single femtosecond laser is used to generate the required 275 nm and 205 nm beams. Measuring and knowing the dissociation cross section of CH₃ radical by 275 nm pump beam, allows the quantification of CH₃ radical concentrations with spatial and time resolution.

AP+EL+PS+TF-ThP-7 Hybrid Electrospun ZnO Nanofiber and ALD ZnO Electron Transport Layers for Near-Infrared Organic Photodiodes, *Anna Leonard*, *Neha Chaturvedi*, *Veena Misra*, North Carolina State University

Organic photodiodes (OPDs) require efficient electron transport layers (ETLs) to achieve charge-selective extraction while minimizing recombination and dark current impacts. In this work, hybrid ZnO ETLs consisting of electrospun ZnO nanofibers and atomic layer deposited (ALD) ZnO were investigated for integration into near-infrared OPDs with a PTB7-Th:IEICO-4F active layer. The inclusion of the electrospun ZnO nanofibers increases the interfacial area between the ETL and active layer and provides enhanced electron transport pathways while the ALD ZnO layer provides film continuity and assists in passivating traps to mitigate defect states introduced by the nanofibers.

Three ETL architectures were investigated: ZnO fibers beneath ALD ZnO, ZnO fibers above ALD ZnO, and ALD ZnO control devices. ZnO nanofibers were fabricated through electrospinning of a zinc acetate dihydrate/polyvinylpyrrolidone (PVP) solution followed by calcination to oxidize the fibers and remove excess PVP. Device performance was evaluated through dark current density vs. voltage (J_{dark} -V) and external quantum efficiency (EQE) measurements. Devices utilizing hybrid nanofiber/ALD ZnO ETLs exhibited reduced initial dark current compared to control devices indicating suppression of leakage pathways and trap-assisted recombination. Enhanced EQE response was observed in devices with fibers deposited above the ALD ZnO layer, suggesting improved interface quality and charge extraction.

These results demonstrate that combining electrospun ZnO nanostructures with ALD ZnO provides a promising method to engineer ETLs for high-performance OPDs and other optoelectronic devices.

AP+EL+PS+TF-ThP-8 Site-Specific and Temperature-Dependent Hydration of Faceted Hematite, *Asare Dua*, Illinois Institute of Technology; *Luke Pretzie*, Purdue University; *Ashley Bielinski*, Argonne National Laboratory; *Yue Li*, Argonne National Lab; *Valentine Novosad*, *Cong Liu*, *Alex Martinson*, Argonne National Laboratory; *Adam Hock*, Illinois Institute of Technology

Alpha hematite ($\alpha\text{-Fe}_2\text{O}_3$) is an abundant metal oxide whose surfaces and interfaces control key processes in catalysis, sensing, and photoelectrochemistry. While the more stable $\alpha\text{-Fe}_2\text{O}_3$ (001) surface is well studied, less stable facets such as (012) and (104) which are more relevant in the aforementioned applications due to higher surface activity remain least studied. Surface sites of $\alpha\text{-Fe}_2\text{O}_3$ (012) and (104), as well as their distinct stability were identified through temperature-dependent hydration by combining in situ infrared reflection absorption spectroscopy (IRAS) with density functional theory (DFT) vibrational analysis. For $\alpha\text{-Fe}_2\text{O}_3$ (012), we found sites that promoted the dissociative and molecular adsorption of D₂O. Dissociatively adsorbed D₂O were either terminal or bridging. In both cases the hydroxyls were either isolated or interacting with nearby species. Protons of bridging hydroxyls ($\mu\text{3a-OD}$ (isolated); $\mu\text{3b-OD}$ (interacting with nearby hydroxyls)) are bound to triply coordinated surface oxygens while terminal hydroxyls ($\mu\text{1a-OD}$ (interacting with nearby hydroxyls)) were bound to octahedral surface Fe atoms ($\text{Fe}_{\text{oct}}^{3+}$). Similar to dissociatively adsorbed D₂O, molecularly adsorbed D₂O was either isolated (D_2Oa) or interacting with nearby hydroxyls (D_2Ob). $\alpha\text{-Fe}_2\text{O}_3$ (104) on the other hand exhibited an isolated doubly coordinated bridging hydroxyl ($\mu\text{2a-OD}$) and an interacting bridging species ($\mu\text{2b-OD}$) rather than triply coordinated bridging hydroxyls. This preference is backed by our DFT calculations which

shows triply coordinated bridging hydroxyls were highly unstable on $\alpha\text{-Fe}_2\text{O}_3$ (104). Results from IRAS showed two isolated terminal hydroxyls, $\mu\text{1a-OD}$ and $\mu\text{1b-OD}$, were present on the surface of $\alpha\text{-Fe}_2\text{O}_3$ (104). This indicates two types of undercoordinated surface atoms existed with the first being undercoordinated tetrahedral Fe atoms ($\text{Fe}_{\text{tet}}^{3+}$) since they're the surface atoms of $\alpha\text{-Fe}_2\text{O}_3$ (104), while the second is from the second layer of Fe atoms which have octahedral geometry. The undercoordination of $\text{Fe}_{\text{oct}}^{3+}$ atoms however suggests oxygen vacancies (V_o) were present and some of these might migrate from surface to the second lattice of $\alpha\text{-Fe}_2\text{O}_3$ (104). Our temperature dependent studies support this hypothesis since the population of $\mu\text{1a-OD}$, $\mu\text{1b-OD}$, and $\mu\text{2a-OD}$ increased with increasing temperature when under vacuum (0.8 torr). The distinct local environments of the sites on $\alpha\text{-Fe}_2\text{O}_3$ (012) and $\alpha\text{-Fe}_2\text{O}_3$ (104) and changes in properties with respect to temperature revealed through this work provide a fundamental tool that may be used to engineer the surface of alpha hematite through site- or facet-selective atomic layer deposition.

AP+EL+PS+TF-ThP-9 Impact of TiN Metal Gate Deposition Process on Oxygen-Vacancy-Related Defects and Electrical Characteristics of HfO₂-Based Gate Stacks, *Sangho Lee*, *Seonghwan Shin*, *Seong Hyun Lee*, *Gustavo Panama*, *Sun Kyu Jung*, *Minkyun Sohn*, *Min-A Park*, *Dongwoo Suh*, Electronics and Telecommunications Research Institute (ETRI), Republic of Korea

Hafnium-oxide-based high-k/metal gate (HKMG) stacks are a cornerstone of advanced CMOS technology. The electrical quality of the high-k dielectric is determined not only by its intrinsic film properties but also by interfacial reactions and defect formation induced during the overlying metal gate deposition. In particular, the TiN capping process, whether by sputtering or plasma-enhanced atomic layer deposition (ALD), can strongly modulate sub-oxide formation and oxygen-vacancy-related defects in the underlying HfO₂. In this study, we investigate how the TiN deposition method and ALD plasma power affect the chemical and electrical characteristics of HfO₂-based MOS capacitors.

TiN electrodes were formed on HfO₂ (4 nm) / chemical oxide SiO₂ (1 nm) / Si stacks by sputtering (7.2 kW) and by plasma-enhanced ALD at 150, 225, and 300 W, using p+ Si substrates for C-V analysis and n++ Si substrates for leakage-current evaluation. X-ray photoelectron spectroscopy revealed that the fraction of sub-stoichiometric HfO_x (Hf₂O₃-like) states increases with ALD plasma power, which is attributed to surface damage from accelerated Ar ions and highly reactive TiCl₄-derived radicals, indicating an elevated oxygen vacancy concentration in the HfO₂ film.

These chemical trends were directly reflected in the electrical behavior. Leakage conduction was governed by trap-assisted tunneling (TAT), and model fitting yielded trap energy levels consistent with the oxygen vacancy level of HfO₂. The sputtered sample exhibited the highest leakage current and trap density, while the trap density decreased monotonically with decreasing ALD plasma power. Furthermore, the interface trap density extracted by the conductance method (1 kHz–1 MHz) decreased from $\sim 1.8 \times 10^{12} \text{ cm}^{-2}\text{eV}^{-1}$ for the sputtered sample to $\sim 9.4 \times 10^{10} \text{ cm}^{-2}\text{eV}^{-1}$ for the 150 W ALD TiN sample, a reduction of more than 19 times. This suggests that oxygen vacancy formation is not confined to the HfO₂ layer but also couples to the SiO₂ interlayer through oxygen scavenging reactions, degrading the Si/SiO₂ interface quality.

These results demonstrate that gate-stack engineering must extend beyond the dielectric itself, since the metal capping process directly governs the defect landscape of the underlying insulators. The proposed power-controlled ALD TiN deposition offers a damage-minimized metallization route potentially applicable to aggressively scaled gate-all-around (GAA) HKMG technologies.

Acknowledgments This research was supported by the National Research Council of Science & Technology (NST) grant by the Korean government (MSIT) (No. GTL24043-000).

AP+EL+PS+TF-ThP-10 Understanding the Influence of Plasma Surface Interactions using MOS Capacitors, *Emily Frederick*, University of Central Florida; *John Murphy*, *Glenn Jernigan*, *David Boris*, *Scott Walton*, U.S. Naval Research Laboratory

Plasma-surface interactions are fundamental to atomic layer processing, driving the structural and chemical changes that enable the growth of high-quality materials or the removal of functionalized layers. However, the effects of these interactions are not limited to the surface. While the penetration depth of charged and reactive neutral species is minimal, photon penetration can be much greater, and the overall energy deposition can extend well into the material. As films are grown or etched by atomic

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layer processes, and devices continue to shrink, it becomes increasingly critical to understand and quantify the impact and depth of changes plasma exposure has on the compositional and electronic properties of thin films, and as a result, functionality of the resulting structures and devices.

In this work, we discuss the influence of plasma exposure on Al_2O_3 films grown by thermal atomic layer deposition (ALD) on p-type silicon substrates. This is accomplished by “interrupting” the growth of the oxide layer with a brief exposure to an argon plasma before resuming growth. Through varying both the depth and duration of this plasma interrupt, we systematically probe the impact plasma treatments have on the capacitance-voltage (CV) characteristics of metal-oxide-semiconductor (MOS) capacitors. When the plasma interrupt is near the oxide-semiconductor interface, we see a pronounced shift in the flatband voltage and a corresponding increase in hysteresis, indicating an increase in trapped charges and a greater density of mobile charge or defect states in the oxide. X-ray photoelectron spectroscopy (XPS) analysis was also done to reveal chemical changes induced in the Al_2O_3 by plasma exposure, thus linking these electrical signatures to underlying compositional changes.

Atomic Scale Processing Mini-Symposium Room 316 - Session AP1+EL+PS+TF-FrM

Thermal and Plasma-Enhanced ALD

Moderators: Sagar Udyavara, LAM Research, Virginia Wheeler, U.S. Naval Research Laboratory

8:15am AP1+EL+PS+TF-FrM-1 Analyzing Plasma-Enhanced ALD of III-Nitrides via Experimental and Theoretical Studies, Steven Allaby, Heba Saleh, Fatih Bayansal, Necmi Biyikli, University of Connecticut INVITED
Research efforts on low-temperature ($T < 300^\circ\text{C}$) synthesis of crystalline III-nitride thin films using plasma-assisted ALD utilized various reactor configurations featuring different plasma sources. While our early III-nitride growth experiments using quartz-based ICP sources resulted in nanocrystalline/amorphous films with elevated oxygen impurities, shifting to stainless-steel based hollow-cathode plasma sources revealed highly (002) oriented polycrystalline III-nitride films on Si(100) and sapphire substrates. Upon further modification of the hollow-cathode plasma source and reactor chamber design, we have achieved monocrystalline AlN, quasi-epitaxial GaN and (002) oriented InN films on sapphire substrates at a common growth temperature of 200°C .

The films were deposited using conventional metal-alkyl precursors (trimethylaluminum, triethylgallium, trimethylindium) and various nitrogen plasmas ($\text{N}_2/\text{H}_2/\text{Ar}$, N_2/H_2 , N_2/Ar , and N_2 -only) as metal precursor and nitrogen co-reactant, respectively. *In-situ* ellipsometry and optical emission spectroscopy (OES) were employed to monitor the surface ligand-exchange reactions, plasma surface interactions, and formed reaction byproducts in real-time. *Ex-situ* spectroscopic ellipsometry measurements revealed the film thickness variation, growth-per-cycle (GPC) and optical properties of the III-nitride films. When compared to reference films grown on Si(100) substrates, growth-per-cycle (GPC) values obtained for III-nitride films on c-plane sapphire substrates showed a notable increase. Moreover, a significant improvement of crystalline order has been observed for all binary nitride films grown on sapphire substrates, achieving or approaching epitaxial quality films. We attribute this significant improvement in crystal quality to the synergistic impact of customized HCP-ALD reactor, large-diameter third-generation hollow-cathode plasma source, and optimized growth conditions (plasma gas mixture, rf-power, pressure).

To gain further insight into how different plasma mixtures and radical species affect III-nitride film quality, we have carried out density functional theory (DFT) based simulations of both metal-organic and plasma half cycles. The computational studies revealed how different adsorbed metal-organic precursor surface groups interact with hydrogen and nitrogen radicals by depicting energetically favorable reaction routes. These results are compared and correlated with the experimental observations, deciphering the critical role of hydrogen radicals.

8:45am AP1+EL+PS+TF-FrM-3 Optical Bandgap and Thermal Stability Driven High- κ Engineering of HfAlO Dielectrics on a 200 mm ALD Platform, Partha Mukhopadhyay, Zuriel Caribe, Ivan Fletcher, Jim Fulford, Tokyo Electron America, USA

This work demonstrates the tunable high- κ , optical, and thermal stability characteristics of hafnium aluminate ($\text{Hf}_x\text{Al}_{1-x}\text{O}$) thin films synthesized using a 200 mm high-volume batch atomic layer deposition (ALD) platform. Precise monolayer-level engineering of $\text{HfO}_2/\text{Al}_2\text{O}_3$ superlattice structures enables controlled Al incorporation, which systematically modulates the optical bandgap, dielectric constant, and electrical structure of the alloy. A key outcome is that increasing Al content systematically increases the optical bandgap of the alloy from 5.5 – 6.5 eV, accompanied by improved film density and structural uniformity, which directly influences the dielectric response and reliability of the material.

At lower Al content (~ 0.31), the alloy exhibits superior dielectric properties with $\kappa > 22$ and a capacitance density of $12.46 \text{ fF}/\mu\text{m}^2$, representing $\sim 29\%$ enhancement over HfO_2 , attributed to suppressed oxygen vacancies and reduced trap density. With increasing Al composition, the widened bandgap increases barrier height, resulting in reduced leakage current and improved dielectric robustness. A higher Al fraction (~ 0.56) demonstrates a breakdown electric field of $\sim 8 \text{ MV}/\text{cm}$, exceeding HfO_2 by over $3 \text{ MV}/\text{cm}$, while maintaining low leakage ($\sim 10^{-6} \text{ A}/\text{cm}^2$) even after 650°C annealing. Notably, increasing Al content significantly enhances the thermal budget of the dielectric stack, stabilizing the amorphous phase and preventing dielectric degradation at elevated temperatures, while preserving relatively high κ values compared to pure Al_2O_3 .

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XPS analysis reveals systematic shifts of Hf4f and Al2p peaks toward higher binding energies with increasing Al concentration, indicating modified bonding characteristics and enhanced ionic contributions, consistent with bandgap widening and improved film stability. These electronic structure changes correlate with reduced surface charge transfer effects and improved dielectric integrity. Additionally, the films exhibit excellent capacitance linearity over applied voltage and stable frequency response, making them highly suitable for RF applications.

The 200 mm batch ALD platform ensures excellent uniformity, repeatability, and scalability for compositionally engineered dielectrics. Overall, this study highlights that Al-content-driven optical bandgap tuning, coupled with improved thermal stability and enhanced breakdown strength, provides a powerful route to customize high- κ behavior in HfAlO. This enables optimized performance trade-offs between capacitance density, leakage, and reliability, making HfAlO a compelling material system for advanced MIM capacitors in BEOL, RF, and memory technologies.

9:00am AP1+EL+PS+TF-FrM-4 Atomic Layer Deposition Metal and Barrier Layers Enabling Next Generation Interconnects, Matthew Weimer, Sara Harris, Forge Nano; Thomas Moffat, NIST-Gaithersburg; Dane Lindblad, Forge Nano; Daniel Josell, NIST-Gaithersburg; Arrelaine Dameron, Forge Nano

Device miniaturization continues to push the technological boundaries of manufacturing processes and integrated circuit (IC) manufacturing must keep pace to meet the design requirements for next-gen transistors. Back end of line (BOEL) fabrication poses several challenges to chip scaling: most notoriously the copper bottleneck in which thick barrier layers and resistance capacitance (RC) delays limit functional interconnect pitch to 21 nanometers [1]. To overcome this critical barrier, low resistivity, conformal metal films have been studied for hybrid metallization; decreasing interconnect resistance and reducing barrier layer thicknesses. As interconnect pitch decreases line-of-sight PVD copper barrier/seed layers experience pinch off and void formation [2]. This work explores the use of ruthenium (Ru) and lower resistivity thermal ALD iridium (Ir) thin films for copper seed layers to enable next generation interconnects. Thermal ALD Ir and Ru deposited at 250°C both demonstrate conformal deposition on 10:1 aspect ratio through glass vias (TGVs) and show void free copper fill using a cyclic pulsed electrochemical deposition process. Deposited in partnership with ALD SiO_2 and TiN layers, this completes a metal barrier seed solution for TSVs. As expected, the primary difference between the Ir and Ru is electrical resistivity. Seed film resistivity as deposited on TGVs was measured using four-point probe; a 10 nm Ru film measured $41 \mu\Omega \cdot \text{cm}$ and a 10 nm Ir film measured $16 \mu\Omega \cdot \text{cm}$. Successful copper electrochemical deposition (ECD) at various ECD potentials, was completed with 10 nm of Ir with (resistivity $16 \mu\Omega \cdot \text{cm}$) and 20 nm of Ru (resistivity $22 \mu\Omega \cdot \text{cm}$) with the full layer stack for these film shown in Figure 1. Reduction in required layer thickness combined with improved electrical properties and demonstrated conformality can serve as crucial steps forward for advanced interconnects and BEOL architecture. Additionally, this work compares Ir film quality as deposited at 250°C and 300°C . Ir deposited at 300°C exhibits improved environmental stability when compared to 250°C Ir as measured with 4-point probe after aging in atmosphere over several months. 300°C Ir also shows a shortened nucleation delay, and optical constants (n and k) more closely aligned to bulk Ir values, as measured with spectroscopic ellipsometry. Ir film characterization for both temperatures including XPS, XRR, XRD and AFM is ongoing, and will be presented

9:15am AP1+EL+PS+TF-FrM-5 Tuning the Mechanical Properties of Silica Aerogels Using Atomic Layer Deposition, Victor Vogt, Ali Madanchi, Katherine Rybkin, Andrés Miranda Mañón, Andrej Lenert, M.D. Thouless, Neil Dasgupta, University of Michigan

Silica aerogels are a key material used in aerospace, catalysis, and energy applications due to their ultralow density and thermal conductivity as well as high solar transparency. Specifically, they show promise as an emerging transparent insulating material in concentrating solar thermal energy generation.¹ In our recent work we have shown that atomic layer deposition (ALD) can be used to improve the thermal stability of silica aerogels at high temperatures of up to 800°C .^{2,3} This was attributed to the ability of ALD to conformally modify the pore structure of the aerogels and form a more thermally stable phase at the surface. However, silica aerogels also suffer from low strength and fracture toughness, restricting their potential applications and making practical implementation difficult due to the risk of brittle fracture in assembly or operation.

In this study, we utilize sub-nanometer ALD treatments to conformally modify silica aerogels with aspect ratios > 100,000:1. We demonstrate a monotonic increase in stiffness with increasing number of ALD cycles, resulting in a ~30-fold increase in elastic modulus after 3 cycles of Al₂O₃ (nominal ~4 Å thickness). This result is investigated using finite element modeling (FEM) of the aerogel nanostructure, which reveals the key factors which drive the large increase in elastic modulus. Furthermore, we show that ALD increases the fracture toughness of the aerogels, resulting in a ~7-fold increase in fracture toughness after 3 ALD cycles. Due to the ultra-thin and conformal nature of the ALD layer, the porous nanostructure is maintained, allowing the aerogels to maintain high solar transparency and low thermal conductivity. This method enables a pathway to broader applications for silica aerogels through improving their mechanical durability while maintaining the favorable properties of low thermal conductivity and high optical transparency over a range of operating temperatures.

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9:30am AP1+EL+PS+TF-FrM-6 Multilayer FUV Coatings Using ALD-Grown LaF₃ and ZrF₄, John Hennessy, Jet Propulsion Laboratory

We report on the targeted development of atomic layer deposition processes for high-refractive-index metal fluorides, specifically focusing on lanthanum fluoride and zirconium fluoride. The primary objective is to integrate these ALD-deposited materials into multilayer optical coatings designed for demanding far-ultraviolet (FUV) applications. By successfully combining ALD processes for lanthanum and zirconium fluorides with established processes for lower-index materials, we enable the fabrication of specialized optical components that are critical to various NASA applications. As a demonstration of this ALD capability, a 16-layer AlF₃/LaF₃ mirror was fabricated and shown to achieve an average reflectance exceeding 90% within a band centered around 140 nm. Finally, we discuss the prospects for increasing coating complexity and further optimizing FUV performance.

9:45am AP1+EL+PS+TF-FrM-7 Controlling the Crystallinity of V₂O₅ Deposited by Plasma-Enhanced Atomic Layer Deposition, Scott Walton, Peter Litwin, Neeraj Nepal, Maria Sales, David Boris, Michael Johnson, Mackenzie Meyer, Virginia Wheeler, Naval Research Laboratory

Plasma-enhanced atomic layer deposition (PEALD) is a low temperature, conformal, layer-by-layer deposition technique that is based on a pair of self-terminating and self-limiting gas-surface half-reactions, in which at least one half-reaction involves species from a plasma. This approach to ALD generally offers the benefit of substantially reduced growth temperatures and greater flexibility in tailoring the gas-phase chemistry to produce amorphous, crystalline, and epitaxial films of varying types and characteristics. Importantly, by carefully controlling the plasma properties, it has been shown that the degree of crystallinity and phase selection is possible.

In this work, we demonstrate the ability to manage the crystallinity of V₂O₅ through careful control of the plasma characteristics during growth in a Kurt J. Lesker 150 LX PEALD system. We employ both plasma diagnostics and material characterization techniques to understand the process-to-structure-property relationship while varying the input power, operating pressure, and gas flow ratio. Optical emission spectroscopy (OES) and Langmuir probe measurements are used to characterize the production and delivery of energetic and reactive species to the growing film surface, while x-ray photoelectron spectroscopy (XPS) and Raman spectroscopy are used to characterize the physico-chemical properties of the V₂O₅ films. We find that by varying the operating pressure in the system during the plasma process, the films can be selectively deposited in an amorphous or crystalline state. We link the transition from amorphous to crystalline material to the energy flux density delivered to the material surface during deposition and derive an estimate of the critical energy flux density necessary for crystallization. Lastly, we discuss these results more broadly

and the applicability of these findings to other material systems. This work is supported by the Naval Research Laboratory base program.

10:00am AP1+EL+PS+TF-FrM-8 Epitaxial Growth of VN on Si₃N₄(0001)/Si(111) Using Molecular Beam Epitaxy, Nuri Oncel, Mehmet Ozdogan, Carlos Munoz, Deniz Kadir, University of North Dakota; Randy Duma, Quantum Design; Rajendra Dulal, University of North Dakota

We report on the epitaxial growth of VN thin films on β-Si₃N₄ (0001)/Si(111) substrates using molecular beam epitaxy. The growth process and resulting film properties were systematically investigated using in-situ reflection high-energy electron diffraction (RHEED) and ex-situ scanning/transmission electron microscopy (S/TEM), X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS), energy-dispersive spectroscopy (EDS), and electrical transport measurements. RHEED and cross-sectional TEM analyses confirm the formation of epitaxial VN(111) films, with growth proceeding via a Volmer–Weber Island mechanism followed by coalescence. Increasing nitrogen flux during deposition significantly improves film continuity, stoichiometry, and crystalline quality. Structural characterization reveals an azimuthally aligned epitaxial relationship without the expected in-plane rotational reconstruction, indicating that interfacial bonding and strain relaxation dominate over simple lattice-mismatch considerations. XPS and EDS measurements confirm near-stoichiometric VN composition with reduced oxygen incorporation at higher nitrogen flux. Electrical measurements demonstrate ohmic transport across the VN/Si₃N₄/Si heterostructure, suggesting efficient carrier transport through the ultrathin dielectric layer via tunneling or defect-assisted conduction. Low-temperature transport measurements reveal a suppressed superconducting transition temperature relative to bulk and oxide-supported VN films, attributed to compressive strain and structural disorder. These results establish Si₃N₄(0001)/Si(111) as a viable platform for integrating epitaxial VN with semiconductor technology and provide insight into nitride–nitride heteroepitaxy and interface-driven property modulation.

Atomic Scale Processing Mini-Symposium

Room 316 - Session AP2+EL+PS+TF-FrM

Enabling Atomic Scale Processing (ALD/ALE) by leveraging new Precursors and Surface Reactions

Moderators: Austin Minnich, California Institute of Technology, Angel Yanguas-Gil, Argonne National Laboratory

10:30am AP2+EL+PS+TF-FrM-10 Plasma-Enhanced ALD of SiO₂ with an Alternative Precursor: From Mechanistic Insight to Area-Selective Deposition, James Jensen, Jay Swarup, James Engstrom, Cornell University

Silicon dioxide (SiO₂) is and has been an extremely important thin film material in microelectronics and related fields. The great majority of approaches to atomic layer deposition (ALD) of SiO₂ involve the use of plasma since, for example, the Si-H bond is resistant to reaction with molecules such as H₂O and O₂. We have examined the use of an alternative Si precursor, which we refer to as di-isopropylamido trisilylamine [DiPA(TSA)], employing an O₂ plasma as the co-reactant. The precursor contains only Si-N and Si-H bonds. We have investigated the process using *in situ* real time quartz crystal microbalance (QCM) techniques in a custom-built hot-wall reactor, which provides detailed insight into the half-reactions, and the presence of nucleation delay. We find that ALD with DiPA(TSA) occurs over a large temperature window, spanning a range of $T = 120 - 285^\circ\text{C}$. The thickness deposited per cycle decreases with increasing temperature, from 4.7 Å-cycle^{-1} at $T = 120^\circ\text{C}$ to 2.9 Å-cycle^{-1} at $T = 285^\circ\text{C}$. These rather large rates of growth from ALD reflect the silicon content in the precursor (three atoms per molecule). From QCM a nearly complete loss of the di-isopropylamido ligand is implicated in the precursor half reaction. The deposited thin films have been characterized using *ex situ* X-ray photoelectron spectroscopy (XPS), X-ray reflectivity (XRR), spectroscopic ellipsometry (SE) and atomic force microscopy (AFM). From XPS we find that the thin films are near stoichiometric (O:Si ~ 1.9) over the entire range of temperatures, while the expected impurities (e.g., C, N) are < 1%. From XRR we find that the thin film density increases from 2.24 g-cm^{-3} at $T = 120^\circ\text{C}$, to 2.38 g-cm^{-3} at $T = 285^\circ\text{C}$, which can be compared to the values for amorphous silica (2.20), and quartz (2.65). From AFM we find that the thin films are continuous and smooth exhibiting an average roughness of 0.54 nm at $T = 120^\circ\text{C}$ (19 nm thick film), and 0.30 nm at $T = 285^\circ\text{C}$ (13 nm thick film). Finally, we have conducted a preliminary set of studies concerning blocking PE-ALD growth of SiO₂ on Al₂O₃ using acetylacetone

(Hacac) and we find the deposition can be blocked for at least 10 cycles, over the temperature range $T = 120\text{--}285^\circ\text{C}$.

10:45am **AP2+EL+PS+TF-FrM-11 Temperature-Dependent Growth Behavior of Aluminum Oxide Using Dimethylaluminum Isopropoxide and Water**, *Azeez O. Musa*, University of Missouri-Columbia; *Hemant Kumar, Campbell Sweet*, University of Missouri, Columbia; *Justin R. Walensky*, University of Missouri-Columbia; *Matthias J. Young*, University of Missouri, Columbia

Area-selective atomic layer deposition (AS-ALD) requires precursors with controlled surface reactivity to enable selective film growth on targeted regions while minimizing unwanted nucleation on non-growth surfaces. Dimethylaluminum isopropoxide (DMAI) has emerged as a promising alternative to trimethylaluminum (TMA) for AS-ALD applications due to its reduced reactivity and non-pyrophoric nature. However, experimental mechanistic studies of DMAI/ H_2O ALD and the influence of deposition temperature on growth behavior remain limited. In this work, the temperature-dependent growth characteristics of Al_2O_3 ALD using DMAI and H_2O were investigated using in situ quartz crystal microbalance (QCM) measurements together with ex situ X-ray photoelectron spectroscopy (XPS), Fourier-transform infrared spectroscopy (FTIR), and spectroscopic ellipsometry. DMAI was synthesized from aluminum isopropoxide and trimethylaluminum in 79% yield and >95% purity, with product formation confirmed by ^1H NMR spectroscopy. Real-time QCM measurements were used to examine precursor adsorption, ligand removal behavior, saturation conditions, and mass gain per cycle (MGPC) between 100 and 200 $^\circ\text{C}$. Al_2O_3 growth at 100 $^\circ\text{C}$ exhibited a lower MGPC ($\sim 19\text{ ng cm}^{-2}\text{ cycle}^{-1}$) and less stable saturation behavior compared to films deposited between 150 and 200 $^\circ\text{C}$, where the average MGPC remained approximately constant at $\sim 29\text{ ng cm}^{-2}\text{ cycle}^{-1}$. Single-cycle QCM analysis further revealed distinct differences in surface reaction behavior between low- and intermediate-temperature growth regimes. These findings provide experimental insight into DMAI/ H_2O ALD surface chemistry and establish growth conditions relevant to the development of DMAI-based AS-ALD processes.

11:00am **AP2+EL+PS+TF-FrM-12 Void-free Thermal ALD BaTiO_3 Thin Films Prepared by Removing Water Dose after Ba Precursor**, *Giayi Chen*, Georgia Institute of Technology, China; *Mark Losego*, Georgia Institute of Technology, USA; *Asif Khan*, Georgia Institute of Technology

This talk will discuss our efforts to develop a robust atomic layer deposition process (ALD) to create ferroelectric BaTiO_3 (BTO) thin films using Bis-(1,2,4 triisopropylcyclopentadienyl)-Barium and Titanium Isopropoxide precursors. Ferroelectric materials are potential candidates for future low voltage RAM and NAND memory because of their reversible two polarization states under low external electric field. While the CMOS compatible gate dielectric materials HfO_2 and $\text{Hf}_{0.5}\text{Zr}_{0.5}\text{O}_2$ are ferroelectric, they have high coercive fields that make it difficult to lower switching voltages below 1 V. Therefore, perovskite ferroelectric materials, like BaTiO_3 are desirable to use for these applications because their coercive voltages can be an order of magnitude lower, approaching 0.1 V. However, these ferroelectric films must be deposited by ALD to match the conformality and small thickness requirements desired for RAM and NAND memory. While reports exist for ALD processes of these materials, most if not all those reports use ALD recipe of “A metal precursor – oxidant – B metal precursor – oxidant”. This talk will present our finding that high quality BTO films can still be deposited with recipe in the form of “ $\text{H}_2\text{O} - \text{Ba} - \text{Ti}$ ” or “ $\text{H}_2\text{O} - \text{Ba} - \text{Ba} - \text{Ti}$ ”. A hypothetical ALD chemistry mechanism is proposed. These reactions can happen at 280 $^\circ\text{C}$, but not 220 $^\circ\text{C}$. Inverting the precursor sequence, such that $\text{H}_2\text{O} - \text{Ti} - \text{Ba}$, does not work. XRD confirms the formation of BTO crystal lattices through these novel recipes. One benefit of these recipes is getting void-free BTO films after annealing, whereas our previous studies show that conventional ALD recipes produce BTO films with voids because of the evaporation of H_2O during annealing. 40 nm BTO film deposited with this dehydration recipe shows high dielectric constant of 72 with low dielectric loss at the magnitude of 10^{-3} .

11:15am **AP2+EL+PS+TF-FrM-13 Optimizing Molybdenum Pentachloride Delivery for Vapor Deposition Processes**, *James Maslar, Vladimir Khromchenko, Berc Kalanyan*, NIST-Gaithersburg

Molybdenum metal has favorable properties for ultrathin layers compared to metals such as copper and tungsten and, therefore, has attracted much recent interest as a potential material for interconnects and gate metallization. A common method for forming ultrathin metal layers in high volume manufacturing is vapor deposition. For vapor deposition of molybdenum metal layers, molybdenum pentachloride is a common precursor. However, molybdenum pentachloride is a solid and can be

difficult to reproducibly deliver from the precursor canister to the deposition surface in a tool, depending on delivery conditions and canister design. Also of concern is the presence of molybdenum oxychloride impurity species in the canister, and whether the oxychlorides are being generated during delivery or are present in the supplied material. The primary goal of this study was to characterize the process space for pulsed delivery of molybdenum pentachloride with respect to overall mass carryover, pulse-to-pulse stability, and impurity evolution. The approach used in this study was to quantify the amount of molybdenum pentachloride and molybdenum oxychlorides in an argon carrier gas as a function of carrier gas flow rate, system pressure, canister temperature, and canister design. Quantification was based on direct absorption measurements in the ultraviolet-visible and infrared spectral regions. The results of this study should facilitate the identification of process conditions that optimize molybdenum pentachloride for different applications.

11:30am **AP2+EL+PS+TF-FrM-14 Atomic Layer Modulation of Ru-Ir Alloy Thin Films for Low-Resistivity Interconnect Applications**, *Yoonseo Choi, Wonjoong Kim, Byungchan Lee, Minhyeok Lee, Han-Bo-Ram Lee*, Incheon National University, Republic of Korea

As semiconductor devices continue to scale down, Ru and Ir have attracted attention as promising candidates to replace Cu interconnects because their short mean free path and low bulk resistivity lead to a low figure-of-merit (FOM). However, ALD-grown single Ru and Ir thin films suffer from increased resistivity at thicknesses comparable to their mean free path due to enhanced electron scattering, particularly surface scattering, and film discontinuity. In this study, a Ru-Ir system was designed and fabricated by atomic layer modulation (ALM) to mitigate the thickness-dependent resistivity. ALM sequentially exposes different precursors to the surface with purge steps between each precursor exposure, followed by co-counter reactant exposure, enabling multicomponent thin films growth within a single atomic-layer cycle. In particular, the ratio of each element is controlled by the surface reactivity, adsorption behavior, and steric hindrance effects of the precursors, making ALM advantageous for forming compositionally uniform Ru-Ir alloy thin films even at reduced thicknesses. In this work, Ru-Ir alloy thin films were deposited at 250 $^\circ\text{C}$ by sequentially pulsing tricarbonyl(trimethylenemethane)ruthenium ($\text{Ru}(\text{TMM})(\text{CO})_3$) and tricarbonyl (1,2,3- η)-1,2,3-tri(tert-butyl)-cyclopropenyl iridium ($\text{C}_{18}\text{H}_{22}\text{IrO}_3$) as Ru and Ir precursors, respectively, with O_2 as the counter reactant. To design the process and understand the composition-control mechanism, density functional theory (DFT) and Monte Carlo (MC) simulations were used to analyze precursor surface reactions and their effects on film composition. In addition, a deep-learning-based framework (IDEAL) was used to explore candidate compositions and crystal structures for Ru-Ir alloy films. X-ray diffraction (XRD) and transmission electron microscopy (TEM) analyses confirmed that ALM-grown Ru-Ir films formed a solid solution without distinct phase separation. The electrical properties varied depending on the alloy composition; while single Ru and Ir films showed resistivity above 40 $\mu\Omega\cdot\text{cm}$ at 7 nm, Ir-rich Ru-Ir films exhibited a relatively low resistivity of 37.6 $\mu\Omega\cdot\text{cm}$ at 5 nm. The high crystallinity of the Ir-rich Ru-Ir films was also consistent with the IDEAL framework prediction. These results demonstrate that ALM-based Ru-Ir alloy thin films can overcome the thickness-scaling limitations of single-component Ru and Ir thin films and have potential as next-generation interconnect materials for replacing Cu.

11:45am **AP2+EL+PS+TF-FrM-15 Pulsed Thermal Etching of Amorphous TiO_2 Thin Films Using MoCl_5** , *Hyuenwoo Yang*, National Institute for Science and Technology (NIST); *James Maslar, Berc Kalanyan*, NIST-Gaithersburg

Gas-phase pulsed etching has emerged as a promising approach for advanced thin-film processing, enabling controlled and potentially low-damage material removal with advantages in process integration over conventional continuous etching methods. Despite growing interest in pulsed etching, including atomic layer etching (ALE), the thermal etching chemistry of metal halides—particularly MoCl_5 —remains largely unexplored for oxide materials. In this work, we investigate the pulsed thermal etching of amorphous TiO_2 thin films by MoCl_5 in a warm-walled viscous-flow reactor. Thickness changes were monitored in real time by in-situ spectroscopic ellipsometry during repeated MoCl_5/Ar cycles, while delivered precursor dose and impurity content were monitored upstream using a non-dispersive UV-vis gas analyzer.

Amorphous TiO_2 films on Si/native SiO_2 exhibited clear thickness loss under repeated MoCl_5 exposure, with etching behavior that depended strongly on substrate temperature, MoCl_5 source temperature, and Ar purge duration. Increasing the MoCl_5 source temperature increased the measured UV-vis

absorbance signal and produced a corresponding increase in TiO₂ etch rate, indicating that precursor delivery is a key parameter controlling the etching behavior. A substrate-temperature-dependent process window was also observed: etching was strongly suppressed at 150 °C and 350 °C, while the highest etch rate was observed near 250 °C. Temperature-switching experiments further showed that the low-temperature suppression was reversible upon heating to 250 °C, whereas exposure at 350 °C led to an etch-inactive state that did not recover after returning to 250 °C.

To our knowledge, this is the first report of TiO₂ etching using pulsed MoCl₅ chemistry. This work establishes an initial process window for MoCl₅-based thermal oxide etching and identifies precursor dose, substrate temperature, delivery history, and purge duration as important variables governing pulsed TiO₂ etching.

Thin Films

Room 321 - Session TF+AP+EUV-FrM

Atomic Scale Patterning and Processing

Moderator: Mark Losego, Georgia Institute of Technology

8:15am TF+AP+EUV-FrM-1 Expanding the Library of Mask Materials for Selective Area Growth by Molecular Beam Epitaxy, Corey White, Subhashree Seth, Daniela Montes, Daniel Bernstein, Tufts University; *Cornell Horne Jr.*, University of Illinois Urbana-Champaign; *Minjoo Larry Lee*, University of Illinois at Urbana Champaign

Selective area growth (SAG) enables laterally structured semiconductor growth as well as seamless encapsulation of foreign mask materials and, thus, presents an appealing approach to the monolithic integration of dissimilar materials. During SAG, III-V growth is carried out at relatively high substrate temperatures and slow growth rates to mitigate the formation of deleterious polycrystalline material on the patterned mask.¹ Historically, SiO₂ and Si₃N₄ have reigned as the most prevalent mask materials for SAG by molecular beam epitaxy (MBE).² However, successfully utilizing new mask materials with unique properties could unlock new applications and functionalities for hybrid structures. Here, we first elucidate the properties of mask materials that determine their suitability for SAG. By comparing SiO₂ masks synthesized by different thin film deposition methods, we find that differences in SiO₂ surface roughness, stoichiometry/density, and hydrocarbon contamination all impact the adsorption of group-III adatoms during epitaxial growth. Demystifying the relationship between mask material properties and the propensity of group-III adatoms to nucleate on the mask during epitaxy not only lays the foundation to achieve SAG under less stringent growth conditions over optimized masks and predict the effects of tool-to-tool variation, but also enables the identification of new, untested mask materials that may also be suitable for SAG. Thus, in this work, we showcase complete selectivity for GaAs growth over ZrO₂ and HfO₂ masks for the first time.³ This is achieved at approximately half of the group-III flux that can be employed for growth over SiO₂ with near-perfect selectivity due to differences in adatom desorption probabilities. Faceting of GaAs grown selectively within ZrO₂ gratings is found to be consistent with nominally identical growth within SiO₂ gratings. Namely, the facets that emerge depend wholly on crystallographic orientation of the dielectric mask patterns with growth within [010]-aligned structures being especially well-suited for epitaxial lateral overgrowth. III-V SAG over ZrO₂ and HfO₂ has applications in on-chip integration of passive and active optical components and deterministic growth of three-dimensional ferroelectric devices, but also demonstrates the exciting potential of further expanding the palette of available mask materials. Further work integrating other novel dielectric and two-dimensional mask materials will be presented at the conference.⁴ F. E. Allegretti and T. Nishinaga, *J. Cryst. Growth* (1995).² D. J. Ironsides et al., *Prog. Quant. Electron.* (2021).³ R. C. White and M. L. Lee, *J. Vac. Sci. Technol. A* (2026).

8:30am TF+AP+EUV-FrM-2 Hybrid Molecular Layer Deposition of Tin-Aminates Toward EUV Resists, Alex Persson, Long Than, Stacey Bent, Stanford University

The leading edge of today's semiconductor manufacturing technology relies on extreme ultraviolet (EUV) lithography, utilizing a short 13.5 nm wavelength – a roughly fourteenfold reduction from the 193 nm ArF sources of previous deep ultraviolet (DUV) lithographic technologies. Use of this wavelength has dramatically reduced achievable critical dimensions, with new high-NA EUV tools resolving half-pitches as fine as 8 nm. However, the development of photoresists capable of translating this resolution into reliably patterned features has not kept pace with the

breakthroughs in EUV exposure tools, making this material challenge a bottleneck to continued semiconductor scaling.

Conventional organic resists absorb EUV photons inefficiently, compounding stochastic failures at the ultrathin film thicknesses that pitch scaling now demands. The present work incorporates tin in an organometallic resist to address the need for better absorption, while amine organic linkers enable a tunable solubility switch using a metal-amine bonding motif not yet reported in such resists. Hybrid molecular layer deposition (MLD) is used to deposit this Sn-amine material one molecular layer at a time through alternating, self-limiting surface reactions between chemical precursors. This vapor-phase deposition technique affords molecular-level control over film thickness and structure, providing smooth and conformal ultrathin films while avoiding the challenges of solvent processing.

This presentation will introduce hybrid MLD procedures for depositing Sn-aminates as a new set of materials using tetrakis(dimethylamino)tin as a tin precursor with ethylenediamine and p-phenylenediamine as amine linkers. The stability of these materials in air and in various developers is reported, providing insight into their solubility switch mechanism and lithographic process optimization. Electron-beam lithography (EBL) is employed as an EUV proxy, and initial results indicate that Sn-aminates act as promising positive-tone e-beam resists in various developers, including HCl, KOH, and TMAH. The effect of deposition temperature on film structure, composition, stability and EBL patterning quality is discussed between 70 °C and 150 °C. Lower temperatures led to higher growth rates and higher organic content in the films, and accelerated film oxidation and decomposition in air. Optimal patterning was found with films deposited at 90 °C using ethylenediamine as the organic linker and developed in 0.02 M KOH, resulting in a dose-to-clear of 12 mC/cm² with a full-pitch of 25 nm. The effect of air-aging and post-exposure baking is also discussed.

8:45am TF+AP+EUV-FrM-3 A Multivariate Approach Based on Residual Gas Analysis for Understanding Model Photoresist Processes Occurring During Exposure, Muhammad Ramzan, University of Mississippi, Pakistan; *Markus Langner*, University of Mississippi, Germany; *Kas Andrie, Meng Zhang, Bernhard Lüttgenau*, Lawrence Berkeley National Laboratory (LBNL); *Won-Il Lee, Md Istiaque Chowdhury, Xinpei Wu*, Brookhaven National Laboratory; *Melinda Lin*, Stony Brook University; *Nikhil Tiwale, Qin Wu, Qi Zhang, Cheng Wang, Ricardo Ruiz, Michael D. Connolly, Oleg Kostko*, Lawrence Berkeley National Laboratory (LBNL); *Chang-Yong Nam*, Stony Brook University/Brookhaven National Laboratory; *Michael J. Eller*, University of Mississippi

Photoresist processes such as photon absorption, electron generation, bond cleavage, crosslinking, and volatile-product formation are key challenges in extreme ultraviolet lithography (EUVL) that need to be understood and resolved. Here, we investigate functional-group-dependent EUV exposure chemistry using simultaneous total electron yield (TEY) and residual gas analysis (RGA) measurements on a library of model polymer photoresists with defined repeat-unit structures, including methacrylate, acid-functional methacrylate, hydroxyl-containing methacrylate, ether, aromatic, heteroaromatic, imide-containing, chemically amplified epoxy, and metal-oxide-infiltrated polymer systems. Principal component analysis (PCA) was applied to the mass spectrometry data from the RGA as an unsupervised dimensionality reduction technique. Firstly, PCA clearly differentiated between low- and high-outgassing photoresists, with high-outgassing photoresists driving the maximum variance in PC1 (24.5 %). Characteristic signals from the PC loading plots were identified and assigned to corresponding photoresists. Two fragment ions, m/z 31 (CH₃O⁺) and m/z 45 (C₂H₅O⁺), were observed during the exposure of polyethylene glycol (PEG) and poly(2-hydroxyethyl methacrylate) (PHEMA). Despite having identical mass-to-charge ratios, they exhibited opposite trends with respect to exposure dose. PEG displayed increasing intensity, whereas PHEMA showed a decrease with increasing exposure dose. This trend was attributed to differences in the scission mechanisms, with PEG undergoing a solubility switch via main-chain scission, while PHEMA undergoes side-chain scission. This allows us to distinguish these two mechanisms by the time-dependent response of characteristic fragment species. We also examined the relationship between TEY and various outgassing species and found that m/z 44, assigned to CO₂, showed a positive correlation (R²=0.99) with TEY in metal-oxide-infiltrated polymer systems, whereas no correlation was observed in PMMA-only resists. Showing that these infiltrated resists exhibit improved electron production and increased outgassing, both of which are critical to improving resist sensitivity. Moving forward, we anticipate our approach will be extremely useful for screening unknown resists and identifying their mechanisms.

9:00am **TF+AP+EUV-FrM-4 A Single Amine Additive as Both Catalyst and Inhibitor for Area-Selective SiO₂ ALD, Jeong-Min Lee, Stacey Bent, Stanford University**

Driven by the demand for atomic-scale precision patterning in advanced semiconductor processing, area-selective atomic layer deposition (AS-ALD) has emerged as a paradigm-shifting technique in nanofabrication, leveraging its bottom-up nature to enable spatially selective and precise film growth. In this work, we report a methodology for achieving accelerated deposition of SiO₂ thin films on SiO₂ surfaces while suppressing growth on metal oxide surfaces using a single additive molecule that acts as both catalyst and inhibitor. To overcome the limited selectivity of conventional SiO₂ AS-ALD processes associated with the use of strong oxidizing agents, we introduce a catalytic SiO₂ ALD process. This process employs a Lewis-base catalyst that both promotes SiO₂ growth with H₂O as a mild reactant at temperatures as low as 100°C, and simultaneously inhibits growth on metal oxide surfaces such as Al₂O₃, TiO₂, RuO_x, and CuO_x. Results show that the catalyzed ALD process using amine additives exhibits a mild inherent growth suppression on metal oxide surfaces—but not on SiO₂—suggesting that interactions between the Lewis-base catalyst and metal oxide surfaces during co-injection with the precursor contribute to the retardation of SiO₂ growth. To further enhance the selectivity between SiO₂ and metal oxide substrates, an amine pretreatment was performed prior to ALD to amplify the blocking capability of the same amines as inhibitors. Comparative evaluation of pyridine and triethylamine revealed different inhibition behaviors depending on the underlying metal oxide surface, with triethylamine providing more effective growth inhibition on Al₂O₃ and TiO₂ surfaces than did pyridine. By combining the inherent growth suppression of the catalyzed ALD process with the additional blocking effect of amine pretreatment, enhanced selectivity for SiO₂ deposition on an SiO₂ growth surface and several metal oxide nongrowth surfaces was achieved. This work provides a new strategy for broadening the applicability of SiO₂ AS-ALD by leveraging the dual functionality of amine additives for both catalytic growth and surface inhibition.

9:15am **TF+AP+EUV-FrM-5 Ultrasonic Level Sensing for ALD Precursors Through the Ampoule Wall, Paul Dreher, Inficon, Liechtenstein**

Precursor inventory is one of the least directly observed state variables in an ALD delivery system. It is normally inferred from run time, wafer count or recipe demand rather than measured, so the estimate drifts over the life of the source and anomalous draw down remains invisible. Where sensing is fitted it usually detects liquid presence at a few fixed heights using an element immersed in the precursor, which quantises the reading and adds a wetted surface to a corrosive high purity path.

We determine remaining liquid height from outside a stainless steel ampoule by ultrasonic pulse echo. A piezoelectric element coupled to the outer face of the vessel base launches a longitudinal pulse through the wall. The acoustic impedance contrast between steel and a typical precursor is large, so most energy is reflected at the wall, but the transmitted fraction is sufficient to produce a resolvable echo from the liquid vapor interface. The vessel wall itself becomes part of the acoustic path and no feedthrough or wetted surface is introduced.

Extracting height is an underdetermined inverse problem. The echo delay gives the product of propagation velocity and path length, while the quantity of interest is the path length alone. Sound velocity in these liquids is strongly temperature dependent with a coefficient specific to each chemistry, and ampoules operate at temperatures up to 200 °C, so a calibration taken at room temperature does not transfer. The chemistry itself may also be unknown to the operator. We show that the velocity can be recovered in situ at operating temperature from the same acquisition, so height is determined with no prior property data for the fluid.

Silicone oil was used as a deliberately unfavorable test case: attenuation is high, the surface echo is correspondingly weak and higher order reverberations within the liquid produce competing arrivals that must be separated. Recovered height agrees with an independent reference to approximately 1 mm with R² = 0.997, continuously across the full range rather than at discrete heights, and the residual is limited by the reference rather than by the sensing geometry. The result is stable from room temperature to 150 °C and reproducible through repeated thermal cycles, with time of flight tracking the velocity change through heating and cooling.

Because the velocity determination responds to the liquid itself rather than to its height, it is also sensitive to changes in composition, suggesting that a single acquisition may report precursor condition alongside inventory.

9:30am **TF+AP+EUV-FrM-6 ALD-Enabled Ag/ZnO Nanostructured PTFE Membranes as Flexible SERS Substrates for Trace Molecular Detection, Halil AKYILDIZ, Sumeyye DILER INAN, Burak SOZEN, Bursa Uludag University, Turkey**

Surface-enhanced Raman scattering (SERS) is widely used for sensitive and label-free detection of molecules, with signal enhancement mainly arising from localized plasmonic effects associated with metallic nanostructures. In this study, we developed a flexible SERS substrate by combining atomic layer deposition (ALD) of ZnO with Ag nanoparticles formed on porous polytetrafluoroethylene (PTFE) membranes. PTFE membranes were first coated with ZnO by ALD, followed by the formation of Ag nanoparticles from a silver acetate solution using UV-assisted photodeposition. The SERS performance of the prepared substrates was evaluated using crystal violet (CV) as a model analyte. CV solutions ranging from 10⁻³ to 10⁻⁸ M were deposited onto the substrates and analyzed by confocal Raman microscopy using a 532 nm excitation laser. The Ag/ZnO-coated PTFE membranes showed a clear SERS response to CV. Characteristic Raman bands at 732, 761, 809, 1175, 1373, 1538, 1588, and 1618 cm⁻¹ were observed, and their intensities decreased as the CV concentration was reduced. The characteristic bands could still be clearly identified at a CV concentration of 10⁻⁷ M, whereas they were no longer distinguishable at 10⁻⁸ M. The intensity of the characteristic band at 1175 cm⁻¹ also showed a clear dependence on CV concentration. The results show that ALD-grown ZnO can be used as an intermediate surface layer for preparing Ag-containing SERS-active PTFE membranes. Combining the flexibility and porous structure of PTFE with ALD surface modification and Ag photodeposition offers a practical approach to producing flexible SERS substrates. The prepared membranes were able to detect CV at concentrations as low as 10⁻⁷ M and may provide a useful platform for further development of flexible Raman-based chemical sensors.

9:45am **TF+AP+EUV-FrM-7 Low Temperature PEALD of Epitaxial AlN on GaN, Ga₂O₃, and Al₂O₃ Surfaces, Jeffrey Woodward, David Boris, Michael Johnson, Daniel Pennachio, U.S. Naval Research Laboratory; Henry Chuang, Naval Research Enterprise Internship Program; Michael Mathews, Emma Rocco, Katie Gann, Benjamin Sekely, Tatyana Feygelson, Jonathan Levine-Miles, U.S. Naval Research Laboratory; Jennifer Hite, University of Florida; Michael Mastro, Virginia Wheeler, Scott Walton, U.S. Naval Research Laboratory**

AlN is a wide-bandgap semiconductor with exceptional thermal conductivity, temperature stability, and piezoelectric properties, making it a promising material for high power electronics and deep ultraviolet optoelectronics in extreme environments. While plasma-enhanced atomic layer deposition (PEALD) of AlN is well-established as a promising back-end-of-line compatible process for passivation, electrical insulation, and thermal management in devices, it typically results in amorphous or polycrystalline films, which have inferior electrical and thermal properties as well as reduced chemical resistances compared to epitaxial AlN. Most reports of epitaxial AlN growth by PEALD involve the use of atomic layer annealing (ALA), in which an Ar plasma exposure is incorporated into each cycle to induce crystallization of the surface.[1] While the effectiveness of ALA is proven, its use also increases cycle duration, which significantly prolongs total process time and can promote impurity incorporation. For these reasons, the growth of epitaxial AlN without ALA is desirable, though this is challenged by the complexity of controlling the plasma properties to achieve suitable growth conditions.

In this work, we demonstrate the growth of 30-50 nm thick epitaxial AlN films on Al₂O₃, GaN, and Ga₂O₃ at 300 °C using PEALD without ALA or thermal annealing. Plasma diagnostics were used to identify favorable conditions which produce ion energy and flux characteristics comparable to those of plasmas used in ALA. The films were characterized using x-ray reflectivity (XRR), high resolution x-ray diffraction (HRXRD), in-plane grazing incidence diffraction (IP-GID), atomic force microscopy (AFM), transmission electron microscopy (TEM), x-ray photoelectron spectroscopy (XPS), and optical measurements. The growth per cycle (GPC) and density ranged from 0.90–1.03 Å and 2.998–3.173 g/cm³, respectively, with deposition on GaN resulting in the highest GPC and density. The topography of the films closely replicated that of the underlying material with roughness from 0.25–1 nm by AFM and 0.83–2 nm by XRR, indicating that the deposition was highly conformal. The epitaxial nature of the AlN (i.e., exhibiting a highly ordered crystalline structure with well-defined orientational relationship to the substrate) is confirmed by HRXRD and IP-GID, which show a single orientation with distinct 6-fold azimuthal rotational symmetry. While the AlN films on Al₂O₃ and Ga₂O₃ exhibit significant mosaicity, the AlN films on

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GaN exhibit low tilt disorder, as evidenced by narrow out-of-plane rocking curves with FWHM= 0.149 degrees (537 arcseconds).

[1] H. Y. Shih et al., Sci. Rep. **7**, 39717 (2017)

10:00am **TF+AP+EUUV-FrM-8 Kinetic Stabilization of Highly Dispersed Rh on MoO₃-Modified Anatase TiO₂(101),** *Sineth Premarathna*, University of Washington; *Zbynek Novotny*, Pacific Northwest National Laboratory; *Charles T. Campbell*, University of Washington; *Zdenek Dohnalek*, Pacific Northwest National Laboratory

Developing approaches to stabilize highly dispersed metals on oxide supports represents one of the grand challenges in catalysis. In this context, the (101) surface of TiO₂ anatase is particularly challenging because it lacks surface defects and has a high propensity for metal clustering. We investigate the role of MoO₃ monomeric clusters, pre-deposited on TiO₂(101), in suppressing clustering of adsorbed Rh, using combined scanning tunneling microscopy (STM) and X-ray photoelectron spectroscopy (XPS). Rh deposited directly on bare TiO₂(101) sinters readily at 300 K, forming nm-sized Rh clusters with apparent heights of 3 Å and larger, serving as a reference system. When Rh is instead deposited on a (2×1)-ordered MoO₃/anatase(101) surface at 163 K, STM reveals significantly smaller, highly dispersed Rh species, with apparent heights of 1.8 Å, which remain stable to 450 K. These species exhibit a higher Rh 3d binding energy of 307.8 eV as compared to 307.2 eV of metallic Rh. Only after annealing to 600 K is significant ripening observed, and the cluster sizes approach those observed for Rh on bare TiO₂(101), while the Rh 3d peak shifts toward lower binding energy. The correlated STM and XPS evolution is consistent with increased final-state screening in Rh 3d peak as Rh clusters grow. Our results indicate that modification with MoO₃, combined with low-temperature deposition, kinetically traps and stabilizes highly dispersed Rh species. Ongoing work aims to identify the Mo-O-Rh binding motifs responsible for this stabilization and to determine whether they can stabilize isolated Rh atoms.

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